Materials Testing IV
(Transportation and Environment)

University of Global Village (UGV),
Barishal

COURSEINFORMATION			
Course Title	Materials Testing IV (Transportation and Environment)	Lecture ContactHours	5.00

No.	Course Outcomes
CO1	**Determine** diverse layouts and interpreting different results accurately.
CO2	**Analyze** plan and model characteristics and behavior under different conditions.
CO3	**Apply** software knowledge to solve engineering problems effectively.
CO4	**Plan** effectively to work individually and in a group.

SI	Content of Course	Class Time (hr)	CLOs
01	1.Determination of Aggregate Impact Value 2.Determination of Aggregate Crushing Value 3.Determination of Ten Percent Fines Value	03	CLO1 CLO 2, CLO3, CLO4
02	4.Determination of Flakiness Index 5.Determination of Elongation Index 6.Determination of Angularity Number	03	CLO1 CLO 2, CLO3, CLO4

03	1.Specific Gravity of Semi-Solid Bituminous Material 2.Loss on Heating of Oil and Asphaltic Compounds 3.Penetration of Bituminous Material 4.Softening Point of Bituminous Material	03	CLO1 CLO 2, CLO3, CLO4
04	5.Solubility of Bituminous Material 6.Ductility of Bituminous Material 7.Flash and Fire Points of Bituminous Material (Cleavelandopen cup)	03	CLO1 CLO 2, CLO3, CLO4
05	Determination of Roadway Capacity	03	CLO1 CLO 2, CLO3, CLO4
06	Determination of Saturation Flow at Traffic Signals	03	CLO1 CLO 2, CLO3, CLO4
07	Determination of CBR	03	CLO1 CLO 2, CLO3, CLO4
08	Marshall Mix Design	03	CLO1 CLO 2, CLO3, CLO4

09	1. Determination of pH of Water 2. Determination of Color of Water 3. Determination of Turbidity of Water 4. Determination of Total Solids, Dissolved Solids and Suspended Solids in Water	03	CLO1 CLO 2, CLO3, CLO4
10	Determination of Carbon dioxide in Water	03	CLO1 CLO 2, CLO3, CLO4
11	1. Determination of Alkalinity of Water 2. Determination of Hardness of Water	03	CLO1 CLO 2, CLO3, CLO4
12	1. Determination of Chloride in Water 2. Determination of Residual Chlorine and Chlorine Demand: Break Point Chlorination	03	CLO1 CLO 2, CLO3, CLO4
13	Determination of Iron in Water	03	CLO1 CLO 2, CLO3, CLO4
14	Estimation of Organic Pollution Load through Determination of BOD and COD	03	CLO1 CLO 2, CLO3, CLO4

15	Chemical Coagulation of Water: Alum Coagulation	03	CLO1 CLO 2, CLO3, CLO4
16	Determination of Arsenic in Water	03	CLO1 CLO 2, CLO3, CLO4
17	Determination of Total Coliform and Fecal Coliform for Microbiological Water Quality Analysis	03	CLO1 CLO 2, CLO3, CLO4

Week	Topic	Teaching Learning Strategy	Assessment Strategy	Corresponding CLOs
01	1.Determination of Aggregate Impact Value 2.Determination of Aggregate Crushing Value 3.Determination of Ten Percent Fines Value	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
02	4.Determination of Flakiness Index 5.Determination of Elongation Index 6.Determination of Angularity Number	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
03	1.Specific Gravity of Semi-Solid Bituminous Material 2.Loss on Heating of Oil	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2,

	and Asphaltic Compounds 3.Penetration of Bituminous 4.Softening Point of Bitumi Material			CLO3, CLO4
04	5.Solubility of Bituminous Material 6.Ductility of Bituminous Material 7.Flash and Fire Points of Bituminous Material (Cleavelandopen cup)	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
05	Determination of Roadway Capacity	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
06	Determination of Saturation Flow at Traffic Signals	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
07	Determination of CBR	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
08	Marshall Mix Design	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
09	1. Determination of pH of Water 2. Determination of Color of Water 3. Determination of Turbidity of Water 4. Determination of Total Solids, Dissolved Solids and Suspended Solids in Water	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
10	Determination of Carbon dioxide in Water	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
11	1.Determination of Alkalinity of Water	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3,

	2. Determination of Hardness of Water			CLO4
12	1. Determination of Chloride in Water 2. Determination of Residual Chlorine and Chlorine Demand: Break Point Chlorination	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
13	Determination of Iron in Water	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
14	Estimation of Organic Pollution Load through Determination of BOD and COD	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
15	Chemical Coagulation of Water: Alum Coagulation	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
16	Determination of Arsenic in Water	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4
17	Determination of Total Coliform and Fecal Coliform for Microbiological Water Quality Analysis	Lecture, Discussion, Experiment	Lab Test, Quiz, Report	CLO1, CLO2, CLO3, CLO4

TABLE OF CONTENTS

Tests on Aggregates

Traffic Engineering

Tests on Bituminous Material

CBR and Bituminous Mix Design

EXPERIMENT NO: 01

DETERMINATION OF AGGREGATE IMPACT VALUE

(BS: 812: 1975 (PART 1, 2, 3))

1.1 GENERAL

The aggregate impact value gives a relative measure of the resistance of an aggregate to “sudden shock or impact”, which in some aggregates differs from its resistance to a slowly applied compressive load. With aggregate of aggregate impact value (AIV) higher than 30 the result may be anomalous. Also aggregate sizes larger than 14 mm are not appropriate to the aggregate impact test.

The standard aggregate impact test shall be made on aggregate passing a 14. mm BS test sieve and retained on a 10.0 mm BS test sieve. If required, or if the standard size is not available, smaller sizes may be tested but owing to the non-homogeneity of aggregates the results are not likely to be the same as those obtained from the standard size. In general, the smaller sizes of aggregate will give a lower impact value but the relationship between the values obtained with different sizes may vary from one aggregate to another.

1.2 APPARATUS

The following apparatus is required.

1.2.1 An impact testing machine of the general form and complying with the followings,

(a) Total mass not more than 60 kg or less than 45 kg.
The machine shall have a circular metal base weighing between 22 kg and 30 kg., with a plane lower surface of not less than 300 mm diameter, and shall be supported on a level and plane concrete or stone block or floor at least 450 mm thick. The machine shall be prevented from rocking either by fixing it to the block or floor or by supporting it on a level and plane metal plate cost into the surface of the block or floor.

(b) A cylinder steel cup having an internal diameter of 102 mm and an internal depth of 50 mm. The walls shall be not less than 6 mm thick and the inner surfaces shall be case hardened. The cup shall be rigidly fastened at the center of the base and be easily removed for emptying.

(c) A metal hammer weighing 13.5 kg to 14.0 kg the lower end of which shall be cylindrical in shape, 100.00 mm diameter and 50 mm long, with a 1.5 mm chamfer at the lower edge, and case hardened. The hammer shall slide freely between vertical guides so arranged that the lower (cylindrical) part of the hammer is above and concentric with the cup.

(d) Means or raising the hammer and allowing it to fall freely between the vertical guides from a height of 380±5 mm on to the test sample in the cup, and means for adjusting the height of fall within 5 mm.

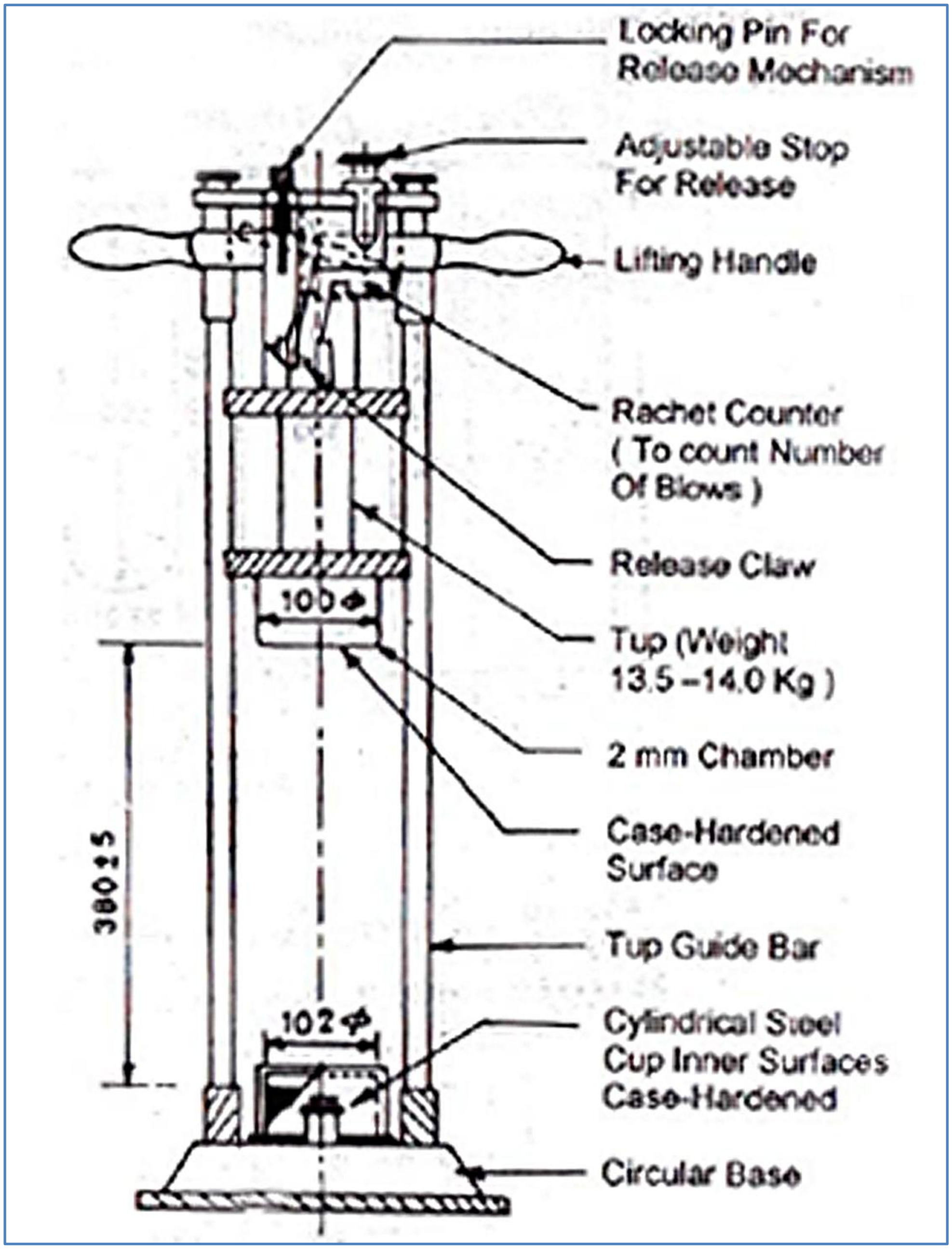


Figure1.1 Aggregate Impact Test machine

(e) Means for supporting the hammer whilst fastening or removing the cup.

NOTE: Some means for automatically recording the number of blow is desirable.

1.2.2 BS test sieves of aperture size 14.0 mm, 10.0 mm and 2.36 mm for a standard test.

1.2.3 A cylindrical metal measure of sufficient rigidity to retain its form under rough usage and with an internal diameter of 75 ± 1 mm and an internal depth of 50± 1 mm.

1.2.4 A straight metal tamping rod of circular cross section, 10 mm diameter, 230 mm long, rounded at one edge.

1.2.5 A balance of capacity not less than 500 gm, and accurate to 0.1 gm.

1.3 PREPARATION OF THE TEST SAMPLE

The material for the standard test shall consist of aggregate passing a 14.0 mm BS test sieve and retained on a 10.00 mm BS test sieve and shall be thoroughly separated on these sieves before testing. For smaller sizes the aggregate shall be prepared in a similar manner using the appropriate sieves given in Table 1. The quantity of aggregate sieved out shall be sufficient for two tests.

The aggregate shall be tested in a surface dry condition. If dried by heating, the period of drying shall not exceed 4 h, the temperature shall not exceed 110^{0}c and the samples shall be cooled to room temperature before testing.

The measure shall be filled about one third full with the aggregate by means of a scoop, the aggregate being discharged from a height not exceeding 50mm above the top of the container. The aggregate shall then be tamped with 25 blows of the rounded end of the tamping rod, each blows being given by allowing the tamping rod to fall freely from a height of about 50 mm above the surface of the aggregate and the blows being evenly distributed over the surface. A further similar quantity of aggregate shall be added in the same manner and a further tamping of 25 times and the surplus aggregate removed by rolling the tamping rod across, and in contact with, the top of the container, any aggregate which impedes its progress being removed by hand and aggregate being added to fill any obvious depressions. The net mass of aggregates in the measure shall be recorded (mass A) and the same mass used for the second test.

1.4 TEST PROCEDURE

Rest the impact machine, without wedging or packing, upon the level plate, black or floor, so that it is rigid and the hammer guide columns are vertical. Fix the cup firmly in position on the base of the machine and place the whole of the test sample in it and compact by a single tamping of 25 strokes of the taming rod as above.

Adjust the height of the hammer so that its lower face is 380± 5 mm above the upper surface of the aggregate in the cup and then allow it to fall freely on to the aggregate. Subject the test sample to a total of 15 such blows, each being delivered act an interval of not less than 1 s. No adjustment for hammer height is required after the first blow.

Then remove the crushed aggregate by holding the cup over a clean tray and hammering on the outside with a suitable rubber mallet until the sample particle s are sufficiently disturbed to enable the mass of the sample to fall freely on to the tray. Transfer fine particles adhering to the inside of the cup and the underside of the hammer to the tray by means of a stiff bristle brush. Sieve the whole of the sample in the tray, for the standard test, on the 2.36 mm BS test sieve until no further significant amount in 1 min. When testing sizes smaller than the standard separate the fines on the appropriate sieve given in the ‘for separating fines’ column in table 1.1.

Weigh the fraction passing and retained on the sieve to an accurately of 0.1 gm (mass B and mass C respectively) and if the total mass B+C is less than the initial mass (mass A) by more than 1 gm, discard the result and make afresh test.
Repeat the whole procedure starting from the beginning using a second sample of the same mass as the first sample.

Table 1.1 Particulars of BS test sieves for testing standard and non standard sizes of aggregates

Sample size	Nominal aperture sizes of BS test sieves complying with the requirements of BS410 (full tolerance)			
	for sample preparation		for separating fines	
	passing			
Non-standard	mm	mm	mm	μ
	28.0	20.0	5.00	-
	20.0	14.0	3.35	-
.Standard	14.0	10.0	2.36	-
Non-standard	10.0	6.30	1.70	-
	6.30	5.00	1.18	-
	5.00	3.35	-	85
	3.35	2.36	-	60

NOTE: Aggregate sizes larger than 14.0 mm are not appropriate to the aggregate impact test.

1.5 CALCULATIONS

The ratio of the mass of fines formed to the total sample mass in each test shall be expressed as a percentage, the result being recorded to the first decimal place.

Percentage fines: $B/A \times 100$

Where,
A is the a mss of surface dry sample, gm
B is the fraction passing the sieve for separating the fines, gm

1.6 REPORTING OF RESULTS

The mean of the two results shall be reported to the nearest whole number as the aggregate impact value.

Experiment No: 01
Determination of Aggregate Impact Value

Name : **Student No**:

Type of material : Brick Chips/ Stone Chips/ Gravels/ Boulder/ Rock
Sample Size : 14 mm to 10 mm
Test Method : Bs 812 (part 3) 1975

Test No Data	1	2
Wt. of Sample (Surface Dry), A gm		
Wt. of materials retained on 2.36 mm sieve, C gm		
Wt. of materials passing 2.36 mm sieve, B gm		
Aggregate Impact Value (%) = B/A x 100% **(to the first decimal place)**		
Average Aggregate Impact Value (AIV) = **(to the nearest whole number)**		

Calculation:

Average Aggregate Impact Value (AIV):

Signature of the Course Teacher

EXPERIMENT NO: 02

DETERMINATION OF AGGREGATE CRUSHING VALUE

(BS: 812: 1975 (PART 1, 2, 3))

2.1 GENERAL

The aggregate crushing value gives a relative measure of the resistance of an aggregate to crushing under a gradually applied compressive load. With aggregate of an aggregate crushing value higher than 30 the result may be anomalous, and in such cases the ten percent fines value (clause 8) should be determined instead.

The standard aggregate crushing test shall be made as described in section 2.3 to section 2.7 on aggregate passing a 14.0 mm BS test sieve and retained on a 10.0 m BS test sieve. If required, or if the standard size of aggregate is not available, the test shall be made according to section 2.8.

2.2 SAMPLING

The sample for this test shall be taken in accordance with clause 5 of part 1 of this standard (BS 812).

2.3 APPARATUS

The following apparatus is required for the standard test.

2.3.1 An open ended steel cylinder of nominal 150 mm internal diameter with plunger and base plate, of the general from and diameter shown in the figure. The surfaces in contact with the aggregate shall be machined and case hardened, and shall be maintained in a smooth condition.

2.3.2 A straight metal tamping rod of circular cross section, 16 mm diameter and 450 mm to 600 mm long. One end shall be rounded.

2.3.3 A balance of at least 3 kg capacity and accurate to 1 gm.

2.3.4 BS test sieves of sizes 14.0 mm, 10.0 mm and 2.36 mm.

2.3.5 A compression testing machine capable of applying a force of 400 KN and which can be operated to give a uniform rate of loading so that this force is reached in 10 minute.

2.3.6 A cylindrical metal measure (optional) for measuring the sample, of sufficient rigidity to remain its form under rough usage and having an internal diameter of 115 mm and an internal depth of 180 mm.
Note. See Table 2.1

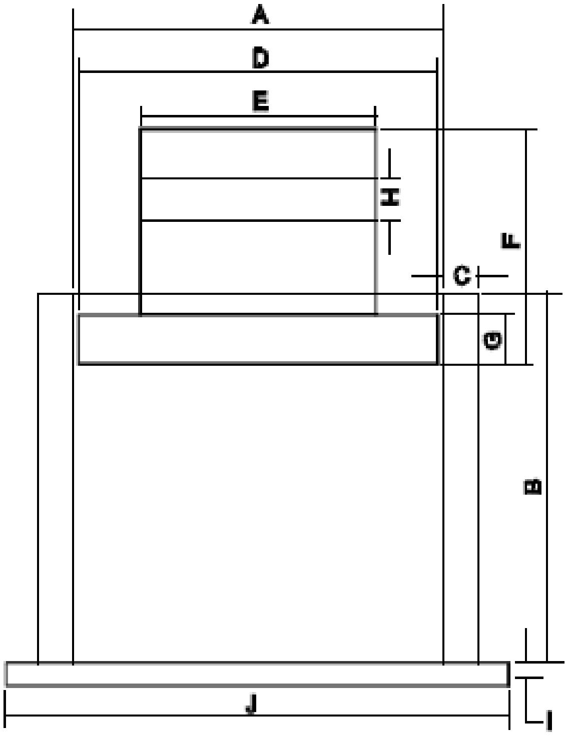


Figure 2.1 Outline of the cylinder and plunger apparatus for the crushing value

Table 2.1: Dimensions of the cylinder and Plunger apparatus for the crushing value.

Component	Dimensions See Figure	Nominal 150 mm internal diameter of cylinder, mm	Nominal 75 mm internal diameter of cylinder mm
Cylinder	Internal diameter, A Internal height, B Minimum wall thickness, C	154±0.5 mm 125 to 140 mm Not less than 16.0 mm	78±0.5 mm 70.0 to 85.0 mm Not less than 8.0 mm
Plunger	Diameter of piston, D Diameter of stem, E Overall length of piston plus stem, F Minimum depth of piston, G Diameter of hole, H	152±0.5 mm 95 to 155 100 to 115 mm Not less than 25.0 20.0±0.1 mm	76.0±0.5 mm 45.0 to 80.0 60.0 to 80.0 mm Not less than 19.0 10.0±0.1 mm
Base-plate	Minimum thickness, I Length of each side of square, J	10 mm 200 to 230 mm	10 mm 110 to 115 mm

NOTE: As a temporary measure, apparatus complying with the requirements of BS 812: 1967 (now withdrawn) shall be deemed to comply with this requirement.

2.4 PREPARATION OF TEST SAMPLE

The material for the standard test shall consist of aggregate passing the 14.0 mm BS test sieve and retained on the 10.0 mm BS test sieve and shall be thoroughly separated on these sieves before testing. The quality of aggregate shall be cooled to room temperature before testing.

The aggregate shall be tested in a surface-dry condition. If dried by heating the period of drying shall not exceed 4 h, the temperature shall not exceed 110^0c and the aggregate shall be cooled to room temperature before testing.

The quantity of aggregate for one test shall be such that the depth of the material in the cylinder shall be 100 mm after tamping.

The appropriate quantity may be found conveniently by filling the cylindrical measure in three layers of approximately equal depth, each layer being tamped 25 times from a height of approximately 50 mm above the surface of the aggregate with the rounded end of the tamping rod and finally leveled off, using the tamping rod as a straight edge.

The mass of material comprising the test sample shall be determined (mass A).

2.5 TEST PROCEDURE

Put the cylinder of the test apparatus in position on the base plate, and add the test sample in thirds, each third being subjected to 25 strikes from the tamping rod distributed evenly over the surface of the layer and dropping from a height approximately 50 mm above the surface of the aggregate. Carefully level the surface of the aggregate and insert the plunger so that it rests horizontally on this surface, taking care to ensure that the plunger does not jam in the cylinder.

Place the Apparatus, with the test sample and plunger in position, between the plates of the testing machine and load it at as uniform a rate as possible so that the required force is reached in 10 minutes. The required force shall be 400 kN.

Release the load and remove the crushed material by holding the cylinder over a clean tray and hammering on the outside with a suitable rubber mallet until the sample particles are sufficiently disturbed to enable the mass of the sample to fall freely on to the tray. Transfer fine particles adhering to the inside of the cylinder, to the base-plate and the underside of the plunger to the tray by means of a stiff bristle brush. Sieve the whole of the sample on the tray on the 2.36 mm BS test sieve until no further significant amount passes in 1 minute. Weight the fraction passing the sieve (mass B). Take care in all of these operations to avoid loose of the fines.

Repeat the whole procedure, starting from the beginning of 2.5, using a second sample of the same mass as the first sample.

2.6 CALCULATION

The ratio of the mass of fines formed to the total mass of the sample in each test shall be expressed as a percentage, the result being recorded to the first decimal place.
Percentage Fines: B/A x 100

Where,
A is the mass of surface dry sample (gm)
B is the mass of the fraction passing the 2.36 mm BS test sieve (gm)

2.7 REPORTING OF RESULTS

The mean of the two results shall be reported to the nearest whole number as the aggregate crushing value.

2.8 DETERMINATION OF AGGREGATE CRUSHING VALUE FOR NON-STANDARD SIZES OF AGGREGATE

2.8.1 General

If required, or if the standard size is not available, tests may be made on aggregates of other sizes larger than the standard up to a size which passes a 28.0 mm BS test sieve, using the standard apparatus. Alternatively tests may be made on aggregates smaller than the standard down to a size which is retained on a 2.36 mm BS test sieve, using either the standard apparatus or that described in 2.8.2 which is referred to as the smaller apparatus.

Owing to the non-homogeneity of aggregates the results of tests on non-standard sizes are not likely to be the same as those obtained from standard tests. In general, the smaller sizes of aggregate will give a lower aggregate crushing value and the larger sizes a higher value, but the relationship between the values obtained will vary from one aggregate to another. However, the results obtained with the smaller apparatus have been found to be slightly higher than those with the standard apparatus and the errors for the smaller sizes of aggregate tested in the smaller apparatus are therefore compensator.

2.8.2 Apparatus

The following apparatus is required for the standard test

2.8.2.1 An open ended steel cylinder, with plunger and base plate, generally as described in 2.3.1, with a nominal internal diameter of 75 mm the general form and dimensions of the cylinder and of the plunger are shown in Fig. 2.1

2.8.2.2 A balance of at least 500g capacity and accurate to 0.2g.

2.8.2.3 *BS 410 test sieves* of appropriate sizes given in Table 1.1 (see section 2.1.3.1 of Part 1 of this standard.)

2.8.2.4 A compression testing machine generally as described in 2.3.5 except that it shall be capable of applying force of 100 KN, and of being operated to' give a uniform rate of loading so that this force is reached in 10 min.

2.8.2.5 A cylindrical metal measure generally as described in 2.3.6 except that it shall have an internal diameter of 57 mm and an internal depth of 90 min.

2.8.3 Preparation of test sample

The material for tests on non-standard sizes shall consist of aggregate passing and retained on corresponding BS test sieves given in Table 1.1.

The procedure shall in other respects follow that given in section 2.4. except that in tests with the smaller apparatus the quantity shall be such that the depth of material in the nominal 75.0 mm internal diameter cylinder shall be 50 mm after taping with the smaller rod. The appropriate quantity may be found conveniently by using the smaller measure and taping rod.

2.8.4 Test procedure

Tests on non-standard sizes shall follow, the procedure given in 2.5 except that, when using the smaller apparatus, the smaller tamping rod shall be used and the total force shall be 100 KN. Take particular care with the larger sizes of aggregate to ensure that the plunger does not jam in the cylinder. Sieve the material removed from the cylinder on the appropriate sieve given in the “for separating fines” column in Table 1.1.

2.8.5 Calculations

Calculations for tests on non-standard sizes shall follow the method given in section2.6 substituting in the description of mass B the test sieve of appropriate size.

2.8.6 Reporting of results

Results of tests on non-standard sizes shall be reported as in 2.7 with, additionally, a report on the size of the aggregate tested and, if smaller than the standard size, the nominal size of the cylinder used in the test.

Experiment No: 02
Determination of Aggregate Crushing Value

Name : **Student No**:

Type of material : Brick Chips/ Stone Chips/ Gravels/ Boulder/ Rock
Sample Size : 14 mm to 10 mm
Test Method : Bs 812 (part 3) 1975

Test No Data	1	2
Wt. of Sample (Surface Dry), A gm		
Wt. of materials retained on 2.36 mm sieve, C gm		
Wt. of materials passing 2.36 mm sieve, B gm		
Aggregate Crushing Value (%) = B/A x 100% **(to the first decimal place)**		
Average Aggregate CrushingValue (ACV) = **(to the nearest whole number)**		

Calculation:

Average Aggregate Crushing Value:

Signature of the Course Teacher

EXPERIMENT NO: 03

DETERMINATION OF THE TEN PERCENT FINES VALUE

(BS: 812: 1975 (PART 1, 2, 3))

3.1 GENERAL

The ten percent fines value gives a measure of the resistance of an aggregate to crushing which is applicable to both weak and strong aggregate.

The standard ten percent fines shall be made as described in section 3.3 to section 3.7 on aggregate passing a 14.0 mm BS test sieve and retained on a 10.0 mm BS test sieve. If required, or if the standard size of aggregate is not available, the test shall be made in accordance with section 3.8.

3.2 SAMPLING

The sample for this test shall be taken in accordance with clause 5 of part 1 of this standard (BS 812).

3.3 APPARATUS

The following apparatus is required for the standard test.
3.3.1 An open ended steel cylinder with plunger, and base plate, as described in section2.3.1.

3.3.2 A tamping rod as described in section 2.3.2.

3.3.3 A balance as described in section 2.3.3.

3.3.4 BS 410 test sieves as described in section 2.3.4.

3.3.5 A compression testing machine as described in section2.3.5, except that the forces which is to be applied may vary from 5 kN to 500 kN.

3.3.6 A cylindrical metal measure as described in section 2.3.6.

3.3.7 (if required; see note in section 3.5), a means of measuring to the nearest 1 mm the reduction in distance between the platens of the testing machine during the test (e.g. a dial gauge).

3.4 PREPARATION OF THE TEST SAMPLE

The preparation of the test sample shall be as described in 2.4 except that, in the case of weak materials, particular care shall be taken not to break the particles when filling the measure and the cylinder.

NOTE: Sufficient test sample for three of more tests may be necessary.

3.5 TEST PROCEDURE

Put the cylinder of the test apparatus in position on the base-plate and add the test sample in thirds, each third being subjected to 25 strokes from the tamping rod distributed evenly over the surface of the layer and dropping from a height approximately 50 mm above the surface of the aggregate, particular care being taken in the case of weak materials not to break the particles. Carefully level the surface of the aggregate and insert the plunger so that it rests horizontally on this surface, taking care to ensure that the plunger does not jam in the cylinder.

Then place the apparatus, with the test sample and plunger in position, between the plates of the testing machine. Apply forces at as uniform a rate as possible so as to cause a total penetration of the plunger in 10 min of about:

a) 15 mm for rounded or partially rounded aggregate (e.g. uncrushed gravels)
b) 20 mm for normal crushed aggregate
c) 24 mm for honeycombed aggregate (e.g. some slags)

The figures may be varied according to the extent of the rounding or honeycombing.
NOTE: When an aggregate impact value is available, the force required for the first ten percent fines test can be estimated by means of the following more conveniently than by the use of the dial gauge.

Required force (KN) = 4000/ Aggregate Impact Value

This value of force will nearly always gives a percentage fines within the required range of 7.5 to 12.5.

Record the maximum force applied to produce the required penetration. Release the force and remove the crushed material by holding the cylinder over a clean tray and hammering on the outside with a suitable rubber mallet until the sample particles are sufficiently disturbed to enable the mass of the sample to fall freely on to the tray. Transfer fine particles adhering to the inside of the cylinder and the underside of the plunger to the tray by means of a still bristle brush. Sieve the whole of the sample in the tray on the 2.36 mm BS test sieve until no further significant amount passes in 1 minute. Weight the fraction passing the sieve, and express this mass as percentage of the mass of the test sample. Normally this percentage of fines will fall within the range 7.5 to 12.5, but if it does not, make a further test loading to a maximum value adjusted as seems appropriate to bring the percentage fines within the range of 7.5 to 12.5. The formula given in 3.6 may be used for calculating the force required.

In all of these operations take care to avoid loss of the fines. Make a repeat test at the maximum force that gives a percentage fines within the range 7.5 to 12.5.

3.6 CALCULATIONS

The mean percentage fines from the two tests at this maximum force shall be used in the following to calculate the force required to produce ten percentage fines.

Force required to produce ten percent fines = 14 x / (y+4)
Where,
x is the maximum force (kN)
y is the mean percentage fines from two tests at "x" KN forces.

3.7 REPORTING AND RESULTS

The force required to produce ten percent fines shall be reported, to the nearest 10 KN for forces of 100 kN or more or the nearest 5 kN for loads of less than 100 kN, as the ten percent fines value.

3.8 DETERMINATION OF THE TEN PERCENT FINES VALUE FOR NON-STANDARD SIZES OF AGGREGATE

3.8.1 General

If required, or if the standard size is not available, test may be made on aggregates of other sizes which pass a 28.0 mm BS test sieve and are retained on a 2.36 mm BS test sieve. Because of the lack of experience of the testing sizes other than the standard it has not been possible to give any indication as to how the results obtained on non-standard sizes would compare with those obtained in the standard test as in the case of the aggregate crushing value.

3.8.2 Apparatus

The apparatus shall be as described in section 3.3.1 to section 3.3.3 and section 3.3.5 to section 3.3.7.

BS test sieves of appropriate sizes shall be as given in Table 1.1.

3.8.3 Preparation of test sample

The material for tests on non-standard sizes shall consist of aggregate passing and retained on corresponding BS test sieves given in Table 1.1.

The procedure shall in other respects follow that given in section 3.4.

3.8.4 Test procedure

Test on non-standard sizes shall follow the procedure given in 3.5 using the appropriate separating sieve given in table 1.1, it should be noted that the penetration of the plunger may not accord with the values given in 3.5.

3.8.5 Calculations

Calculations for non-standard sizes shall follow the method given in 3.6.

3.8.6 Reporting of results

Results of tests on non-standard sizes shall be reported as in 3.7 with, additionally, a report on the size of the aggregate tested.

Experiment No: 03
Determination of the Ten Percent Fines Value

Name : **Student No**:

Type of material : Brick Chips/ Stone Chips/ Gravels/ Boulder/ Rock
Sample Size : 14 mm to 10 mm
Test Method : Bs 812 (part 3) 1975

Force required to produce T.F.V = 4000kN / A.I.V = kN.
Used Force = x = kN

Test No / Data	1	2	3 (if required)
Wt. of Sample (Surface Dry), A gm			
Wt. of materials retained on 2.36 mm sieve, C gm			
Wt. of materials passing 2.36 mm sieve, B gm			
Percent Fines = B/A x 100% (to the first decimal place)			
Mean percentage fines (y), at x = kN force			

Calculation:

Ten Percent Fines Value (T.F.V) = 14x / (y+4) =
(to the nearest 5 kN, if < 100 kN
& 10kN, if ≥ 100 kN)

Ten Percent Fines Value (T.F.V):

Signature of the Course Teacher

EXPERIMENT NO: 04

DETERMINATION OF FLAKINESS INDEX

(BS: 812: 1975 (PART 1, 2, 3))

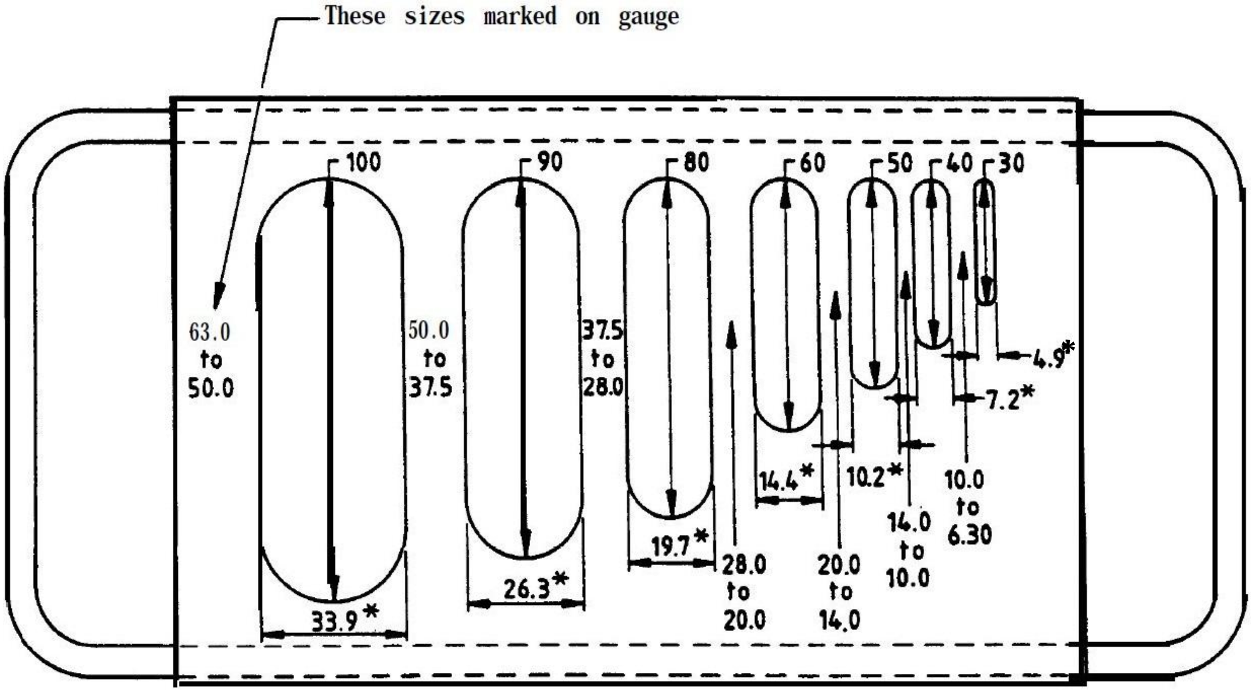
4.1 GENERAL

This method is based on the classification of aggregate particles as flaky when they have a thickness (smallest dimension) of less than 0.6 of their nominal size, this size being taken as the mean of the limiting sieve apertures used for determining the size fraction in which the particle occurs. The flakiness index of an aggregate sample is found by separating the flaky particles and expressing their mass as a percentage of the mass of the sample tested. The test is not applicable to material passing a 6.30 mm BS test sieve or retained on a 63.0 mm BS test sieve.

4.2 APPARATUS

The following apparatus is required.

4.2.1 A metal thickness gauge of the pattern shown in figure 4.1 or special sieves having elongated apertures shown in figure 4.2. The width of the apertures and the thickness of the sheet used in the gauge or sieve shall be as specified in the following figures.



Dimensions are in mm
(Tolerances are given on essential dimensions as shown)

Figure 4.1 Thickness gauge

Figure 4.2 Sieves having elongated apertures

4.2.2 BS test sieves as shown in Table 4.1 (see also 7.1.3.1 of BS 812 Part 1:1975).

4.2.3 A balance accurate to 0.5% of the mass of the test sample.

4.3 SAMPLE FOR TEST

The sample for this test shall be taken in accordance with clause 5 of this part of this standard. It shall comply with the appropriate minimum mass given in Table 4.1, for sieve analysis with due allowance for later rejection of the particles retained on a 63.0 mm BS test sieve and passing 6.30 mm BS test sieve. The sample shall be taken from the laboratory sample by quartering or by means of a sample divider as described in 5.2.4 of BS 812 Part 1. Before testing it shall be brought to a dry condition in accordance with 5.1.4 of BS 812 Part 1.

Table 4.1: Dimensions of thickness gauges

Aggregate size fraction		Thickness gauge Width of slot of (Average x 0.6)	Minimum mass for subdivision
BS test sieve nominal aperture size			
100% passing	100% retained		
mm	mm	mm	Kg
63.0	50.0	33.9 ±0.3	50
50.0	37.5	26.3 ±0.3	35
37.5	28.0	19.7 ±0.3	15
28.0	20.0	14.4 ±0.15	5
20.0	14.0	10.2 ±0.15	2
14.0	10.0	7.2 ±0.10	1
10.0	6.30	4.9 ±0.1	0.5

4.4 PROCEDURE

Carry out a sieve analysis in accordance with BS 812: section 105.1using the sieve given in Table 4.1.

Discard all aggregate retained on the 63.0 mm BS test sieve and all aggregate passing the 6.30 mm BS test sieve.

Then weigh each of the individual size fractions retained on the sieves, other than the 63 mm BS test sieve and store them in separate trays with their size marked on the trays.

NOTE: Where the mass of any size fraction is considered to be excessive, i.e. more than the appropriate mass given in Table 4.1. Provided that the mass of the sub-divided fraction is not less than half the appropriate mass given in Table 4.1. Under such circumstances the rest of the procedure should be suitably modified and the appropriate correction factor applied to determine the mass of flaky particles that would have been obtained had the whole of the original size fraction been gauged.

From the sums of the masses of the fractions in the trays (M_1), calculate the individual percentage retained on each of the various sieves. Discard any fraction of which the mass is 5% of less of mass M_1. Record the mass remaining (M_2).

Gauge each fraction by one of the following procedures:

(a) Using the gauge: select the thickness gauge appropriate to the size-fraction under test (see Table 4.1) and gauge each particle separately by hand, or

(b) Using the special sieves: select the special sieve appropriate for the size-fraction under test. Place the whole of the size-fraction into the sieve which shall then be shaken until the majority of flaky particles have passed through the slots. Then gauge the particles retained individually by hand.

Combine and weigh all the particles passing the gauges or special sieves (M_3).

4.5 CALCULATIONS AND REPORTING

Flakiness index = $M_3/M_2 \times 100$

The flakiness index shall be reported to the nearest whole number. The sieve analysis obtained in this test shall also be reported.

Experiment No: 04
Determination of the Flakiness Index

Name : **Student No**:

Type of material : Brick Chips/ Stone Chips/ Gravels/ Boulder/ Rock
Test Method : Bs 812 (part 1) Clause 7.3 & 7.4

Sieve size (mm)	Gauge size used, (mm)	Wt. of the material retained (gm)	Percent of the material retained	Check if greater than 5% (ok/ not ok)	Flaky Particles (amount passed) gm
63.0	-	x	x		
50.0	33.9				
37.5	26.3				
28.0	19.7				
20.0	14.4				
14.0	10.2				
10.0	7.2				
6.3	4.9				
		M_1 =	**M_2 =**		**M_3 =**

Calculation:

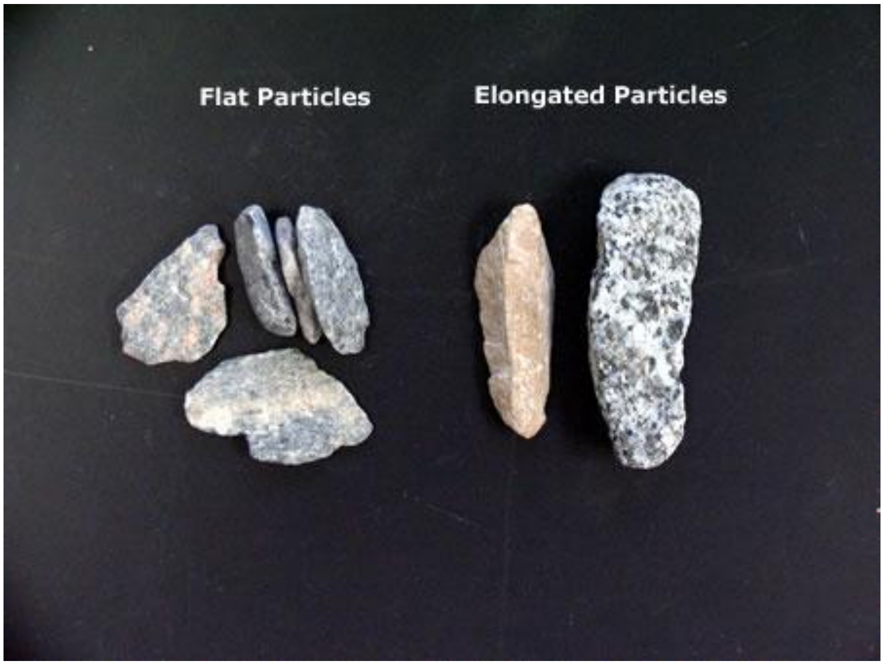
Flakiness index (F.I) = M_3/M_2 x 100% = % (to the nearest whole number)

Signature of the Course Teacher

EXPERIMENT NO: 05

DETERMINATION OF ELONGATION INDEX

(BS: 812: 1975 (PART 1, 2, 3))



5.1 GENERAL

This method is based on the classification of aggregate particles as elongated when they have a length (greatest dimension) of more than 1.8 of their nominal size, this size being taken as the mean of the limiting sieve apertures used for determining the size fraction in which the particle occurs.

The elongation index of an aggregate sample is found by separating the elongated particles and expressing their mass as a percentage, of the mass of the sample tested. The test is not applicable to material passing a 6.30 mm BS test sieve or retained on a 50 mm BS test sieve.

5.2 APPARATUS

The following apparatus is required.

5.2.1 A metal length gauge of the pattern shown in figure 5.1. The gauge lengths shall be those specified in the length gauge column of table 5.1.

5.2.2 BS test sieves as shown in Table 5.1 as appropriate (see also 7.1.3.1 of BS Part 1:1975).

5.2.3 A balance accurate to 0.5% of the mass of the test sample.

5.3 SAMPLE FOR TEST

The sample for this test shall be taken in accordance with clause 5 of this Part of this standard. It shall comply with the appropriate minimum mass given in Table 5 of BS 812 Part 1:1975, for sieve analysis, with due allowance for later rejection of particles retained on a 50.0 mm BS test sieve and passing a 6.30 mm BS test sieve. The sample shall be taken from the laboratory sample by quartering or by means of a sample divider as described in 5.2.4 of BS 812 Part 1:1975. Before testing it shall be brought to a dry condition in accordance with 7.1.4 of BS 812 Part 1:1975.

Table 5.1: Dimensions of length gauges

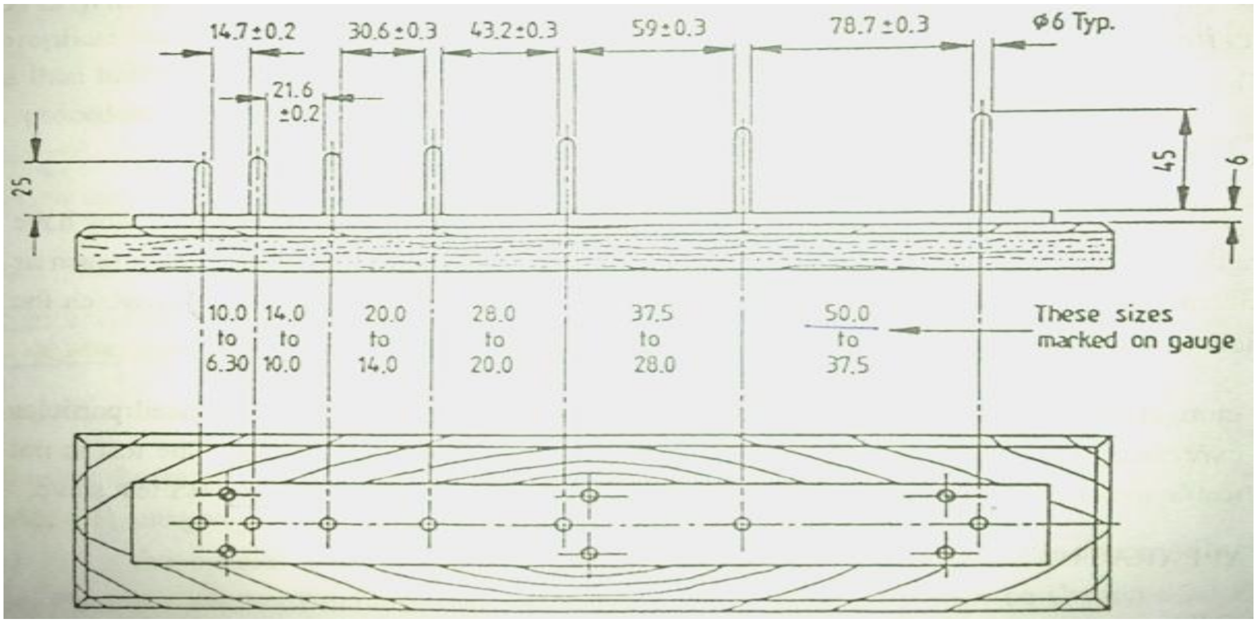
Aggregate size fraction		Length gauge Gap between pins (Average x 1.8)	Minimum mass for subdivision
BS test sieve nominal aperture size			
100% passing	100% retained		
mm	mm	mm	Kg
63.0	50.0	-	50
50.0	37.5	78.0 ± 0.3	35
37.5	28.0	59.0 ± 0.3	15
28.0	20.0	43.2 ± 0.3	5
20.0	14.0	30.6 ± 0.3	2
14.0	10.0	21.6 ± 0.2	1
10.0	6.30	14.7 ± 0.2	0.5

5.4 PROCEDURE

Carry out a sieve analysis in accordance with BS 812: section 105.2using the sieves shown in Table 5.1.

Discard all aggregate retained on the 50.0 mm BS test sieve and all aggregate passing the 6.30 mm BS test sieve.

Weigh and store each of the individual size-fractions retained on the other sieves in separate trays with their size marked on the tray.



Dimensions are in mm
(Tolerances are given on essential dimensions as shown)

Figure 5.1 Metal length gauge

NOTE: Where the mass of any size fraction is considered to be excessive, i.e. more than the appropriate mass given in Table 5.1, the fraction may be subdivided by the methods described in 5.2.4 of BS 812 Part 1:1975 provided that the mass of the subdivided fraction is not less than half the appropriate mass given in Table 5.1. Under such circumstances the rest of the procedure should be suitably modified and the appropriate correction factor applied to determine the mass of elongated particles that would have been obtained had the whole of the original size-fraction been gauged.

From the sum of the masses of the fractions in the trays (M_1), calculate the individual percentages retained on each of the various sieves. Discard any fraction whose mass is 5% or less of mass M_1. Record the mass remaining (M_2).

Select the length gauge appropriate to the size-fraction under test (see Table 5.1) and gauge each particle separately by hand. Elongated particles are those whose greatest dimension prevents them from passing through the gauge. Combine and weigh all elongated particles (M_3).

5.5 CALCULATIONS AND REPORTING

Elongation index = $M_3/M_2 \times 100$

The elongation index shall be reported to the nearest whole number. The sieve analysis obtained in this test shall also be reported.

Experiment No: 05
Determination of the Elongation Index

Name : **Student No**:

Type of material : Brick Chips/ Stone Chips/ Gravels/ Boulder/ Rock
Test Method : Bs 812 (part 1) Clause 7.3 & 7.4

Sieve size (mm)	Gauge size used, (mm)	Wt. of the material retained (gm)	Percent of the material retained	Check if greater than 5% (ok/ not ok)	Elongated Particles (amount retained) gm
50.0	-	x	x		
37.5	78.0				
28.0	59.0				
20.0	43.2				
14.0	30.6				
10.0	21.6				
6.3	14.7				
		M_1 =	**M_2 =**		**M_3 =**

Calculation:

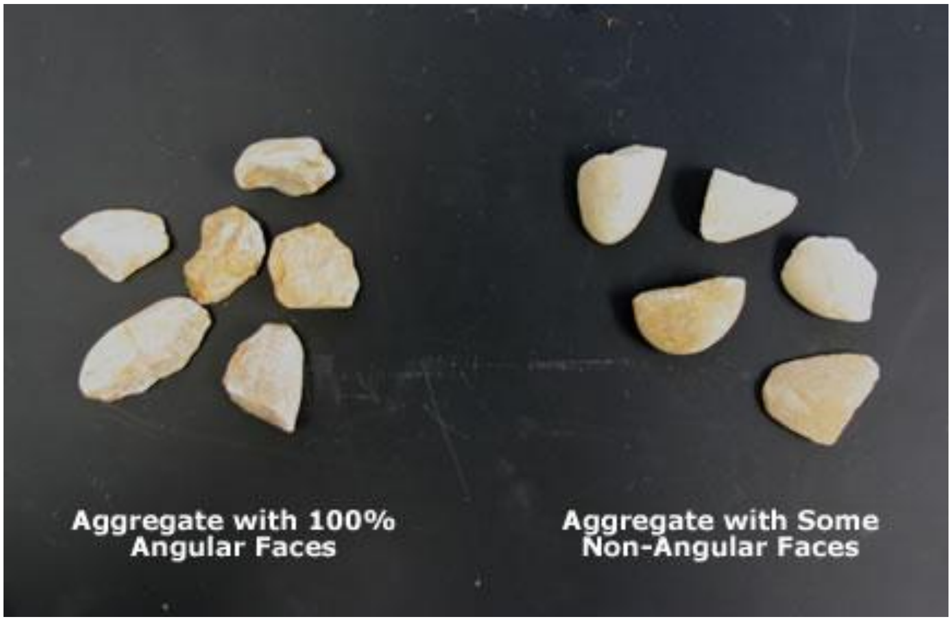
Elongation index (E.I) = M_3/M_2 x 100% = % (to the nearest whole number)

Signature of the Course Teacher

EXPERIMENT NO: 06

DETERMINATION OF ANGULARITY NUMBER

(BS: 812: 1975 (PART 1, 2, 3))



6.1 GENERAL

The angularity number is determined from the proportion of voids in a sample of aggregate after compaction in the specified manner. This property is used mainly in the design of mix proportions and in research.

Angularity or absence of rounding of the particles of an aggregate is a property which is of importance because it affects the ease of handling of a mixture of aggregate and binder (e.g. the workability of concrete) or the stability of mixtures that rely on the interlocking of the particles. The least angular (most rounded) aggregates are found to have about 33% voids and the angularity number is defined as the amount by which the percentage of voids exceeds 33. The angularity number ranges from 0 to about 12.

Since considerably more compactive effort is used than in the test for bulk density and voids (see 6.1 of part 2) the percentage of voids will be different. Weaker aggregates may be crushed during compaction and the results will be anomalous if this method is applied to any aggregate which breaks down during the test.

6.2 SAMPLING

The sample for this test shall be taken in accordance with clause 5 of this part of this standard.

6.3 APPARATUS

The following apparatus is required:

6.3.1 A metal cylinder closed at one end , of about 0.003 m^3 volume, the diameter and height of which should be approximately equal (e.g. 150 mm and 150 mm).

The cylinder shall be made from metal of a thickness not less than 3 mm and shall be of sufficient rigidity to retain its shape under rough usage.

6.3.2 A straight metal tamping rod of circular cross section 16 mm in diameter and 600 mm long rounded at one end.

6.3.3 A balance or scale of capacity 10 kg, accurate to 1 g.

6.3.4 A metal scoop approximately 200 mm x 120 mm x. 50 mm (i.e. about 1 liter heaped capacity).

6.3.5 BS perforated plate test sieves from 20.0 mm to 5.0 mm aperture size (but see note to section 3.5.5(a)).

6.4 CALIBRATION OF THE CYLINDER

The cylinder shall be calibrated by determining to the nearest gram the mass of water at $20^0 \pm 2^0$C required to fill it up so that no meniscus is present above the rim of the container (mass C).

6.5 PREPARATION OF THE TEST SAMPLE

The sets sample shall be prepared as follows.

a) The amount of aggregate available shall be sufficient to provide, after separation on the appropriate pair of sieves, at least 10 kg of the predominant size as determined by sieve analysis on the BS test sieves.

The test sample shall consist of aggregate retained between the appropriate pair of BS test sieves from the following list:

20.0 mm and 14.0 mm; 14.0 mm and 10.0 mm; 10.0 mm and 6.3 mm; 6.3 mm and 5.0 mm.

Note: In testing aggregates larger than 20.0 mm the volume of the cylinder should be greater than 0.003 m^3, but for aggregates smaller than 5.0 mm a smaller cylinder may be used. The procedures should be the same as with the 0.003 m^3 cylinder, except that the amount of compactive effort (mass) should be proportioned to the volume of the cylinder used.

b) The aggregate to be tested shall be dried for at least 24 hr in shallow trays in a well ventilated oven at a temperature of 105 ± 5^0C, cooled in an air tight container and tested.

6.6 TEST PROCEDURE

Fill the scoop and heap it to overflowing with the aggregate, which shall be placed in the cylinder by allowing it to slide gently off the scoop from the least height possible.

Subject the aggregate in the cylinder to 100 blows of the tamping rod at a rate of about two blows per second. Apply each blow by holding the rod vertical with its rounded end 50 mm above the surface of the aggregate and releasing it so that it falls freely. Do not apply any force to the rod. Evenly distribute the 100 blows over the surface of the aggregate.

Repeat the process of filling and tamping exactly as described above with a second and third layer of aggregate; the third layer shall contain just sufficient aggregate to fill the cylinder level with the top edge before tamping.

After the third layer of aggregate has been tamped, fill the cylinder to overflowing, and strike off the aggregate level with the top, using the tamping rod as a straight-edge.

Then add individual pieces of aggregate and roll them in, to the surface by rolling the tamping rod across the upper edge of the cylinder, and continue this finishing process as, long as the aggregate does not lift the rod off the edge of the cylinder on either side. Do not push in or otherwise force down the aggregate, and apply no downward pressure to the tamping rod, which shall roll in contact with the metal on both sides of the cylinder. Then weigh the aggregate in the cylinder to the nearest 5 g.

Make three separate determinations and calculate the mean mass of aggregate in the cylinder (mass M). If the result of any one determination differs from the mean by more than 25 g, three additional determinations shall immediately be made on the same material, and the determinations calculated (mass M).

6.7 CALCULATIONS

The angularity number of the aggregate shall be calculated from the equation:

Angularity number = $67 - \frac{100\ \mathrm{M}}{\mathrm{CG}}$

Where,

M is the mean mass of aggregate in the cylinder (g);
C is the mass of water required to fill the cylinder (g);
G is the relative density on an oven-dried basis of the aggregate determined in accordance with clause 5 of part 2 of this standard.

6.8 REPORTING OF RESULTS

The angularity number shall be reported to the nearest whole number.

Experiment No: 06
Determination of Angularity Number

Name : **Student No**:

Type of material : Brick Chips/ Stone Chips/ Gravels
Test Method : Bs 812; Part 1 Clause: 7.5 & BS 812, Part 2 Clause: 5

Serial No.	Mass of Aggregate in Cylinder (gm)	Mean Mass of Aggregate in the Cylinder, (M) (gm)	Relative Density of the Aggregate (Oven dry basis) (G)	Water Required to Fill the Cylinder, (C) (gm)	Angularity Number (A.N)
1					
2					
3					

Calculation:

Angularity Number (A.N) = $67 - \frac{100\,M}{CG} =$ **(to the nearest whole number)**

Signature of the Course Teacher

EXPERIMENT NO: 07

DETERMINATION OF ROADWAY CAPACITY

(Highway Capacity Manual (HCM)-1994, FHA, USA)

7.1 INTRODUCTION

The capacity of a roadway is its ability to accommodate traffic. It is usually expressed as the number of vehicles that can pass a given point in a certain time at a given speed.

Of course roadways are not ideal and prevailing roadway and traffic conditions those reduce ability of a road to accommodate traffic must be taken into consideration in highway capacity estimation. In determining highway capacities for uninterrupted flow conditions the general procedure, described below, is to apply appropriate empirically based adjustments for prevailing roadway and traffic conditions. The capacity of a given section of roadway stated either as unidirectional or both directions for a two lane or three lane roadway, may be defined as maximum number of vehicle that has a reasonable expectation of passing over a given section of roadway during a given time period under prevailing roadway and traffic condition.

While the maximum number of vehicle that can be accommodated remains fixed under similar roadway and traffic conditions, there is a range of lesser volumes which can be handled under differing operating conditions. Operation at capacity provides the maximum volume, but as both volume and congestion decrease there is an improvement in the level of service.

Level of service denotes any of an infinite number of differing combinations of operating conditions that can occur on a given lane or roadway when it is accommodating various traffic volumes.

Six levels of service (LOS), Level A through F, define full range of driving conditions from the best to worst in that order.

Level of service “A” represent free flow at low densities with no restriction due to traffic conditions. Level “B”, the lower limit of which is often used for the design of rural highways, is the zone of stable flow with some slight restriction of driver freedom. Level “C” denotes some of stable flow with more marked restriction in driver’s selection of speed and with reduced ability to pass. The conditions of level “D” reflect little freedom for driver maneuverability and condition approaches unstable flow. Level “E” area of unstable flow, is the zone of low operating speed and volume. Level “F” is the level of service provided by the familiar traffic jam, with frequent interruptions and breakdown of flow as well as volumes below capacity, coupled with low operating speed.

7.2 CAPACITY UNDER UNINTERRUPTED CONDITION

The capacity of roadway under uninterrupted flow condition can be obtained by modifying the capacity of the roadway under ideal conditions. Table 7.1 shows capacity of highway under ideal conditions according to Highway Capacity Manual (HCM)-1994 (USA).

These ideal conditions comprise uninterrupted flow, with no interference by side traffic or obstructions, a vehicle stream composed solely of passenger vehicles, 12 ft wide traffic lane, and should be capable of providing operation at 70 mph, with no restriction of passing sight distance.

Table 7.1: Capacity of roadway as per HCM-1994

Roadway Type	Capacity (passenger Vehicle/hr)
Multi-lane Free Flow	2000 per lane
Two lane, two way	2000 total both direction
Three lane , two way	4000 total both direction

The adjustment factors used in modifying the capacity under ideal conditions to give capacity and service volumes under prevailing conditions may be grouped under

1. Roadway factors

2. Traffic factors

Table 7.2 gives the scale of operating characteristics established for various levels of service on 2 lane highways and summarizes general level of service criteria during uninterrupted flow conditions. Included, in addition to operating speeds and basic volume/capacity ratios for ideal alignment, are approximate measures of the influences of passing sight distance, expressed as a percentage of the total section that is adequate if greater than 1500 ft and of average highway speeds on volume/capacity ratios.

Table 7.2: Operating characteristics for various levels of service on 2 lane highways and general level of service criteria during uninterrupted flow conditions as per HCM-1994

Level of Service	Traffic Flow Condition		Passing Sight Distance (%) >1500 ft	Basic Limiting Value for AHS* of 70 mph	Maximum service value under ideal condition
	Description	Operating Speed (mph)			
A	Free Flow	≥ 60	100	0.20	400
			80	0.18	
			60	0.15	
			40	0.12	
			20	0.08	
			0	0.04	
B	Stable	≥ 50	100	0.45	900
			80	0.42	
			60	0.38	
			40	0.34	
			20	0.30	
			0	0.24	
C	Approaching unstable flow	≥40	100	0.70	1400
			80	0.68	
			60	0.65	

			40 20 0	0.62 0.59 0.54	
D	Approaching unstable flow	≥ 35	100 80 60 40 20 0	0.85 0.84 0.83 0.82 0.81 0.80	1700
E	Unstable Flow	30	Not Applicable	<=<1	2000
F	Forced Flow	<30	Not Applicable	Not Meaningful	Widely Variable (0 to capacity)

*AHS = Average highway speed

7.2.1 Roadway factors

The roadway factors make allowances for the effects of design elements such as lane width, lateral clearances at the edge of lanes, alignment and grades. Table 7.3 shows the effect of reduced lane widths on capacity and Table 7.4 shows the effect of edge clearance. Under both conditions the driver has a feeling of restricted movement with limited clearance resulting reduced capacity and lower level of service.

Capacity of a roadway is also highly influence by width of shoulder. For safe operation and to develop full traffic capacity well maintained shoulder is required. Outside shoulder should be at least 10 ft preferably 12 ft and free of all obstructions for heavily traveled, high speed roadways. Fully implemented, firm and smooth shoulders increase traffic lane-width by 2 ft.

Table 7.3: Effect of lane width on capacity for uninterrupted flow conditions as per HCM-1994

Lane Width (ft)	**Capacity %**	
	Two-lane Roadways	**Multi Lane Roadways**
12	100	100
11	88	97
10	81	91
09	76	81

Table 7.4: Effective roadway width due to restricted lateral clearance under uninterrupted flow as per HCM-1994

Clearance from Pavement to Observation, Both Sides (ft)	**Effective Width of Two 12-ft lane (ft)**	**Capacity of Two 12-ft Lanes (%)**
6	24	100
4	22	92
2	20	83
0	17	72

7.2.2 Traffic factors

In addition to roadway factors, traffic factors (such as many trucks and buses in the traffic stream and variation of flow) affect the capacity and service columns of a road way. Trucks and busses, because of their restricted maneuverability, reduce the number of vehicles that a facility can handle. This reduction in vehicle is represented by the term passenger can equivalent (PCE), which indicates the equivalent number of passenger cars that have been displaced by the presence of each truck or bus, Table 7.5 shows passenger car equivalents for different types of vehicles as mentioned by the Roads and Highways Department.

Table 7.5: PCU values for different types of vehicles

Vehicle Type	Passenger Car Unit (PCU) Value
Truck	3
Bus	3
Mini-bus	2
Passenger car	1
Utility	1
C.N.G, Baby taxi	0.75
Motorcycle	0.5
Bicycle	0.5
Cycle Rickshaw	2

Bullock Cart	4

(Source: Geometric Design Standards for Roads & Highways Department, Draft version-4, October 2004, Page-4)

The method of estimation of highway capacity described in this article is applicable to rural highway situation only. In urban conditions, the roadway capacity mainly depends on the discharge capacity of the intersections. The commonly used intersections are: signalized, priority-controlled un-signalized and roundabout/traffic circles. However, considering the difficulties in arranging a field data collection trip to a representative rural highway section, required data will be collected from city roads. Therefore, it is suggested that an intersection free long stretch of road should be taken as the field site in order to get closer to the rural highway situation.

EXAMPLE

Find the capacity of a roadway section for the following data:

1. Roadway pattern : Two lane two way
2. Lane width : 11 ft
3. Shoulder condition: 4 ft on both side of roadway smooth and well maintained
4. Operating speed : 40 mph
5. % of passing sight distances: 80
6. Level of service C

Solution:

From Table 7.1 and Table 7.2
Capacity of two lane two way = 2000× 0.68 = 1360 veh/hr.

From Table 7.3,
Capacity reduction factor for 11 ft lane width =0.88

From Table 7.4 :
Capacity reduction factor for 4 ft shoulder = 0.92
Therefore actual roadway capacity = 1360×0.88×0.92 = 1101 passenger veh/hr. (total in both direction)

Experiment No: 07
Determination of Roadway Capacity

Name : **Student No**:

Find the capacity of a roadway section for the following data:

1. Roadway pattern :

2. Lane width :

3. Shoulder condition :

4. Operating speed :

5. % of passing sight distances :

6. Level of service :

Calculation:

Signature of the Course Teacher

EXPERIMENT NO: 08

A METHOD FOR MEASURING SATURATION FLOW AT TRAFFIC SIGNALS

(THE ROAD NOTE 34)

8.1 INTRODUCTION

When the green period at a traffic signal commences vehicles take a few seconds to accelerate to normal running speed, but after this initial period the queue discharges at a more or less constant rate. This rate is called the saturation flow and is usually expressed in vehicles per hour of green time. While the signal is green, vehicles continue to pass through the intersection at the saturation rate of flow, subject to the existence of stable queue. Some vehicles, but not all, make use of the amber period to cross the intersection and the average discharge rate falls to zero toward the end of this period.

The green plus the amber period can thus be divided, theoretically, into an “effective green time” during which traffic flows at the ‘saturation’ rate and a ‘ lost time’ during which no flow occurs. The Road Note 34 describes a practical method of measuring saturation flow and lost time. The method consists of recording the number of vehicles discharged from a waiting queue in successive short intervals of the green period. The Note gives practical hints on how to decide when saturation flow ceases and on the treatment of non-standard intersections.

The analysis of data from a typical field data sheet is followed step by step. Passenger Car Equivalence of vehicles is given in order to be able to convert the saturation flow to passenger car units if the composition of the traffic is known.

8.2 BASIS OF THE ROAD NOTE 34 METHOD

In this method, the time “green period” refers to the green plus the amber period, i.e. the period when no red signal is shown.

A typical example of the variation of flow past the stop-line in successive short intervals of a fully saturated green period (i.e. a green period during which the queue is not fully discharged) is given in Figure 8.1. The effects on flow of starting delays and of the amber period are clearly shown. The average level of flow in these saturated intervals of the green period but excluding the beginning and end intervals is taken as the saturation flow. The graph can be simplified to the rectangular form shown by the broken line, the height of which is equal to the saturation flow and the area to the total number of vehicles discharged during a fully saturated green period. The width of the rectangle is called the effective green time and the difference between the actual green period (including amber) and this quantity is the lost time. This method of representing the discharge of the queue simplifies the calculation of delay and capacity because the number of vehicles discharged in a fully saturated green period is then directly proportional to the effective green time. Values calculated on this basis have been found to be in good agreement with those observed.

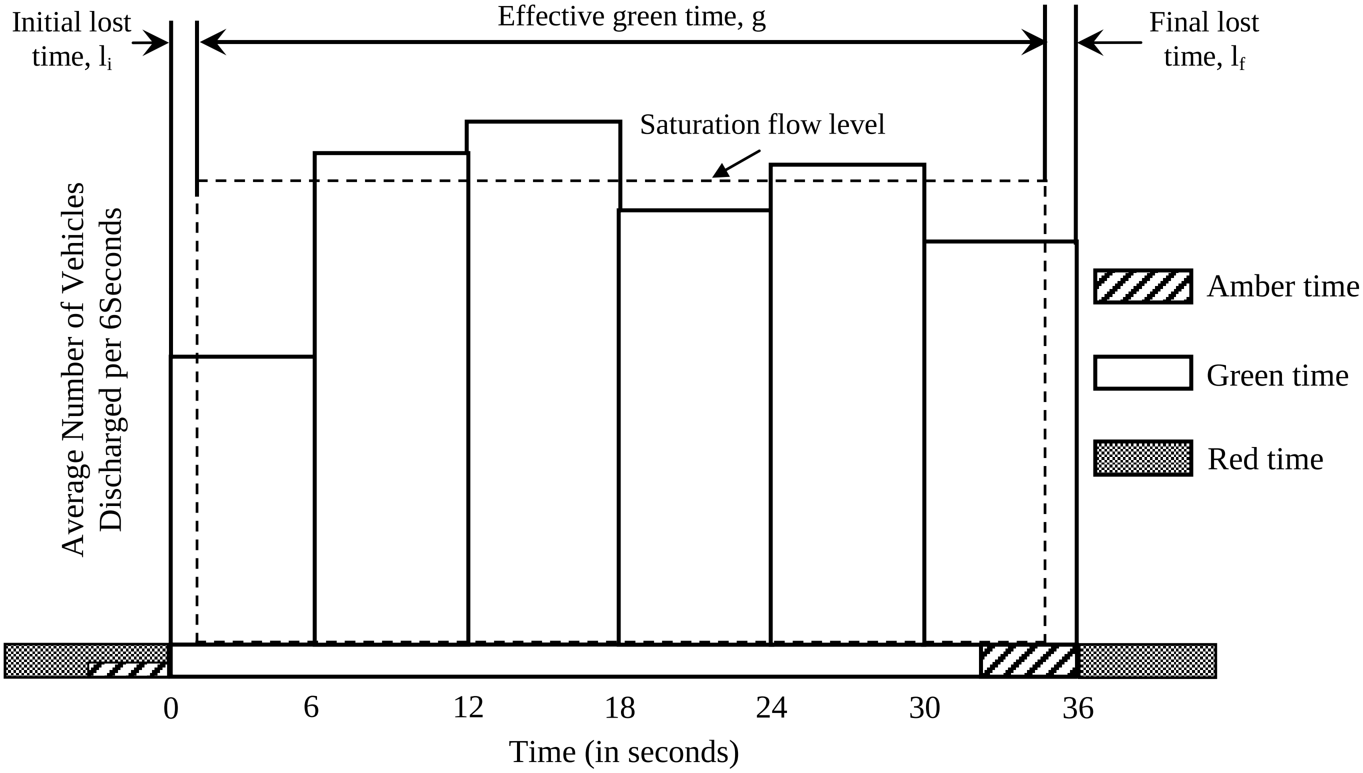


Figure 8.1 A typical example for fixed-time signals showing the variation of average discharge rate during a fully saturated green period

8.3 DATA COLLECTION PROCEDURE

- ❖ Select a suitable approach of an intersection where
  - flow condition is at the saturation level
  - at the road side and near the stop line of the approach there is a convenient place for collecting data

❖ Take total cycle time and green + amber period. Divide the combined green plus amber time by 6 in order to find the number of 6 sec intervals and the duration of last interval.

❖ Counting should be taken at the stop line (if there is no slop line a convenient reference line should be marked on the road).

❖ Start counting at the commencement of green signal and continue till the end of amber period.

❖ For each 6 sec interval, the classified vehicle counts should be recorded on the given form.

(See Table 8.1 for vehicle classification)

- For counting purpose, when rear wheel of a vehicle will cross the stop line, it should be included in the count for that particular interval.

- Recording of the flows should be discontinued, when the flow is no longer at the saturation level. (End of saturation level means — when queue disappears and vehicles discharges without stopping).

- Although the counting must stop at the end of the amber, any vehicle crossing on the red must be included in the last interval.

- Any vehicle that cross the observation point but fails to complete their journey through the intersection must not be counted until the next green period has started.

- Repeat vehicle counts for at least 10 cycles.

- Using Table 8.3, convert vehicles in terms of PCU values for each interval.

- Determine average PCU for each interval

- Draw histogram (i.e. discharge profile)

- Determine saturation flow by taking the height of the rectangle in each interval (excluding the first and last)

- Calculate lost times, $l = t - (n/s)$

  Where, n = no. of vehicles discharge in initial/final interval (in PCU)
  s = saturation flow, PCU/sec
  t = duration of initial/final interval (in seconds)

- Calculate effective green lime, $g = G+A-(l_i + l_f)$

  Where, G = observed green period
  A = observed amber period
  l_i = Initial lost time
  l_f = final lost time

Approach Capacity = $g/c \times s$

Where, c = cycle time

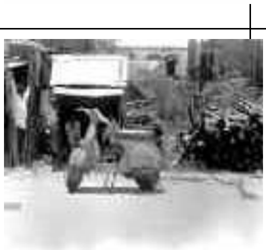
Table 8.1: Short description of various types of vehicles.

Category	Type	Description
1	Heavy Truck	Three or more axles. Includes multi-axle tandem trucks, container carriers and other articulated vehicles.
2	Medium Truck	All 2-axle rigid trucks over three tonnes payload. Typical medium trucks are the Hindustan Bedford, “English” Bedford and Hino trucks of about 10 tonnes gross vehicle weight. Agricultural tractors and trailers are also included in this category.
3	Light Truck	Small trucks up to 3 tonnes payload. The most typical example is the Jeep based conversion.
4	Large Bus	More than 40 seats on 36 foot or longer chassis. Includes double decker buses.
5	Minibus	Between 16 and 39 seats. Typical minibuses are the TATA 909 and Hindustan Mascot.
6	Microbus	Up to 16 seats. Typical microbuses are the 12/15 seat Toyota Hi-ace, and the Mitsubishi L300.
7	Utility	Pick-ups, jeeps and four wheels drive vehicles, such as Pajero’s and Land Rover’s.
8	Car/Taxi	All types of car used either for personal or taxi services.
9	Baby-taxi	Includes Baby taxi and Mishuks
10	Tempo	Auto-Tempo and Auto-Vans.
11	Motor Cycle	All two wheeled motorized vehicles.
12	Bicycle	All pedal cycles.
13	Rickshaw Standard	Three wheeled cycle rickshaws (not rickshaw vans)
14	Rickshaw Van	Rickshaw vans
15	Cart	All animal and manually drawn/pushed carts.

(Source: Roads and highways department)

Table 8.2: Vehicle identification sheet

No.	CATEGORY	CHARACTERISTICS	TYPICAL VEHICLES (TRUCKS AND BUSES)		
1	HEAVY TRUCK	3 OR MORE AXLES			
2	MEDIUM TRUCK	2 AXLES OVER THREE TONNES UNLOADED WEIGHT			
3	LIGHT TRUCK	2 AXLES UNDER THREE TONNES UNLOADED WEIGHT			
4	LARGE BUS	OVER 39 SEATS			
5	MINI BUS	16-39 SEATS			
No.	CATEGORY	CHARACTERISTICS	TYPICAL VEHICLES (LIGHT MOTORISED VEHICLES)		
6	MICROBUS	LESS THAN 16 SEATS			

7	UTILITY	PICK UPS AND FOUR WHEEL DRIVE VEHICLES			
8	CAR	ALL CARS AND TAXIS			
9	AUTO RICKSHAW	ALL THREE WHEELED MOTORISED VEHICLES			
10	MOTOR CYCLE	ALL TWO WHEELED MOTORISED VEHICLES			
No.	CATEGORY	CHARACTERISTICS	TYPICAL VEHICLES (NON MOTORISED VEHICLES)		
11	BICYCLE	PUSH BICYCLE			
12	CYCLE RICKSHAW	ALL THREE WHEELED NON MOTORISED VEHICLES			
13	CART	ALL ANIMAL AND PERSON DRAWN/PUSHED CARTS			

(Source: Roads and highways department)

Table 8.3 (a): PCU estimates without non-motorized vehicles in the traffic stream

Proportion of NMV 0%	6 seconds intervals			
	3.5m	7.0m	10.5m	14.0 m
Motor-Cycle	0.02	0.06	0.03	0.01
Auto-Rickshaw	0.70	0.36	0.41	0.44
Tempo	0.76	0.41	0.51	0.55
Mini- Bus/Truck	1.43	1.45	1.53	1.40
Truck	1.99	1.97	2.12	2.28
Bus	1.96	1.95	1.99	2.09

(Source: Roads and highways department)

Table 8.3 (b): PCU estimates with 10% non-motorized vehicles in the traffic stream

Proportion of NMV 10%	6 seconds intervals		
	7.0m	10.5m	14.0 m
Cycle	0.18	0.59	0.63
Rickshaw	1.23	1.12	0.91
Push-cart	2.77	2.76	2.24
Motor-Cycle	0.01	0.11	0.00
Auto-Rickshaw	0.20	0.22	0.30
Tempo	0.27	0.28	0.38
Mini- Bus/Truck	1.02	1.12	1.18
Truck	1.69	1.98	2.07
Bus	1.45	1.93	1.77

(Source: Roads and highways department)

Table 8.3 (c): PCU estimates with 20% non-motorized vehicles in the traffic stream

Proportion of NMV 20%	6 seconds intervals		
	7.0 m	10.5 m	14.0 m
Cycle	0.31	0.41	0.38
Rickshaw	0.76	0.81	0.85
Push-cart	1.48	1.58	1.71
Motor-Cycle	0.64	0.12	0.04
Auto-Rickshaw	0.24	0.36	0.37
Tempo	0.32	0.42	0.41
Mini- Bus/Truck	1.01	1.06	1.04
Truck	1.25	1.14	1.66
Bus	1.14	1.16	1.23

(Source: Roads and highways department)

Table 8.3 (d): PCU estimates with 30% non-motorized vehicles in the traffic stream

Proportion of NMV 30%	6 seconds intervals		
	7.0 m	10.5 m	14.0 m
Cycle	0.19	0.24	0.21
Rickshaw	0.60	0.53	0.48
Push-cart	1.28	1.71	1.10
Motor-Cycle	0.22	0.24	0.06
Auto-Rickshaw	0.17	0.29	0.22
Tempo	0.23	0.36	0.38
Mini- Bus/Truck	0.84	0.92	0.90
Truck	1.69	1.46	1.59
Bus	1.41	1.60	1.66

(Source: Roads and highways department)

Experiment No: 08

A Method for Measuring Saturation Flow at Traffic Signals

Name : **Student No**:

TRAFFIC COUNT FOR SATURATION FLOW CALCULATION
OF ……………………..…………………… INTERSECTION

C =
G =
A =

No. of vehicles per …….. Sec interval		1	2	3	4	5	No. of Vehicles in total 5 cycle	PCU factor	Converted PCU in total 5 cycle	Total PCU	Sample	Average
0												
…………												
………….												

....												
....												
....												
....												

……........												
………….												
………….												
………….												
………….												

EXPERIMENT NO: 09

SPECIFIC GRAVITY OF SEMI-SOLID BITUMINOUS MATERIAL

(AASHTO DESIGNATION : T 228-93
ASTM DESIGNATION : D 70-76)

1. SCOPE

1.1 This method covers the determination of the specific gravity of semi-solid bituminous materials, asphalt cements, and soil tar pitches by use of a pycnometer.

2. SPECIFIC GRAVITY

2.1 The specific gravity of semi-solid bituminous materials, asphalt cements, and soft tar pitches shall be expressed as the ratio of the mass of a given volume of the material at 25^0C(77^0F) or at 15.6°C (60°F) to that of an equal volume of water at the same temperature, and shall be expressed thus:

Specific gravity, 25/25°C (77/77^0F) or 15.6/15.6°C (60/60°F)

3. APPARATUS

3.1 Pycnometer, glass, consisting of a cylindrical or conical vessel carefully ground to receive an accurately fitting glass stopper 22 to 26 mm in diameter. The stopper shall be provided with a hole 1.0 to 2.0 mm in diameter, centrally located in reference to the vertical axis. The top surface of the stopper shall be smooth and substantially plane and the lower surface shall be concave in order to allow all air to escape through the bore. The height of the concave section shall be 4.0 to 18.0 mm at the centre. The stoppered pycnometer shall have a capacity of 24 to 30 ml, and shall weigh not more than 40 g.

3.2 Water Bath- Constant temperature, capable of maintaining the temperature within 0.1^0C (0.2^0F) of the test temperature.

3.3 Thermometers- Calibrated liquid-in-glass of suitable range with graduations at least every 0.2°F (0.1°C) and a maximum scale error of 0.2°F (0.1°C) as prescribed in ASTM specification on El. Thermometers commonly used are 63°F or 63°C. Any other thermometer of equal accuracy may be used.
NOTE-1: Other ASTM thermometers (such as the ASTM 17°C) which have sub-divisions and scale errors equal to or smaller than those specified for the ASTM 63^0C and 63^0F may also be used.

3.4 Balance - a balance conforming to the requirements of M 231, Class B.

4. MATERIALS

4.1 Distilled Water - Freshly boiled and cooled distilled water shall be used to fill the pycnometer and the beaker.
NOTE-2: For the purpose of this lest, freshly boiled and cooled distilled, aemineralized or deionized water may be used.

5. PREPARATION OF EQUIPMENT

5.1 Partially fill a 600 ml or larger Griffin low-form beaker with freshly boiled and cooled distilled water to a level that will allow the top of the pycnometer to be immersed to a depth of not less than 40 mm.

5.2 Partially immerse the beaker in the water bath to a depth sufficient to allow the bottom of the beaker to be immersed to a depth of not less than 100 mm, while the top of the beaker is above the water level of the bath. Clamp the beaker in place.

5.3 Maintain the temperature of the water bath within 0.1°C (0.2°F) of the test temperature.

6. CALIBRATION OF PYCNOMETER

6.1 Thoroughly clean, dry, and weigh the pycnometer to the nearest 1 mg. Designate this mass as “A”.

6.2 Fill the pycnometer with freshly boiled distilled water at test temperature and place the stopper in the pycnometer. Do not allow any air bubbles to remain in the pycnometer.

6.3 Allow the pycnometer to remain in the water for a period of not less than 30 min. Remove the pycnometer, immediately dry the top of the stopper with one stroke of a dry towel (Note 3), then quickly dry the remaining outside area of the pycnometer and weigh to the nearest 1 mg. Designate the mass of the pycnometer plus water as “B”.

Note-3: Do not re-dry the top of the stopper even if a small droplet of water forms due to expansion. If the top is dried at the instant of removing the pycnometer from the water, the proper mass of the contents at the test temperature will be recorded. If moisture condenses on the pycnometer during weighing, quickly re-dry the outside of the pycnometer (excluding the top) before recording the mass.
Note-4: Calibration should be done at the specific temperature. A pycnometer calibrated at one temperature cannot be used at a different temperature without recalibration, at that temperature.

Table 9.1: Precision of specific Gravity Data for Semi-Solid Bituminous Materials

	deg C (deg F)	Single-Operator			Multi laboratory		
		Degrees of Freedom	(IS)	(D2S)	Degrees of Freedom	(LS)	(D2S)
Asphalt	15.6(60)	54	0.0011	0.0032	24	0.0018	0.0051
	25.0(77)	54	0.00080	0.0023	24	0.0024	0.0068
Soft tar pitch	15.6(60)	72	0.0013	0.0038	27	0.0029	0.0083
	25.0(77)	72	0.00083	0.0023	27	0.0017	0.0048
Pooled	15.6(60)	114	0.0013	0.0035	51	0.0024	0.0067
	25.0(77)	114	0.00082	0.0023	51	0.0019	0.0053

7. PROCEDURE

7.1 Preparation of Sample - Heat the sample with care, stirring to prevent local overheating, until the sample has become sufficiently fluid to pour. In no case should the temperature be raised to more than 56^0C (100°F) above the expected softening point for tar, or to more than 111°C (200°F) above the expected softening point for asphalt. Do not heat for more than 30 minutes over a flame or hot plate or for more than 2 hours in an oven, and avoid incorporating air bubbles in the sample.

7.2 Pour enough sample into the clean, dry, warmed pycnometer to fill it about three-fourth to its capacity. Take precautions to keep the material from touching the sides of the pycnometer above the final level, and to prevent the inclusion of air bubbles (Note 5). Allow the pycnometer and its contents to cool to ambient temperature for a period of not less than 40 minutes, and weigh with the stopper to the nearest 1 mg. Designate the mass of the pycnometer plus sample as “C”.

NOTE-5: If any air bubbles are inadvertently included, remove by brushing the surface of the asphalt in the pycnometer with a high "soft" flame of a Bunsen burner. In order to avoid overheating, do not allow the flame to remain in contact with the asphalt more than a few seconds at any one time.

7.3 Fill the pycnometer with freshly boiled distilled water at test temperature and place the stopper in the pycnometer. Do not allow any air bubbles to remain in the pycnometer.

7.4 Allow the pycnometer to remain in the water bath for a period of not less than 30 minutes. Remove the pycnometer from the bath. Dry and weigh using the same technique as that employed in Section 6.3. Designate this mass of pycnometer plus sample plus water as “D”.

8. CALCULATIONS

8.1 Calculate the specific gravity to the nearest third decimal as follows:

$$\text{Specific gravity} = \frac{(C-A)}{[(B-A)-(D-C)]}$$

Where :

A = mass of pycnometer (plus stopper)
B = mass of pycnometer filled with water
C = mass of pycnometer partially filled with asphalt, and
D = mass of pycnometer plus asphalt plus water

9. REPORT

9.1 Report the specific gravity to the nearest third decimal at 25/25°C (77°F) or 15.6/15.6^{0}C (60/60°F).

10. PRECISION

10.1 Single-Operator Precision:

10.1.1 The single-operator standard deviation for semi-solid bituminous materials tested at 15.6^0 C (60^{0}F) has been found to be 0.0013 (Note 6). Therefore, results of two properly conducted tests by the same operator should not differ by more than 0.002 (Note 6).

10.2 Multilaboratory Precision:

10.2.1 The multilaboratory standard deviation for semi-solid bituminous materials tested at 15.6^0 C (60^{0}F) has been found to be 0.0024 (Note 6). Therefore, results of two properly conducted tests from two different laboratories on samples of the same material should not differ by more than 0.007 (Note 6).

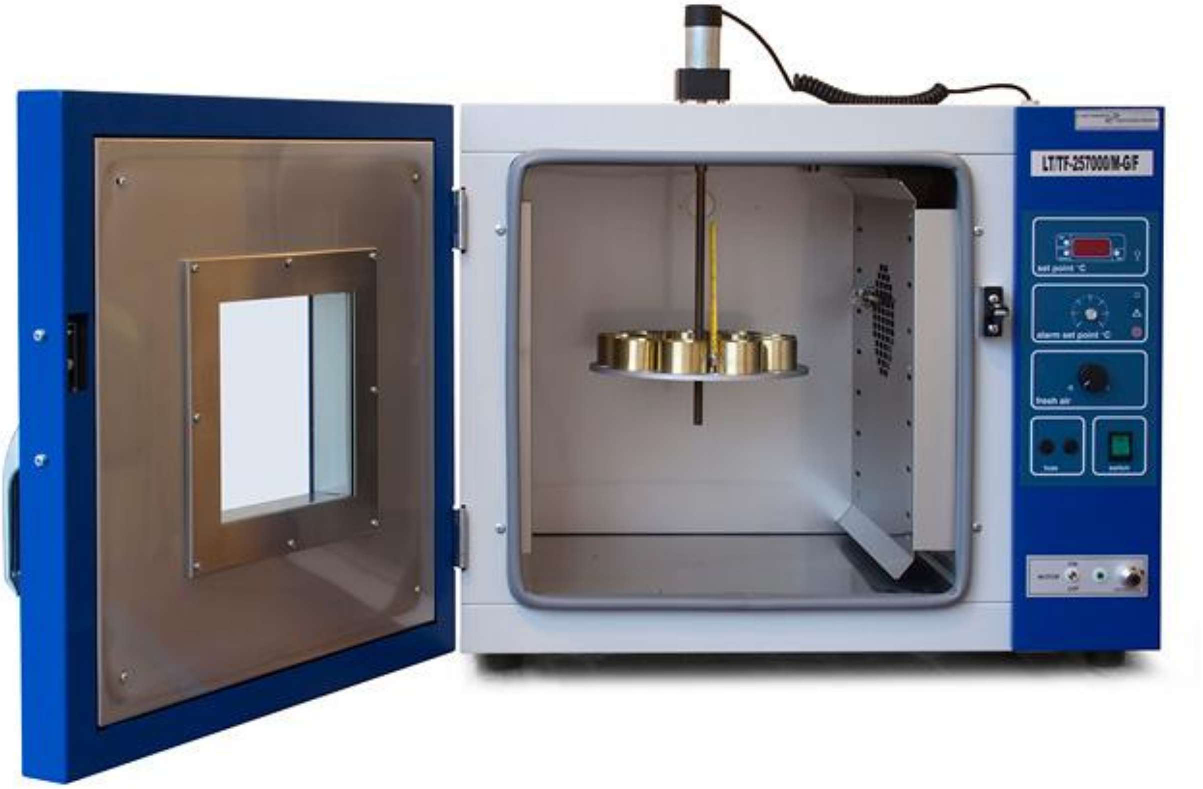
10.2.2 For materials tested at 25^{0}C (77^{0}F) the standard deviation has been found to be 0.0019 (Note 6). Therefore, results of two properly conducted tests from two different laboratories on samples of the same material should not differ by more than 0.005 (Note 6).

NOTE-6: These numbers represent, respectively, the (IS) and (D2S) limits as describe in AASHTO Recommended Practice R2, for Preparing Precision Statements for Test Methods for Construction Materials.

EXPERIMENT NO: 10

LOSS ON HEATING OF OIL AND ASPHALTIC COMPOUND

(AASHTO DESIGNATION : T 47-83 (1993)
ASTM DESIGNATION : D 6-80)



1. SCOPE

1.1 This method covers the determination of the loss in mass (exclusive of water) of oil and asphaltic compounds when heated as herein after prescribed.

2. REFERENCED DOCUMENTS

2.1 ASTM Standards:
E1 Specification for ASTM Thermometers
E145 Specification for gravity-convection and forced-ventilation ovens

3. APPARATUS

3.1 Oven: The oven shall be electrically heated and shall conform to the performance requirements of ASTM specification E 145, for Gravity-Convection and Forced-Ventilation Ovens. It shall comply with the following requirements.

3.1.1 Construction: The oven shall be rectangular with minimum interior dimensions of 330 mm (13 in.) in each direction. The oven shall have in front a tightly fitting hinged door, which shall provide a clear opening substantially the same as the interior height and width of the oven. The door may contain a window with dimensions of a least 100 by 100 mm (4 by 4 in.), and with two sheets of glass separated by an air space, through which a vertical thermometer located in the oven, may be read without opening the door, or the oven may be provided with an inner glass door, through which the thermometer may be observed on opening the outer door momentarily. The oven shall be adequately ventilated by convection currents of air and for this purpose shall be provided with openings for the entrance of air and for the exit of heated air and vapors.

3.1.2 Rotating Shelf: The oven shall be provided with a circular metal shelf having a minimum diameter of 250 mm (9.8 in.). The shelf shall suspend by a vertical shaft and centered with respect to the horizontal interior dimensions. The shelf shall be provided with a mechanical means for rotating it at the rate of 5 to 6 rpm. The shelf shall be vertically located as close to the center of the oven as permitted by compliance with the requirements regarding to thermometer placement.

3.2 Thermometer: An ASTM Loss on Heating Thermometer graduated in Celsius degrees, having a range from 155 to 170°C, and conforming to the requirements for Thermometer as prescribed in the ASTM Specification E1, for ASTM Thermometers.

3.3 Container: The container in which the sample is to be tested shall be of metal or glass, cylindrical in shape, and shall have a flat bottom. It’s inside diameter and depth shall be 55mm (2.17 in.) and 35 mm (1.38 in.) respectively.

4. PROCEDURE

4.1 First test the material under examination for water and if water is present, remove it by suitable methods of dehydration before subjecting the material to the loss on heating test, or obtain another sample that is free from water.

4.2 Place 50.0±0.5 gm of the sample of the water free material in a container, cool the sample to room temperature and weigh to the nearest 0.01 gm. Bring the oven to a temperature of 163°C (325°F) and place the container with the weighed sample on recesses if the recommended shelf is used. Close the oven and rotate the shelf during the entire test at a rate of 5 to 6 rpm. Maintain the temperature at 163 ± 1°C (325±1.8°F) for 5 hrs, start counting the time when the temperature reaches 162°C, and in no case shall the total time that a sample is in the oven be more than 5h and 15 min. At the conclusion of the heating period, remove the sample from the oven, cool to room temperature, and weigh to the nearest 0.01 gm.

5. CALCULATIONS

5.1 Calculate the percentage loss to the nearest second decimal as follows:

$$\% \text{ loss} = [(A-B)/A'] * 100$$

Where, A= initial weight of the container plus sample
B = final weight of the container plus sample after heating
A′ = initial weight of the sample

6. PRECAUTIONS

6.1 Under ordinary circumstances a number of samples having about the same degree volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. When extreme accuracy is required not more than one material should be tested at one time and duplicate samples of it should be placed simultaneously in the oven to check the accuracy of result. Samples showing evidences of foaming during the test shall be rejected.

7. REPRODUCIBILITY OF RESULTS

7.1 Up to 5 percent loss in mass the results obtained may be considered as correct within 0.5. Above 5 percent loss in mass, the numerical limit of error increases 0.01 for every 0.5 percent increase in loss by volatilization as follows:

Table 10.1: Numerical limit of error for loss above 5 percent

Volatilization Loss (%)	Numerical Correction	True Volatilization Loss, (%)
5.0	± 0.50	4.50 to 5.55
5.5	± 0.51	4.99 to 6.01
6.0	± 0.52	5.48 to 6.52
10.0	± 0.60	9.40 to 10.60
15.0	± 0.70	14.30 to 15.70
25.0	± 0.90	24.10 to 25.90
40.0	± 1.20	38.80 to 41.20

EXPERIMENT NO: 11

PENETRATION OF BITUMINOUS MATERIAL

(AASHTO DESIGNATION : T 49-93′
ASTM DESIGNATION : D 5-86)

1. SCOPE

1.1 This test method covers determination of the penetration of semi-solid and solid bituminous materials. Materials having penetrations below 350 can be tested by the standard apparatus and procedure described. Materials having penetrations between 350 and 500 can be determined using the special apparatus and modifications given in Section 9.3.

1.2 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. REFERENCED DOCUMENTS

2.1 AASHTO standards:

T 53 Softening Point of Asphalt (Bitumen) and Tar in Ethylene Glycol (Ring-and-Ball)

2.2 ASTM Standards:

C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials
E1 Specification for ASTM Thermometers
E77 Method for Verification and Calibration of Liquid in Glass Thermometers.

2.3 ANSI Standard

B 46.1 Surface Texture
IP Standard Thermometers

3. DESCRIPTION OF TERM

3.1 The penetration of a bituminous material is the distance in tenths of a millimeter that a standard needle penetrates vertically into a sample of the material under fixed conditions of temperature, load and time.

4. SUMMARY OF METHOD

4.1 The sample is melted and cooled under controlled conditions. The penetration is measured with a penetrometer by means of which a standard needle is applied to the sample under specific conditions.

5. SIGNIFICANCE AND USE

5.1 The penetration test is used as a measure of consistency. Higher values of penetration indicate softer consistency.

6. APPARATUS

6.1 Penetration Apparatus - Any apparatus permitting movements of the spindle without appreciable friction and which is accurately calibrated to yield results in accordance With the description of the term penetration (see section 3.1) will be acceptable. The surface on which the sample container rests shall be flat and the axis of the plunger shall be at approximately 90 degrees to this surface. The spindle shall be detachable without the use of special toots, for checking its mass. When the needle is mounted in a ferrule, the mass of the moving spindle shall be 47.5 ± 0.05 g. Regardless of the type of mounting of the needle, the total mass of the needle and spindle assembly shall be 50.0 ± g. Weights of 50.0 ± 0.05 g and 100.0 ± 0.05 g shall be provided for total loads of 100 g and 200 g (0.9 N and 2 N), depending upon the conditions of test to be applied.

NOTE: 1- The detachability of the spindle prescribed here is intended to apply to penetration apparatus acquired after January 1, 1976. Penetration apparatus acquired before January 1, 1976, may conform either to this standard or to the previous standard (T. 49-74).

6.2 Needle- The needle, Figure 11.1, shall be made from fully hardened and tempered stainless steel, grade 440 C or equal HRC 54 to 60. It shall be approximately 50 mm (2 in.) in length and 1.00 to 1.02 mm (0.039 to 0.040 in) in diameter. It shall be symmetrically tapered at one end to a cone whose angle shall be within the range of 8.7 to 9.7 deg over the entire length from full needle diameter, and whose axis shall be coincident with the needle axis within 0.0127 mm (0.005 in.) maximum run out (total indicator reading).

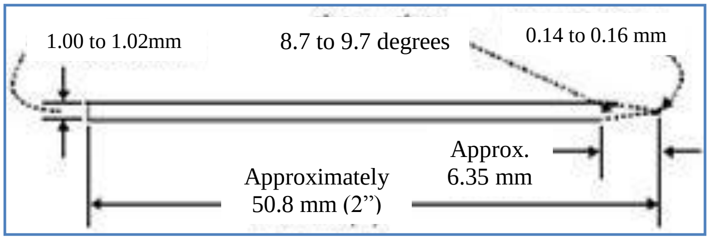


Figure 11.1 Needle for penetration test

After tapering, the point shall be ground off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm (0.0055 to 0.0063 in) in diameter. The truncation shall be square with the needle axis within 2 degree, and the edge shall be sharp and free from burrs.

6.2.1 When the surface texture of the tapered cone surface is measured in accordance with American National Standards Institute Standard B46.1, the surface roughness height shall be 0.2 to 0.3 μm (8 to 12 μ in.) arithmetic average.

6.2.2 The exposed length of the needle when mounted in the chuck of the penetration apparatus or in a ferrule shall be approximately 40 to 45 mm (1.57 to 1.77 in). When the needle is mounted in a ferrule, the ferrule shall be a cylindrical rod, 3.20 ± 0.05 mm (0.126 ± 0.002 in) in diameter and approximately 38 mm (1.5 in) long, made of stainless steel or brass, in which the needle shall be rigidly and coaxially mounted. The weight of the ferrule needle assembly shall be 2.50 ± 0.05 g. (A drill hole is permissible at the end of the ferrule to control weight). Individual identification markings shall be placed on the ferrule of each needle; the same markings shall not be repeated by a manufacturer within a 3 year period.
NOTE 2- The manufacturer or commercial laboratories will certify the test needles for conformance to the permissible variations.

6.3 Container- A container, in which the sample is tested, made of metal or glass cylindrical in shape, and having a flat bottom. The container to be used for materials having a penetration of 200 or less shall have a nominal capacity of 3 oz (90 ml). Its inside dimensions shall be essentially as follows: 55 mm (2.17 in) in diameter and 35 mm (1.38 in) in depth. The container to be used for materials having a penetration over 200 shall have a nominal capacity of 6 oz (175 ml). Its inside dimensions shall be essentially as follows: 70 mm (2.75 in) in diameter and 45 mm (1.77 in) in depth.
NOTE 3- Containers known as tin boxes or as seamless ointment boxes may be obtained in dimensions conforming to the above requirements.

6.4 Water Bath - A water bath maintained at a temperature varying not more than 0.1°C (0.2°F) from the temperature of the test. The volume of water shall not be less than 10 liters. The bath shall have a perforated shelf supported in a position not less than 50 mm from the bottom of the bath and not less than 100 mm below the liquid level in the bath. The water in the bath shall be substantially free from oil and slime or other organic growth. Brine may be used in the water bath for determinations at low temperatures. If penetration tests are to be made without removing the sample from the bath, a shelf strong enough to support the penetration apparatus shall be provided.

NOTE 4- The use of distilled, demineralized or deionized water is recommended for the bath. Care should be taken to avoid contamination of the bath water by surface active agents, release agents or other chemicals as their presence may affect the penetration values obtained.

6.5 Transfer Dish for Container- When used; the transfer dish for the container shall be a cylinder with a flat bottom made of glass, metal or plastic. It shall be provided with some means which will ensure a firm bearing and prevent rocking of the container. It shall have a minimum inside diameter of 90 mm (3.5 in) and a minimum depth above the bottom bearing of 55 mm (2.17 in).

NOTE 5- A magnetic strip in the bottom of the transfer dish may be used to prevent the ointment tin from rocking.

6.6 Thermometers for Water Bath- Calibrated Liquid-in-glass thermometers of suitable range with subdivisions and maximum scale error of 0.1°C (0.2°F) or any other thermometric device of equal accuracy, precision, and sensitivity shall be used.

The following thermometers conforming to the requirements of ASTM Specification E1, ASTM Thermometers are required:

6.6.1 For tests at 25°C (77°F) use an ASTM Saybolt Viscosity Thermometer 17°C (or 17°F) having a range of 19 to 27°C (66 to 80°F). The thermometer shall be immersed in the bath 150±15 mm.

6.6.2 For tests at 0°C (32°F) and 4°C (39.2°F) use ASTM Precision Thermometer 63°C (or 63°F) having a range of -8 to + 32°C (18 to 89°F). The thermometer shall be immersed in the bath 150±15 mm.

6.6.3 For tests at 46.1°C (115°F) use ASTM Precision Thermometer 64°C (or 64°F) having a range of 25 to 55°C (77 to 131°F). The thermometer shall be immersed in the bath 150 ± 15 mm.

6.6.4 Since the accuracy of the test results is dependent upon closely controlled temperature conditions, the thermometer used for the water bath should be calibrated by ASTM E77, Inspection, Test and standardization of Etched Stem Liquid-in-Glass thermometers.

6.7 Timing Device- For hand-operated penetrometers any convenient timing device such as an electric timer, a stop watch, or other spring-activated device may be used provided it is graduated in 0.1 second or less and is accurate to within ± 0.1 second for a 60 second interval. An audible second counter adjusted to provide 1 beat each 0.5 second may also be used. The time for a count interval must be 5 ± 0.1 second. Any automatic timing device attached to a penetrometer must be accurately calibrated to provide the desired test interval within ± 0.1 second.

6.8 Heater- An oven or hot plate, heated by electricity or gas, shall be provided for heating samples.

7. PREPARATION OF SAMPLE

7.1 Heat the sample with care to prevent local overheating until it has become fluid. Then with constant stirring, raise the temperature of the asphalt sample not more than 100°C or l80°F above its expected softening point or the tar pitch sample not more than 56°C or 100°F above its softening point determined in accordance with the Method of test for Softening Point of Bituminous Materials (Ring and Ball Method), T 53. Avoid the inclusion of air bubbles. To reach the pouring temperature, do not heat the softened sample more than 30 minutes.

Then pour it into the sample container to a depth such that, when cooled to the temperature of test the depth of the sample is at least 10 mm greater than the depth to which the needle is expected to penetrate. Pour separate samples for each variation in test conditions.

7.2 Loosely cover each container and its contents as a protection against dust, and allow to cool in an atmosphere at a temperature not higher than 30°C or 86°F and not lower than 20°C or 68°F for not less than $1\frac{1}{2}$hours nor more than 2 hours when the sample is in a 175 ml (6 oz) container and for not less than 1 nor more than $1\ \frac{1}{2}$hours when the sample is in a 90 ml (3 oz) container. Then place the sample in the water bath maintained at the prescribed temperature of test, along with the transfer dish if used, and allow it to remain for not less than $1\ \frac{1}{2}$ hours nor more than 2 hours when the sample is in the 175 ml (6 oz) container, and for not less than 1 nor more than $1\frac{1}{2}$ hours when the sample is in a 90 ml (3 oz) container.

8. TEST CONDITIONS

8.1 Where the conditions of test are not specifically mentioned, the temperature, load, and time are understood to be 25°C (77°F), 100 g, 5 second, respectively.

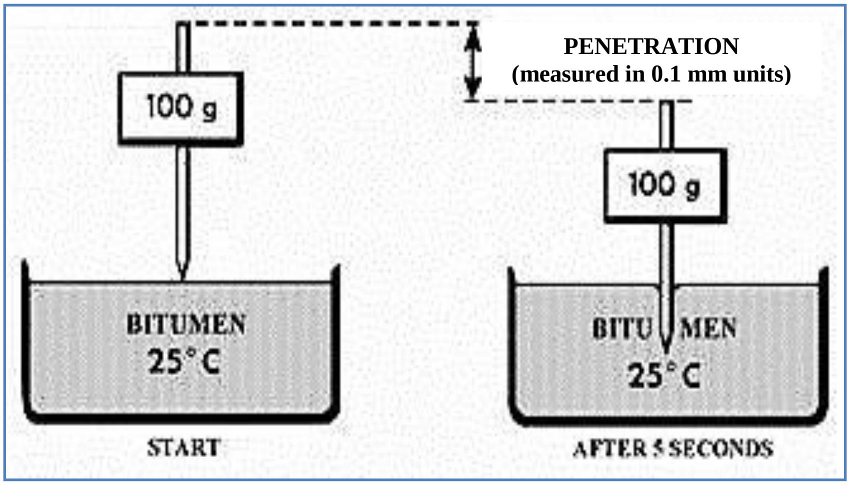


Figure 11.2 Penetration test

Other conditions of temperature, load and time may be used for special testing, such as:

Table 11.1: Temperature and Time for different loads of penetration needle assembly.

Temperature	Load, g	Time
0°C/(32°F)	200	60
4°C/(39.2°F)	200	60
46.1^{0}C/(115^{0}F)	50	5

In such cases, the specific conditions of test shall be reported.

9. PROCEDURES

9.1 Examine the needle holder and guide to establish the absence of water and other extraneous matter. Clean a penetration needle with toluene or other suitable solvent, dry with a clean cloth, and insert the needle in the penetrometer. Unless otherwise specified, place the 50 g weight above the needle, making the total load of 100 g ± 0.1g for the needle and attachment. If tests are made with the penetration apparatus mounted in the bath, place the sample container directly on the submerged stand of the penetration apparatus. If tests are made with the sample in the bath and the penetration apparatus outside the bath, place the containers on the shelf provided in the bath. In the above procedures the container shall be kept completely submerged during the complete test. If tests are made using the transfer dish with the penetration apparatus outside the bath, place the sample in a dish filled with water from the bath to a depth to cover completely the sample container. Then place the transfer dish containing the sample on the stand on the penetration apparatus and penetrate immediately. In each case, adjust the needle loaded with the specified weight to just make contact with the surface of the sample. Accomplish this by making contact of the actual needle point with its image reflected by the surface of the sample from a properly placed source of light (Note 8). Either note the reading of the dial or bring the pointer to zero. Then quickly release the needle for the specified period of time and adjust the instrument to measure the distance penetrated. Observe the sample container as the needle is applied, and if any movement of the container is noted, ignore the result.

NOTE 6- For certain types of asphalt erratic results are sometimes obtained. When this occurs, pre-treat the needles by immersing them for 5 minutes in a 1 percent solution of olene acid prior to drying and running the test.

NOTE 7- For reference tests, penetrations at temperature other than 25^0C (77^0F) should be made without removing the sample from the bath.

NOTE 8- The positioning of the needle can be materially aided by using an illuminated methyl methacrylate rod.

9.2 Make at least three penetrations at points on the surface of the sample not less than 10 mm (3/8 in) from the side of the container and not less than 10 mm (3/8 in) apart. If the transfer dish is used, return the dish and sample to the water bath after each penetration. Before each test, clean the needle with a clean cloth moistened with toluene or other suitable solvent to remove all adhering bitumen, and then wipe with a clean dry cloth. For penetration values greater than 200, use at least three needles, leaving them in the sample until completion of the penetrations.

9.3 The needles, containers, and other conditions described in this method provide for determinations of penetrations up to 350. However, the method may be used for direct determinations up to 500 provided special containers and needles are used. The container shall be at least 60 mm in depth. The overall volume of material in the container should not exceed 125 ml to permit proper temperature adjustment of the sample.

9.3.1 Specially made needles for such determination shall meet all the requirements of Section 6.2 for dimensions and weight except that the minimum exposed length of the needle shall be 50 mm.

9.3.2 An approximation of the penetration of such high penetration materials may also be obtained by determining the penetration using the standard needle and 6 oz container but with a 50 g loading. The penetration is then calculated by multiplying the result for the 50 g load by the square root of 2. That is:

Penetration under 100g load = (Penetration under 50g load) x 1.414

The report of results obtained by this procedure shall indicate the basis of the test.

10. REPORT

10.1 Report to the nearest whole unit the average of at least three penetrations whose values do not differ by more than the amount shown:

Table 11.2: Limit of penetration values for different penetration range.

Penetration	0-49	50-149	150-249	≥ 250
Maximum difference between highest and lowest determinations	2	4	6	8

10.1.1 If the appropriate tolerance is exceeded, ignore all results and repeat the test.

11. PRECISION

11.1 Repeatability- Two results obtained by the same operator on the same

	Standard Deviation Within Laboratory	Standard Deviation Between Laboratory
Asphalt at 25^0C (77^0F) below 50 penetration	0.35 units	1.4 units
Asphalt at 25^0C (77^0F) 50 penetration and above	1.1 percent of their mean	2.8 percent of their mean
Tar pitches at 25^0C (77^0F)	5.2 percent of their mean	1.4 units

Sample in the same apparatus, and on different days should be considered suspect if they differ by more than the following amounts:

Asphalt at 25^0C (77^0F) below 50 penetration	1 units
Asphalt at 25^0C (77^0F) 50 penetration and above	3 percent of their mean
Tar pitches at 25^0C (77^0F)	15 percent of their mean

For pitches estimates of precision are based on results from 2 pitches with penetration of 7 and 24 estimates may not be applicable to appreciably harder or softer materials

11.2 Reproducibility- Two results obtained by different operators in different laboratories on different days should be considered suspect if they differ by more than the following amounts:

Asphalt at 25^0C (77^0F) below 50 penetration	4 units
Asphalt at 25^0C (77^0F) 50 penetration and above	8 percent of their mean

Tar pitches at 25^0C (77^0F)	4 units*

NOTE: 9- Values show above was obtained by multiplying the estimates of the following population standard deviation (0'p) by 2 v 2.

These estimates of precision are based on the following data (see table entitled "Estimates of Precision"):

	Asphalt at below 50 penetration	Asphalt 50 penetration and above	Tar pitches
Samples	2	7	2
Laboratories	16	27	19
Replicates per sample	3	3	3
Degrees of freedom:			
Within laboratories	32	185	38
Between laboratories	14	89	17

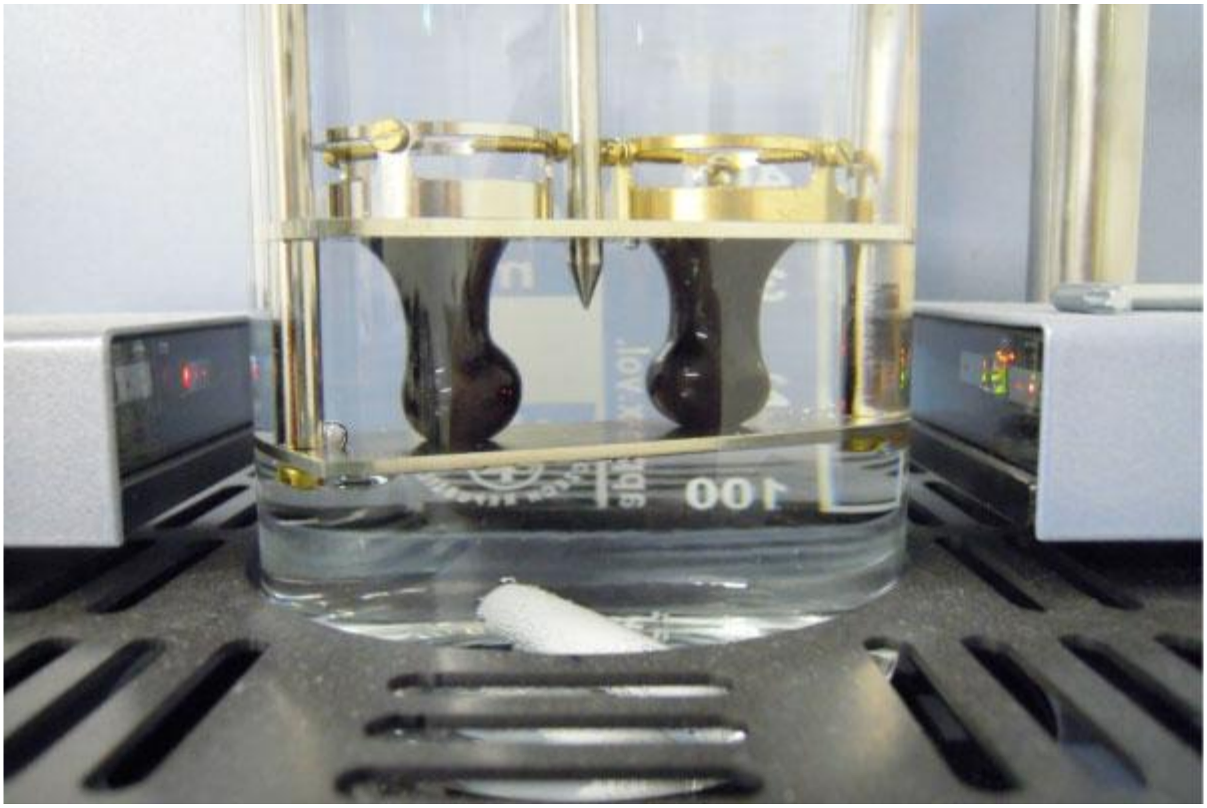
---

* For definitions of terms and recommended used of precision indexes see ASTM Recommended Practice E77, for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material.

EXPERIMENT NO: 12

SOFTENING POINT OF BITUMINOUS MATERIAL (RING AND BALL METHOD)

(AASHTO DESIGNATION : T 53-92
ASTM DESIGNATION : D 36-89)



1. SCOPE

1.1 The ring and ball softening point is extensively used to evaluate the consistency of bituminous binders. It is a very simple one, consisting of placing a 3/8 in diameter steel ball on a binder sample placed in a steel ring and immersed in a water bath. Heat is applied to the water and its temperature is raised until a value is reached when the test sample has become sufficiently soft to allow the ball, enveloped in binder to fall down. The water temperature at which this occurs is called the ring and ball softening point.

The softening point is not a melting point; bituminous binders do not melt but instead gradually change from semi-solids to liquids on the application of heat. It is useful for determining the temperature susceptibilities of bitumen which are to be used in thick films, such as in crack fillers. When two bitumen have the same penetration value, the one with the higher softening point is normally less susceptible to temperature changes.

2. REFERENCED DOCUMENTS

2.1 Standards

- C 670 Practices for Preparing Precision Statements for Test Methods for Construction materials
- E 1 Specification for ASTM Thermometers
- T 40 Methods of Sampling Bituminous Materials
- T 48 Test Method for Flash and Fire Points by Cleveland Open cup

3. APPARATUS AND MATERIALS

3.1 Ring- A brass ring of 15.875 mm (5/8 in) inside diameter, 6.35 mm (1/4 in) depth and thickness of wall is 2.38 mm (3/32 in). This ring shall be attached in a convenient manner to a brass with (diameter 1.85 mm = 0.072 in).

3.2 Ball - A steel ball 9.53 mm (3/8 in) in diameter having a mass of 3.50 ± 0.05 g.

3.3 Container - A glass vessel, not less than 8.5 cm (3.34 in) in diameter and measuring 10.5 cm (4.13 in.) in depth from the bottom of the flare (a 600 ml beaker, low form, meets this requirement).

Figure 12.1 Ring and Ball apparatus

3.4 Thermometer - ASTM Low Softening point Thermometer having a range of -2 to +80°C or 30°to 180°F is specified.

4. REAGENTS AND MATERIALS

4.1 Bath liquids:
4.1.1 Freshly boiled distilled water.
4.1.2 USP Glycerin, or
4.1.3 Ethyl Glycol, with a boiling point between 195 and 197°C (383 and 387°F).

5. PREPARATION OF SAMPLE

Melt and thoroughly stir the sample avoiding incorporating air bubbles in the mass and then pour it into the ring. The ring, while being filled, should rest on a brass plate which has been amalgamated to prevent the bituminous material from adhering to it. Allow the excess material to cool for 1 hr then cut it off cleanly with a slightly heated knife.

6. PROCEDURE FOR MATERIALS HAVING SOFTENING POINTS 80^{0}C (176^{0}F) OR BELOW

6.1 Fill the glass vessel to a depth of substantially 8.25 cm (3.25 in) with freshly boiled, distilled water at 5°C (41°F).

6.2 Suspend the ring containing the sample in the water so that the lower surface of the filled ring is exactly 2.54 cm (1 in) above the bottom of the glass vessel and its upper surface is 5.08 cm (2 in) below the surface of the water.

6.3 Place the ball in the water but not on the specimen.

6.4 Suspend the thermometer so that the bottom of the bulb is level with the bottom of the ring and within 0.635 cm (3/4 in) but not touching the ring. Maintain the temperature of the water at 5°C (41°F) for 15 min.

6.5 With suitable force, place the ball in the center of the upper surface of the bitumen in the ring, thus completing the assembly.

6.6 Apply the heat in such a manner that the temperature of the water is raised 5°C (9°F) each minute.

7. SOFTENING POINT

Report the temperature recorded by the thermometer at the instant the bituminous material touches the bottom of the glass vessel as the softening point. No correction shall be made for emergent stem of the thermometer.

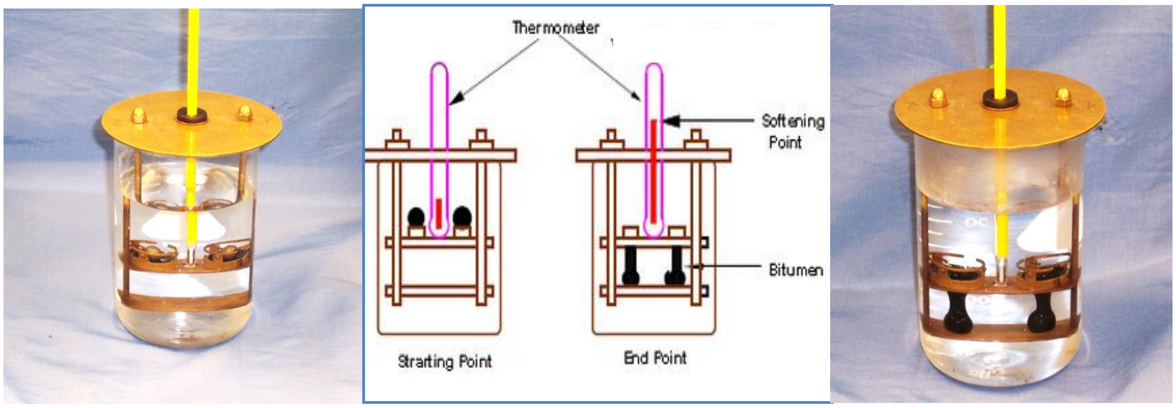


Figure 12.2 Illustration of softening point

8. PERMISSIBLE VARIATION IN RISE OF TEMPERATURE

The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three minutes shall be 0.5°C (0.9°F). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

9. PROCEDURE FOR MATERIALS HAVING SOFTENING POINTS ABOVE 80^{0}C (176^{0}F)

Thermometer- An ASTM high softening point Thermometer having a range of 30 to 200°C or 85 to 392°F is specified Modifications for Hard Materials.

Employ the same procedure as described above except that U.S.P., Glycerin shall be used instead of water, and the starting point of the Glycerin bath shall be 32°C (89.6°F). Bring the bath to this temperature and thoroughly agitate it, then place the apparatus and specimens in the bath, which shall be maintained, under agitation at the starting temperature for 15 min. In applying the heat, place the ring apparatus of the center of the container and place the burner midway between the center and edge of the beaker away from the specimen.

10. PRECAUTIONS

10.1 The use of freshly boiled distilled water is essential as otherwise air bubbles may form on the specimen and affect the accuracy of the results. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results.

A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the bituminous material from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning.

11. ACCURACY

The limit of accuracy of the test is 0.5°C (0.9°F).

EXPERIMENT NO: 13

SOLUBILITY OF BITUMINOUS MATERIAL

(AASHTO DESIGNATION : T 44-93
ASTM DESIGNATION : D 2042-81)

1. SCOPE

1.1 This method covers the determination of the degree of solubility in trichloroethylene or 1, 1, 1 trichloroethylene of asphalt minerals having little or no mineral matter.

2. SUMMARY OF METHOD

2.1 The sample is dissolved in trichloroethylene or 1, 1, 1 trichloroethylene and filtered through a filter mat. The insoluble material is washed, dried, and weighed.

3. SIGNIFICANCE AND USE

3.1 This method is a measure of the solubility of asphalt in trichloroethylene or 1, 1, 1 trichloroethylene. The portion that is soluble in trichloroethylene or 1, 1, 1 trichloroethylene represents the active cementing constituents.

4. APPARATUS AND MATERIALS

4.1 The assembly of the filtering apparatus is illustrated in Figure 1. Details of the component parts are as follows:

Figure 13.1 Component parts of the filtering apparatus

4.1.1 Gooch Crucible, glazed inside and outside with the exception of outside bottom surface. The approximate dimensions shall be a diameter of 44 mm at top, tapering to 36 mm at bottom and a depth of 28 mm.

4.1.2 Glass Fiber Pad of 3.7 cm in diameter.

4.1.3 Filter Flask, heavy-wall, with side tube, 250 ml, capacity or larger.

4.1.4 Filter Tube, 40 to 42 mm inside diameter.

4.1.5 Rubber Tubing or Adapter, for holding the Gooch crucible on the filter tube.

NOTE 1- Other suitable assemblies permitting vacuum filtration with a Gooch crucible may be used.

4.2 Erlenmeyer Flask, 125 ml, or other suitable container.

4.3 Oven, capable of maintaining a temperature or $110 \pm 5^{\circ}C$ ($230 \pm 9^{\circ}F$).

4.4 Desiccator, of suitable size, charged with an effective desiccant.

4.5 Analytical Balance, class A conforming to the requirements of an AASHTO Specification M 231.

5. SOLVENT

5.1 Technical grade, Type 1, Trichloroethylene or technical grade 1,1,1 trichloroethylene.

6. SAFETY PRECAUTIONS

6.1 "Trichloroethylene and 1,1,1 trichloroethylene are toxic material and strict adherence to instructions in Material Safety Data Sheets are to be followed". Caution: Trichloroethylene and 1,1,1 trichloroethylene in the presence of heat and moisture may form acids that are extremely corrosive.

7. PREPARATION OF GOOCH CRUCIBLE

7.1 Assemble the filtering apparatus. Place filter pad into the gooch crucible, moisten the pad with solvent and seat firmly in the bottom of the crucible with light suction. Place in an oven at $110 \pm 5^{\circ}C$ ($230 \pm 9^{\circ} F$) for at least 20 minutes, cool in desiccator and weigh to the nearest 0.1 mg. Repeat the drying and weighing until constant mass (± 0.3 mg) is obtained. Store in a desiccator until ready for use.

8. SAMPLE PREPARATION

8.1 If the sample is not fluid, heat to any convenient temperature, but in any case not more than 100°C or 180°F above the softening point.

9. PROCEDURE

9.1 Note safety precautions in Section 6. Transfer approximately 2 g of the sample into a tared 25 ml Erlenmeyer flask or other suitable container. Allow the container and its contents to cool to ambient temperatures and weigh to the nearest 1 mg. Add 100 ml of the trichloroethylene or 1,1,1 trichloroethylene to the container in small portions with continuous agitation until all lumps disappear and no undissolved sample adhere to the container. Stopper the flask or otherwise cover the container and set aside for at least 15 minutes.

Normally the temperature at which this test is run is not critical and it may be performed at the laboratory air temperature. For referee tests, however, the flask and sample in solution shall be placed in a water bath maintained at 37.8 ± 0.25°C (100 ± 0.5°F), for 1 hour before filtering.

9.2 Place the previously prepared and weighed Gooch crucible in the filtering tube.

Wet the filter pad with a small portion of clean solvent and decant the solution through the filter pad of the crucible with light suction.

When the insoluble matter is appreciable, retain as much of it as possible in the container until the solution has drained through the filter pad. Wash the container with a small amount of solvent and, using a stream of solvent from a wash bottle, transfer all insoluble matter to the crucible. Use a "policeman" if necessary to remove any insoluble matter adhering to the container, rinse the policeman and the container, thoroughly wash the insoluble matter in the crucible with solvent until the filtrate is substantially colorless, and then apply strong suction to remove the remaining solvent. Remove the crucible from the tube and wash the bottom free of any dissolved matter. Place in an oven at 110 ± 5°C (230 ± 9°F), for at least 20 min. Cool in a desiccator and weigh to the nearest 0.1 mg. Repeat the drying and weighing until constant weight (± 0.3 mg) is obtained. Fiber glass filter pads should be used only one time.

10. CALCULATIONS AND REPORT

10.1 Calculate either the total percentage of insoluble matter or the percentage of the sample soluble in the solvent used as follows:

$$\text{Insoluble, percent} = A/B \times 100$$

$$\text{Soluble, percent} = 100 - (A/B \times 100)$$

Where,
A = total weight insoluble, and
B = total weight of sample.

10.1.1 For percentage of insoluble less than 1.0, report to the nearest 0.01 percent; for percentage of insoluble 1.0 or more report to the nearest 0.1 percent.

11. PRECISION

11.1 estimates of standard deviation for this procedure and the criteria for judging the acceptability of results (95 percent confidence level) are indicated in the table entitled "Standard Deviations".

The estimates of standard deviation are based on the following:

	Asphalts
Materials	4
Replications	3
Solvents	4
Laboratories	26
Degree of freedom:	
within-laboratory	159
between Laboratory variability	81
Standard deviation (S) of data:	
within-laboratory variability	0.035
between laboratory variability	0.090

EXPERIMENT NO: 14

DUCTILITY OF BITUMINOUS MATERIAL

(AASHTO DESIGNATION : T 51-93
ASTM DESIGNATION : D 113-79)

1. SCOPE

1.1 The ductility of a bituminous material is measured by the distance to which it will elongated before breaking when two ends of a briquette specimen of the material, of the form describe in Section 2, are pulled apart at a specified speed and at a specified temperature. Unless otherwise specified, the test shall be made at a temperature of $77^\circ \pm 0.9^\circ F$ ($25^\circ \pm 0.5^\circ C$) and with a speed of 5 cm/min, + 5.0 percent. At other temperatures the speed should be specified.

2. APPARATUS

2.1 Mould - the mould shall be similar in design to that shown in Figure 14.1. Dimensions shown Figure 1 shall be as given with the permissible variations indicated. The mould shall be made of brass, the ends b and b' being known as clips, and the parts a and a' as sides of the mould. The dimensions of the mould shall be such that, when properly assembled, it will form a briquette specimen having the following dimensions:

Table 14.1: Dimensions of Ductility Test apparatus

Total length	7.45 to 7.55 cm
Distance between clips	2.97 to 3.03 cm
Width at mouth of clip	1.98 to 2.02 cm
Width at minimum cross section (halfway between clips)	0.99 to 1.01 cm
Thickness throughout	0.99 to 1.01 cm

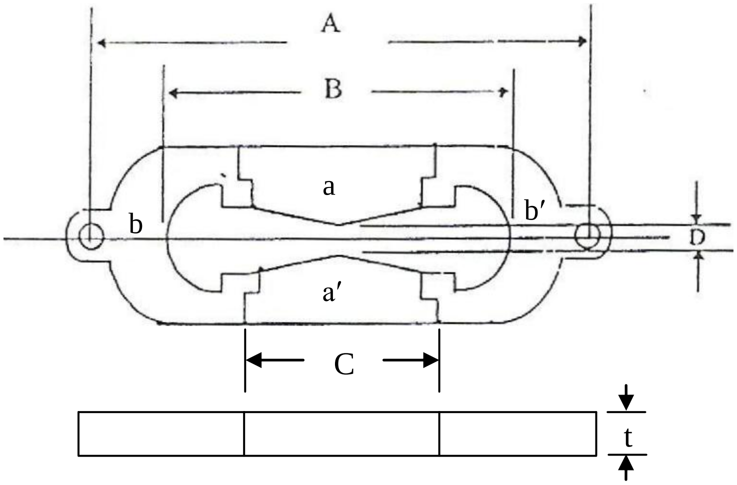


Figure 14.1 Mold for Ductility Test Specimen

Table 14.2: Dimensions of different parts of Ductility Test apparatus shown in Figure 14.1

A	Distance between centers	111.5 to 113.5 mm
B	Total length of briquette	74.5 to 75.5 mm
D	Width at minimum cross section	9.9 to 10.1 mm
t	Thickness	9.9 to 10.1 mm

2.2 Water Bath - The water bath shall be maintained at the specified test temperature, varying not more than 0.18°F (0.1°C) from this temperature. They volume of water shall be not less than 10 liters, and the specimen shall be immersed to a depth of not less than 10 cm and shall be supported on a perforated shelf not less than 5 cm from the bottom of the bath.

2.3 Testing Machine - For pulling the briquette of bituminous material apart, any apparatus may be used which is so constructed that the specimen will be continuously immersed in water as specified in Section 3.3, while the two clips are pulled apart at a uniform speed, as specified, without under vibration. Figure 14.2 shows the ductility testing machine.

Figure 14.2 Ductility Testing Machine

2.4 Thermometer - A thermometer having a range as shown below and conforming to the requirements prescribed in Specification E 1 for Standard Thermometer.

Table 14.3: Specifics of Standard Thermometer for Ductility Test.

Temperature Range	ASTM Thermometer No
-8 to 32°C	63°C
18 to 89°F	63°F

3. PROCEDURE

3.1 Molding Test Specimen - Heat the sample with care to prevent local overheating until it has become sufficiently fluid to pour. Strain the melted sample through a No. 50 sieve conforming to ASTM Specification E11, for Wire cloth Sieves for Testing Purposes, and, after a thorough stirring pour it into the mold. Assemble the mold on a brass plate and, to prevent the material under test from sticking, thoroughly amalgamate the surface of the plate and interior surface of the sides a and a', Figure 1, of the mold or coat with a mixture of glycerin and dextrin, talc, or china clay. The plate upon which the mold is placed shall be perfectly flat and level so that the bottom surface of the mold will touch it throughout. In filling the mold, take care not to disarrange the parts and thus distort the briquette. In filling, pour the material in a thin stream back and forth from end to end of the mold until the mold is more than level full. Let the mold containing the material cool to room temperature or a period of from 30 to 40 min and then place it in the water bath maintained at the specified temperature of test for 30 min; then cut off the excess bitumen with a hot straight edged putty knife or spatula to make the mold just level full as shown in Figure 14.3.

Figure 14.3 Molding and Cutting of Test Specimen

3.1.1 Caution - Careless handling of mercury will create a definite health hazard. The rules prescribed as follows should be observed at all times:

1. Store the mercury in a closed jug in a cool place.
2. Strictly avoid spilling any mercury.
3. Remove mercury vapors by working under a ventilated hood.
4. Keep amalgamated brass plates and other apparatus at no higher than normal room temperature.

3.2 Keeping Specimen at Standard Temperature - Place the brass plate and mold, with briquette specimen, in the water bath and keep at the specified temperature for a period of from 85 to 95 min. Then remove the briquette from plate, detach the side pieces, and immediately test the briquette.

3.3 Testing - Attach the rings at each end of the clips to the pins or hooks in the testing machine and pull the two clips apart at a uniform speed as specified until the briquette ruptures as shown in Figure 14.4.

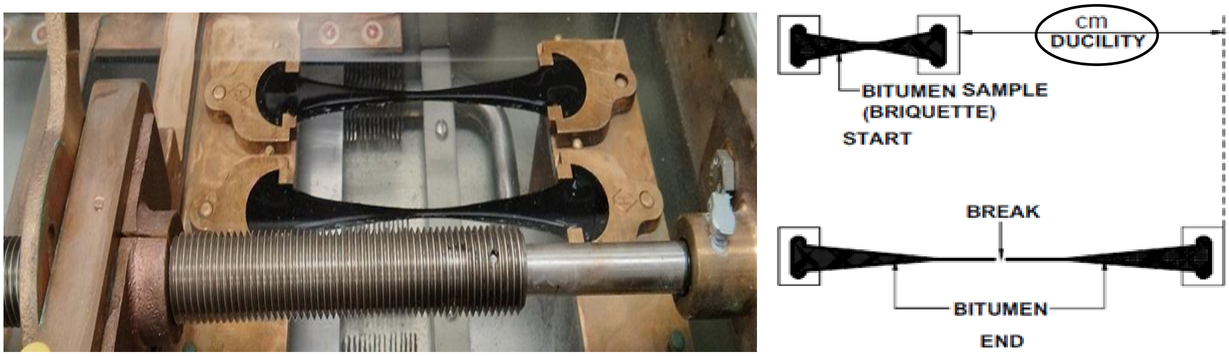


Figure 14.4 Ductility Testing

A variation of + 5 percent from the speed specified will be permissible. Measure the distance in centimeters through which the clips have been pulled to produce rupture. While the test is being made, the water in the tank of the testing machine shall cover the specimen both above and below it by at least 2.5 cm and shall be kept continuously at the temperature specified within +0.9°F (0.5°C).

4. REPORT

4.1 A normal test is one in which the material between the clips pulls out to a point or thread until rupture occurs at the point where the thread has practically no cross sectional area. Report the average of three normal tests as the ductility of the sample.

4.2 If the bituminous material comes in contact with the surface of the water or the bottom of the bath, the test shall not be considered normal. Adjust the specific gravity of the bath by the addition of either methyl alcohol or sodium chloride so that the bituminous material neither comes to the surface of the water, nor touches the bottom of the bath at any time during the test.

4.3 If a normal tests not obtainable on three terms, report the ductility as being unobtainable under the conditions of the test.

5. PRECAUTIONS

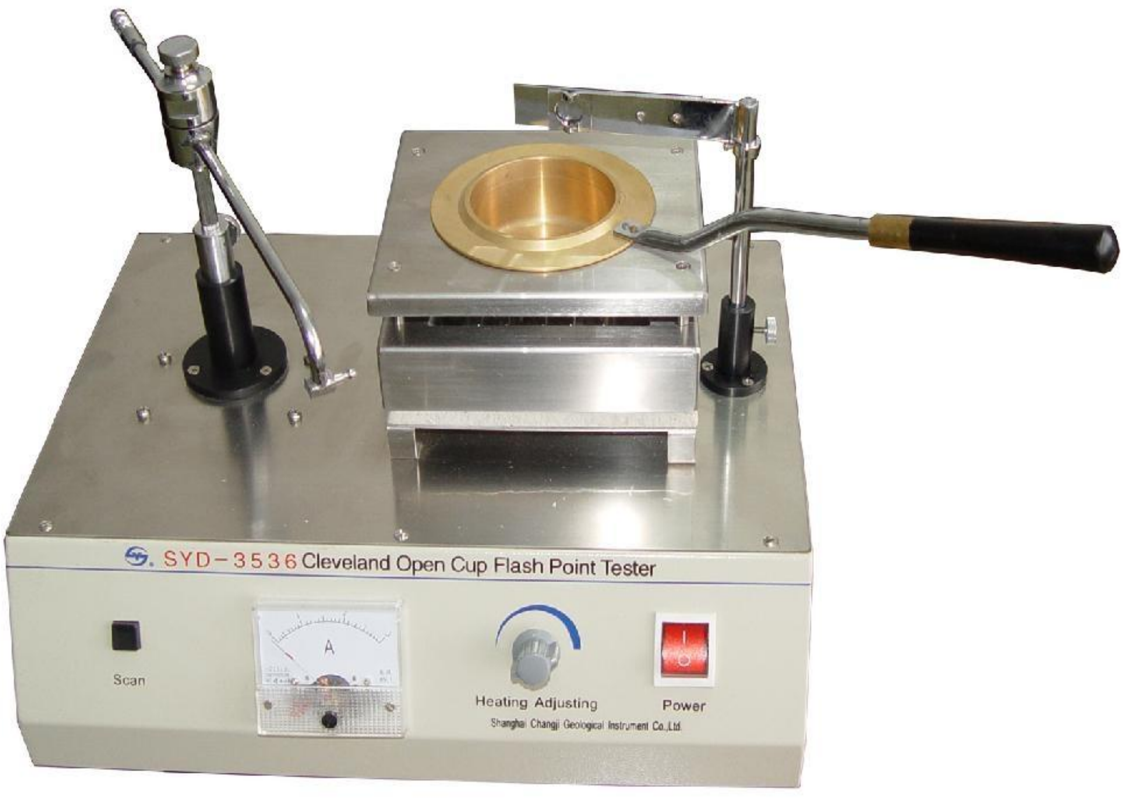
5.1 Owing to possible danger to health if mercury is handled carelessly, the following rules should be observed at all times:

5.1.1 Store the mercury in a closed jug in a cool place.
5.1.2 Strictly avoid spilling any mercury.
5.1.3 Remove mercury vapors by working under a suitable hood with good ventilation.
5.1.4 Keep amalgamated brass plates and other apparatus at not above normal room temperature.

EXPERIMENT NO: 15

FLASH AND FIRE POINTS OF BITUMINOUS MATERIAL (CLEVELAND OPEN CUP METHOD)

(AASHTO DESIGNATION : T 48-91
ASTM DESIGNATION : D 92-85)



1. SCOPE AND SIGNIFICANCE

This method describes a test procedure for determining the flash and fire points (Cleveland Open Cup Tester) of all petroleum products except fuel oils and those having an open cup flash below 175^0F. The flash point is the temperature at which a bituminous material, during heating, will evolve vapors that will temporarily ignites or flash when a small flame is brought in contact with them. The fire point is the temperature at which the evolved vapors will ignite and continue to burn.

To make the test, the material is heated in an open cup, and at intervals a small flame is applied near its surface. The lowest temperature at which application of the test flame causes the vapors to ignite is recorded as the flash point while the temperature at which the vapors ignited and burn for at least 5 seconds is recorded as the fire point. The flash and fire point test is purely a safety test. It indicates the maximum temperature to which the material can be safely heated.

NOTE 1- It is the practice in the United Kingdom and in any other countries to use IP Method 35, Flash Point (Open) and Fire Points by Means of the Pensky-Martens Apparatus unless T73. Test flash point by Pensky-Martens Closed Tester is specified. This Method may occasionally be specified for the determination of the fire point of a fuel oil. For the determination of Flash points of fuel oils, use AASHTO T 73 IP 34, T 73 should be used when it is desired to determine the possible presence of small but significant concentrations of lower flash points substances which may escape detection by T 48. T79, Flash Point with Tag Open Cup Apparatus, may be employed if the flash point is below 79°C (175^0F) as determined by T 48.

2. REFERENCED DOCUMENTS

2.1 AASHTO Standards:
  T 73 Flash Point by Pensky-Martens Closed Tester
  T 79 Flash Point with Tag Open-Cup Tester

2.2 ASTM Standards:
  E 1 Specification for ASTM Thermometers

2.3 Other Methods:
  IP Method 35 Flash Point (Open) and Fire Point by Means of the pensky-Martens Apparatus.

3. APPARATUS AND MATERIALS

3.1 Cleveland Open Tester - The apparatus consists of the test cup, heating plate, test flame applicator, heater, and support as shown in Figure 15.1.

Figure 15.1 Cleveland Open Cup Tester

3.2 Shield - A shield 18 inch (46 cm) square and 24 in, (61 cm) high, is recommended but not essential.

3.3 Thermometer - ASTM thermometer having a range of 20^{0}F to 760^{0}F (-6°C to + 400°C).

4. PROCEDURE

4.1 Support the tester on a level steady table in a draft free room or compartment and shield the spot of the tester from strong light by any suitable means.

4.2 Clean the cup with an appropriate solvent and remove all gums, carbon deposit, and oxide coating from the inside of the cup with fine steel wool until a bright metallic surface is presented.

4.3 Support the thermometer in a vertical position with the bottom of the bulb 1/4 inch (0.635 cm) from the bottom of the cup and above a point halfway between the center and back of the cup.
Note 2- The immersion line engraved on the thermometer will be 5/64 inch (0.20 cm) below level of the rim of the cup when the thermometer is properly positioned.

4.4 Fill the cup at any convenient temperature (Note 3) so that the top of the meniscus is exactly at the filling line. When too much sample has been added to the cup, remove the excess, using a spoon or other suitable device; however, if there is sample on the outside of the apparatus, empty, clean. Destroy any air bubbles appear on the surface of the sample.

Note 3- Viscous samples should be heated until they are reasonably fluid before being poured in to the cup; however, the temperature during heating must not exceed 100°F (65°C) below the probable flash point.

4.5 Light the test flame and adjust it to a diameter of 1/8 to 3/16 in. (0.08 cm).

4.6 Apply heat initially so that the rate of temperature rise of the sample is 25 to 30^0F (13.9 to 16.7°C) per minute. When the sample temperature is approximately 100°F (56^0C) below the anticipated flash point, decrease the heat so that the rate of temperature rise for the last 50^0F (27.8°C) before the flash point is 10 + 1°F (5.5 + 0.6°C) per minute.

4.7 Starting at least 50°F (2.8°C) mark pass the test flame across the center of the cup, at right angles to the diameter which passes through the thermometer. With a smooth, continuous motion apply the flame either in a straight line or along the circumference of a circle having a radius of at least 6 inch (15 cm). The center of the test flame must move in a plane not more than 5/6" inch (0.2 cm) above the plane of the upper edge of the cup. The time consumed in passing the test flame across the cup shall be about 1 sec.

4.8 Record as the flash point the temperature read on the thermometer when a flash appear at any point on the surface of the sample but do not confuse the true flash with the bluish halo that sometimes surrounds the test flame.

4.9 To determine the fire point, continue heating so that the sample temperature increases at rate of 10 ± 1°F (5.5± 0.6^0C) per minute. Continue the application of the test flame at 5^0F (2.8°C) intervals until the vapor ignites and continues to burn for at least 5 sec. Record the temperature at this point as the fire point.

5. CALCULATION AND REPORT

5.1 Observe and record the barometric pressure at the time of the test. When the pressure differs from 760 mm Hg, correct the flash or fire point, or both, by means of the following equations:

- Corrected flash or fire point, or both = F + 0.06(760-P) or
- Corrected flash or fire point, or both = C + 0.03(760-P)

Where:

F = observed flash or fire point, or both, to the nearest 5°F
C = observed flash or fire point, or both, to the nearest 2°C.
P = barometric pressure, mm Hg.

EXPERIMENT NO: 16

STANDARD TEST METHOD FOR CBR (CALIFORNIA BEARING RATIO) OF LABORATORY COMPACTED SOILS[1]

(ASTM DESIGNATION : D 1883-99)

1. SCOPE

1.1 This test method covers the determination of the CBR (California Bearing Ratio) of pavement sub-grade, sub-base and base/course materials from laboratory compacted specimens. The test method is primarily intended for but not limited to, evaluating the strength of cohesive materials having maximum particle sizes less than ¾ inch (19 mm).

NOTE 1 The agency performing this test can be evaluated in accordance with Practice D 3740. Notwithstanding statements on precision and bias contained in this Standard: The precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies which meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing. Users of this method are cautioned that compliance with Practice D 3740 does not in itself assure reliable testing. Reliable testing depends on many factors; Practice D 3740 provides a means of evaluating some of those factors.

1.2 When materials having maximum particle sizes greater than ¾ inch (19 mm) are to be tested, this test method provides for modifying the gradation of the material so that the material used for tests all passes the ¾ inch sieve while the total gravel (No. 4 to 3 in.) fraction remains the same. While traditionally this method of specimen preparation has been used to avoid the error inherent in testing materials containing large particles in the CBR test apparatus, the modified material may have significantly different strength properties than the original material. However, a large experience base has developed using this test method for materials for which the gradation has been modified, and satisfactory design methods are in use based on the results of tests using this procedure.

1.3 Past practice has shown that CBR results for those materials having substantial percentages of particles retained on the No. 4 sieve are more variable than for finer materials. Consequently, more trials may be required for these materials to establish a reliable CBR.

1.4 This test method provides for the determination of the CBR of a material at optimum water content or a range of water content from a specified compaction test and a specified dry unit weight. The dry unit weight is usually given as a percentage of maximum dry unit weight from the compaction tests of Test Methods D 698 or D 1557.

1.5 The agency requesting the test shall specify the water content or range of water content and the dry unit weight for which the CBR is desired.

1.6 Unless specified otherwise by the requesting agency, or unless it has been shown to have no effect on test results for the material being tested, all specimens shall be soaked prior to penetration.

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.08 on Special and Construction Control Tests. Current edition approved Feb. 10, 1999. Published May 1999; Originally published as D 1883-61T; Last previous edition D 1883-94.

1.7 For the determination of CBR of field compacted materials, see Test Method D 4429.

1.8 The values stated in inch-pound units are to be regarded as the standard. The SI equivalents shown in parentheses may be approximate.

1.9 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. REFERENCED DOCUMENTS

2.1 ASTM Standards:

D 422 Test Method for Particle-Size Analysis of Soils[2]
D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]
D 698 Test Method for Laboratory Compaction Characteristics of Soil Using Standard Effort (12,400 ft-lbf/ft^3 (600 kN-m/m^3))[2]
D 1557 Test Method for Laboratory Compaction Characteristics of Soil Using Modified Effort (56,000 ft-lbf/ft^3 (2,700 kN-m/m^3))[2]
D 2168 Test Methods for Calibration of Laboratory Mechanical-Rammer Soil Compactors[2]
D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock[2]
D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]
D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[2]
D 3740 Practice for Minimum Requirements of Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction[2]
D 4318 Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils[2]
D 4429 Test Method for CBR (California Bearing Ratios) of Soils in Place[2]

3. SUMMARY OF TEST METHOD

3.1 For tests performed on materials compacted to one water content, three specimens are prepared. The specimens are compacted using three different compaction efforts to obtain unit weights both above and below the desired unit weight. After allowing specimens to take on water by soaking, or other specified treatment such as curing, each specimen is subjected to penetration by a cylindrical rod. Results of stress (load) versus penetration depth are plotted to determine the CBR for each specimen. The CBR at the specified density is determined from a graph of CBR versus dry unit weight.

[2] Annual Book of ASTM Standards, Vol 04.08.

3.2 For tests in which the result is to be determined for a water content range, a series of specimens at each of three compaction efforts are prepared over the range of water content of interest. The compaction efforts are chosen to produce unit weights above and below the desired unit weight. After allowing the specimens to take on water by soaking, or other specified treatment such as curing, each specimen is penetrated. Results are plotted to obtain the CBR for each specimen. A plot of CBR versus unit weight for each water content is made to determine the minimum CBR for the water content range of interest.

4. SIGNIFICANCE AND USE

4.1 This test method is used to evaluate the potential strength of sub-grade, sub-base and base course material, including recycled materials for use in road and airfield pavements. The CBR value obtained in this test forms an integral part of several flexible pavement design methods.

4.2 For applications where the effect of compaction water content on CBR is small, such as cohesion less, coarse grained materials, or where an allowance is made for the effect of differing compaction water contents in the design procedure, the CBR may be determined at the optimum water content of a specified compaction effort. The dry unit weight specified is normally the minimum percent compaction allowed by the using agency’s field compaction specification.

4.3 For applications where the effect of compaction water content on CBR is unknown or where it is desired to account for its effect, the CBR is determined for a range of water content, usually the range of water content permitted for field compaction by using agency’s field compaction specification.

4.4 The criteria for test specimen preparation of self cementing (and other) materials which gain strength with time must be based on a geotechnical engineering evaluation. As directed by the engineer, self cementing materials shall be properly cured until bearing ratios representing long term service conditions can be measured.

5. APPARATUS

5.1 Loading Machine: The loading machine shall be equipped with a movable head or base that travels at a uniform (not pulsating) rate of 0.05 in. (1.27 mm)/min for use in forcing the penetration piston into the specimen. The machine shall be equipped with a load indicating device that can be read to 10 lbf (44 N) or less. The minimum capacity of the loading machine shall be based on the requirements indicated in Table 16.1.

Table 16.1: Minimum Load Capacity

Maximum Measurable CBR	Minimum Load Capacity	
	lbf	kN
20	2500	11.2
50	5000	22.3
> 50	10000	44.5

5.2 Mold: The mold shall be a rigid metal cylinder with an inside diameter of 6 ± 0.026 inch (152.4 ± 0.66 mm) and a height of 7 ± 0.018 inch (177.8 ± 0.46 mm). It shall be provided with a metal extension collar at least 2.0 inch (50.8 mm) in height and a metal base plate having at least twenty eight $\frac{1}{16}$inch (1.59 mm) diameter holes uniformly spaced over the plate within the inside circumference of the mold. When assembled with spacer disc in place in the bottom of the mold, the mold shall have an internal volume (excluding extension collar) of 0.075 ± 0.0009 ft (2124 ± 25 cm). Figure 16.1 shows a satisfactory mold design. A calibration procedure should be used to confirm the actual volume of the mold with the spacer disk inserted. Suitable calibrations are contained in Test Methods D 698 and D 1557.

5.3 Spacer Disk: A circular metal spacer disc (see Figure 16.1) having a minimum outside diameter of $5\frac{15}{16}$inches (150.8 mm) but no greater than will allow the spacer to easily slip into the mold. The spacer disc shall be 2.416 ± 0.005 inch (61.37 ± 0.127 mm) in height.

5.4 Rammer: A rammer as specified in either Test Methods D 698 or D 1557 except that if a mechanical rammer is used it must be equipped with a circular foot, and when so equipped, must provide a means for distributing the rammer blows uniformly over the surface of the soil when compacting in a 6 inch (152.4 mm) diameter mold. The mechanical rammer must be calibrated and adjusted in accordance with Test Methods D 2168.

5.5 Expansion Measuring Apparatus: An adjustable metal stem and perforated metal plate, similar in configuration to that shown in Figure 16.1. The perforated plate shall be $5\frac{7}{8}$ to $5\frac{15}{16}$ inch (149.23 to 150.81 mm) in diameter and have at least forty two $\frac{1}{16}$ inch (1.59 mm) diameter holes uniformly spaced over the plate. A metal tripod to support the dial gage for measuring the amount of swell during soaking is also required.

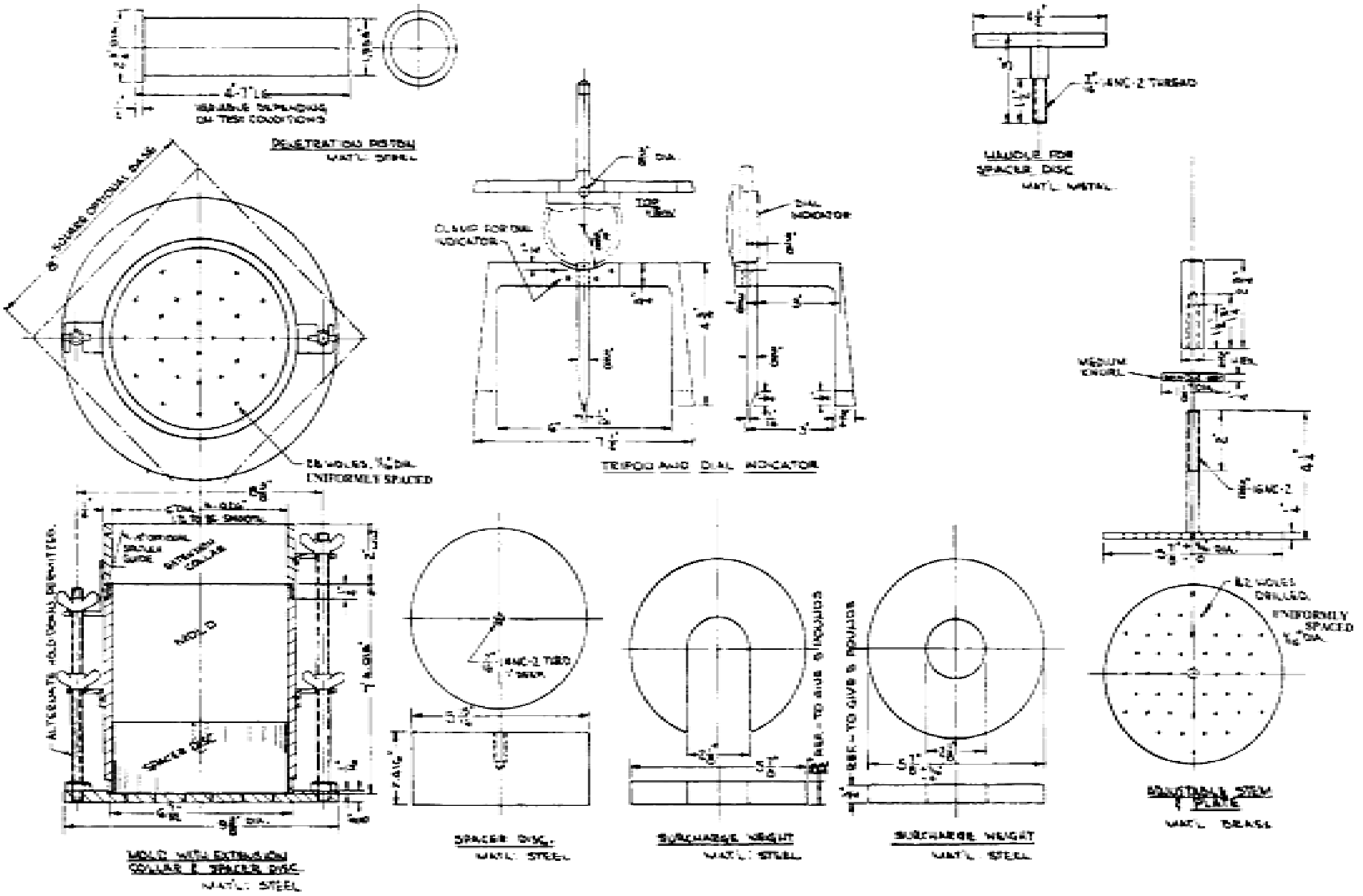


Figure 16.1 Bearing Ratio Test Apparatus (See Table 16.2 for metric equivalents)

5.6 Weights: One or two annular metal weights having a total mass of 4.54 ± 0.02 kg and slotted metal weights each having masses of 2.27 ± 0.02 kg. The annular weight shall be $5\frac{7}{8}$ to $5\frac{15}{16}$inches (149.23 to 150.81 mm) in diameter and shall have a center hole of approximately $2\frac{1}{8}$inch (53.98 mm).

5.7 Penetration Piston: A metal piston 1.954 ± 0.005 inch (49.63 ± 0.13 mm) in diameter and not less than 4 inch (101.6 mm) long (see Figure 16.1). If, from an operational standpoint, it is advantageous to use a piston of greater length, the longer piston may be used.

5.8 Gages: Two dial gages reading to 0.001 inch (0.025 mm) with a range of 0.200 minimum.

5.9 Miscellaneous Apparatus: Other general apparatus such as a mixing bowl, straightedge, scales, soaking tank or pan, oven, fast filtering high wet strength filter paper, dishes, and 2inch, $\frac{3}{4}$inch and No. 4 sieves.

Table 16.2: Metric Equivalents

Inch-Pound Units, in.	Metric Equivalent, mm	Inch-Pound Units, in.	Metric Equivalent, mm	Inch-Pound Units, in.	Metric Equivalent, mm
0.003	0.076	19⁄32	15.08	3 1⁄2	88.90
0.005	0.127	5⁄8	15.88	3 3⁄4	95.25
0.135	3.43	3⁄4	19.10	4 1⁄4	108.0
0.201	5.11	15⁄16	23.81	4 1⁄2	114.3
0.4375	11.11	1	25.40	4 3⁄4	120.7
0.4378	11.12	1 1⁄8	28.58	5 7⁄8	149.2
0.510	12.95	1 1⁄4	31.8	5 15⁄16	150.8
0.633	16.08	1 3⁄8	34.9	6	152.0
1.370	34.60	1 1⁄2	38.10	6 7⁄32	158.0
1.375	34.93	1 3⁄4	44.5	6 1⁄2	165.1
1.954	49.63	1 13⁄16	46.04	7	177.8
2.416	61.37	1 15⁄16	49.21	7 1⁄2	190.1
1⁄16	1.59	2	50.80	8 3⁄8	212.7
7⁄32	5.56	2 1⁄8	53.98	8 1⁄2	215.9
1⁄4	6.35	2 1⁄5	55.9	9 3⁄8	238.1
3⁄8	9.53	2 1⁄4	57.2	14 1⁄4	362.0
7⁄16	11.11	2 1⁄2	63.50	18	457.2
15⁄32	11.91	2 3⁄4	69.85	32 1⁄4	719.2
1⁄2	12.70	2 31⁄32	75.41	36 5⁄8	930.3
17⁄32	13.49	3	76.20	39	990.6

Inch-Pound Units, lb	Metric Equivalent, kg	Inch-Pound Units, psi	Metric Equivalent, MPa
0.04	0.02	200	1.4
0.05	0.02	400	2.8
0.12	0.05	600	4.1
0.59	0.27	800	5.5
0.71	0.32	1000	6.9
0.75	0.34	1200	8.3
3.20	1.45	1400	9.7
5.00	2.27		
10.00	4.54		

6. SAMPLE

6.1 The sample shall be handled and specimen(s) for compaction shall be prepared in accordance with the procedures given in Test Methods D 698 or D 1557 for compaction in a 6 inch (152.4 mm) mold except as follows:

6.1.1 If all material passes a ¾ inch (19 mm) sieve, the entire gradation shall be used for preparing specimens for compaction without modification. If there is material retained on the ¾inch (19 mm) sieve, the material retained on the ¾inch (19 mm) sieve shall be removed and replaced by an equal amount of material passing the ¾inch (19 mm) sieve and retained on the No. 4 sieve obtained by separation from portions of the sample not otherwise used for testing.

7. TEST SPECIMENS

7.1 Bearing Ratio at Optimum Water Content Only: Using material prepared as described in 6.1, conduct a control compaction test with a sufficient number of test specimens to definitely establish the optimum water content for the soil using the compaction method specified, either Test Methods D 698 or D 1557. A previously performed compaction test on the same material may be substituted for the compaction test just described, provided that if the sample contains material retained on the ¾inch (19 mm) sieve, soil prepared as described in 6.1 is used (Note 2).

NOTE 2 Maximum dry unit weight obtained from a compaction test performed in a 4 inch (101.6 mm) diameter mold may be slightly greater than the maximum dry unit weight obtained from compaction in the 6 inch (152.4 mm) compaction mold or CBR mold.

7.1.1 For cases where the CBR is desired at 100 % maximum dry unit weight and optimum water content, compact a specimen using the specified compaction procedure, either Test Methods D 698 or D 1557, from soil prepared to within ±0.5 percentage point of optimum water content in accordance with Test Method D 2216.

7.1.2 Where the CBR is desired at optimum water content and some percentage of maximum dry unit weight, compact three specimens from soil prepared to within ±0.5 percentage point of optimum water content and using the specified compaction but using a different number of blows per layer for each specimen. The number of blows per layer shall be varied as necessary to prepare specimens having unit weights above and below the desired value. Typically, if the CBR for soil at 95 % of maximum dry unit is desired, specimens compacted using 56, 25, and 10 blows per layer is satisfactory. Penetration shall be performed on each of these specimens.

7.2 Bearing Ratio for a Range of Water Content: Prepare specimens in a manner similar to that described in 7.1 except that each specimen used to develop the compaction curve shall be penetrated. In addition, the complete water content unit weight relation for the 25 blows and 10 blows per layer compactions shall be developed and each test specimen compacted shall be penetrated. Perform all compaction in the CBR mold. In cases where the specified unit weight is at or near 100% maximum dry unit weight, it will be necessary to include a compaction effort greater than 56 blows per layer (Note 3).

NOTE 3 A semi-log log plot of dry unit weight versus compaction effort usually gives a straight line relation when compaction effort in ft-lb/ft^3 *is plotted on the log scale. This type of plot is useful in establishing the compaction effort and number of blows per layer needed to bracket the specified dry unit weight and water content range.*

7.2.1 If the sample is to be soaked, take a representative sample of the material, for the determination of moisture, at the beginning of compaction and another sample of the remaining material after compaction. Use Test Method D 2216 to determine the moisture content. If the sample is not to be soaked, take a moisture content sample in accordance with Test Methods D 698 or D 1557 if the average moisture content is desired.

7.2.2 Clamp the mold (with extension collar attached) to the base plate with the hole for the extraction handle facing down. Insert the spacer disk over the base plate and place a disk of filter paper on top of the spacer disk. Compact the soil water mixture into the mold in accordance with 7.1, 7.1.1, or 7.1.2.

7.2.3 Remove the extension collar and carefully trim the compacted soil even with the top of the mold by means of a straightedge. Patch with smaller size material any holes that may have developed in the surface by the removal of coarse material. Remove the perforated base plate and spacer disk, weigh, and record the mass of the mold plus compacted soil. Place a disk of coarse filter paper on the perforated base plate, invert the mold and compacted soil, and clamp the perforated base plate to the mold with compacted soil in contact with the filter paper.

7.2.4 Place the surcharge weights on the perforated plate and adjustable stem assembly and carefully lower onto the compacted soil specimen in the mold. Apply a surcharge equal to the weight of the base material and pavement within 2.27 kg (5 lb), but in no case shall the total weight used be less than 4.54 kg (10 lb). If no pavement weight is specified, use 4.54 kg. Immerse the mold and weights in water allowing free access of water to the top and bottom of the specimen. Take initial measurements for swell and allow the specimen to soak for 96 hr. Maintain a constant water level during this period. A shorter immersion period is permissible for fine grained soils or granular soils that take up moisture readily, if tests show that the shorter period does not affect the results. At the end of 96 hr, take final swell measurements and calculate the swell as a percentage of the initial height of the specimen.

7.2.5 Remove the free water and allow the specimen to drain downward for 15 min. Take care not to disturb the surface of the specimen during the removal of the water. It may be necessary to tilt the specimen in order to remove the surface water. Remove the weights, perforated plate, and filter paper, and determine and record the mass.

8. PROCEDURE FOR BEARING TEST

8.1 Place a surcharge of weights on the specimen sufficient to produce an intensity of loading equal to the weight of the base material. If no pavement weight is specified, use 4.54 kg mass. If the specimen has been soaked previously, the surcharge shall be equal to that used during the soaking period. To prevent upheaval of soil into the hole of the surcharge weights, place the 2.27

kg annular weight on the soil surface prior to seating the penetration piston, after which place the remainder of the surcharge weights.

8.2 Seat the penetration piston with the smallest possible load, but in no case in excess of 10 lbf (44 N). Set both the stress and penetration gages to zero. This initial load is required to ensure satisfactory seating of the piston and shall be considered as the zero load when determining the load penetration relation. Anchor the strain gage to the load measuring device, if possible; in no case attach it to the testing machines support bars (legs).

NOTE 4 At high loads the supports may torque and affect the reading of the penetration gage. Checking the depth of piston penetration is one means of checking for erroneous strain indications.

8.3 Apply the load on the penetration piston so that the rate of penetration is approximately 0.05 inch (1.27 mm)/min. Record the load readings at penetrations of 0.025 inch (0.64 mm), 0.050 inch (1.27 mm), 0.075 inch (1.91 mm), 0.100 inch (2.54 mm), 0.125 inch (3.18 mm), 0.150 inch (3.81 mm), 0.175 inch (4.45 mm), 0.200 inch (5.08 mm), 0.300 inch (7.62 mm), 0.400 inch (10.16 mm) and 0.500 inch (12.70 mm). Note the maximum load and penetration if it occurs for a penetration of less than 0.500 inch (12.70 mm). With manually operated loading devices, it may be necessary to take load readings at closer intervals to control the rate of penetration. Measure the depth of piston penetration into the soil by putting a ruler into the indentation and measuring the difference from the top of the soil to the bottom of the indentation. If the depth does not closely match the depth of penetration gage, determine the cause and test a new sample.

8.4 Remove the soil from the mold and determine the moisture content of the top 1 inch (25.4 mm) layer. Take a moisture content sample in accordance with Test Methods D 698 or D 1557 if the average moisture content is desired. Each moisture content sample shall weigh not less than 100 g for fine grained soils nor less than 500 g for granular soils.

NOTE 5 The load readings at penetrations of over 0.300 inch (7.6 mm) may be omitted if the testing machine’s capacity has been reached.

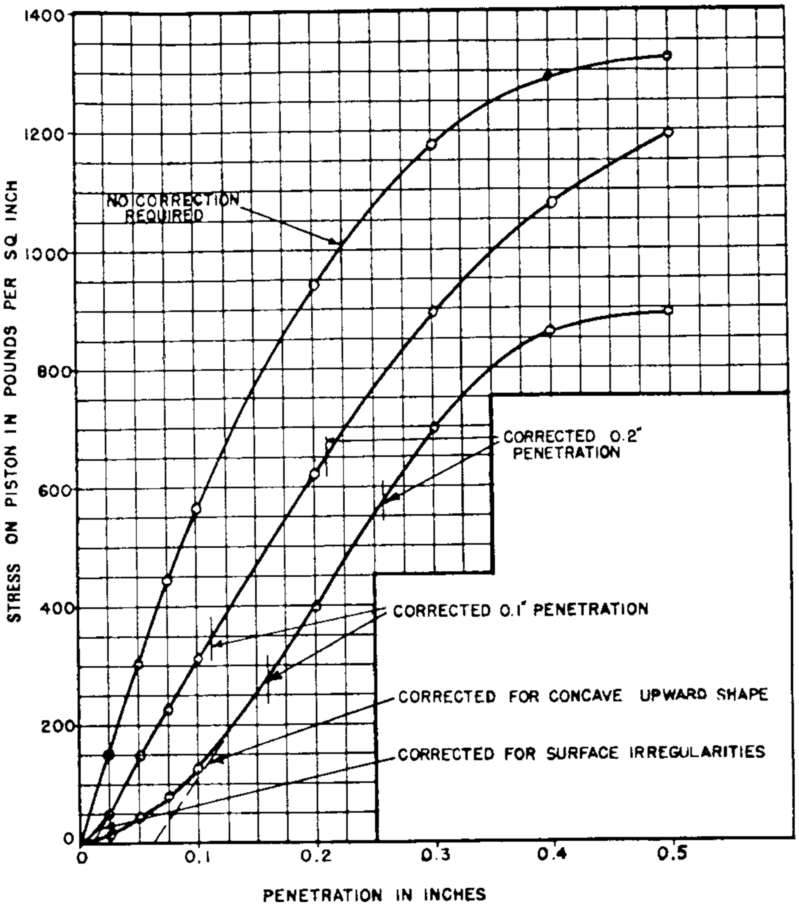
9. CALCULATION

9.1 Load-Penetration Curve: Calculate the penetration stress in pounds per square inch or mega pascals and plot the stress-penetration curve. In some instances, the stress-penetration curve may be concave upward initially, because of surface irregularities or other causes, and in such cases the zero point shall be adjusted as shown in Figure 16.2.

NOTE 6 Figure 16.2 should be used as an example of correction of load-penetration curves only. It is not meant to imply that the 0.2 inch penetration is always more than the 0.1 inch penetration.

9.2 Bearing Ratio: Using corrected stress values taken from the stress penetration curve for 0.100 inch (2.54 mm) and 0.200 inch (5.08 mm) penetrations, calculate the bearing ratios for each by dividing the corrected stresses by the standard stresses of 1000 psi (6.9 MPa) and 1500 psi (10.3 MPa) respectively, and multiplying by 100. Also, calculate the bearing ratios for the maximum stress, if the penetration is less than 0.200 inch (5.08 mm) interpolating the standard stress. The bearing ratio reported for the soil is normally the one at 0.100 inch (2.54 mm) penetration. When the ratio at 0.200 inch (5.08 mm) penetration is greater, rerun the test. If the check test gives a similar result, use the bearing ratio at 0.200 inch (5.08 mm) penetration.

NOTE 7 If bearing ratio values at penetrations of 0.300 inch (7.62 mm), 0.400 inch (10.16 mm) and 0.500 inch (12.7 mm) are desired, the corrected stress values of these penetrations should be divided by the standard stresses of 1900 psi (13.1 MPa), 2300 psi (15.9 MPa), 2600 psi (17.9 MPa), respectively, and multiplied by 100.



NOTE: See Table 16.2 for metric equivalents

Figure 16.2 Correction of Load-Penetration Curves

9.3 Design CBR for One Water Content Only: Using the data obtained from the three specimens, plot the CBR versus molded dry unit weight relation as illustrated in Figure 16.3. Determine the design CBR at the percentage of the maximum dry unit weight requested.

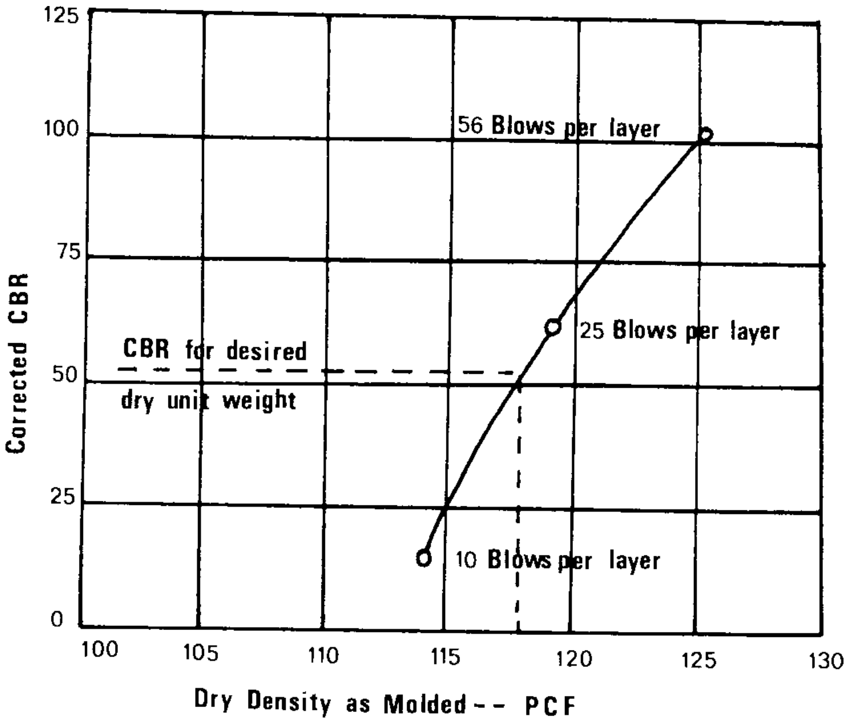
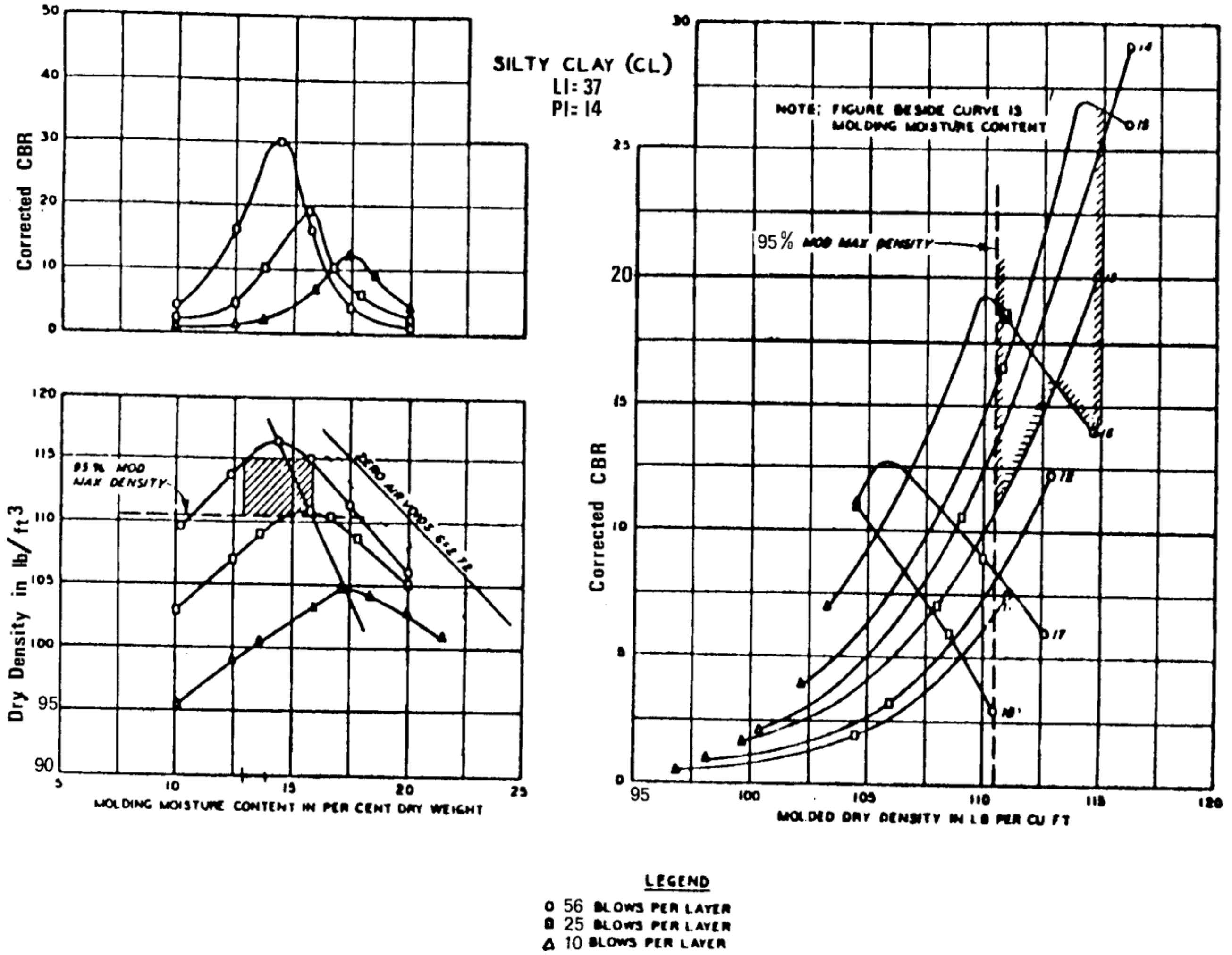


Figure 16.3 Dry density Vs. CBR

9.4 Design CBR for Water Content Range: Plot the data from the tests at the three compactive efforts as shown in Figure 16.4. The data plotted as shown represents the response of the soil over the range of water content specified. Select the CBR for reporting as the lowest CBR within the specified water content range having a dry unit weight between the specified minimum and the dry unit weight produced by compaction within the water content range.



NOTE Surcharge = 50 lb soaking and penetration. All samples soaked top and bottom four days. All samples compacted in 5 layers, 10-lb hammer, 18-in. drop in CBR mold.

Figure 16.4 Determining CBR for Water Content Range and Minimum Dry Unit Weight

10. REPORT

10.1 The report shall include the following:

10.1.1 Method used for preparation and compaction of specimen: Test Methods D 698 or D 1557, or other, with description.
10.1.2 Condition of sample (unsoaked or soaked).
10.1.3 Dry density (unit weight) of sample before soaking, kg/m^3 (lb/ft^3).
10.1.4 Dry density (unit weight) of sample after soaking kg/m^3 (lb/ft^3).
10.1.5 Moisture content of sample in percent:

10.1.5.1 Before compaction.
10.1.5.2 After compaction.
10.1.5.3 Top 1 inch (25.4 mm) layer after soaking.
10.1.5.4 Average after soaking.
10.1.6 Swell (percentage of initial height).
10.1.7 Bearing ratio of sample (unsoaked or soaked), percent.
10.1.8 Surcharge amount.
10.1.9 Any special sample preparation and testing procedures (for example: for self cementing materials).
10.1.10 Sample identification (location, boring number, etc.).
10.1.11 Any pertinent testing done to identify the sample such as: soil classifications per Test Method D 2487, visual classification per Practice D 2488, Atterberg limits per Test Method D 4318, gradation per Method D 422 etc.
10.1.12 The percent material retained on the 19 mm sieve for those cases where scalping and replacement is used.

11. PRECISION AND BIAS

11.1 No available methods provide absolute values for the soil bearing strength derived by this test method; therefore, there is no meaningful way to obtain an evaluation of bias.

11.2 At present, sufficient data for determining the precision of this test method has not been gathered. Users are encouraged to submit data to the subcommittee for inclusion in the statement. One user, based on seven repetitions, has developed a IS % of 8.2 % (compacted per Test Method D 698) and 5.9 % (compacted per Test Method D 1557). See Appendix X1 for the data used.

Experiment No: 16
Standard Test Method for CBR of Laboratory Compacted Soils

Name: **Student No**:

Mold: Height of Mold = Diameter of Mold = **Spacer Disk :** Height of Spacer Disk = Compacted Height of Sample = Volume of Compacted Sample =	**Calibration of Proving Ring:** …………………………………………………… y = load in lb. x = load dial reading

Bulk Density of Sample:

Weight of Soil + Mold =

Weight of Mold =

Weight of Soil

Bulk Density = (Weight of Soil/Volume of Compacted Soil) =

Moisture Content of Sample:

Wet Weight of (Container + Soil) =

Dry Weight of (Container + Soil) =

Weight of Container =

Weight of wet soil, W_w =

Weight of dry soil, W_d=

Moisture Content = $(W_w - W_d)/W_d$ =

Penetration Stress Calculation

Diameter of Penetration Plunger =

Area of Penetration Plunger =

1	**2**	**3**	**4**
Penetration (inch)	**Proving Ring Dial Reading**	**Piston Load (lb)**	**Penetration Stress (psi)**
0			
0.025			
0.05			
0.075			
0.1			
0.125			
0.15			
0.175			
0.2			
0.3			
0.4			
0.5			

CBR $_{0.1^{//}}$ =

CBR $_{0.2^{//}}$ =

Signature of Course Teacher

EXPERIMENT NO: 17

ASPHALT CONCRETE MIX DESIGN BY MARSHALL METHOD

(ASPHALT INSTITUTE MANUAL SERIES NO. 2 (MS-2), Ch. 4 & Ch. 6, SIXTH EDITION 1997)

VOLUMETRIC PROPERTIES OF COMPACTED PAVING MIXTURES

1.1 GENERAL

The volumetric properties of a compacted paving mixture [air voids (V_a), voids in the mineral aggregate (VMA), voids filled with asphalt (VFA), and effective asphalt content (P_{be})) provide some indication of the mixture's probable pavement service performance. The intent of laboratory compaction is to simulate the in place density of HMA after it has endured several years of traffic. How well the laboratory compaction procedure simulates either the compacted state immediately after construction or after years of service can be determined by comparing the properties of an undisturbed sample removed from a pavement with the properties of a sample of the same paving mixture competed in the laboratory.

It is necessary to understand the definitions and analytical procedures described here to be able to make informed decisions concerning the selection of the design asphalt mixture. The information here applies to both paving mixtures that have been compacted in the laboratory, and to undisturbed samples that have been removed from a pavement in the field.

A comparison of field and laboratory compacted mix properties has been made in several research studies. Statistical analysis of these data has failed to establish one laboratory compaction method that consistently produces the closest simulation to the field for all of the measured properties. However, there is a trend toward the gyratory method of compaction based on these findings and other subjective factors. This is a very complicated issue. Compaction method, level of compaction, structural concerns, construction conditions and other influences can all make a difference in these comparisons. Assuming that a reasonable degree of simulation is achieved by whatever compaction procedures are used, it is universally agreed that the air void analysis is an important part of mix design.

1.2 DEFINITIONS

Mineral aggregate is porous and can absorb water and asphalt to a variable degree. Furthermore, the ratio of water to asphalt absorption varies with each aggregate. The three methods of measuring aggregate specific gravity take these variations into consideration. The methods are ASTM bulk, ASTM apparent and effective specific gravities. The differences among the specific gravities come from different definitions of aggregate volume.

Bulk Specific Gravity, G_{sb} - the ratio of the weight in air of a unit volume of permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of equal density of an equal volume of gas free distilled water at a stated temperature. See Figure 17.1.

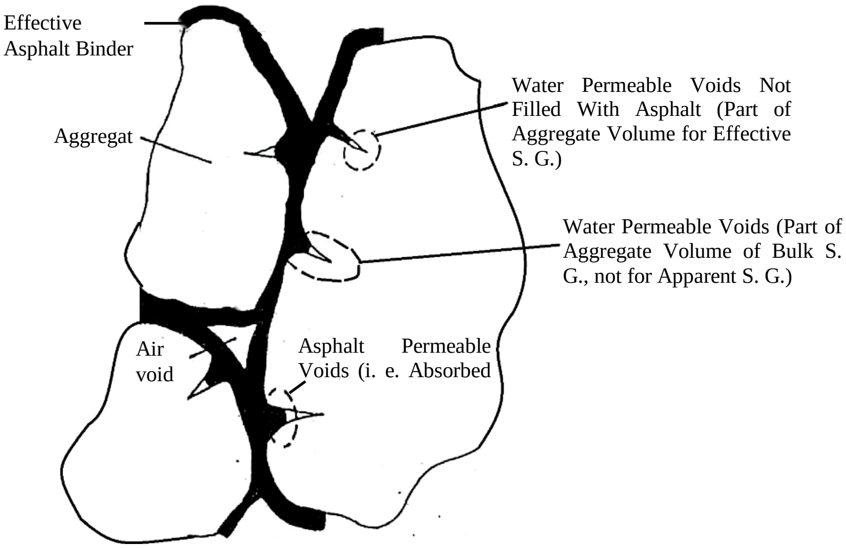


Figure 17.1 Illustrating bulk, effective, and apparent specific gravities; air voids and effective, asphalt content in compacted asphalt paving mixture

Apparent Specific Gravity, G_{sa} - the ratio of the weight in air of a unit volume of an impermeable material at a stated temperature to the weight in air of equal density of an equal volume of gas free distilled water at a stated temperature. See Figure 17.1.

Effective Specific Gravity, G_{se} - the ratio of the weight in air of a unit volume of a permeable material (excluding voids permeable to asphalt) at a stated temperature to the weight in air of equal density of an equal volume of gas free distilled water at a stated temperature. See Figure 17.1.

NOTE: The accuracy of specific gravity measurements for mix design is important. Unless specific gravities are determined to four significant figures (three decimal places) an error in air voids value of as much as 0.8 percent can occur. Therefore, the Asphalt Institute recommends the use of weigh scales whose sensitivity will allow a mix batch weighing 1000 to 5000 grams to be measured to an accuracy of 0.1 gram.

V_{ma} = Volume of voids in mineral aggregate
V_{mb} = Bulk volume of compacted mix
V_{mm} = Void less volume of paving mix
V_{fa} = Volume of voids filled with asphalt
V_a = Volume of air voids
V_b = Volume of asphalt
V_{ba} = Volume of absorbed asphalt
V_{sb} = Volume of mineral aggregate (by bulk specific gravity)
V_{se} = Volume of mineral aggregate (by effective specific gravity)

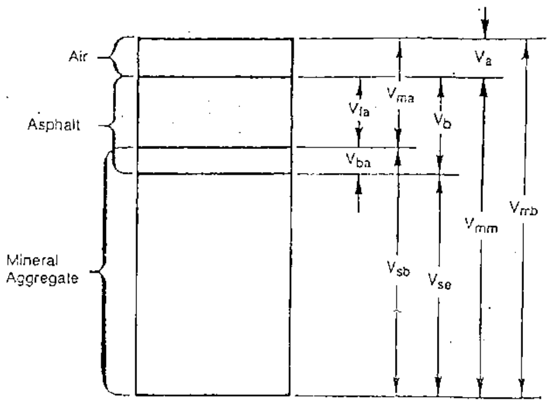


Figure 17.2 Representation of volumes in a compacted asphalt specimen

The definitions for voids in the mineral aggregate (VMA), effective asphalt content (P_{be}), air voids (V_a) and voids filled with asphalt (VFA) are:

Voids in the Mineral Aggregate, VMA - the volume of inter granular void space between the aggregate particles of a compacted paving mixture that includes the air voids and the effective asphalt content, expressed as a percent of the total volume of the sample. See Figure 17.2.

Effective Asphalt Content, P_{be} - the total asphalt content of a paving mixture minus the portion of asphalt that is lost by absorption into the aggregate particles. See Figure 17.2.

Air Voids, V_a - the total volume of the small pockets of air between the coated aggregate particles throughout a compacted paving mixture, expressed as percent of the bulk volume of the compacted paving mixture. See Figure 17.2.

Voids Filled with Asphalt, VFA - the portion of the volume of intergranular void space between the aggregate particles (VMA) that is occupied by the effective asphalt. See Figure 17.2.

The Asphalt Institute recommends that VMA values for compacted paving mixtures should be calculated in terms of the aggregate's bulk specific gravity, G_{sb}. The effective specific gravity should be the basis for calculating the air voids in a compacted asphalt paving mixture.

Table 17.1 illustrates that the type of aggregate specific gravity used in the analysis of a compacted paving mixture can have a very dramatic effect on the values reported for air voids and VMA. These differences are enough to make it appear that a mixture may satisfy or fail the design criteria for air voids and VMA depending on the aggregate specific gravity used for analysis. Asphalt Institute mix design criteria do not apply unless VMA calculations are made using bulk specific gravity and air void content calculations are made using effective specific gravity.

Voids in the mineral aggregate (VMA) and air voids (V_a) are expressed as percent by volume of the paving mixture. Voids filled with asphalt (VFA) is the percentage of VMA that is filled by the effective asphalt. Depending on how asphalt content is specified, the effective asphalt content may be expressed either as percent by weight of the total weight of the paving mixture, or as percent by weight of the aggregate in the paving mixture.

Because air voids, VMA and VFA are volume quantities and therefore cannot be weighed, a paving mixture must first be designed or analyzed on a volume basis. For design purposes, this volume approach can easily be changed over to a weight basis to provide a job mix formula.

1.3 OUTLINE OF PROCEDURE FOR ANALYZING A COMPACTED PAVING MIXTURE

This list delineates all the measurements and calculations needed for a void analysis:
(a) Measure the bulk specific gravities of the coarse aggregate (AASHTO T 85 or ASTM C 127) and of the fine aggregate (AASHTO T 84 or ASTM C 128).
(b) Measure the specific gravity of the asphalt cement (AASHTO T 228 or ASTMD 70) and of the mineral filler (AASHTO T 100 or ASTM D 854).
(c) Calculate the bulk specific gravity of the aggregate combination in the paving mixture.
(d) Measure the maximum specific gravity of the loose paving mixture (ASTMD 2041).
(e) Measure the bulk specific gravity of the compacted paving mixture (ASTM D 1188 or ASTM D 2726).
(f) Calculate the effective specific gravity of the aggregate.
(g) Calculate the maximum specific gravity of the mix at other asphalt contents.
(h) Calculate the asphalt absorption of the aggregate.

Table 17.1: Influence of type of specific gravity on determination of VMA, VFA and air voids

Specific gravity Employed for Aggregate		Allowance For Absorption of Asphalt by Aggregate	Void Properties Compacted Mixture		
			Percent Voids in Mineral Aggregate	Percent Air Voids	Percent Voids Filled With Asphalt
ASTM Bulk	2.651	Yes	13.6	1.1	92
ASTM Bulk	2.651	No	13.6	-0.8	106
ASTM Bulk (sat. surf. dry)	2.716	Yes	15.6	3.2	79
ASTM Bulk (sat. surf. dry)	2.716	No	15.6	1.3	92
ASTM Apparent	2.834	No	19.1	4.9	74
Effective	2.708	No	15.4	1.1	93

Bulk Specific Gravity of Compacted Mixture, G_{mb}	2.436
Density of Compacted Mixture, W_r, Mg/m^3 (lb/ft^3)	2.435 (152.0)
Asphalt Content, percent by weight of total mix	5.9
Asphalt Absorbed by Aggregate Particles, percent	0.8
Specific Gravity of Asphalt Cement, G_b	1.011

(i) Calculate the effective asphalt content of the paving mixture.
(j) Calculate the percent voids in the mineral aggregate in the compacted paving mixture.

(k) Calculate the percent air voids in the compacted paving mixture.
(l) Calculate the percent voids filled with asphalt.
Equations for these calculations are found in Articles 1.5 through 1.11 and their application may be expedited by use of the appropriate worksheet.

1.4 PAVING MIXTURE DATA FOR SAMPLE CALCULATIONS

Table 17.2 illustrates the basic data for a sample of paving mixture. These design data are used in the sample calculations used in the remainder of this chapter.

1.5 BULK SPECIFIC GRAVITY OF AGGREGATE

When the total aggregate consists of separate fractions of coarse aggregate, fine aggregate and mineral filler, all having different specific gravities, the bulk specific gravity for the total aggregate is calculated using:

$$G_{sb} = \frac{P_1 + P_2 + \ldots + P_n}{P_1/G_1 + P_2/G_2 + \ldots + P_n/G_n}$$

Where, G_{sb}= bulk specific gravity for the total aggregate
P_1, P_2, P_n= individual percentages by weight of aggregate
G_1, G_2, G_n= individual bulk specific gravities of aggregate

Table 17.2: Basic data for sample of paving mixture:

a) Constituents

Material	Specific Gravity				Mix Composition	
		Bulk	AASHTO Method	ASTM Method	Percent By Weight of Total Mix	Percent By Weight of Total Aggregate
Asphalt Cement	1.030 (G_b)		T 228	D 70	5.3 (P_b)	5.6 (P_b)
Coarse Aggregate		2.716 (G_1)	T 85	C 127	47.4 (P_1)	50.0 (P_1)
Fine Aggregate		2.689 (G_2)	T 84	C 128	47.3 (P_2)	50.0 (P_2)
Mineral Filler	----		T 100	D 854	----	----

b) Paving Mixture

Bulk specific gravity of compacted paving mixture sample, G_{mb} (ASTM D 2726) = 2.442
Maximum specific gravity of paving mixture sample, G_{mm} (ASTM D 2041) = 2.535

The bulk specific gravity of mineral filler is difficult to determine accurately. However, if the apparent specific gravity of the filler is substituted, the error is usually negligible.
Using the data in Table 17.2:

$$G_{sb} = \frac{50.0 + 50.0}{50.0/2.716 + 50.0/2.689} = 2.703$$

1.6 EFFECTIVE SPECIFIC GRAVITY OF AGGREGATE

When based on the maximum specific gravity of a paving mixture, G_{mm}, as measured using ASTM D 2041, the effective specific gravity of the aggregate, G_{se}, includes all void spaces in the aggregate particles except those that absorb asphalt. G_{se} is determined using:

$$G_{se} = \frac{P_{mm} - P_b}{P_{mm}/G_{mm} - P_b/G_b}$$

Where, G_{se} = effective specific gravity of aggregate
G_{mm} = maximum specific gravity (ASTM D 2041) of paving mixture (no air voids)
P_{mm} = percent by weight of total loose mixture = 100
P_b = asphalt content at which ASTM D 2041 test was performed, percent by total weight of mixture
G_b= specific gravity of asphalt

Using the data in Table 17.2:

$$G_{se} = \frac{100 - 5.3}{100/2.535 - 5.3/1.030} = 2.761$$

NOTE: The volume of asphalt binder absorbed by an aggregate is almost invariably less than the volume of water absorbed. Consequently, the value for the effective specific gravity of an aggregate should be between its bulk and apparent specific gravities. When the effective specific gravity falls outside these limits, its value must be assumed to be incorrect. The calculations, the maximum specific gravity of the total mix by ASTM D 2041, and the composition of the mix in terms of aggregate and total asphalt content should then be rechecked for the source of the error.
The apparent specific gravity, G_{sa}, of the total aggregate can be calculated by the same formula as the bulk by using the apparent specific gravity of each aggregate constituent.

1.7 MAXIMUM SPECIFIC GRAVITY OF MIXTURES WITH DIFFERENT ASPHALT CONTENTS

In designing a paving mixture with a given aggregate, the maximum specific gravity, G_{mm}, at each asphalt content is needed to calculate the percentage of air voids for each asphalt content. While the maximum specific gravity can be determined for each asphalt content by ASTM D 2041, the precision of the test is best when the mixture is close to the design asphalt content. Also it is preferable to measure the maximum specific gravity in duplicate or triplicate. After calculating the effective specific gravity of the aggregate from each measured maximum specific gravity (see Article 1.6) and averaging the G_{se} results, the maximum specific gravity for any other asphalt content can be obtained as shown below. For all practical purposes, the effective specific gravity of the aggregate is constant because the asphalt absorption does not vary appreciably with variations in asphalt content.

$$G_{mm} = \frac{P_{mm}}{P_s/G_{se} + P_b/G_b}$$

Where, G_{mm} = maximum specific gravity of paving mixture (no air voids)
P_{mm} = percent by weight of total loose mixture = 100
P_s = aggregate content, percent by total weight of mixture
P_b = asphalt content, percent by total weight of mixture
G_{se} = effective specific gravity of aggregate
G_b = specific gravity of asphalt

Using the specific gravity data from Table 17.2, and the effective specific gravity, G_{se}, determined in article 1.6, the G_{mm} at an asphalt content, P_b, of 4 percent would be:

$$G_{mm} = \frac{100}{96/2.761 + 4/1.030}$$

1.8 ASPHALT ABSORPTION

Absorption is expressed as a percentage by weight of aggregate rather than as a percentage by total weight of mixture. Asphalt absorption, P_{ba}, is determined using:

$$P_{ba} = 100\ \frac{G_{se} - G_{sb}}{G_{se}G_{sb}} G_b$$

Where, P_{ba} = absorbed asphalt, percent by weight of aggregate
G_{se} = effective specific gravity of aggregate
G_{sb}= bulk specific gravity of aggregate
G_b= specific gravity of asphalt

Using the bulk and effective aggregate specific gravities determined in Articles 1.5 and 1. 6 and the asphalt specific gravity from Table 17.2:

$$P_{ba} = 100 \left(\frac{2.761 - 2.703}{2.761 \times 2.703}\right) 1.030 = 0.8$$

1.9 EFFECTIVE ASPHALT CONTENT OF A PAVING MIXTURE

The effective asphalt content, P_{be}, of a paving mixture is the total asphalt content minus the quantity of asphalt lost by absorption into the aggregate particles. It is the portion of the total asphalt content that remains as a coating on the outside of the aggregate particles and it is the asphalt content which governs the performance of an asphalt paving mixture. The formula is:

$$P_{be} = P_b - \frac{P_{ba}}{100} P_s$$

Where, P_{be} = effective asphalt content, percent by total weight of mixture
P_b = asphalt content, percent by total weight of mixture
P_{ba} = absorbed asphalt, percent by weight of aggregate
P_s = aggregate content, percent by total weight of mixture
Using the data from Table 17.2 and Article 1.8:

$$P_{be} = 5.3 - \frac{0.8}{100} \times 94.7 = 4.5$$

1.10 PERCENT VMA IN COMPACTED PAVING MIXTURE

The voids in the mineral aggregate, VMA, are defined as the inter granular void space between the aggregate particles in a compacted paving mixture that includes the air voids and the effective asphalt content, expressed as a percent of the total volume. The VMA is calculated on the basis of the bulk specific gravity of the aggregate and is expressed as a percentage of the bulk volume of the compacted paving mixture. Therefore, the VMA can be calculated by subtracting the volume of the aggregate determined by its bulk specific gravity from the bulk volume of the compacted paving mixture. A method of calculation is illustrated for each type of mixture percentage content.

If the mix composition is determined as percent by weight of total mixture:

$$VMA = 100 - \frac{G_{mb} P_s}{G_{sb}}$$

Where, VMA = voids in mineral aggregate, percent of bulk volume
G_{sb} = bulk specific gravity of total aggregate
G_{mb} = bulk specific gravity of compacted mixture (AASHTO T 166; ASTM D 1188 or
D 2726)
P_s = aggregate content, percent by total weight of mixture

Using the data from Table 17.2 and Article 1.5:

$$VMA = 100 - \frac{2.442 \times 94.7}{2.703} = 14.4$$

Or if the mix composition is determined as percent by weight of aggregate:

$$VMA = 100 - \frac{G_{mb}}{G_{sb}} \times \frac{100}{100 + P_b} 100$$

Where, P_b = asphalt content, percent by weight of aggregate
Using the data from Table 17.2 and Article 1.5:

$$VMA = 100 - \frac{2.442}{2.703} \times \frac{100}{100 + 5.6} \times 100 = 14.4$$

1.11 PERCENT AIR VOIDS IN COMPACTED PAVING MIXTURE

The air voids, V_a, in the total compacted paving mixture consist of the small air spaces between the coated aggregate particles. The volume percentage of air voids in a compacted mixture can be determined using:

$$V_a = 100 \times \frac{G_{mm} - G_{mb}}{G_{mm}}$$

Where, V_a = air voids in compacted mixture, percent of total volume
G_{mm} = maximum specific gravity of paving mixture (as determined in Article 1.7 or as measured directly for a paving mixture by ASTM D 2041)
G_{mb} = bulk specific gravity of compacted mixture (AASHTO T 166; ASTM D 1188 or D 2726)
Using data from Table 17.2:

$$V_a = 100 \times \frac{2.535 - 2.442}{2.535} = 3.7$$

1.12 PERCENT VFA IN COMPACTED PAVING MIXTURE

The voids filled with asphalt, VFA, is the percentage of the inter-granular void space between the aggregate particles (VMA) that are filled with asphalt. VFA, not including the absorbed asphalt, is determined using:

$$VFA = \frac{100\,(VMA - V_a)}{VMA}$$

Where, VFA = voids filled with asphalt, percent of VMA
VMA = voids in mineral aggregate, percent of bulk volume
V_a = air voids in compacted mixture, percent of total volume
Using the data from Table 17.2 and Articles 1.10 and 1.11:

$$VFA = 100 \times \frac{14.4 - 3.7}{14.4} = 74.3 \text{ percent}$$

MARSHALL METHOD OF MIX DESIGN

A. GENERAL

2.1 DEVELOPMENT AND APPLICATION

The concepts of the Marshall method of designing paving mixtures were formulated by Bruce Marshall, a former Bituminous Engineer with the Mississippi State Highway Department. The U.S. Army Corps of Engineers, through extensive research and correlation studies, improved and added certain features to Marshall's test procedure, and ultimately developed mix design criteria. The Marshall test procedures have been standardized by the American Society for Testing and Materials. Procedures are given by ASTM D 1559, *Resistance to Plastic Flow of Bituminous Mixtures Using Marshall Apparatus*.*Testing procedures presented here are basically the same as those of the ASTM method.

The original Marshall method is applicable only to hot mix asphalt (HMA) paving mixtures containing aggregates with maximum sizes of 25 mm (1 inch) or less. A modified Marshall method has been proposed for aggregates with maximum sizes up to 38 mm (1.5 inch). The differences between this proposed method and the Original are discussed in Article 2.16. The Marshall method is intended for laboratory design and field control (Chapter 8, MS-2) of asphalt hot mix dense graded paving mixtures. Because the Marshall stability test is empirical in nature, the meaning of the results in terms of estimating relative field behavior is lost when any modification is made to the standard procedures. An example of such modification is preparing specimens from reheated or remolded materials.

**AASHTO T 245 "Resistance to plastic Flow of Bituminous Mixtures Using Marshall Apparatus" agrees with ASTM D 1559 except for provision for mechanically operated hammer. AASHTO T 245 Par. 2.3 Note 2- Instead of hand operated hammer and associated equipment, a mechanically operated hammer may be used provided it has been calibrated to give results comparable to the hand operated hammer.*

2.2 OUTLINE OF METHOD

The procedure for the Marshall method starts with the preparation of test specimens. Steps preliminary to specimen preparation are:

(a) All materials proposed for use meet the physical requirements of the project specifications.
(b)Aggregate blend combinations meet the gradation requirements of the project specifications.
(c) For performing density and voids analyses, the bulk specific gravity of all aggregates used in the blend and the specific gravity of the asphalt cement are determined.

These requirements are matters of routine testing, specifications, and laboratory technique that must be considered for any mix design method. Refer to Chapter 3 (MS-2), "Evaluation of Aggregate Gradation", for the preparation and analysis of aggregates.

The Marshall method uses standard test specimens of 64 mm ($2\frac{1}{2}$ in.) height x 102mm (4 in.) diameter. These are prepared using a specified procedure for heating, mixing and compacting the asphalt aggregate mixture. The two principal features of the Marshall method of mix design are a density-voids analysis and a stability-flow test of the compacted test specimens.

The stability the test specimen is the maximum load resistance in Newton (lb.) that the standard test specimen will develop at 60^0 C (140^0 F) when tested as outlined.

The flow value is the total movement or strain, in units of 0.25 mm (1/100 in.) occurring in the specimen between no load and the point of maximum load during the stability test.

B. PREPARATION OF TEST SPECIMENS

2.3 GENERAL

In determining the design asphalt content for a particular blend or gradation of aggregates by the Marshall method, a series of test specimens is prepared for a range of different asphalt contents so that the test data curves show well defined relationships. Tests should be planned on the basis of ½ percent increments of asphalt content, with at least two asphalt contents above the expected design value and at least two below this value.

The "expected design" asphalt content can be based on any or all of these sources: experience, computational formula, or performing the centrifuge kerosene equivalency and oil soak tests in the Hveem procedure. Another quick method to arrive at a starting point is to use the dust-to-asphalt ratio guideline (0.6 to 1.2). The expected design asphalt content, in percent by total weight of mix, could then be estimated to be approximately equivalent to the percentage of aggregate in the final gradation passing the 75 μm (No. 200) sieve.

One example of a computational formula is this equation:

$$P = 0.035a + 0.045b + Kc + F$$

Where: P = approximate asphalt content of mix, percent by weight of mix

a = percent* of mineral aggregate retained on 2.36mm (No. 8) sieve

b = percent* of mineral aggregate passing the 2.36mm (No. 8) sieve and retained on the 75 μm (No. 200) sieve

c = percent of mineral aggregate passing 75 μm (No. 200) sieve

K = 0.15 for 11-15 percent passing 75 μm (No. 200) sieve

0.18 for 6-10 percent passing 75 μm (No. 200) sieve

0.20 for 5 percent or less passing 75 μm (No. 200) sieve

F = 0 to 2.0 percent. Based on absorption of light or heavy aggregate. In the absence of other data, a value of 0.7 is suggested.

---

[*] Expressed as a whole number

To provide adequate data, at least three test specimens are prepared for each asphalt content selected. Therefore, a Marshall mix design using six different asphalt contents will normally require at least eighteen test specimens. Each test specimen will usually require approximately 1.2 kg (2.7 lb) of aggregate. Assuming some minor waste, the minimum aggregate requirements for one series of test specimens of a given blend and gradation will be approximately 23 kg (50 lb). About four liters (one gallon) of asphalt cement will be adequate.

2.4 EQUIPMENT

The equipment required for the preparation of test specimens is:

(a) Flat bottom metal pans for heating aggregates.
(b) Round metal pans, approximately 4 liter (4 qt.) capacity, for mixing asphalt and aggregate.
(c) Oven and Hot Plate, preferably thermostatically controlled, for heating aggregates, asphalt, and equipment.
(d) Scoop for batching aggregates.
(e) Containers: gill-type tins, beakers, pouring pots, or sauce pans, for heating asphalt.
(f) Thermometers: armored, glass, or dial type with metal stem, 10°C (50°F) to 235°C (450°F), for determining temperature of aggregates, asphalt and asphalt mixtures.
(g) Balances: 5 kg capacity, sensitive to 1 g, for weighing aggregates and asphalt and 2 kg capacity, sensitive 0.1 g, for weighing compacted specimens.
(h) Large Mixing Spoon or small trowel.
(i) Large spatula.
(j) Mechanical Mixer (optional): commercial bread dough mixer 4 liter (4 qt.) capacity or larger, equipped with two metal mixing bowls and two wire stirrers.
(k) Compaction Pedestal (Figure 17.3), consisting of a 200 x 200 x 460 mm (8 x 8 x 18 in.) wooden post capped with a 305 x 305 x 25 mm (12 x 12 x 1 in.) steel plate. The wooden post should be oak, pine or other wood having a dry weight of 670 to 770 kg/m^3 (42 to 48 pcf). The wooden post should be secured by four angle brackets to a solid concrete slab. The steel cap should be firmly fastened to the post. The pedestal should be installed so that the post is plumb, the cap level, and the entire assembly free from movement during compaction.
*(l) Compaction Mold, consisting of a base plate, forming mold, and collar extension. The forming mold has an inside diameter of 101.6 mm (4 in.) and a height of approximately 75 mm (3 in.); the base plate and collar extension are designed to be interchangeable with either end of the forming mold.
*(m) Compaction Hammer, consisting of a flat circular tamping face. 98.4 mm (3 $^7/_8$ in.) in diameter and equipped with a 4.5 kg (10 lb.) weight constructed to obtain a specified 457 mm (18 in.) height of drop.
*(n) Mold Holder, consisting of spring tension device designed to hold compaction mold centered in place on compaction pedestal.

(o) Paper disks, 100mm (4 in.), for compaction.

(p) Steel specimen extractor, in the form of a disk with a diameter not less than 100 mm (3.95 in.) and 13 mm (0.5 in.) thick for extruding compacted specimens from mold.

(q) Welders gloves for handling hot equipment. Rubber gloves for removing specimens from water bath.

(r) Marking Crayons, for identifying test specimens.

*Marshall test apparatus should conform to requirements of ASTM D 1559

(Note: See additional equipment requirements in Article 2.7)

Mechanical compaction hammer assembly

Manual compaction hammer assembly

Figure 17.3 Pedestal, hammer and mold used in preparing Marshall test specimens

2.5 PREPARATION OF TEST SPECIMENS

These steps are recommended for preparing Marshall test specimens:

(a) Number of Specimens - Prepare at least three specimens for each combination of aggregates and asphalt content.

(b) Preparation of Aggregates - Dry aggregates to constant weight at 105°C to 110°C(220°F to 230°F) and separate the aggregates by dry sieving into the desired size fractions. These size fractions are recommended:

25.0 to 19.0 mm (1 to $^3/_4$ in.)
19.0 to 9.5 mm ($^3/_4$ to $^3/_8$ in.)
9.5 to 4.75 mm ($^3/_8$ in. to No. 4)
4.75 to 2.36 mm (No. 4 to No. 8)
passing 2.36 mm (No. 8)

(c) Determination of Mixing and Compaction Temperature - The temperature to which the asphalt must be heated to produce viscosities of 170 ± 20 centistokes kinematic and 280 ± 30 centistokes kinematic shall be established as the mixing temperature and compaction temperature, respectively. These temperatures can be estimated from a plot of the viscosity (log-log centistokes scale) versus temperature (log degrees Rankine scale, ^{0}R=^{0}F+459.7) relationship for the asphalt cement to be used. An example plot is shown in Figure 17.4.

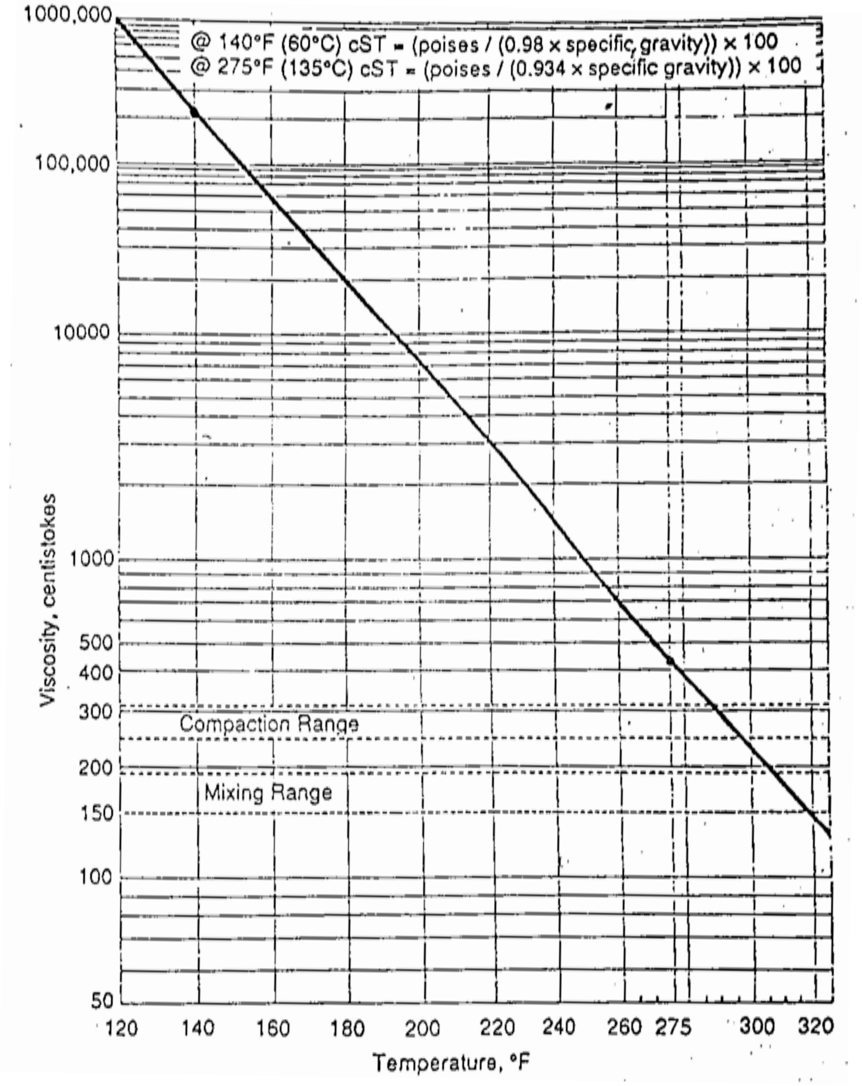


Figure 17.4 Determination of Mixing and Compaction Temperatures

(d) Preparation of Mold and Hammer - Thoroughly clean the specimen mold assembly and the face of the compaction hammer and heat them in a water bath or on the hot plate to a temperature between 95°C and 150°C (200°F and 300°F). Place a piece of filter or waxed paper, cut to size, in the bottom of the mold before the mixture is placed in the mold.

(e) Preparation of Mixtures - Weigh into separate pans for each test specimen the amount of each size fraction required to produce a batch that will result in a compacted specimen 63.5 ± 1.27 mm (2.5 ± 0.05 in.) in height. This will normally be about 1.2 kg (2.7 lb.). It is generally desirable to prepare a trial specimen prior to preparing the aggregate batches. If the trial specimen height falls outside the limits, the amount of aggregate used for the specimen may be adjusted using:

For International System of Units (SI),

*Adjusted mass of aggregate -63.5 (mass of aggregate used) / Specimen height (mm) obtained

U.S. Customary Units,

*Adjusted weight of aggregate - 2.5 (weight of aggregate used) / Specimen height (in.) obtained

Place the pans in the oven or on the hot plate and heat to a temperature not exceeding 28°C (50°F) above the mixing temperature specified in (c). (If a hot plate is used, provision should be made for dead space, baffle plate, or a sand bath beneath the pans and the hot plate to prevent local overheating)Charge the mixing bowl with heated aggregates and dry mix thoroughly. Form a crater in the dry blended aggregate and weigh the required amount of asphalt cement into the mixture in accordance with the calculated batch weights. At this point the temperature of the aggregate and asphalt must be within the limits of the mixing temperature established in paragraph (c). Asphalt cement should not be held at mixing temperatures for more than one hour before using. Mix the aggregate and asphalt cement, preferably with a mechanical mixer or by hand with a trowel, as quickly and thoroughly as possible to yield a mixture having a uniform distribution of asphalt.

Note: Currently, there is no standardized or recommended procedure for aging or curing the mixture prior to Marshall compaction. A number of suggested methods have been proposed; however, a consensus of opinion has not yet been reached. The Hveem procedure recommendsa 2 to 3 hour cure period to allow for both aging and absorption to occur. If severe climates or absorptive aggregates are involved, some consideration should be given to this behavior.

(f) Packing the Mold - Place a paper disk in the mold. Place the entire batch in the mold, spade the mixture vigorously with a heated spatula or trowel 15 times around the perimeter and ten times over the interior. Smooth the surface to a slightly rounded shape. The temperature of the mixture immediately prior to compaction shall be within the limits of the compaction temperature established in paragraph (c); otherwise, it shall be discarded. In no case shall the mixture be reheated.

(g) Compaction of Specimens - Place a paper on top of the mix and place the mold assembly on the compaction pedestal in the mold holder. As specified according to the design traffic category (see Table 17.4), apply either 35, 50 or 75 blows with the compaction hammer using a free fall of 457 mm (18 in.). Hold the axis of the compaction hammer as nearly perpendicular to the base of the mold assembly as possible during compaction. Remove the base plate and collar, and reverse and reassemble the mold. Apply the same number of compaction blows to the face of the reversed specimen. After compaction, remove the base plate and the paper disks and allow the specimen to cool in air until no deformation will result when removing it from the mold. When more rapid cooling is desired, electric fans may be used, but not water unless the specimen is in a plastic bag. Remove the specimen from the mold by means of an extrusion jack or other compression device, then place on a smooth level surface until ready for testing. Normally, specimens are allowed to cool overnight.

Note: ASTM D1559 specifies that hand lifting of a flat faced compaction hammer be used for specimen compaction. If variations (e.g. mechanical lift, slanted face, and rotating base) of the flat face, hand-lifted hammer are used, correlations with the standard Marshall compaction procedure must be made.

C. TEST PROCEDURE

2.6 GENERAL

In the Marshall method, each compacted test specimen is subjected to these tests and analysis in the order listed:

(a) Bulk Specific Gravity Determination
(b) Stability and Flow Test
(c) Density and Voids Analysis

2.7 EQUIPMENT

The equipment required for the testing of the 102 mm (4 in.) diameter x 64 mm (2 $^{1}/_{2}$ in.) height specimens is:

*(a) Marshall Testing Machine, a compression testing device. It is designed to apply loads to test specimens through cylindrical segment testing heads (inside radius of curvature of 51 mm (2 in.)) at a constant rate of vertical strain of 51 mm (2 in.) per minute. Two perpendicular guide posts are included to allow the two segments to maintain horizontal positioning and free vertical movement during the test. It is equipped with a calibrated proving ring for determining the applied testing load, a Marshall stability testing head for use in testing the specimen, and a Marshall flow meter for determining the amount of strain at the maximum load in the test. A universal testing machine equipped with suitable load and deformation indicating devices may be used instead of the Marshall testing frame.

**Marshall test apparatus should conform to requirements of ASTM D 1559*

(b) Water Bath, at least 150 mm (6 in.) deep and thermostatically controlled to 60°C ± 1°C (140°F±1.8°F). The tank should have a perforated false bottom or be equipped with a shelf for suspending specimens at least 50 mm (2 in.) above the bottom of the bath.

2.8 BULK SPECIFIC GRAVITY DETERMINATION

The bulk-specific gravity test may be performed as soon as the freshly compacted specimens have cooled to room temperature. This test is performed according to ASTM D 1188, *Bulk Specific Gravity of Compacted Bituminous Mixtures Using Paraffin-coated Specimens* or ASTM D 2726,*Bulk Specific Gravity of Compacted Bituminous Mixtures Using Saturated Surface-Dry Specimens*.

2.9 STABILITY AND FLOW TESTS

After the bulk specific gravity of the test specimens have been determined, the stability and flow tests are performed:

(a) Immerse specimen in water bath at 60°C ± 1°C (140°F ± 1.8°F) for 30 to 40 minutes before test.

(b) If not using an automatic recording device (as shown in Figure 17.5). "Zero" the flow meter by inserting a 101.6 mm (4.00 in.) diameter metal cylinder in the testing head, placing the flow meter over the guide rod and adjusting the flow meter to read "zero."

Note: This adjustment should be made on the guide post marked with an "0" and with the side of the upper segment of the testing head marked with an "0" being placed on the same side as the guide post so marked. The same assembly of testing head and flow meter must then be used in testing the specimens. Specimens should be 101.6± 0.25 mm (4.00 in. ± 0.01 in.); otherwise, an initial and final reading of flow meter is required for the determination of the flow value.

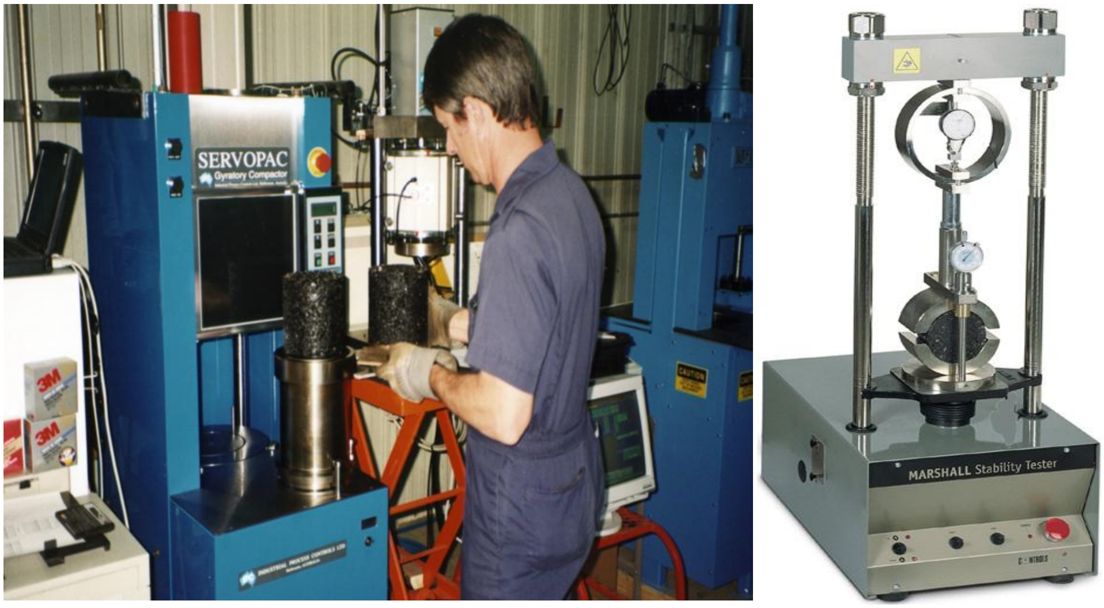


Figure 17.5 Marshall stability and flow test

(c) Thoroughly clean the inside surfaces of testing head. Temperature of head shall be maintained between 21.1°to 37.8°C (70° to 100°F) using a water bath when required. Lubricate guide rods with a thin film of oil so that upper test head will slide freely without binding. If a proving ring is used to measure applied load, check to see that dial indicator is firmly fixed and "zeroed" for the "no-load" position.

(d) With testing apparatus ready, remove test specimen from water bath and carefully dry surface. Place specimen in lower testing head and center; then fit upper testing head into position and center complete assembly in loading device. Place flow meter over marked guide rod as noted in (b) above.

(e) Apply testing load to specimen at constant rate of deformation, 51 mm (2 in.) per minute, until failure occurs. The point of failure is defined by the maximum load reading obtained. The total number of Newtons (lb.) required to produce failure of the specimen shall be recorded as its Marshall stability value.

(f) While the stability test is in progress, if not using an automatic recording device, hold the flow meter firmly in position over guide rod and remove as the load begins to decrease, take reading and record. This reading is the flow value for the specimen, expressed in units of 0.25 mm (1/100 in.). For example, if the specimen deformed 3.8 mm (0.15 in.) the flow value is 15.

(g) The entire procedure for both the stability and flow measurements, starting with the removal of the specimen from the water bath, shall be completed within a period of thirty seconds.

2.10 DENSITY AND VOIDS ANALYSIS

After the completion of the stability and flow test, a density and voids analysis is made for each series of test specimens.

(The calculations for the voids analysis are fully described in previous chapter)

(a) Average the bulk specific gravity values for all test specimens of given asphalt content; values obviously in error shall not be included in the average. These values of bulk specific gravity shall be used in further computations of voids data.

(b) Determine the average unit weight for each asphalt content by multiplying the average bulk specific gravity value by the density of water (1,000 kg/m^3 (62.4 pcf)).

(c) Determine the theoretical maximum specific gravity (ASTM D 2041) for at least two asphalt contents, preferably on mixes at or near the design asphalt content. An average value for the effective specific gravity of the total aggregate is then calculated from these values. This value may then be used for calculation of the maximum specific gravity of mixtures with different asphalt contents, as discussed in previous chapter.

(d) Using the effective and bulk specific gravity of the total aggregate, the average bulk specific gravities of the compacted mix, the specific gravity of the asphalt, and the maximum specific gravity of the mix determined above in (c), calculate the percent absorbed asphalt by weight of dry aggregate, percent air voids (V_a), percent voids filled with asphalt (VFA) and percent voids in mineral aggregate (VMA). These values and calculations are more fully described in previous chapter.

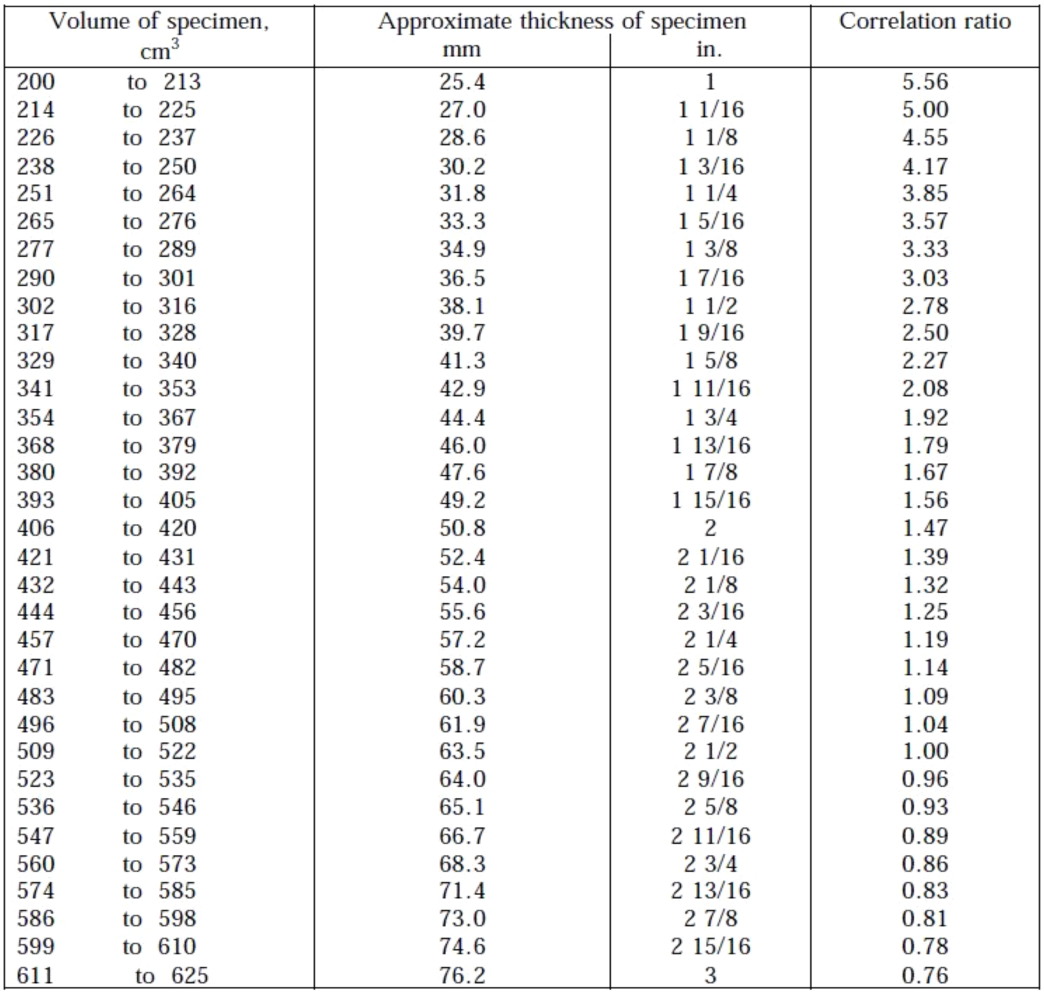
D. INTERPRETATION OF TEST DATA

2.11 PREPARATION OF TEST DATA

Prepare the stability and flow values and void data:

(a) Measured stability values for specimens that depart from the standard 63.5 mm (2 $^1/_2$ in.) thickness shall be converted to an equivalent 63.5 mm (2 $^1/_2$ in.) value by means of a conversion factor. Applicable correlation ratios to convert the measured stability values are set forth in Table 17.3. Note that the conversion may be made on the basis of either measured thickness or measured volume.

Table 17.3: Stability correlation ratios



Volume of specimen, cm^3	Approximate thickness of specimen mm	in.	Correlation ratio
200 to 213	25.4	1	5.56
214 to 225	27.0	1 1/16	5.00
226 to 237	28.6	1 1/8	4.55
238 to 250	30.2	1 3/16	4.17
251 to 264	31.8	1 1/4	3.85
265 to 276	33.3	1 5/16	3.57
277 to 289	34.9	1 3/8	3.33
290 to 301	36.5	1 7/16	3.03
302 to 316	38.1	1 1/2	2.78
317 to 328	39.7	1 9/16	2.50
329 to 340	41.3	1 5/8	2.27
341 to 353	42.9	1 11/16	2.08
354 to 367	44.4	1 3/4	1.92
368 to 379	46.0	1 13/16	1.79
380 to 392	47.6	1 7/8	1.67
393 to 405	49.2	1 15/16	1.56
406 to 420	50.8	2	1.47
421 to 431	52.4	2 1/16	1.39
432 to 443	54.0	2 1/8	1.32
444 to 456	55.6	2 3/16	1.25
457 to 470	57.2	2 1/4	1.19
471 to 482	58.7	2 5/16	1.14
483 to 495	60.3	2 3/8	1.09
496 to 508	61.9	2 7/16	1.04
509 to 522	63.5	2 1/2	1.00
523 to 535	64.0	2 9/16	0.96
536 to 546	65.1	2 5/8	0.93
547 to 559	66.7	2 11/16	0.89
560 to 573	68.3	2 3/4	0.86
574 to 585	71.4	2 13/16	0.83
586 to 598	73.0	2 7/8	0.81
599 to 610	74.6	2 15/16	0.78
611 to 625	76.2	3	0.76

NOTES:
1. The measured stability of a specimen multiplied by the ratio for the thickness of the specimen equals the corrected stability for a 63.5 mm (2 $^1/_2$ in.) specimen.
2. Volume-thickness relationship is based on a specimen diameter of 101.6 mm (4 in.).

(b) Average the flow values and the final converted stability values for all specimens of a given asphalt content. Values that are obviously in error shall not be included in the average.

(c) Prepare a separate graphical plot for these values and connects plotted points with a smooth curve that obtains the "best fit" for all values, as illustrated in Figure 17.6:

- Stability vs. Asphalt Content
- Flow vs. Asphalt Content
- Unit Weight of Total Mix vs. Asphalt Content
- Percent Air Voids (V_a) vs. Asphalt Content
- Percent Voids Filled with Asphalt (VFA) vs. Asphalt Content
- Percent Voids in Mineral Aggregate (VMA) vs. Asphalt Content

These graphs are used to determine the design asphalt content of the mix.

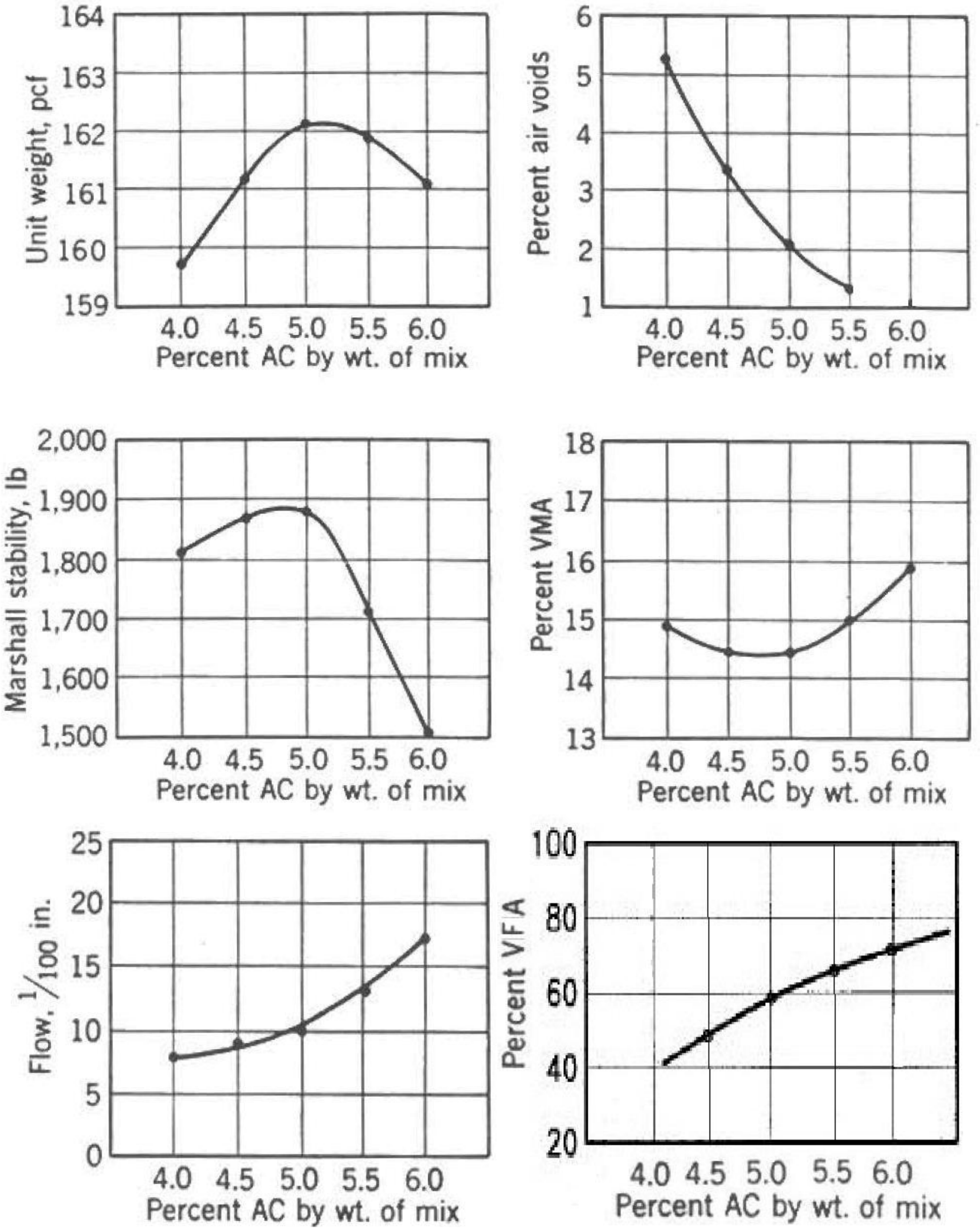


Figure 17.6 Test property curves for hot-mix design data by the Marshall method
(Source: "Highway Materials" by Robert D. Kerbs & Richard D. Walker. Figure 12.3, Page-398)

2.12 TRENDS AND RELATIONS OF TEST DATA

By examining the test property curves plotted for Article 2.11, information can be learned about the sensitivity of the mixture to asphalt content. The test property curves have been found to follow a reasonably consistent pattern for dense-graded asphalt paving mixes, but variations will and do occur. Trends generally noted are:

(a) The stability value increases with increasing asphalt content up to a maximum after which the stability decreases.

(b) The flow value consistently increases with increasing asphalt content.

(c) The curve for unit weight of total mix follows the trend similar to the stability curve, except that the maximum unit weight normally (but not always) occurs at slightly higher asphalt content than the maximum stability.

(d) The percent of air voids, V_a, steadily decreases with increasing asphalt content, ultimately approaching a minimum void content.

(e) The percent voids in the mineral aggregate, VMA, generally decreases, to a minimum value then increases with increasing asphalt content.

(f) The percent voids filled with asphalt, VFA, steadily increases with increasing asphalt content, because the VMA is being filled with asphalt.

2.13 CRITERIA FOR SATISFACTORY PAVING MIX

Deciding whether the asphalt paving mix will be satisfactory at the selected design asphalt content is guided by applying certain limiting criteria to the mixture test data. The Marshall method mix design criteria in Table 17.4 are recommended by the Asphalt Institute.

Table 17.4: Marshall mix design criteria

Marshall Method Mix Criteria	Light Traffic Surface & Base		Medium Traffic Surface & Base		Heavy Traffic Surface & Base	
Compaction, Number of blows on each end of specimen	35		50		75	
Stability, N (lb)	3336 (750)	–	5338 (1200)	–	8006 (1800)	–
Flow, 0.25 mm (0.01 in)	8	18	8	16	8	14
Percent Air Voids	3	5	3	5	3	5
Percent Voids in Mineral (VMA)	See Table 17.5					
Percent Voids Filled with Asphalt (VFA)	70	80	65	78	65	75

NOTES

1. All criteria, not just stability value alone, must be considered in designing an asphalt pavement mix. Hot mix asphalt bases that do not meet the criteria when tested at 60^0 C (140^0 F) are satisfactory if they meet the criteria when tested at 38^0C (100^0F) and are placed 100 mm (4 inches) or more below the surface. This recommendation applies only to regions having range of climate conditions similar to those prevailing throughout most of the United States. A different lower test temperature may be considered in regions having more extreme climate conditions.
2. Traffic classification: Light Traffic condition resulting in a Design EAL < 10^4
   Medium Traffic condition resulting in a Design EAL between 10^4 and 10^6
   Heavy Traffic condition resulting in a Design EAL > 10^6

3. Laboratory compaction efforts should closely approach the maximum density obtained in the pavement under traffic.
4. The flow value refers to the point where the load begins to decrease.
5. The portion of asphalt cement lost by absorption into the aggregate particles must be allowed for when calculating percent of air voids.
6. Percent voids in the mineral aggregate is to be calculated on the basis of the ASTM bulk specific gravity for the aggregates.

Table 17.5: Minimum percent voids in mineral aggregate (VMA)

Nominal Maximum Particle Size 1,2		Minimum VMA, Percent		
		Design Air Voids, Percent 3		
mm	in	3.0	4.0	5.0
1.18	No. 16	21.5	22.5	23.5
2.36	No. 8	19	20	21
4.75	No. 4	16	17	18
9.5	3/8	14	15	16
12.5	½	13	14	15
19	¾	12	13	14
25	1	11	12	13
37.5	1.5	10	11	12
50	2	9.5	10.5	11.5
63	2.5	9	10	11

1. Standard specification for Wire Cloth Sieves for testing purposes. ASTM E11 (AASHTO M92)
2. The nominal maximum particle size larger than the first sieve to return more than 10 percent.
3. Interpolate minimum voids in the mineral aggregate (VMA) for design air voids between those listed.

2.14 DETERMINATION OF PRELIMINARY DESIGN ASPHALT CONTENT

The design asphalt content of the asphalt paving mix is selected by considering all of the data discussed previously. As an initial starting point, the Asphalt Institute recommends choosing the asphalt content at the median of the percent air voids limits, which is four percent. All of the calculated and measured mix properties at this asphalt content should then be evaluated by comparing them to the mix design criteria in Table 2.2. If all of the criteria are met, then this is the preliminary design asphalt content. If all of the design criteria are not met, then some adjustment or compromise is necessary or the mix may need to be redesigned. A number of considerations are discussed in the next article that should be weighed even if all the design criteria are met.

2.15 SELECTION OF FINAL MIX DESIGN

The final selected mix design is usually the most economical one that will satisfactorily meet all of the established criteria. However the mix should not be designed to optimize one particular property. Mixes with abnormally high values of stability are often less desirable because pavements with such mixes tend to be less durable and may crack prematurely under heavy volumes of traffic. This situation is especially critical where the base and sub-grade materials beneath the pavement are weak and permit moderate to relatively high deflections under the actual traffic.

The design asphalt content should be a compromise selected to balance all of the mix properties. Normally the mix design criteria will produce a narrow range of acceptable asphalt contents that pass all of the guidelines as shown by the example in Figure 17.7. The asphalt content selection can be adjusted within this narrow range to achieve a mix property that will satisfy a requirement of a specific project.

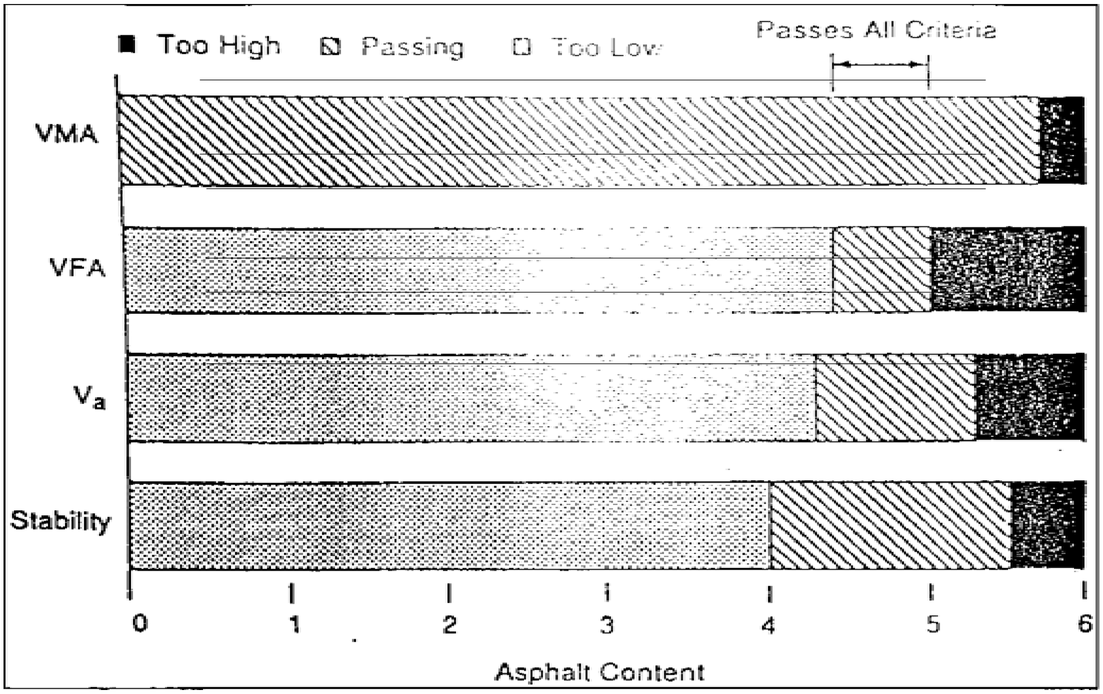


Figure 17.7 An example of the narrow range of acceptable asphalt contents

Different properties are more critical for different circumstances, depending on traffic, structure, climate, construction equipment and other factors. Therefore the balancing process is not the same for every pavement and every mix design. These are some considerations for adjustment that should be evaluated prior to establishing the final design asphalt content:

Evaluation of VMA Curve

In many cases the most difficult mix design property to achieve is a minimum amount of voids in the mineral aggregate. The goal is to furnish enough space for the asphalt cement so it can provide adequate adhesion to bind the aggregate particles, but without bleeding when temperatures rise and the asphalt expands. Normally the curve exhibits a flattened U-shape, decreasing to a minimum value and then increasing with increasing asphalt content, shown in Figure 17.8 (a).

This dependency of VMA on asphalt content appears to be a contradiction to the definition. One might expect the VMA to remain constant with varying asphalt content, thinking that the air voids would simply be displaced by asphalt cement. In reality the total volume changes across the range of asphalt contents; the assumption of a constant unit volume is not accurate. With the increase in asphalt the mix actually becomes more workable and compacts more easily, meaning more weight can be compressed into less volume. Therefore, up to a point, the bulk density of the mix increases and the VMA decreases.

At some point as the asphalt content increases (the bottom of the U-shaped curve) the VMA begins to increase because relatively more dense material (aggregate) is displaced and pushed apart by the less dense material (asphalt cement). It is recommended that asphalt contents on the "wet" or right-hand increasing side of this VMA curve be avoided, even if the minimum air void and VMA criteria is met. Design asphalt contents in this range have a tendency to bleed and/or exhibit plastic flow when placed in the field. Any amount of additional compaction from traffic leads to inadequate room for asphalt expansion, loss of aggregate to aggregate contact, and eventually rutting and shoving in high traffic areas. Ideally, the design asphalt content should be selected slightly to the left of the low point of the VMA curve, provided none of the other mixture criteria are violated.

In some mixes, the bottom of the U-shaped VMA curve is very flat, meaning that the compacted mixture is not as sensitive to asphalt content in this range as some other factors. In the normal range of asphalt contents, compact ability is influenced more by aggregate properties. However, at some point the quantity of asphalt will become critical to the behavior of the mix and the effect of asphalt will dominate as the VMA increases drastically.

When the bottom of the U-shaped VMA curve falls below the minimum criteria level required for the nominal maximum aggregate size of the mix [Figure 17.8 (b)], this is an indication that changes to the job mix formula are necessary. Specifically, the aggregate grading should be modified to provide additional VMA.

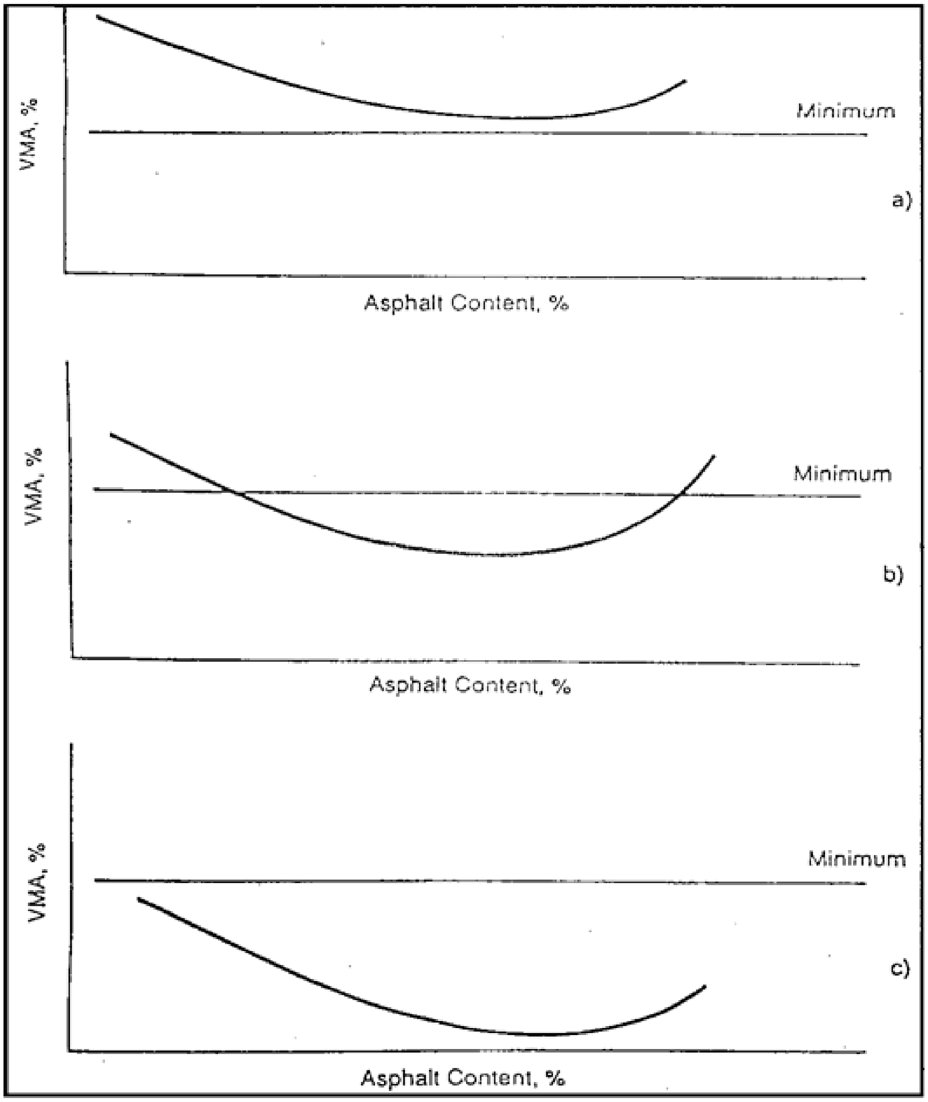


Figure 17.8 Relationship between VMA and specification limit

The design asphalt content should not be selected at the extremes of the acceptable range even though the minimum criteria are met. On the left hand side, the mix would be too dry, prone to segregation and would probably be too high in air voids. On the right hand side, the mix would be expected to rut.

If the minimum VMA criteria is completely violated over the entire asphalt content range [curve is completely below minimum, Figure 17.8 (c)], a significant redesign and/or change in material sources is warranted.

Effect of Compaction Level

At the same asphalt content, both air voids (V_a) and voids in the mineral aggregate (VMA) decrease with higher compactive effort. The three levels of compaction of the Marshall mix procedure can be used to illustrate the consequences of this fact. As shown in Figure 17.9 (a), not only do the magnitudes of the values change but the asphalt content value at the minimum VMA shifts.

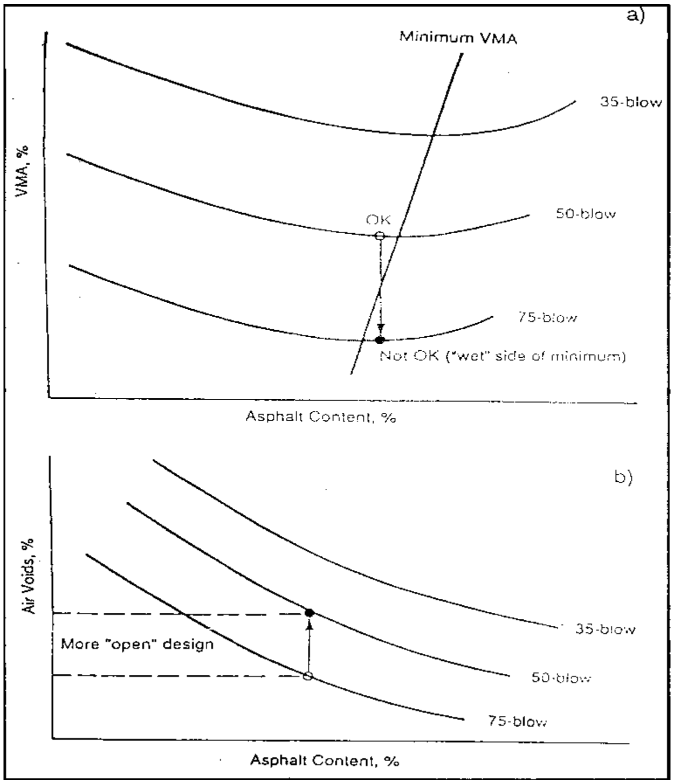


Figure 17.9 Effect of Marshall compactive effort on VMA and air voids

If a mix is designed slightly to the left of minimum VMA at a compaction level of 50 blows and the pavement actually endures heavier traffic than expected (closer to 75 blow design level); then, the same asphalts content now plots on the right hand or "wet" side of the minimum VMA point for a mix designed using 75 blow compaction. Ultimately, a mix susceptible to rutting is the result.

This scenario can also work in the opposite direction. If a mix, designed at a compaction level of 75 blows as shown in Figure 17.9 (b), is placed in a pavement with much lower volumes of traffic, then the final percentage of air voids (V_a) will be considerably higher than planned. This condition could lead to a more open, permeable mix allowing air and water to pass through easily. The consequence of this situation is a mix that hardens prematurely, becomes brittle and cracks at an early age or the aggregate ravels out of the mix because of the loss of asphalt adhesion. This condition may also lead to stripping.

For this reason, it is important that the compactive effort used to simulate the design traffic expected in the pavement be selected accordingly in the laboratory. Also, the mixture must be constructed with appropriate compaction equipment in the field to produce adequate initial density regardless of climatic conditions.

It is also important to note that the VMA criteria do not change based on the level of compaction. The reasoning for having sufficient VMA (providing space for the asphalt and air voids) is consistent regardless of the traffic level for which the mixture is being designed.

Effect of Air Voids

It should be emphasized that the design range of air voids (3 to 5 percent) is the level desired after several years of traffic. This goal does not vary with traffic as seen in Table 17.4; the laboratory compactive effort is supposed to be selected for the expected traffic. This design air void range will normally be achieved if the mix is designed at the correct compactive effort and the percent air voids after construction is about 8 percent. Some consolidation with traffic is expected and desired.

The consequence of a change in any factor or any detour in the procedure that offsets the total process will be a loss of performance or service life. It has been shown that mixtures that ultimately consolidate to less than three percent air voids can be expected to rut and shove if placed in heavy traffic locations. Several factors may contribute to this occurrence, such as an arbitrary or accidental increase in asphalt content at the mixing facility or an increased amount of ultra fine particles passing the 75 µm (No. 200) sieve beyond that used in the laboratory, which will act as an asphalt extender. Similarly, problems can occur if the final air void content is above five percent or if the pavement is constructed with over eight percent air voids initially. Brittleness, premature cracking, raveling and stripping are all possible under these conditions. The overall objective is to limit adjustments of the design asphalt content to less than 0.5 percent air voids from the median of the design criteria (four percent), especially on the low side of the range and to verify that the plant mix closely resembles the laboratory mix.

Effect of Voids Filled with Asphalt

Although VFA, VMA and V_a are all interrelated and only two of the values are necessary to solve for the other, including the VFA criteria helps prevent the design of mixes with marginally acceptable VMA. The main effect of the VFA criteria is to limit maximum levels of VMA and subsequently maximum levels of asphalt content. VFA also restricts the allowable air void content for mixes that are near the minimum VMA criteria. Mixes designed for lower traffic volumes will not pass the VFA criteria with a relatively high percent air voids (five percent) even though the air void criteria range is met. The purpose is to avoid less durable mixes in light traffic situations.

Mixes designed for heavy traffic will not pass the VFA criteria with relatively low percent air voids (less than 3.5 percent) even though that amount of air voids is within the acceptable range. Because low air void contents can be very critical in terms of permanent deformation (as discussed previously), the VFA criteria helps to avoid those mixes that would be susceptible to rutting in heavy traffic situations.

The VFA criteria provide an additional factor of safety in the design and construction process in terms of performance. Since changes can occur between the design stage and actual construction, an increased margin for error is desirable.

Influence of Structure and Climate

Mix design is a compromise of many factors. The asphalt content that provides the best overall performance in addition to passing the previously discussed conventional criteria would be considered the design value. The CAMAS computer program contained in the Asphalt Institute *Computer Assisted Asphalt Mix Analysis System package*, provides an additional tool for evaluating the predicted performance of a specific mix placed in a particular situation. The various mathematical models contained in the program have not been fully verified and the program is currently considered only a research tool. However, models are included for examining fatigue life, subgrade deformation and asphalt concrete deformation of the pavement and mix for the actual climatic and traffic conditions. If any of these levels of performance are not acceptable, then either the mix or the structure could be modified and a subsequent evaluation performed.

The decision making process for selecting the design asphalt content in the mix varies with the circumstances involved in the specific case. Depending on the particular structure or agency policy, certain factors may be more important than others. Although it was found that it is not feasible, suitable, or practical to directly trade-off thickness for better mix compaction or a change in asphalt content, there are other advantages to integrating structural design and mix design.

The type of structure can alter the engineer's evaluation in many ways. For example, in an asphalt concrete overlay of a Portland cement concrete pavement, there would be little concern for fatigue, since the tensile strains in the bottom of the AC overlay would be minimal. This is also true for the subgrade deformation related to the compressive strain on the top of the subgrade. The main consideration would be how to limit the AC rutting as well as any supplementary treatments for minimizing reflective cracking. In this particular case, it may be worthwhile to look at the effects of altering the compactive effort in the lab and field while changing the asphalt content. Depending on the environmental conditions during construction, it may be worthwhile to use heavier rollers or a longer period of rolling to achieve more or the same density with less asphalt in the mix. Mixes with asphalt contents on the high side of the acceptable range are usually avoided in this situation.

In an asphalt pavement, all three performance indicators need to be evaluated in terms of future maintenance. Initially, it is important that the subgrade be adequately protected by the structure; the number of allowable repetitions based on subgrade deformation should exceed that expected or the pavement's performance may have little to do with proper mix design. In some cases, depending on location and traffic volume, the engineer may consider whether cracking or rutting is less of a future maintenance concern and the mix design can be selected accordingly. With all other factors being equal, mixes with asphalt contents on the high side of the range are less prone to cracking because of the additional flexibility. Similarly, mixes on the low side of the range are less susceptible to rutting.

Finally, climate can have a major impact on mix and pavement performance for a given pavement structure. Mix designs do not usually consider this factor except in selecting the category or grade of asphalt cement. Table 17.6 gives recommended asphalt grades for various temperature conditions.

Table 17.6: Selecting Asphalt Grade

Temperature Condition	Asphalt Grades	
Cold, Mean Annual Temperature $\leq 7^{0}$ C (45^{0} F)	AC-5 AR-2000 120/150 pen.	AC-10 AR-4000 85/100 pen.
Warm, Mean Annual Temperature between 7^{0} C (45^{0} F) and 24^{0} C (75^{0} F)	AC-10 AR-4000 85/100 pen.	AC-20 AR-8000 60/70 pen.
Hot, Mean Annual Temperature between $\geq 24^{0}$ C (75^{0} F)	AC-20 AR-8000 60/70 pen.	AC-40 AR-16000 40/50 pen.

In hot climates, harder, more viscous asphalts are normally used to obtain more stability from asphalt adhesion as well as from aggregate interlock. If the mix is designed and constructed to maximize aggregate to aggregate contact, then the properties of the asphalt cement are less important. Regardless, asphalt contents on the low side of the acceptable range are recommended for these areas.

In colder climates, softer, less viscous asphalts are recommended to produce a mix which is less susceptible to low temperature shrinkage cracking. Rutting is less of a concern; therefore, additional stability from asphalt adhesion is not necessary. Usually asphalt contents on the high side of the acceptable range are recommended to furnish a mix which is more elastic and resilient.

Specific Project Conditions

The season of the year when the pavement is being constructed can be another factor to be considered when selecting the final design asphalt content. Summer paving would usually call for lower asphalt contents, while fall or early spring construction would dictate higher asphalt contents to assist compaction in cooler temperatures. Any shift in asphalt content is only a minor amount within the narrow range that passes all the previous criteria.

The amount and handling of traffic can also influence the final decision. If the actual traffic is at the low or high end of the broad traffic categories for selecting the laboratory compactive effort and mix design criteria, then the asphalt content could be slightly modified accordingly. Higher traffic areas would demand the lower asphalt contents within the acceptable range. Mixes to be used in overlay situations with reduced lane detours, where the pavement will undergo severe loading concentrations such as highly channelized wheel passes, very slow speeds or steep upgrades, demand additional attention in all phases of production. The design asphalt content should be selected from the low end of the acceptable range and initial compaction requirements must be met. Traffic should be held off of the pavement as long as possible while the mix is cooling to normal temperatures. This cooling allows the asphalt to contribute more to the mix stability and less as a compaction lubricant.

2.16 MODIFIED MARSHALL METHOD FOR LARGE AGGREGATE

A modified Marshall method has been developed by Kandhal of the National Center for Asphalt Technology for mixes composed of aggregates with maximum size up to 38 mm (1.5 in.). This procedure is documented in draft form in the 1990 Proceedings of the Association of Asphalt Paving Technologists (AAPT). The procedure is basically the same as the original method except for these differences that are due to the larger specimen size that is used:

(a) The hammer weighs 10.2 kg (22.5 lb.) and has a 149.4 mm (5.88 in.) flat tamping face. Only a mechanically operated device is used for the same 457 mm (18 in.) drop height.

(b) The specimen has a 152.4 mm (6 in.) diameter by 95.2 mm (3.75 in.) height.

(c) The batch weights are typically 4 kg.

(d) The equipment for compacting and testing (molds and breaking heads) are proportionately larger to accommodate the larger specimens.

(e) The mix is placed in the mold in two approximately equal increments, with spading performed after each increment to avoid honey combing.

(f) The number of blows needed for the larger specimen is 1.5 times (75 or 112 blows) that required of the smaller specimen (50 or 75 blows) to obtain equivalent compaction.

(g) The design criteria should be modified as well. The minimum stability should be 2.25 times and the range of flow values should be 1.5 times the same criteria listed in Table 2.2 for the normal sized specimens.

(h) Similar to the normal procedure, these values should be used to convert the measured stability values to an equivalent value for a specimen with a 95.2 mm (3.75 in.) thickness, if the actual thickness varies:

Table 17.7: Height of Marshall specimen and Correlation Ratio

Approximate Height		Specimen Volume	Correlation Ratio
(mm)	(in.)	(cc)	
88.9	$3\,^{1}/_{2}$	1608 to 1626	1.12
90.5	$3\,^{9}/_{16}$	1637 to 1665	1.09
92.1	$3\,^{5}/_{8}$	1666 to 1694	1.06
93.7	$3\,^{11}/_{16}$	1695 to 1723	1.03
95.2	$3\,^{3}/_{4}$	1724 to 1752	1.00
96.8	$3\,^{13}/_{16}$	1753 to 1781	0.97
98.4	$3\,^{7}/_{8}$	1782 to 1810	0.95
100.0	$3\,^{15}/_{16}$	1811 to 1839	0.92
101.6	4	1840 to 1868	0.90

Composition of Paving Mixtures
(ASTM D3515) for Marshal Method of Mix Design

Type of Mix: Dense Mix
Mix Designation: ¾" (19 mm)
Course Aggregate: Stone Chips
Bitumen: 80/100 pen. grade Bitumen
Fine Aggregate and Mineral Filler: Fine Fraction of Course Aggregate
Traffic Category: Medium Traffic (Compaction: 50 blows per face)

Sieve Size	% Passing	% Retained (Cumulative)	% Retained (Individual)	Batch Weight (gm)
1 in (25 mm)	100	0	0	0
¾ in (19 mm)	95	5	5	57
$^3/_8$ in (9.5 mm)	68	32	27	312
# 4 (4.75 mm)	50	50	18	208
# 8 (2.36 mm)	36	64	14	162
# 50 (300 µm)	12	88	24	277
# 200 (75 µm)	5	95	7	81
M.F.	0	100	5	58
Total			**100**	**1155**

<u>% of Bitumen (of Total Mix), P_b</u>

4.0% = 48.13 gm, 4.5% = 54.42 gm, 5.0% = 60.79 gm
5.5% = 67.22 gm, 6.0% = 73.72 gm

<u>Specific Gravity</u>

C.A. (1 inch - #8) (G_1) : 2.68 (ASTM C-127)
F.A. (#8 - #200) (G_2) : 2.68 (ASTM C-128)
M.F. (Passing #200) (G_3) : 2.77 (ASTM D-854)
Bitumen (G_b) : 1.02 (ASTM D-5)

<u>Mixing Criteria:</u>

Temperature: 148 – 153^0 ⇨ 150^0 C, Time: 2.5 min.
Compaction Temp.: 138 – 142^0 ⇨ 140^0 C
Oven Temp. for Bitumen and Aggregate: 155^0 C, Time: 2 hrs.

Experiment No: 17

Determination of Optimum Asphalt Content by Marshall Method of Mix Design

Name :

Student No:

Asphalt content (P_b)%	Specimen wt. in air (W_a) gm	Specimen wt. in water (W_w) gm	Specimen wt. in air (SSD) (W_s) gm	G_{mb}	Unit wt. (lb/cft) G_{mb}x62.4	G_{mm}	P_S	G_{sb}	%V_a	%VMA	%VFA	Height of specimen (mm)	Correction factor	Stability observed (lbs.)	Corrected stability (lbs.)	Flow value ($^1/_{100}$in.)

$G_{mb} = \frac{W_a}{Ws - W_w}$; $G_{mm} = \frac{P_{mm}}{\frac{P_s}{G_{se}} + \frac{P_b}{G_b}}$; $P_s = 100 - P_b$; $P_{mm} = 100$; $G_{se} = \frac{P_{mm} - P_b}{\frac{P_{mm}}{G_{mm}} - \frac{P_b}{G_b}}$; $G_{sb} = \frac{P_1 + P_2 + P_3}{\frac{P_1}{G_1} + \frac{P_2}{G_2} + \frac{P_3}{G_3}}$

$\%V_a = 100\frac{G_{mm} - G_{mb}}{G_{mm}}$; $\%\text{VMA} = 100 - \frac{G_{mb} * P_s}{G_{sb}}$; $\%\text{VFA} = \frac{\%VMA - \%V_a}{\%VMA} * 100$

Appendix 1
Lab Report Format

1. Each student shall submit a separate report on each test. All reports shall be written on A4 (210mmX297mm) paper and be bound in a file. On the cover of the file the following items must be present.
    I. Subject
    i.e. Transportation Engg. Sessional-I, CE354
    II. Name and Roll No. of the student
    III. Section and Group No.
    IV. Level Term and Session
2. An index sheet containing Experiment no, Experiment name, Date of performance & submission and Remarks is to be placed just after the cover page.
3. On the first page of the report of each experiment tthe following must be written neatly:
    I. The test Number
    II. The title of the test
    III. Student's name and roll no.
    IV. Section and group no.
    V. Level term and session
4. Report should be brief but self-explanatory. Advantage should be taken of tabular and graphical methods of presenting data. In addition to the subject matter, clarity, conciseness, method of presentation, legibility and neatness of the report will be received for considering in the grading of a report. Lack of neatness shall be sufficient cause for rejection of report. Copying of any report will result in cancellation of all reports concerned.

Arrangement of **the Report**

Objective: Statement of the objective of the test.

Scope of test: A brief statement of purpose of the test must be included.

Apparatus: Any equipment used for the first time, should be described with neat sketches and its operation principle.

Procedure: All important steps in testing procedure must be explained.

Data sheet: A complete laboratory data sheet previously signed by the teacher, must accompany the report.

Calculation: All equations or formula used should be clearly stated with each term containing in it. All calculation should be submitted in concise form.

Graph (if any): Necessary graph/graphs (if any) for the test must be included. Graph should be neat and self explanatory.

Result: The results of the tests should be submitted in tabular or preferably in graphical form whenever possible.

Discussion: A brief discussion of all the salient features of the tests, as in the tables and diagrams should be included. The test results must be compared with pertinent data given any books or publications and definite conclusion should be drawn. Answer to the question given at the end of each test should be submitted with the report.

Reports on the test in a particular class shall be submitted in the next available class. Fifty Percent will be deducted from the total marks for the late (with a maximum two weeks) late submission of the report. Any student who, without satisfactory reasons for excuse, fails to submit a report within two weeks of the date it is due, is subject to dismissal of the particular report.

<u>Warning:</u>

Student should be careful about their own safety and safety of the equipment .Care must be taken during handling of hazardous materials, chemicals electricity, Fire etc. which may be used during testing. A FIRST AID "box is available in the laboratory as a necessary aid for the treating minor injuries.

Appendix 2
Lab Instruction

Important

The students must read carefully the general instructions as stated below before starting laboratory sessional classes.

General

1. Every student must be regular and active in the class. Test should be completed during the assigned laboratory period. If any student is unable to attend the class during an assigned laboratory period for valid reason, the student must see the concerned teacher within that week. The teacher may make special arrangement to make up the test. Failure to report in time may result in cancellation of that particular test.
2. For the systematic performance of tests, the students will be divided into several groups. The teacher will see that each students of a group gets opportunity to perform major operations in a test. All the members of a group will be responsible for proper conduct and completion of a test and submission of a data sheet and reports within a specified time.
3. For proper utilization of the limited time in the laboratory, the students are advised to come prepared in the class. The preparations should be include :

   I. A thorough study of the assigned tests and laboratory procedures
   II. Preparation of proposed data sheet for approval of the teacher
   III. Completion of previous test reports for submission to the teacher. Reports are to be prepared by each student individually
4. Before Roll call in each class, the students will submit their previous test report to the teacher, failure to do so may result in deduction of marks or cancellation of the particular test.
5. The teacher will give any special instruction to the groups whenever needed by them
6. The teacher may call any or all the students from each group who might be required to answer questions regarding the test procedures, apparatus and other fundamental of theory behind the particular test. Their answers will add to the credit or discredit to their grading.
7. Care should be taken so that no instrument is misplaced, broken or lost. Any breakage, damage or loss of apparatus must be reported immediately to the teacher. Damage or loss due to carelessness will be charged to the responsible group.
8. Expensive materials should used carefully to minimize the unnecessary loss.
9. No student shall handed or operate any instrument other than those assigned to his group without prior permission of the teacher.

10. After completion of experiment the instrument or apparatus are to be cleaned and return to the laboratory instructor.
11. No student is allowed to leave the laboratory without taking prior consent of the teacher.
12. After each experiment is performed the data sheet is to be signed by the respective teacher.

Reference

1. BS: 812: 1975 (PART 1, 2, 3)
“Methods for sampling and testing of mineral aggregates, sands and fillers.”

2. Highway capacity manual (HCM)-1994, USA.

3. The Road Note 34

4. ASTM

5. AASHTO

6. Asphalt institute manual series no. 2 (MS-2), Ch. 4 & Ch. 6, sixth edition, 1997.

Table of Contents

Class Schedule

Class Weeks	Scheduled Task
Week -1	Introduction & Water sample collection method
Week -2	Exp. No. - 1,2,3 & 4
Week -3	Exp. No.- 5,6 & 7
Week -4	Exp. No.- 8 & 9
Week -5	Exp. No.- 10
Week -6	Exp. No.- 11
Week -7	Exp. No.- 12
Week -8	**Midterm Quiz and Viva** (Water sampling & Exp. No. – 1 to 9)
Week -9	Exp. No.- 13 & 14
Week -10	Exp. No.-15
Week -11	Class Project -1: Model Test for Drinking Water Treatment
Week -12	**Final Quiz and Viva** (Exp. No. – 10 to 15)

Introduction:
Methods of Water Sampling for Lab Analysis

Water quality is the physical, chemical and biological characteristics of water. It is a measure of the condition of water relative to the requirements of one or more biotic species and or to any human need or purpose. It is most frequently used by reference to a set of standards against which compliance can be assessed. The most common standards used to assess water quality relate to health of ecosystems, safety of human contact and drinking water.

What is Water Quality Analysis?
Water quality standards are put in place to ensure the efficient use of water for a designated purpose. The parameters for water quality are determined by the intended use. Work in the area of water quality tends to be focused on water that is treated for human consumption, industrial use, or in the environment. Water quality analysis is to measure the required parameters of water, following standard methods, to check whether they are in accordance with the standard.

Why Water Quality Analysis is required?
Water quality analysis is required mainly for monitoring purpose. Some importance of such assessment includes:

1. To check whether the water quality is in compliance with the standards, and hence, suitable or not for the designated use.
2. To monitor the efficiency of a system, working for water quality maintenance
3. To check if any upgradation / change of an existing system is required and to decide what changes should take place.
4. To monitor whether water quality is in compliance with rules and regulations.

On-site Water Quality Analysis
The complexity of water quality as a subject is reflected in the many types of measurements of water quality indicators. The most accurate measurements of water quality are made on-site, because water exists in equilibrium with its surroundings. Measurements commonly made on-site and in direct contact with the water source in question include - temperature, pH, dissolved oxygen, conductivity, oxygen reduction potential (ORP) and turbidity..

Water Quality Analysis in Lab: Collection of water sample
More complex measurements are often made in a laboratory requiring a water sample to be collected, preserved, transported, and analyzed at another location. The process of water sampling introduces two significant problems-

A common cause of error in water quality analysis is improper sampling. The results of a water quality analysis of a sample show only what is in the sample. For the results to be meaningful, the sample *must be representative* i.e., it must contain essentially the same constituents as the body of water from which it was taken.

Many water sources vary with time and with location. The measurement of interest may vary seasonally or from day to night or in response to some activity of man or natural populations of aquatic plants and animals.

The measurement of interest may vary with distances from the water boundary with overlying atmosphere and underlying or confining soil. The sampler must determine if a single time and location meets the needs of the investigation, or if the water use of interest can be satisfactorily assessed by averaged values with time and/or location, or if critical maxima and minima require individual measurements over a range of times, locations and/or events.

The sample collection procedure must assure correct weighting of individual sampling times and locations where averaging is appropriate.

The second problem occurs as the sample is removed from the water source and begins to establish chemical equilibrium with its new surroundings - the sample container. Sample containers must be made of materials with minimal reactivity with substances to be measured; and pre-cleaning of sample containers is important. The water sample may dissolve part of the sample container and any residue on that container, or chemicals dissolved in the water sample may sorb onto the sample container and remain there when the water is poured out for analysis.

The collected sample should be small enough in volume to be transported conveniently and yet large enough for analytical purposes while still accurately representing the material being sampled.

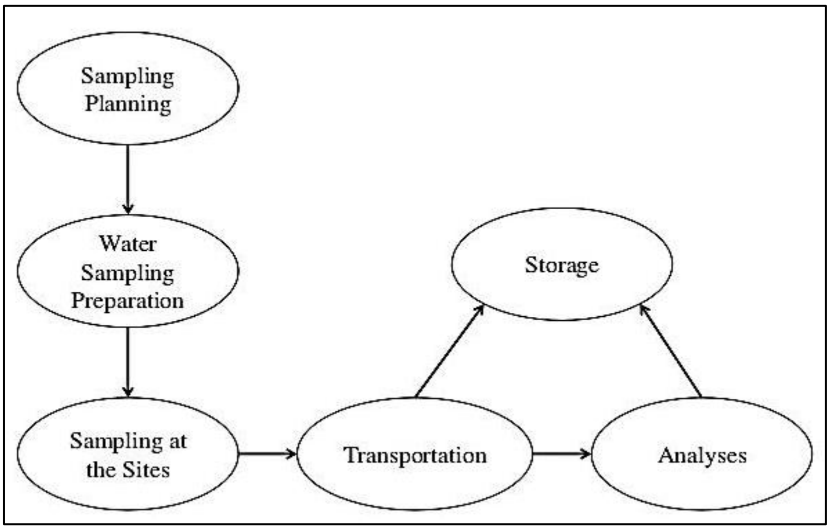
Location of sampling points

The following general criteria are usually applicable:

- Sampling points should be selected such that the samples taken are representative of the different sources from which water is obtained by the public or enters the system.
- These points should include those that yield samples representative of the conditions at the most unfavourable sources or places in the supply system, particularly points of possible contamination such as unprotected sources, loops, reservoirs, low-pressure zones, ends of the system, etc.
- Sampling points should be uniformly distributed throughout a piped distribution system, taking population distribution into account; the number of sampling points should be proportional to the number of links or branches.
- The points chosen should generally yield samples that are representative of the system as a whole and of its main components. Sampling points should be located in such a way that water can be sampled from reserve tanks and reservoirs, etc.
- In systems with more than one water source, the locations of the sampling points should take account of the number of inhabitants served by each source.
- There should be at least one sampling point directly after the clean-water outlet from each treatment plant.

Requirements for Sampling:

- Meet the requirements of the sampling program.
- Handle the sample carefully so that it does not deteriorate or become contaminated or compromised before it is analyzed.
- Ensure sampling all equipment are clean and quality assured before use.
- Use sample containers that are clean and free of contaminants
- Rinse the bag/bottle at least twice with the sample water prior to filling and closing
- Fill bag/bottle as full as possible. Half-filling leaves more room for oxygen which will promote degradation of your sample.
- If sampling a body of running water, point the mouth of the bag upstream and your hands downstream to avoid contamination.
- If sampling from a water faucet, run the faucet for 1 minute before obtaining a sample.
- Make records of every sample collected and identify every bottle e.g., take notes and photographs, fill out tags, etc.
- Place the sample into appropriate, labeled containers.
- All samples must be preserved as soon as practically possible.



Water Sampling Flow Chart

General Water Sampling Techniques

Water sampling can be done in any of the following three methods depending on test requirements:

1. Grab sampling
2. Composite sampling
3. Integrated sampling

1. Grab sampling

A grab sample is a discrete sample which is collected at a specific location at a certain point in any specific time. a grab sample reflects performance only at the point in time that the sample was collected, and then only if the sample was properly collected- such as pH, dissolved oxygen, chlorine residual, nitrites and temperature of a particular point and time. If the environmental medium varies spatially or temporally e.g.in case of waste effluents, then a single grab sample is not representative and more samples need to be collected at suitable time intervals and analyzed separately.

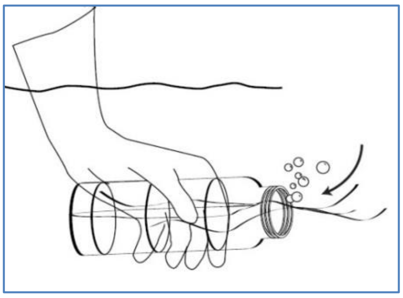
2. Composite sampling

Composite sample is a mixture of grab samples collected at one sampling point at different times. Water being sampled is collected in a common container over the sampling period. The analysis of this material, collected over a period of time, will therefore represent the average performance of a wastewater treatment plant during the collection period.

This is particularly useful in water bodies that have a lot of chemical variability either over space or over short time periods. Composite samples are often used to reduce the cost of analyzing a large number of samples.

3. Integrated sampling

Integrated sample is a mixture of grab samples collected from different points simultaneously and mixed in equal volumes. The points may be horizontal or vertical variation. For example, river, stream or reservoir or lake that varies in composition across the width and depth.



Surface Water Sampling Techniques

When the water source is accessible

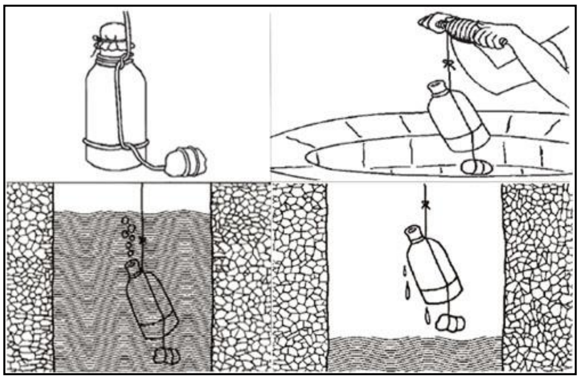
Rinse the sampling vessel with water on site 3~4 times. Care must be taken to avoid contaminating water to be sampled during rinsing.

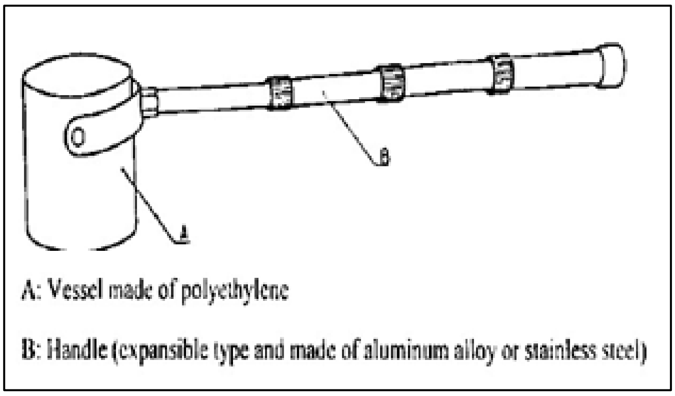
Submerge the sampling vessel gently, fill it with the water sample and close it tightly.

If the collected water sample may be frozen, leave some space for expansion equivalent to about 10% of the sampling vessel.

When the water source is inaccessible

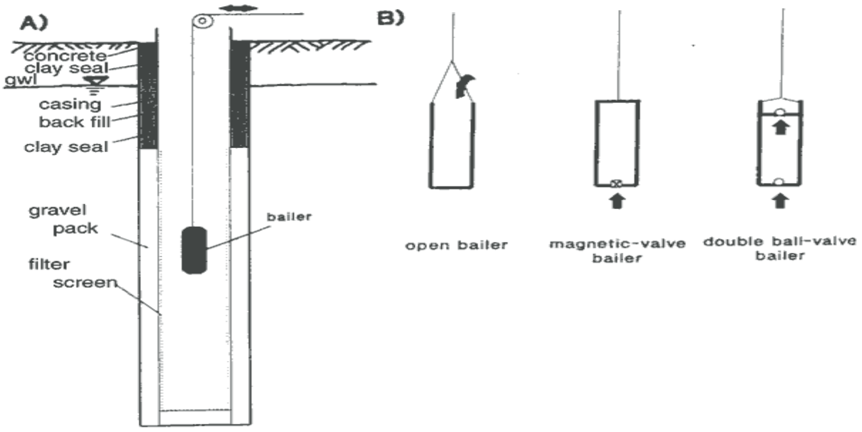
A rope attached to the bucket is often used. Scoops with adjustable shafts are convenient. Items made of synthetic resins such as polypropylene can also be used.





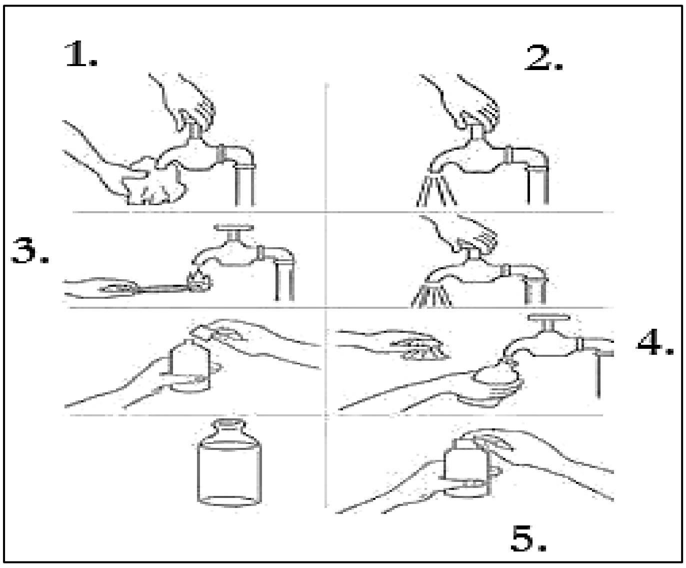
Groundwater (from well)

A bailer is a hollow tube used to retrieve groundwater samples from monitoring wells. Bailers are tied to a piece of rope or a piece of wire and lowered into the water column. Once lowered, the bailer uses a simple ball check valve to seal at the bottom in order to pull up a sample of the groundwater table.



Sampling water from a tap for microbiological analysis

1. Carefully clean and disinfect the inside and outside of the tap.
2. Open the tap and let water flow for 2-3 minutes or until the water temperature has stabilized.
3. Turn off the tap and sterilize the spout by heating it with a blow lamp, gas torch or by igniting a piece of cotton wool soaked with methylated spirits until any water in the tap boils.
4. Take care not to allow the container to touch the tap.
5. Take a water sample with the sample container
   (Details shown in figure below)



Sample labeling

Labeling is an important part in sampling program. The following information should be included in the label. Use water proof ink to record all the information:

- Date and time of sampling
- Sample field code
- Sampling point
- Nature of sample: Effluent / Surface water / Ground water / Others
- Type of sample (Grab/Composite/Integrated)
- Pre-treatment or preservation carried out on the sample
- Any special notes for the analyst
- Name and sign of sample collector.

Sample Preservation

There is usually a delay between the collection and analysis of a sample. The nature of the sample can be changed during this period. Therefore proper preservation is required in the way to laboratory after collection, and in the laboratory up to when analysis starts.

Complete and unequivocal preservation of samples- whether domestic wastewater, industrial wastes, or natural waters, is practically impossible as because - complete stability for every constituent never can be achieved.

At best, preservation techniques only retard chemical (especially, hydrolysis of constituents) and biological changes that inevitably continue after sample collection. No single method of preservation is entirely satisfactory; the preservative is chosen with due regard to the determinations to be made. Commonly used preservation methods are - pH control, chemical addition, the use of amber and opaque bottles, refrigeration, filtration, and freezing.

Experiment No. 1

Determination of pH of Water

Introduction

pH is a measure of the acidic or alkaline condition of water. It is a way of expressing the hydrogen ion concentration or more preciously, the hydrogen ion activity. pH is defined as follows:

$$\text{pH} = -\log\{H^+\} \qquad (1)$$

Where, $\{H^+\}$ is the concentration or activity of hydrogen ion or proton in moles/liter (M).

Water dissociates to form hydrogen ion (H^+) and hydroxyl ion (OH^-) according to the following equation:

$$H_2O = H^+ + OH^- \qquad (2)$$

At equilibrium, we can write,

$$K_w = \frac{\{H^+\}\{OH^-\}}{\{H_2O\}} \qquad (3)$$

Since concentration of water is extremely large (approximately 55.5 moles/L) and is diminished very little by the slight degree of ionization, it may be considered as a constant and its activity is taken as 1.0. Thus, Eq- 3 can be written as:

$$K_w = \{H^+\}\{OH^-\} \qquad (4)$$

Where, K_w = Equilibrium Constant

At 25^0C de-ionized or distilled water dissociates to yield 10^{-7}M of hydrogen ion (H^+) and 10^{-7}M of hydroxyl ion (OH^-). Therefore for pure water at 25^0C, $K_w = 10^{-7} \times 10^{-7} = 10^{-14}$. This is known as the ion product of water or ionization constant for water. Hence, according to Eq.- 1, pH of deionized water is equal to 7.0. The pH is usually represented by a scale ranging from 0 to 14, with 7 being neutral. pH of less than 7 indicate acidity, whereas a pH of greater than 7 indicates a base.

Groundwater is often found to be slightly acidic due to the presence of excess carbon dioxide. Aeration removes carbon dioxide and hence causes a rise in pH value. However, some natural waters are sometimes found to be slightly alkaline due to the presence of bicarbonate and, less often, carbonate. Water with pH outside the desirable neutral range may exhibit sour taste and accelerate the corrosion of metallic plumbing fittings and hot water services.

Environmental Significance

Since pH can be affected by chemicals in the water, pH is an important indicator of water that is changing chemically. A controlled value of pH is desired in water supplies, sewage treatment and chemical process plants. In water supply pH is important for coagulation, disinfection, water softening and corrosion control. In biological treatment of wastewater, pH is an important parameter, since organisms involved in treatment plants are operative within a certain pH range. According to Bangladesh Environment Conservation Rules (1997), drinking water guideline value for pH is 6.5 - 8.5.

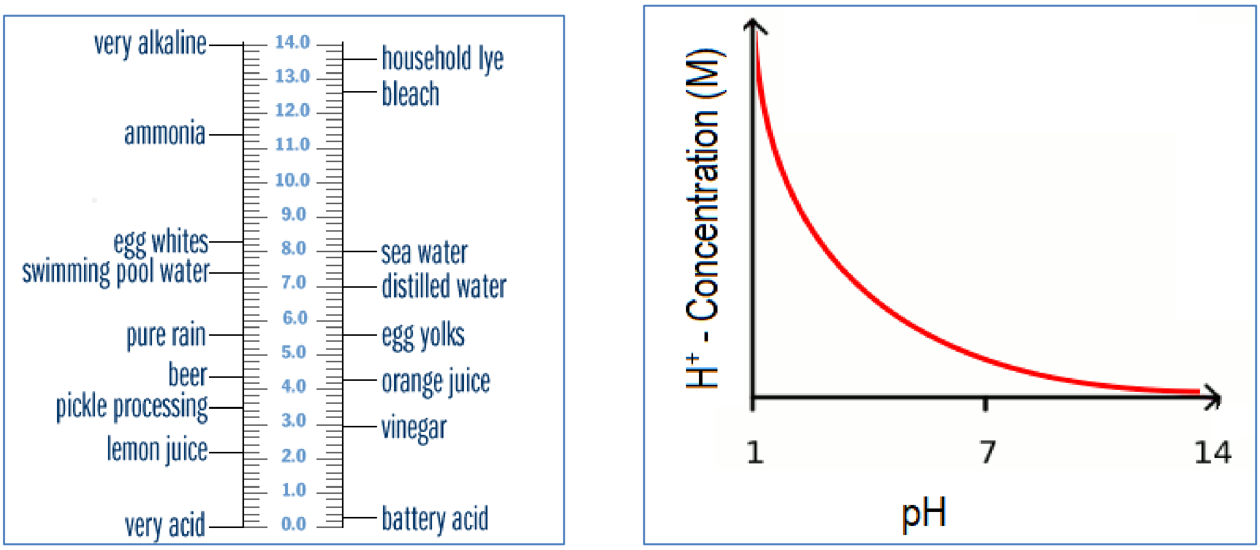


Figure-1.1 (a): pH scale

(b): H^+ ion activity vs pH

Theory on Experimental Method

Measurement of pH is carried out either colorimetrically (using pH indicator paper) or by electrochemical method (i.e., using a H^+ ion sensitive pH probe/meter). The second method is more expensive but precise whereas first one is cheap but serious erroneous result may produce due to interference of color, turbidity, high saline content, etc. However, before measuring pH with pH meter it is necessary to calibrate (standardize) the instrument using some buffer solutions of known pH value.

Reagent

- Standard pH solutions (buffer) for calibration of pH meter.

Apparatus

- pH meter

Figure-1.2: Digital pH meter

Procedure

1. Calibrate the pH meter using standard pH solutions. The calibration procedure would depend on the pH range of interest.
2. Take about 100 ml of the test sample in a beaker. Do not to agitate the sample to prevent exchange of gases between the sample and the atmosphere.
3. Insert pH meter in the sample. Allow sometime for attainment of equilibrium. Turn on the pH meter and take reading of pH.

Experiment No. 2

Determination of Color of Water

Introduction

Pure water should not pose any color. Color in water may result from the presence of natural metallic ions (iron and manganese),humus and peat materials, plankton, weeds, and industrial wastes. Impurities in water may exist either in the colloidal from or in suspended state. Color caused by dissolved and colloidal substances is referred as "true color" and that caused by suspended matter, in addition to dissolved and colloidal matters, is called "apparent color" as it can be easily removed by filtration. Ground water may show color due to the presence of iron compounds.

Environmental Significance

Though presence of color in water is not always harmful to human but in most cases it is. Even if the water is not harmful, aesthetically people do not prefer to use water with color. Moreover, disinfection by chlorination of water containing natural organics (which produces color) results in the formation of tri-halomethanes including chloroform and a range of other chlorinated organic substances leading to problems which is a major concern in water treatment. So it is important to limit the color of water for domestic supplies. According to Bangladesh Environment Conservation Rules (1997), drinking water guideline value for color is 15 Pt-Co Unit.

Theory on Experimental Method

Color is determined by visual comparison of the sample with known concentrations of colored solutions. Comparison also may be made with special, properly calibrated glass color disks. The platinum-cobalt method of measuring color is the standard method; the unit (Pt-Co Units) of color being that produced by 1 mg platinum–cobalt salt/L in the form of the chloro-platinate ion which is approximate the yellow-brown color of natural waters. The platinum-cobalt method is useful for measuring color of potable water and of water in which color is due to naturally occurring materials. It is not applicable to most highly colored industrial wastewaters.

In the laboratory color of water is usually measured using spectrophotometer which uses light intensity of a specific wavelength (455 ηm). The color test measures (inversely) an optical property of water sample which result from the absorption of light of specific wavelength by the soluble color substances present in water (Figure-2.2). Before measuring the color of water it is necessary to plot standard calibration curve for color using different standard platinum-cobalt solutions of known concentrations within the range of interest.

Reagent

- Standard potassium chloro-platinate solution

Apparatus

- Spectrophotometer (HACH, DR 4000U)
- Filtration system including filter paper, funnel, holder, beaker etc.

Fig.-2.1: HACH, DR 4000U spectrophotometer

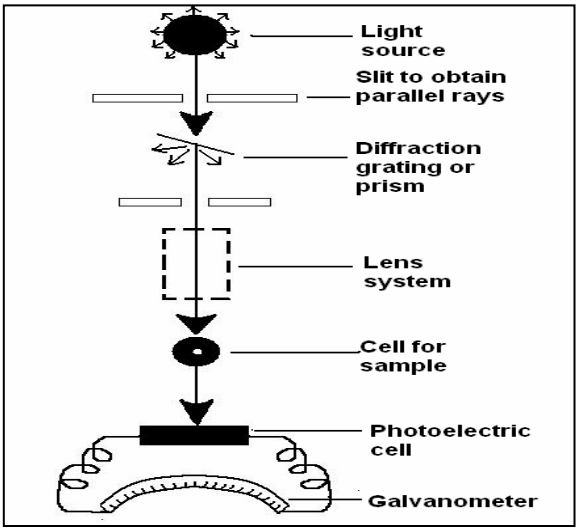


Fig. -2.2: Optical property used in measurement of color in water using spectrophotometer

Procedure

1. Prepare standard samples having color within a specific range by mixing different concentration of standard potassium chloro-platinate solution with distilled water. Using these samples to prepare a color calibration curve (absorbance vs color concentration) for the spectrophotometer.
2. Place a filter paper properly on a funnel and put a clean beaker below the funnel. Pour raw test sample on filter. Filtrate water is collected in the beaker. Discard initial portion of filtrate water. Take 50-mL of filtered test sample in a beaker.
3. Tale 50 –mL raw sample in another beaker. That gives the apparent color present in water sample.
4. Take 50-mL distilled water in another beaker. Use this sample as blank for color measurement.
5. Set the spectrophotometer to determine color concentration of the sample.
6. Put the blank sample inside the spectrophotometer cell and set the reading “zero”.
7. Bring out the blank sample and place the filtrate sample inside the spectrophotometer
8. After a while the display shows the true color concentration of the sample.
9. Repeat procedure 5 to 7 for the raw water sample and record the apparent color value.

Experiment No. 3

Determination of Turbidity of Water

Introduction

Turbidity is a measure of the degree to which the water loses its transparency due to the presence of suspended particulates. In other words the term "turbid" is applied to water containing suspended matter that interferes with passage of light through the water or in which visual depth is restricted. Turbidity may be caused by a wide variety of suspended substances of various sizes ranging in size from colloidal to coarse particles, depending on the degree of turbulence. In rivers under flood conditions, most of the turbidity will be due to relatively coarse particles whereas in lakes and other waters existing under relatively quiescent conditions, most of the turbidity will be due to colloidal and extremely fine particles.

Environmental Significance

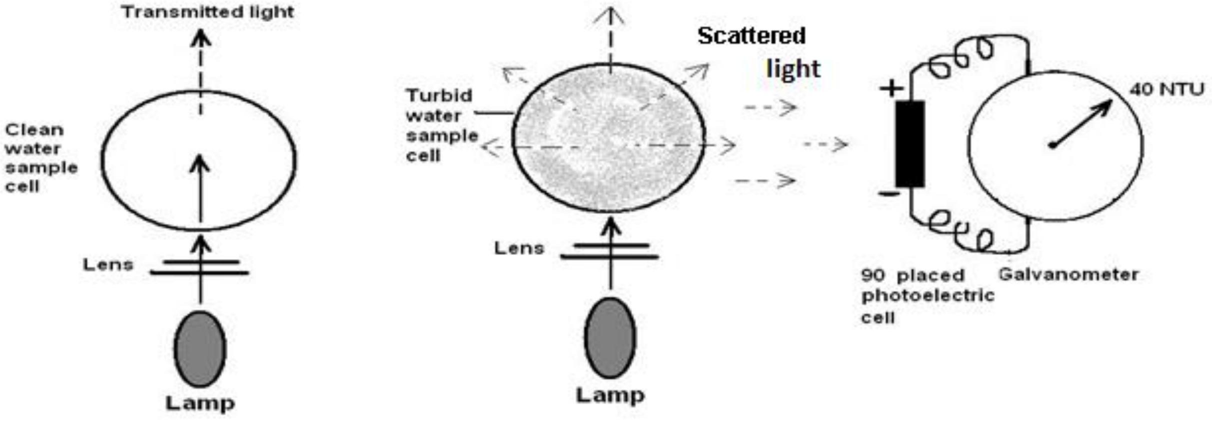
Turbidity is important for water supply engineers as turbid water is not aesthetically acceptable to people. For filtration process, turbid water is not suitable as it causes quick clogging of filter bed and thus requires the use of pre-treatment plant. Turbidity is also an important parameter in disinfection process. Disinfection is usually accomplished by means of chlorine, ozone, or chlorine dioxide. To be effective, there must be contact between the agent and the organisms to be killed. However, in cases in which turbidity is caused by municipal wastewater solids, many of the pathogenic organisms may be encased in the particles and protected from the disinfectant. In addition, the suspended particles absorb heat from the sunlight, making turbid waters become warmer, and so reducing the concentration of oxygen in the water. The suspended particles scatter the light, thus decreasing the photosynthetic activity of plants and algae, which contributes to lowering the concentration even more. According to Bangladesh Environment Conservation Rules (1997), drinking water standard for Turbidity is 10 NTU (Nephelometric turbidity unit).

Theory on Experimental Method

Turbidity is usually measured in Nephelometric turbidity units (NTU) or Jackson turbidity units (JTU). Both methods involve measurement of turbidity using either a nephelometer or Jackson turbidimeter to compare a reference solution to the sample. Nephlometer is now practiced as more reliable, sensitive, and easier to use instrument that depend upon the principle of nephelometry. The second method was a rather crude instrument in which the turbidity of a suspension was measured by the depth of suspension through which the outline of a flame from a standard candle just disappeared.

Jackson turbidimeter used silica as a standard reference material (1 mg SiO_2/L = 1 unit of turbidity). Whereas in nephelometer, a light source illuminates the sample and one or more photoelectric detectors are used with a readout device to indicate the intensity of scattered light at right angle to the path of the incident light(Fig.-1).In nephlometer standardized preparations of formazin polymer as standard reference material. In additions to formazin standards, commercially available standards such as styrene divinylbenzene beads are also available. Turbidity value as low as 0.02 NTU can be determined by this procedure, provided that water with sufficiently low turbidity can be obtained for use in instrument calibration.

Samples with turbidity greater than 40 NTU are diluted with turbidity-free water until values within the range of 30 to 40 NTU are obtained. The turbidity is then determined by multiplying the measured by the dilution factor.



(a) (b)

Fig.-3.1: Measurement of turbidity using Nephelometric turbidimeter; (a) for clean water sample, (b) for turbid water

Reagent

- Formazin polymer standards

Apparatus

- DR LANGE Turbidimeter instrument set

Fig.-3.2: Turbidimeter

Procedure

1. Standardized the turbidimeter using formazin standards.
2. Fill the clean sample cell with the test sample and place it in the sample cell holder. Cover the sample cell with the light shield.
3. Turn on the switch and read the turbidity value directly from the screen.

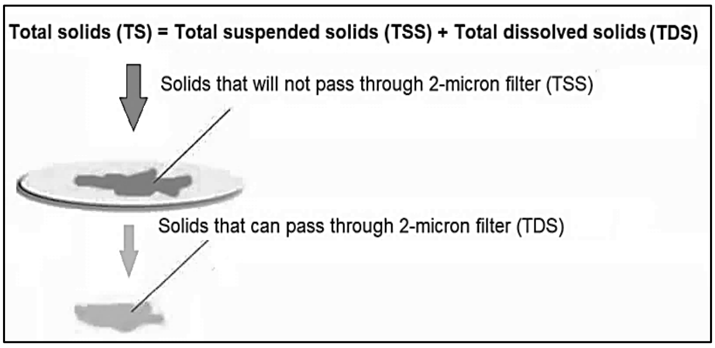
Experiment No. 4

Determination of Total Solids, Dissolved Solids and Suspended Solids in Water

---

Introduction

Total Solids (TS) refer to the matter that remains as residue upon evaporation and drying at 103 -105°C. Total solids (TS) include total suspended solids (TSS) - the portion of total solids retained by a filter, and total dissolved solids (TDS) - the portion that passes through the filter. Solids are incorporated in water from its source (e.g., minerals with which it has been in contact).



In potable water, most of the solids remain in dissolved form and consists of inorganic salts, small amounts of organic matter, and dissolved gases. The total dissolved solids content of potable water usually ranges from 20 to1000 mg/L. Groundwater usually has higher dissolved solids.

Environmental Significance

To determine whether water is suitable for domestic purpose, it is required to know how much solid it contains. According to Bangladesh Environment Conservation Rules (1997), potable water should not contain more than 1000 mg/L of total dissolved solids (TDS). Water with high dissolved solids generally is of inferior palatability and may induce unfavourable physiological reaction in the user. Concentrations of TDS above 500 mg/L result in excessive scaling in water pipes, water heaters, boilers and household appliances. Water high in suspended solids may be aesthetically unsatisfactory for such purposes as bathing.

Theory on Experimental Method

Water has a boiling point of 100°C. It may rise by a few degrees due to the presence of impurities. When water with solids is kept at 103 -105°C for 24 hours, the water gets evaporated and the solids either in dissolved state or in suspended state remain as residue.

A rapid assessment of the dissolved solids content of water can be obtained by specific conductance measurements. Such measurement indicates the capacity of a sample to carry an electrical current, which in turn is related to the concentration of ionized substances in the water. Most dissolved inorganic substances in water are in ionized form and so contribute to the specific conductance.

Although the nature of the various ions, their relative concentrations, and the ionic strength of the water affect this measurement, such measurement can give practical estimate of the dissolved mineral content of water. The TDS content can be approximated by multiplying the specific conductance in micro Siemens per cm (µS/cm) by an empirical factor varying from 0.55 to 0.90 depending on the chemical composition of the TDS.

Apparatus

- Beaker (150 mL) : 2 pcs
- Measuring cylinder (100 mL) : 1 pc
- Funnel : 1 pc
- Dropper : 1pc
- Filter paper (2 µm) : 2 pcs

Procedure

Measurement of Total Solids (TS):

1. Take a clear dry 150 - mL capacity glass beaker (which was kept at 103^{o}C in oven for 1 hour) and put appropriate identification mark on it. Weigh the empty beaker (W_1).
2. Thoroughly mix the sample. Measure 100 - mL of the sample using the measuring cylinder and pour in to the beaker.
3. Place the beaker in an oven maintained at 103^{o}C for 24 hours. After 24 hours, cool the beaker and weigh. Measure the total weight of solids residue and beaker (W_2).
4. Calculate total solids (TS) as follows:

$$\text{Total solids, TS (mg/L)} = \frac{(W_2 - W_1)\,(\text{mg}) \times 1000}{\text{volume of sample taken}\,(\text{mL})}$$

Measurement of Dissolved Solids(TS) :

1. Same as above (step-1 of TS measurement).
2. Take a 100 mL of sample and filter it through a double layered filter paper and collect the filtrate in a beaker.
3. Repeat the same procedure as in steps- 3 and 4 of TS measurement and determine the dissolved solids content as follows:

$$\text{Total Dissolved Solids, TDS (mg/L)} = \frac{\text{solid residue}\,(\text{mg}) \times 1000}{\text{volume of sample taken}\,(\text{mL})}$$

Measurement of Suspended Solids:

Total Suspended Solids, TSS (mg/L) = TS (mg/L) - TDS (mg/L)

Experiment No. 5

Determination of Carbon dioxide in Water

Introduction

The total carbon dioxide present in water is the sum of the free carbon dioxide and the carbon dioxide existing in the form of bicarbonate and carbonate ions. The presence of free carbon dioxide in water is caused by the decay of organic matter, dissolution of carbon dioxide from underground sources, and the dissolution from the atmosphere. Since the carbon dioxide content of the normal atmosphere is quite low (less than 0.04% by volume), this is not a source of appreciable carbon dioxide except where flue gases containing a high percentage of carbon dioxide are brought in relatively close contact with the water. Surface waters usually contain less than 10 ppm (mg/L) of free CO_2, while some ground water sources are found to contain free carbon dioxide exceeding 50 ppm (mg/L).

Environmental Significance

Carbon dioxide gas on solution in water produces carbonic acid (Equation-1) resulting in lowering of pH value and thus produces corrosive characteristic in water. Severe corrosion of heat exchanger, pipes, valves, etc. can result where appreciable quantities of free carbon dioxide are present in water. Corrosion from carbon dioxide in boiler system is most frequently encountered in steam and return lines. Even though the boiler feed water contains no free carbon dioxide, appreciable concentration of this corrosive gas may come from carbonate and bicarbonate of the boiler feed water. Although Bangladesh Environment Conservation Rules (ECR, 1997) does not set any limit for its presence in drinking water, limits for carbon dioxide has been prescribed for many industrial uses.

Theory on Experimental Method

This test for determination of free carbon dioxide in water involves titration of the water sample with standard sodium hydroxide solutions in the presence of phenolphthalein indicator (which changes color from colorless to pink at a pH of about 8.3). Carbon dioxide reacts with sodium hydroxide to form sodium bicarbonate with a consequent increase in pH value. The reaction of sodium hydroxide with free carbon dioxide reaches a completion at a pH of about 8.3. As soon as this pH is reached, a slight pink color is developed and this is taken as end point of reaction. The reactions may be considered to occur as follows:

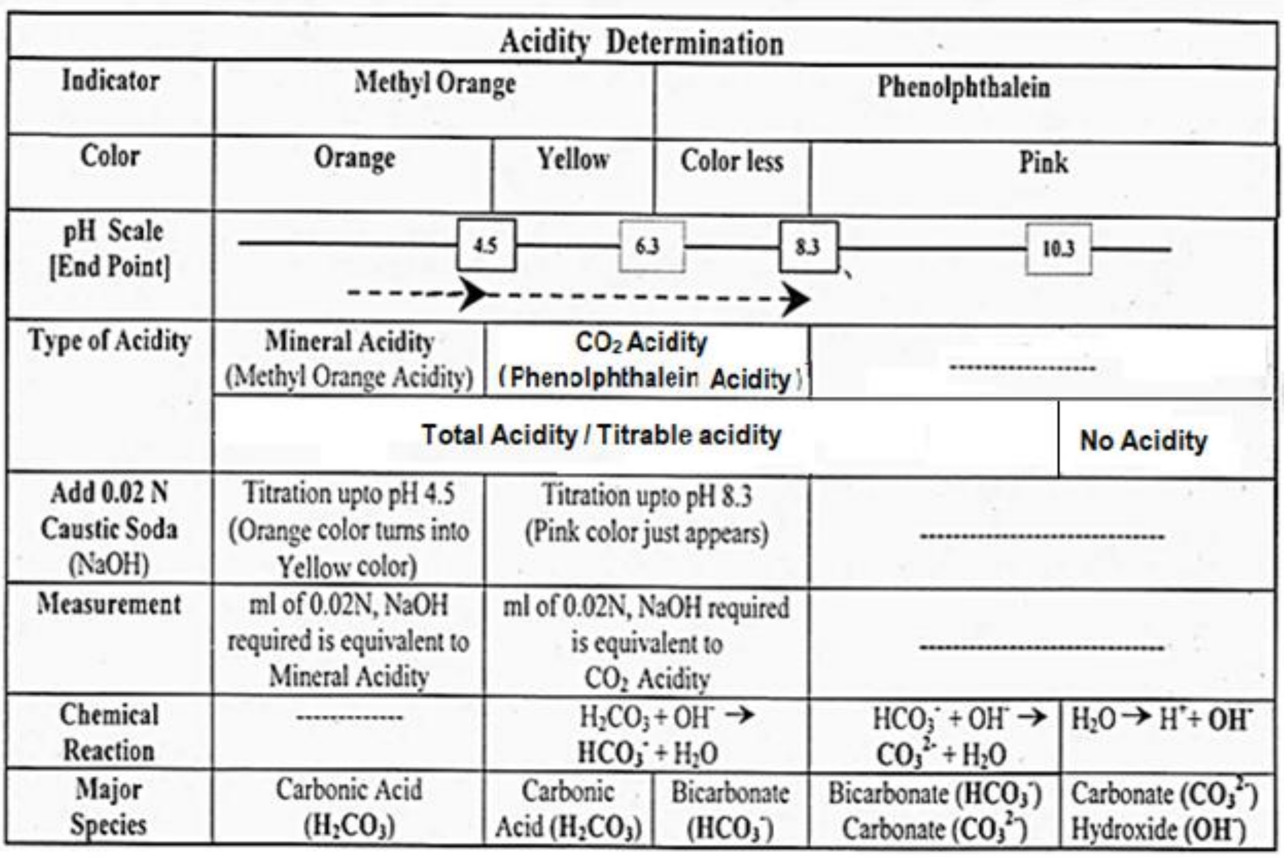
$$CO_2 + H_2O = H_2CO_3 \quad (1)$$
$$H_2CO_3 + NaOH = HCO_3^- + Na^+ + H_2O \quad (2)$$
$$HCO_3- + NaOH = CO_3^{2-} + Na^+ + H_2O \quad (3)$$

Determination of CO_2-Acidity

Phenolphthalein acidity (often called CO_2 acidity) of water is defined as the amount of standard base (usually $\frac{1}{50}$ N NaOH) required raising the pH of a sample of water to the phenolphthalein end point of 8.3. CO_2 acidity is expressed as $CaCO_3$ (calcium carbonate required to neutralize H_2CO_3)

Hence, Acidity could be easily determined from the results of CO_2 determination as follows:

$$\text{Phenolphthalein Acidity as mg/L } CaCO_3 = \frac{CO_2\ (\text{mg/L}) \times 50}{44} \quad (4)$$



Acidity Determination					
Indicator	**Methyl Orange**		**Phenolphthalein**		
Color	**Orange**	**Yellow**	**Color less**	**Pink**	
pH Scale [End Point]	4.5	6.3	8.3	10.3	
Type of Acidity	**Mineral Acidity** (Methyl Orange Acidity)	**CO$_2$ Acidity** (Phenolphthalein Acidity)		-----------------	
	Total Acidity / Titrable acidity				**No Acidity**
Add 0.02 N Caustic Soda (NaOH)	Titration upto pH 4.5 (Orange color turns into Yellow color)	Titration upto pH 8.3 (Pink color just appears)		----------------------------	
Measurement	ml of 0.02N, NaOH required is equivalent to Mineral Acidity	ml of 0.02N, NaOH required is equivalent to CO$_2$ Acidity		----------------------------	
Chemical Reaction	------------	$H_2CO_3 + OH^- \rightarrow HCO_3^- + H_2O$		$HCO_3^- + OH^- \rightarrow CO_3^{2-} + H_2O$	$H_2O \rightarrow H^+ + OH^-$
Major Species	Carbonic Acid (H_2CO_3)	Carbonic Acid (H_2CO_3)	Bicarbonate (HCO_3^-)	Bicarbonate (HCO_3^-) Carbonate (CO_3^{2-})	Carbonate (CO_3^{2-}) Hydroxide (OH^-)

Reagents

- Phenolphthalein indicator
- Standard $\frac{1}{44}$N sodium hydroxide

Apparatus

- Beaker : 2 pc
- Measuring cylinder : 1 pc.
- Dropper : 1 pc.
- Stirrer : 1 pc.
- Burette : 1 pc.

Procedure

1. Take 100 mL of sample into a beaker and same quantity of distilled water into another. Add 10 drops of Phenolphthalein indicator to each. If a pink color develops, no carbon dioxide is present in the water sample. Otherwise, go to the next step.

2. Add $\frac{1}{44}$N sodium hydroxide solution from a burette to the sample and stir gently until a slight permanent pink color appears as compared with the distilled water. Measure the mL of sodium hydroxide required for titration.

Calculation

Free CO_2 in water (mg/L) = Multiplying Factor (MF) x mL of $\frac{1}{44}$ N NaOH added

Where, MF = $$\frac{\text{normality of NaOH x equivalent wt.of } CO_2 \text{ x} 1000}{\text{mL of sample taken}}$$

Phenolphthalein acidity as $CaCO_3$ (mg/L) – use equation (4)

Note:
Since excess CO_2, if present in water, easily escapes to the atmosphere, tests for presence of CO_2 in water should be performed immediately after collection of water sample, especially for groundwater samples which usually contain high carbon dioxide. If this is not possible, the sample bottle should be completely filled and capped and the sample should be kept at a temperature lower than that at which it was collected.
Where the free CO_2 of the water sample is high, there may be some loss of carbon dioxide to the atmosphere during the titration process. To check this, it is advisable to secure a second sample. Complete determination of CO_2 on the first sample. Then take the second sample (100 mL) and immediately add the full amount of $\frac{1}{44}$ N sodium hydroxide solution used in the titration of the first sample. Add 10 drops of phenolphthalein indicator and if the sample remains colorless, add additional $\frac{1}{44}$N sodium hydroxide to the end point (till the slight pink color appears) and accepting this second test as more accurate titration.

Determination of Alkalinity of Water

Introduction

Alkalinity is the measure of water's capacity to absorb hydrogen ion (neutralize acids) without significant pH change. The term acid neutralizing capacity (ANC) is sometimes used. The alkalinity of natural waters is due primarily to the salts of weak acids, although weak or strong bases may also contribute and such substances act as buffer to resist a drop in pH resulting from acid additions. Bicarbonates represent the major form of alkalinity, since they are formed in considerable amounts from the action of CO_2 upon basic materials in the soil.

$$CO_2 + CaCO_3 + H_2O = Ca^{2+} + 2HCO_3^- \qquad (1)$$

Other salts of weak acids, such as borates, silicates, and phosphates, may be present in small amounts. A few organic acids, e.g., humic acid, form salts that add to the alkalinity of natural waters.

Although many materials may contribute to the alkalinity of water, three major types of chemical constituents cause the major portion of the alkalinity in natural waters: (i) hydroxides, (ii) carbonates, and (iii) bicarbonates. For most practical purposes, alkalinity due to other materials in natural waters is insignificant and may be ignored. The predominance of different carbonate species (closed system) is shown in Fig- 1.

$$\text{Total Alkalinity} = \Sigma\,[\,HCO_3^- + 2CO_3^{2-} + OH^- - H^+\,]$$

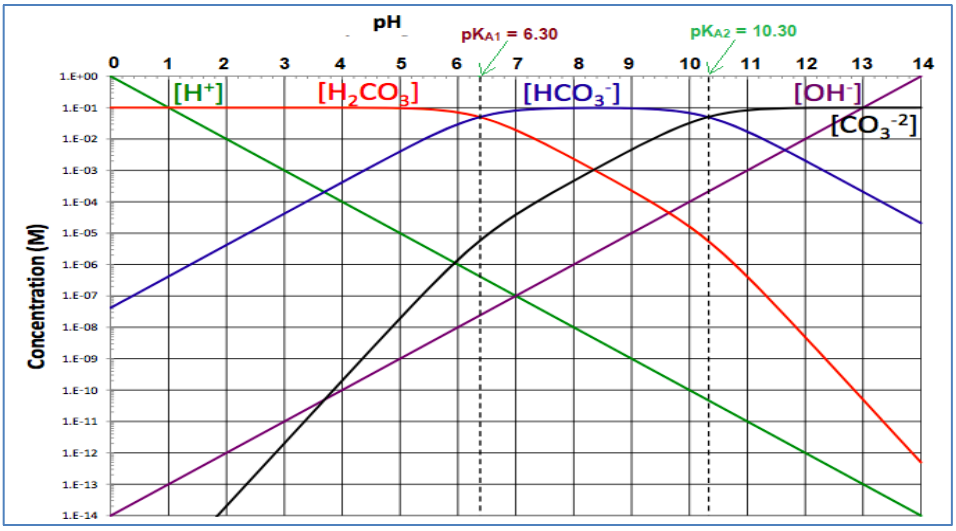


Fig.- 6.1: Logarithmic concentration diagram for 0.01 M Carbonic Acid (Source: Sawyer et al., 1994)

Environmental Significance

As mentioned earlier alkalinity is a measure of the buffer capacity and in this sense is used to a great extent in wastewater treatment practice. Alkalinity is very important in iron removal from water by oxidation-precipitation process. It has been found that rate of oxidation of dissolved ferrous iron (Fe^{2+}) into insoluble ferric iron (Fe^{3+}) is very slow for low alkaline waters (alkalinity less than 130 mg/L as $CaCO_3$). Because the alkalinity of many waters is primarily a function of carbonate, bicarbonate and hydroxide content, it is taken as an indication of the concentration of these constituents.

Alkalinity of water has little public health significance. Highly alkaline waters are usually unpalatable. Typical alkalinity of different natural water sources are given in the table below:

Water sources	**Alkalinity (mg/L as $CaCO_3$)**
Rainwater	< 10
Typical surface water	20 - 200
Surface water in regions with alkaline soils	100 - 500
Groundwater	50 - 1000
Seawater	100 - 500

Theory on Experimental Method

Alkalinity is measured volumetrically by titration with $\frac{1}{50}$ N(i.e.,0.02 N) H_2SO_4 and is reported in terms of equivalent $CaCO_3$. For samples with initial pH above 8.3, the titration is made in two steps-

i) In the first step, the titration is conducted until the pH is lowered to 8.3, the point at which phenolphthalein indicator turns from pink to colorless.

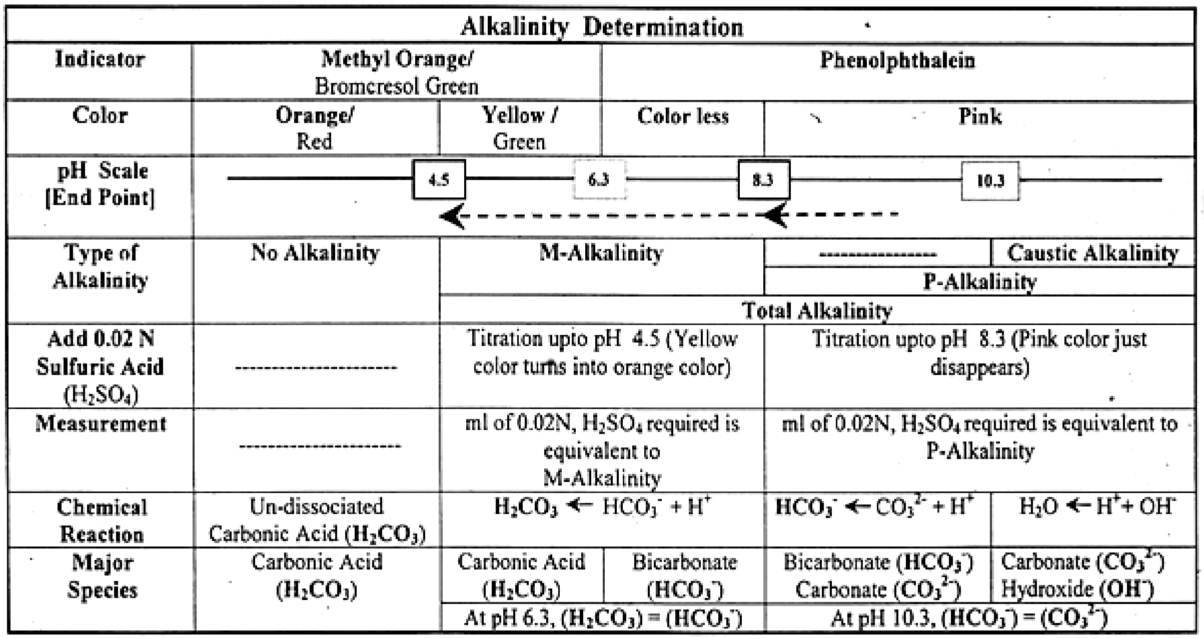
ii) The second phase of the titration is conducted until the pH is lowered to about 4.5, corresponding to the methyl orange (or bromocresol green) end point.

When the pH of a sample is less than 8.3, a single titration is made to a pH of 4.5. The first end point of pH 8.3 corresponds to the equivalence point for the conversion of carbonate to bicarbonate (Eq.-1,Figure-1). When alkalinity is measured to the phenolphthalein end point (pH 8.3), it is called phenolphthalein alkalinity, which is due to the presence of either hydroxide or carbonate or both.

$$CO_3^{2-} + H^+ = HCO_3^- \qquad (2)$$

The use of a pH of about 4.5 for the end point for the second step of the titration corresponds approximately to the equivalence point for the conversion of bicarbonate ion to carbonic acid (Eq. 2, Fig. 1). When alkalinity is measured to the methyl orange (or bromocresol green) end point, it is called methyl orange alkalinity or total alkalinity, which is due to the presence of hydroxide, carbonate, and bicarbonate.

$$HCO_3^- + H^+ = CO_3^{2-} \qquad (3)$$



Alkalinity Determination					
Indicator	**Methyl Orange/** Bromcresol Green		**Phenolphthalein**		
Color	**Orange/ Red**	**Yellow / Green**	**Color less**	**Pink**	
pH Scale [End Point]	4.5	6.3	8.3	10.3	
Type of Alkalinity	**No Alkalinity**	**M-Alkalinity**		---------------	**Caustic Alkalinity**
				P-Alkalinity	
		Total Alkalinity			
Add 0.02 N Sulfuric Acid (H_2SO_4)	---------------------	Titration upto pH 4.5 (Yellow color turns into orange color)		Titration upto pH 8.3 (Pink color just disappears)	
Measurement	---------------------	ml of 0.02N, H_2SO_4 required is equivalent to M-Alkalinity		ml of 0.02N, H_2SO_4 required is equivalent to P-Alkalinity	
Chemical Reaction	Un-dissociated Carbonic Acid (H_2CO_3)	$H_2CO_3 \leftarrow HCO_3^- + H^+$		$HCO_3^- \leftarrow CO_3^{2-} + H^+$	$H_2O \leftarrow H^+ + OH^-$
Major Species	Carbonic Acid (H_2CO_3)	Carbonic Acid (H_2CO_3)	Bicarbonate (HCO_3^-)	Bicarbonate (HCO_3^-) Carbonate (CO_3^{2-})	Carbonate (CO_3^{2-}) Hydroxide (OH^-)
		At pH 6.3, (H_2CO_3) = (HCO_3^-)		At pH 10.3, (HCO_3^-) = (CO_3^{2-})	

Reagents

- Standard 0.02N sulfuric acid
- Phenolphthalein indicator

- Methyl orange indicator

Apparatus

- Beaker	: 2 pcs
- Measuring cylinder	: 1pc
- Dropper	: 1pc
- Stirrer	: 1pc

Procedure

1. Take 100 mL of the sample into one beaker and the same amount of distilled water into another beaker.
2. Measure pH of the sample with a pH meter.
3. Add 3 drops of phenolphthalein indicator to each. If the sample becomes pink, go to step- 3. Otherwise, go to step- 4.
4. Add 0.02N H_2SO_4 acid from a burette until the pink color just disappears. Record the mL of the acid used.
5. Add 3 drops of methyl orange indicator to each beaker. If the sample becomes yellow, add 0.02N H_2SO_4 acid until the first change in color is noted. The end point is a slight orange tinge. Record the mL of acid used.

Calculation

Phenolphthalein Alkalinity (mg/L as $CaCO_3$)

= Multiplying Factor (MF) x mL of 0.02N H_2SO_4 (added up to pH 8.3) (4)

and, Total Alkalinity (mg/L as $CaCO_3$)

= MF x mL of 0.02N H_2SO_4 (added upto pH approx. 4.5) (5)

Where, MF = $$\frac{\text{normality of } H_2SO_4 \text{ x equivalent wt.of } CaCO_3 \text{ x1000}}{\text{mL of sample taken}}$$

Calculation of Hydroxide, Carbonate and Bicarbonate Alkalinity

In water analysis it is often desirable to know the kinds and amounts of the various forms of alkalinity present. This information is especially needed in water-softening processes and in boiler-water analysis. The most common approach is computation of the concentrations of the different alkalinity forms using various equilibrium equations for carbonic acid. This method gives reasonably accurate results for constituents, even when present in fractional mg/L range. A total alkalinity, as well as a pH measurement, is required. In addition, a dissolved solids measurement to correct for ion activity and a temperature measurement for the selection of a proper equilibrium constant are needed. However, reasonably accurate results are obtained without these two data if the dissolved solids concentration is not too high and water temperature is around 25°C. Description of all three procedures is available in Sawyer et al. (1994). The following section describes only the third procedure to determine hydroxide, carbonate and bicarbonate alkalinities.

Calculation from Equilibrium Equations

The relevant equilibrium equations are as follows:

$$H_2O = H^+ + OH^- \qquad (6a)$$

$$K_w = \{H^+\}\{OH^-\} = 10^{-14} \text{ (at } 25^oC) \qquad (6b)$$

$$H_2CO_3 = H^+ + HCO_3^- \qquad (7a)$$

$$K_{A1} = \frac{\{H^+\}\{HCO_3^-\}}{\{H_2CO_3\}} = 10^{-6.3} \text{ (at } 25^oC) \qquad (7b)$$

$$HCO_3^- = H^+ + CO_3^{2-} \qquad (8a)$$

$$K_{A2} = \frac{\{H^+\}\{CO_3^{2-}\}}{\{HCO_3^-\}} = 10^{-10.3} \text{ (at } 25^oC) \qquad (8b)$$

The alkalinity of water can be expressed as follows:
Total alkalinity (mg/L as $CaCO_3$)/50,000 = $\sum[\,(HCO_3^-) + 2(CO_3^{2-}) + (OH^-) - (H^+)\,]$ (9)

From the above equations, the following expressions for different alkalinities can be derived:

Carbonate alkalinity (mg/L as $CaCO_3$)

$$= \frac{50{,}000\ \left\{\left(\frac{\text{Alkalinity}}{50{,}000} + H^+\right) - \left(\frac{K_w}{H^+}\right)\right\}}{\left[1 + \left(\frac{H^+}{2K_{A2}}\right)\right]} \qquad (10)$$

Bicarbonate alkalinity (mg/L as $CaCO_3$)

$$= \frac{50{,}000\ \left\{\left(\frac{\text{Alkalinity}}{50{,}000} + H^+\right) - \left(\frac{K_w}{H^+}\right)\right\}}{\left[1 + \left(\frac{2K_{A2}}{H^+}\right)\right]} \qquad (11)$$

It should be noted that the equilibrium constants vary with temperature and activities of ions vary with concentration of ions in water. If temperature of water varies significantly from 25^oC and ionic strength of water is high (i.e., high concentration dissolved ions), appropriate corrections need to be incorporated in the above equations. Otherwise these equations would give reasonably accurate results.

Sometimes actual concentrations of carbonate and bicarbonate ions may be desired. Conversions to mg/L of CO_3^{2-} and HCO_3^- are as follows:

mg/L CO_3^{2-} = mg/L carbonate alkalinity x 0.6 (12)
mg/L HCO_3^- = mg/L bicarbonate alkalinity x 1.22 (13)

Molar concentrations may be obtained as follows:

(CO_3^{2-}) = (mg/L CO_3^{2-}) / 60,000 (14)
(HCO_3^-) = (mg/L HCO_3^-) / 61,000 (15)

Experiment No. 7

Determination of Hardness of Water

Introduction

Hard waters are generally considered to be those waters that require considerable amounts of soap to produce foam or lather and that also produce scale in hot-water pipes, heaters, boilers, and other units in which the temperature of water is increased substantially. The hardness of water varies considerably from place to place. In general, surface water is softer than groundwater. The hardness of water reflects the nature of the geological formations with which it has been in contact.

Hardness is caused by multivalent metallic cations. Such cations are capable of reacting with soap to form precipitates and with certain anions present in water to form scale. The principal hardness causing cations are the divalent calcium, magnesium, strontium, ferrous iron, and manganous ions. These cations and the important anions with they are associated are shown in Table-1 in the order of their relative abundance in natural waters. Aluminum and ferric ions are sometimes considered as contributing to the hardness of water. However, their solubility is so limited at pH values of natural waters that ionic concentrations are negligible. The hardness of water is derived largely from contact with the soil and rock formations.

Table- 7.1. **:** Principal hardness causing cations and the major anions associated with them

Cations causing Hardness	Associated Anions
Ca^{2+}	HCO_3^-
Mg^{2+}	SO_4^{2-}
Sr^{2+}	Cl^-
Fe^{2+}	NO_3^-
Mn^{2+}	SiO_3^{2-}

Hardness caused by each cation can be calculated as follows:

$$\text{Hardness (mg/L as CaCO}_3\text{)} = \frac{M^{2+}\ (\text{mg/L}) \times 50}{\text{Equivalent weight of } M^{2+}} \quad \text{--(1)}$$

Where, M^{2+} = concentration of divalent metal cation (mg/L) and 50 is the equivalent weight of $CaCO_3$.

$$\text{Total Hardness} = \sum\left[\frac{50}{20.04}Ca^{++} + \frac{50}{12.15}Mg^{++} + \frac{50}{27.92}Fe^{++} + \frac{50}{27.46}Mn^{++}\right] \text{--(2)}$$

$$= \sum[2.49\,Ca^{++} + 4.12\,Mg^{++} + 1.79\,Fe^{++} + 1.82\,Mn^{++}] \quad \text{--(3)}$$

Environmental Significance

Hard waters are as satisfactory for human consumption as soft waters. Because of their adverse action with soap, however, their use for cleansing purpose is quite unsatisfactory, unless soap costs are disregarded. Soap consumption by hard waters represents an economic loss to the water user. Sodium soaps react with multivalent metallic cations to form a precipitate, thereby losing their surfactant properties. In recent years these problems have been largely alleviated by the development of soaps and detergents that do not react with hardness.

Boiler scale, the result of the carbonate hardness precipitation, may cause considerable economic loss through fouling of water heater and hot water pipes. Changes in pH in the water distribution systems may also result in deposits of precipitates. Bicarbonates begin to convert to the less soluble carbonates at pH values above 9.0.

Magnesium hardness, particularly associated with the sulfate ion has a laxative effect on persons unaccustomed to it. Magnesium concentrations of less than 50 mg/L are desirable in potable waters, although many public water supplies exceed the amount. Calcium hardness presents no public health problem. In fact, hard water is apparently beneficial to the human cardiovascular system. Water can be generally classified in terms of the degree of hardness as follows:

Table-7.2: Water quality with respect to hardness

Water Quality	Hardness (mg/L as $CaCO_3$)
Soft	<50
Moderately hard	50-150
Hard	150-300
Very hard	>300

Theory on Experimental Method

Hardness is usually expressed in terms of $CaCO_3$. Perhaps the most accurate method of determining hardness is by a calculation based upon the divalent ions found through complete cation analysis (Equation– 1 to 3).This method is preferred where complete analyses are available.

However, complete analyses of metal ions are not always available and in laboratory total hardness is usually determined by the *EDTA Titrimetric Method*. This method yields very precise and accurate results and is the method of choice in most laboratories.

The EDTA Titrimetric method involves the use of solutions of ethylene-diamine-tetra-acetic acid (EDTA) or its sodium salt as the titrating agent. These compounds are chelating agents (A chemical compound in the form of a heterocyclic ring, containing a metal ion attached by coordinate bonds to at least two non-metal ions) and thus form extremely stable complexes with Ca^{2+}, Mg^{2+} and other divalent cations causing hardness, as shown in Eq. -4.

$$M^{2+} + \text{EDTA} = [\text{M . EDTA}]_{\text{complex}} \qquad \text{--(4)}$$

The successful use of EDTA for determining hardness depends upon having an indicator present to show when EDTA is present in excess, or when all the ions causing hardness have been complexed.

The Eriochrome Black T dye serves as an excellent indicator to show when all the hardness ions have form complex with EDTA. When a small amount of Eriochrome Black T, having a blue color, is added to hard water with a pH of about 10.0, it combines with a few of the $Ca^{2+}$and Mg^{2+} ions to form a weak complex ions which produces wine red in color in the solution (Eq.-5).

$$M^{2+} + \text{Eriochrome Black T} = \underset{\textit{(wine red color)}}{(\text{M . Eriochrome Black T})_{\text{complex}}} \qquad \text{--(5)}$$

During titration with EDTA, all free hardness ions are complexed according to Eq. 2. Finally, the EDTA disrupts the weak wine red complex compounds (M . Eriochrome Black T) and forms more stable complex with the divalent ions. This action frees the Eriochrome Black T indicator, and the wine red color changes to a distinct blue colour (Eq.- 4), indicating the end of titration.

$$\underset{\textit{(wine red color)}}{(\text{M.Eriochrome Black T})_{\text{complex}}} + \text{EDTA} = (\text{M.EDTA})_{\text{complex}} + \underset{\textit{(blue color)}}{\text{Eriochrome Black T}} \quad \text{--(6)}$$

Reagents

- Buffer solution (for attaining a pH close to 10.0).
- Eriochrome Black T dye.
- Standard EDTA solution
- Auto titrator (Hach)

Apparatus

- Beaker : 1 pcs
- Measuring cylinder : 1pc
- Dropper : 1pc
- Stirrer : 1pc
- HACH Auto-titration device: 1 set

Procedure

1. Take 50 mL sample in a 150 mL beaker.
2. Add one ml of standard buffer solution (supplied by HACH) to raise the pH of water sample to about 10.0 (Note: If 100 mL sample is taken, add 2 mL buffer).

3. Add one packet of Eriochrome Black T dye (supplied by HACH) indicator to the beaker. The sample would turn wine-red (if hardness is present).
4. Fit the cartridge containing standard EDTA solution to the titrator device (supplied by HACH).
5. Turn the flow control knob of the device until the solution starts to come out of the tube fitted to the cartridge. Take initial reading of the counter.
6. Immerse the tube fitted to the cartridge into the water sample and start titrating (under constant stirring) by turning the flow control knob of the auto-titrator. Continue until the wine-red color of the sample changes to blue. Take final reading of the counter.

Calculation

Total hardness (mg/L as $CaCO_3$) = MF x (initial counter reading - final counter reading)

$$\text{Where, MF} = \frac{\text{normality of EDTA x equivalent wt. of } CaCO_3 \text{ x 1000}}{\text{mL of sample taken}}$$

(Normality of EDTA titrant used in the lab is 0.002 N)

Therefore for 100 mL sample,

Total hardness (mg/L as $CaCO_3$) = initial counter reading - final counter reading

And for 50 mL sample,

Total hardness (mg/L as $CaCO_3$) = 2 x (initial counter reading - final counter reading)

Types of Hardness

In addition to total hardness, it is often desirable and sometimes necessary to know the type of hardness present. Hardness is classified in two ways:

(1) With respect to the metallic ion (calcium and magnesium hardness), and

(2) With respect to the anions associated with the metallic ions (carbonate and non-carbonate hardness).

Calcium and Magnesium Hardness

Calcium and magnesium cause by far the greatest portion of the hardness occurring in natural waters. Sometimes, it is important to know the amounts of calcium and magnesium hardness in water. For example, it is necessary to know the magnesium hardness or the amount of Mg^{2+} in order to calculate lime requirement in lime-soda ash softening. The calcium and magnesium hardness may be calculated from the complete chemical analysis. If calcium hardness is determined, magnesium hardness is obtained by subtracting calcium hardness from total hardness as follows:

$$\text{Total hardness - calcium hardness = magnesium hardness} \quad (5)$$

Carbonate and Non-carbonate Hardness

The part of total hardness that is chemically equivalent to the bicarbonate plus carbonate alkalinities present in water is considered to be carbonate hardness. Since alkalinity and hardness are both expressed in terms of $CaCO_3$, the carbonate hardness can be found as follows:

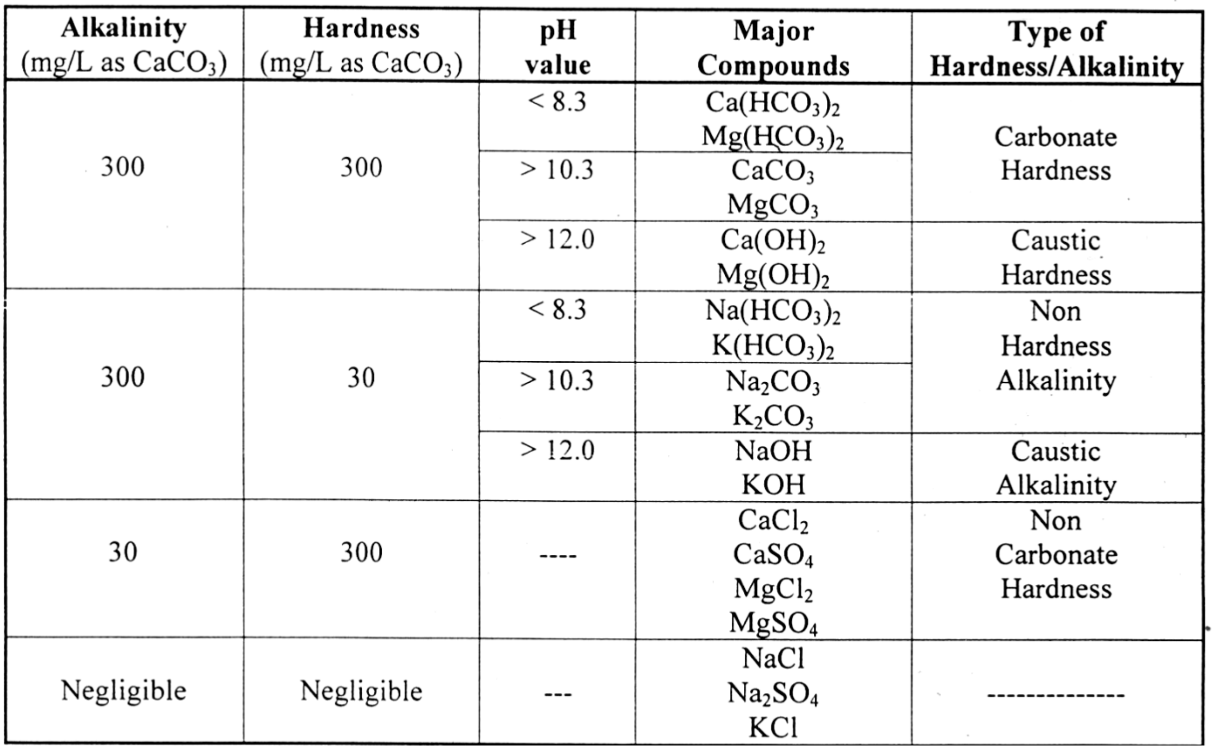
When alkalinity < total hardness,

$$\text{Carbonate hardness (mg/L) = alkalinity (mg/L)} \quad (6)$$

When alkalinity ≥ total hardness,

$$\text{Carbonate hardness (mg/L) = total hardness (mg/L)} \quad (7)$$

Table -3: Different Forms of Hardness Causing Compounds Depending on pH, Harness and Alkalinity



Alkalinity (mg/L as $CaCO_3$)	**Hardness** (mg/L as $CaCO_3$)	**pH value**	**Major Compounds**	**Type of Hardness/Alkalinity**
300	300	< 8.3	$Ca(HCO_3)_2$ $Mg(HCO_3)_2$	Carbonate Hardness
		> 10.3	$CaCO_3$ $MgCO_3$	
		> 12.0	$Ca(OH)_2$ $Mg(OH)_2$	Caustic Hardness
300	30	< 8.3	$Na(HCO_3)_2$ $K(HCO_3)_2$	Non Hardness Alkalinity
		> 10.3	Na_2CO_3 K_2CO_3	
		> 12.0	NaOH KOH	Caustic Alkalinity
30	300	----	$CaCl_2$ $CaSO_4$ $MgCl_2$ $MgSO_4$	Non Carbonate Hardness
Negligible	Negligible	---	NaCl Na_2SO_4 KCl	--------------

Carbonate hardness is of particular interest because the bicarbonate and carbonate ions with which it is associated tend to precipitate this portion of the hardness at elevated temperatures such as occur in boilers or during the softening process with lime. Carbonate hardness was formerly called *temporary hardness* because it can be caused to precipitate by prolonged boiling (Eq.- 8).

$$Ca^{2+} + 2HCO_3^- = CaCO_3\,(s) + CO_2 + H_2O \qquad (8)$$
$$Ca^{2+} + 2HCO_3^- + Ca(OH)_2 = 2CaCO_3\,(s) + 2H_2O \qquad (9)$$

The amount of hardness which is in excess of the carbonate hardness is called non-carbonate hardness, and can be estimated as follows:

Non-carbonate hardness (NCH) = total hardness - carbonate hardness (10)

Non-carbonate hardness was formerly called permanent hardness because it cannot be removed or precipitated by boiling. Non-carbonate hardness cations are associated with sulfate, chloride, and nitrate anions.

Pseudo-Hardness

Sea, brackish, and other waters that contain appreciable amounts of Na^+ interfere with the normal behavior of soap because of the common ion effect. Sodium is not a hardness-causing cation, and so this action which it exhibits when present in water in high concentration is termed *pseudo-hardness*.

Experiment No. 8

Determination of Chloride in Water

Introduction

Chlorides occur in all natural waters in widely varying concentration. The chloride ion is highly mobile and concentrations in water are not affected by chemical reactions. Hence chloride does not biodegrade, readily precipitate, volatilize, or bioaccumulate. The chloride content normally increases as the mineral content increases. Upland and mountain supplies usually are quite low in chlorides, whereas river and groundwater usually have a considerable amount. Sea and ocean waters represent the residues resulting from partial evaporation of natural waters that flow into them and chloride levels are very high.

Chlorides gain access to natural waters in many ways. The solvent power of water dissolves chlorides from topsoil and deeper formations. Spray from the ocean is carried inland as droplets or as minute salt crystals, which result from evaporation of the water in the droplets. These sources constantly replenish the chlorides in inland areas where they fall. Ocean and seawaters invade the rivers that drain into them, particularly the deeper rivers. The salt water, being denser, flows upstream under the fresh water, which is flowing downstream. There is a constant intermixing of the salt water with the fresh water above. Groundwater in areas adjacent to the ocean is in hydrostatic balance with seawater. Over-pumping of groundwater produces a difference in hydrostatic head in favor of the seawater, and it introduce into the fresh water area. Such intrusion has occurred in many areas of the coastal southern region of Bangladesh.

Human excreta, particularly the urine, contain chloride in an amount about equal to the chlorides consumed with flood and water. This amount average about 6 gm of chlorides per person per day and increases the amount of Cl^- in municipal wastewater about 15 mg/L above that of the carriage water. Thus, wastewater effluents add considerable chlorides to receiving streams. Many industrial wastes (e.g., tannery waste) also contain appreciable amount of chlorides.

Environmental Significance

Chlorides in reasonable concentrations are not harmful to human. At concentrations above 250 mg/L they give a salty taste to water, which is objectionable to many people. However, The taste threshold of the chloride anion in water is dependent on the associated cation. Taste thresholds for sodium chloride and calcium chloride in water are in the range 200–300 mg/L. The taste of coffee is affected if it is made with water having a chloride concentration above 400 mg/L as NaCl or 530 mg/L as $CaCl_2$. Again, in many areas of the world where water supplies are scarce, source containing as much as 2,000 mg/L are used for domestic purposes without the development of adverse effects, once the human system becomes adapted to the water.

According to Bangladesh Environment Conservation Rules (1997), drinking water standard for chloride is 150 - 600 mg/L; but for coastal regions of Bangladesh, the limit has been relaxed to 1000 mg/L.

Before the development of bacteriological testing procedures, chloride and nitrogen determination is being used for detecting contamination of groundwater by wastewater. Chlorides are used to some extent as tracers in sanitary engineering practice; however, they have been replaced to a great extent by organic dyes. Moreover measurement of total salinity and Cl^- is also important for irrigation water.

Theory on Experimental Method

A number of suitable analytical techniques are available for chloride in water:

i. Mohr method – silver nitrate titration with potassium as the chromate indicator,
ii. Mercuric nitrate method- mercuric nitrate titration with diphenyl-carbazone as the indicator.

iii. Potentiometric titration with silver nitrate,
iv. Automated iron(iii) mercury(ii) thiocyanate colorimetry,
v. Chloride ion-selective electrode,
vi. Silver colorimetry, and
vii. Ion chromatography.

In this class chloride is determined using Mohr method. This method involves simple titration process. However, in Mohr method color change occurs gradually and true end point (reddish brown precipitate) cannot be detected readily in solutions containing low concentrations of chloride (below 20 ppm) and even in higher concentrations, satisfactory titrations can be made only by a highly skilled technologist..
The "Standard Method" recommends a 0.0141 N silver nitrate solution which corresponds to $\frac{1}{71}$ N solution or a solution in which each millilitre is equivalent to 0.50 mg of chloride ion.

In the titration, chloride is precipitated as a white silver chloride (Eq. -1)

$$Ag^+ + Cl^- = AgCl \text{ (white precipitate)} \qquad (1)$$

The end point of titration cannot be detected visually unless an indicator capable of demonstrating the presence of excess Ag^+ is present. The indicator normally used is potassium chromate, which supplies chromate ions. As the concentration of Cl^- ions becomes exhausted, the silver ion concentration increases and a reddish brown precipitate of silver chromate is formed (Eq.- 2).

$$2Ag^+ + CrO_4^{2-} = Ag_2CrO_4 \text{ (reddish brown precipitate)} \qquad (2)$$

This is taken as evidence that all chloride has been precipitated. Since an excess Ag^+ is needed to produce a visible amount of Ag_2CrO_4, the indicator error is subtracted from all titrations.

Several precautions must be observed in the determination of chloride in order to ensure accuracy:

1. A uniform sample size must be used, preferably 100 ml (or 50 mL), so that ionic concentrations needed to indicate the end point will be constant.
2. The pH must be in the range of 7 to 8 because Ag^+ is precipitated as AgOH at high pH levels and the CrO_4^{2-} is converted to $Cr_2O_7^{2-}$ at low pH levels.
3. A definite amount of indicator must be used to provide a certain concentration of CrO_4^-, otherwise Ag_2CrO_4 may form too soon or not soon enough.

The indicator error or blank varies somewhat with the ability of individuals to detect a noticeable color change. The usual range is 0.2 to 0.4 mL of titrant. An error of 0.2 mL will be used in the class.

Reagents

- Potassium chromate indicator
- Silver nitrate solution (0.0141 N)

Apparatus

- Measuring cylinder (100 mL)	: 1 pc.
- Beaker	: 1 pc.
- Dropper	: 1 pc.
- Stirrer	: 1 pc.

Procedure

1. Take 50 mL of the sample in a beaker and add 5 drops (about 1 mL) of potassium chromate indicator to it.
2. Add standard (0.0141 N) silver nitrate solution to the sample from a burette, a few drops at a time, with constant stirring until the first permanent reddish color appears. This can be determined by comparison with distilled water blank. Record the mL of silver nitrate used.
3. If more than 7 or 8 mL of silver nitrate solution are required, the entire procedure should be repeated using a smaller sample diluted to 50 mL with distilled water.

Calculation

Chloride, Cl^- (mg/L) = (mL of $AgNO_3$ used - “error”/“blank”) x Multiplying Factor (MF) --- (1)

Where, MF = $\dfrac{\text{normality of } AgNO_3 \text{ x equivalent wt.of } Cl^- \text{ x} 1000}{\text{mL of sample taken}}$

If a diluted sample is used for chloride measurement,

Chloride (Cl^-) concentration of original sample (mg/L) = Cl^- measured in Eq.-1 x (DF) --- (2)

Where,

Dilution Factor (DF) = $\dfrac{\text{Volume (mL) of diluted water sample (mix)}}{\text{Volume (mL) of original sample in the mix}}$ ---- - (3)

Experiment No. 9

Determination of Total Iron in Water

Introduction

Iron is the second most abundant metal in the earth's crust, of which it accounts for about 5%. Iron is mainly present in water in two forms: either the soluble ferrous iron or the insoluble ferric iron. Iron exists in soils and minerals as insoluble ferric oxide/hydroxide and iron sulphide (pyrite). In some areas, it also occurs as ferrous carbonate, which is very slightly soluble. Since groundwater usually contains significant amounts of carbon dioxide, appreciable amounts of ferrous carbonate may be dissolved according to eq.-1.

$$FeCO_3 + CO_2 + H_2O = Fe^{2+} + 2HCO_3^- \qquad (1)$$

If reducing (anaerobic) conditions exist in groundwater environment, the insoluble ferric iron [Fe^{3+}] is reduced to more soluble ferrous iron [Fe^{2+}] and iron concentration in water increases. Water containing ferrous iron is clear and colorless because the iron is completely dissolved. When exposed to air it oxidized to ferric form and cause reddish brown precipitates in water. Manganese in water also shows similar behavior. Thus, iron, as well as manganese, creates serious problems in public water supplies, specially in the system which uses groundwater as the source of water supply.

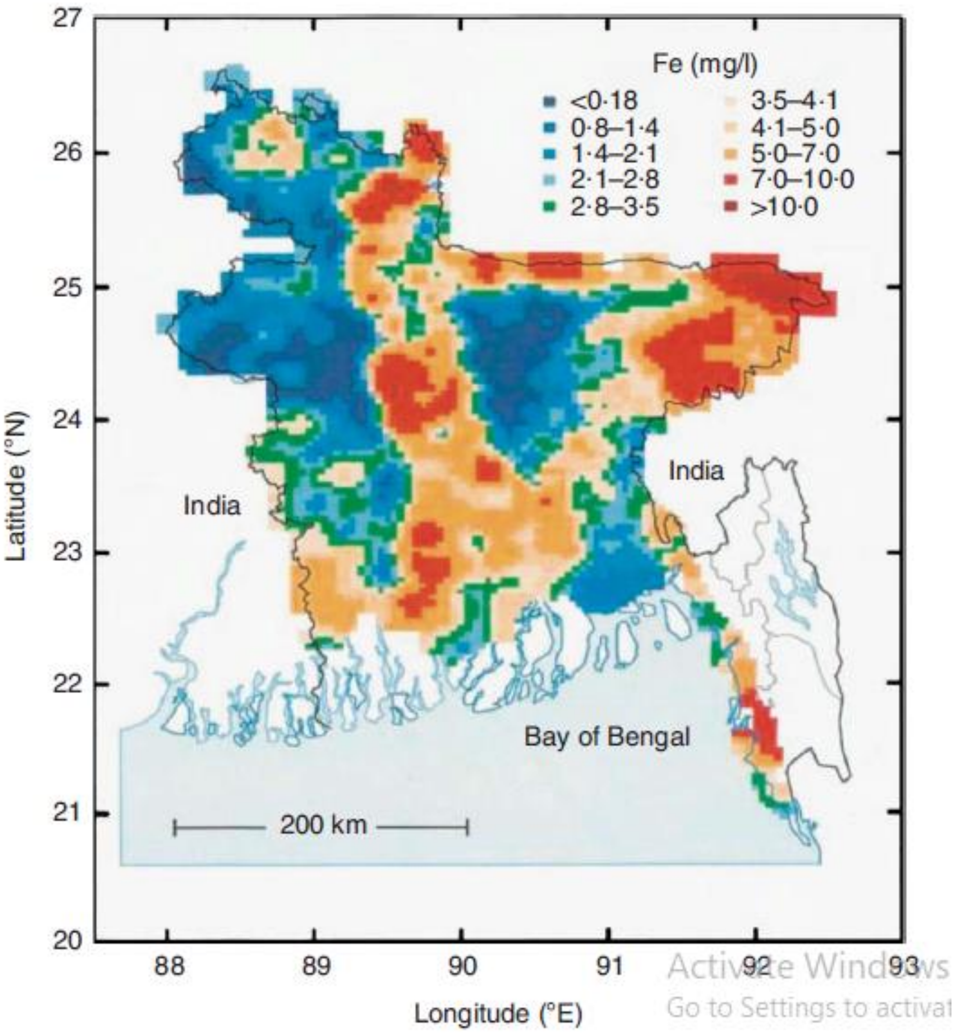


Fig-9.1: Iron concentration in groundwater in Bangladesh (British Geological Survey 2001)

Not only arsenic (As), but also iron (Fe) contamination in groundwater is now a vital problem in Bangladesh. According to the National Drinking Water Quality Survey (NDWQS) in 2009, approximately 70% and 61% of drinking water sources in Bangladesh, mainly groundwater, have been found to contain elevated level of iron (>0.3 mg/L) and manganese (>0.1 mg/L), respectively. According to the 2001 British Geological Survey (Figure 9.1), either side of the Brahmaputra basin, south of the Ganges basin and north-eastern haor regions are predominantly high groundwater iron areas, while the Barind and Madhupur tracts are the areas with predominantly low iron in groundwater. In several areas of Bangladesh, groundwater withdrawals are causing a large Fe contamination in groundwater levels during dry season.

Environmental Significance

As far as is known, human suffer no harmful effects from drinking waters containing iron and manganese. Such waters, when exposed to the air so that oxygen can enter, become turbid and highly unacceptable from the aesthetic viewpoint owing to the oxidation of iron and manganese to the Fe^{3+}, and Mn^{4+} states which form colloidal precipitates. The rates of oxidation are not rapid, and thus reduced forms can persist for some time in aerated waters. This is especially true when the pH is below 6 with iron oxidation and below 9 with manganese oxidation. The rates may be increased by the presence of certain inorganic catalysts through the action of microorganisms.

Both iron and manganese interfere with laundering operations, impart objectionable stains to plumbing fixtures and cause difficulties in distribution systems by supporting growths of iron bacteria. Iron also imparts a taste to water, which is detectable at very low concentrations. According to Bangladesh Environment Conservation Rules (1997), drinking water standard for iron is 0.3 - 1.0 mg/L.

Theory on Experimental Method

Ferric iron is seldom found in true solution in natural waters, unless they are highly acidic, because of the formation of insoluble ferric hydroxides. Ferrous iron is more likely to be found in true solution, although it is easily oxidized to the ferric state and precipitated in alkaline waters as ferric hydroxide.

Since some iron may exist as iron hydroxide precipitates, therefore it is necessary to bring precipitated form(s) of iron back in to solution before oxidizing total iron content in water. For this hydrochloric acid is added to the test sample to dissolve the insoluble ferric forms.

For determination of total iron by the following procedure, it must be ensured that all iron exists in ferric form (Fe^{3+}). This is most readily accomplished by using potassium permanganate, an oxidizing agent.

$$5Fe^{2+} + MnO_4^- + 8H^+ = 5Fe^{3+} + Mn^{2+} + 4H_2O \qquad (2)$$

Ferric iron is determined by producing a red-colored iron compound, ferric thiocyanate, by the addition of potassium thiocyanate (Eq.-3).

$$Fe^{3+} + 3\,KCNS = \underset{\text{(red)}}{Fe(CNS)_3} + 3\,K^+ \qquad (3)$$

The quantity of ferric iron is determined by comparison with the red color produced by standard iron solutions.

Reagents

- Hydrochloric acid
- Potassium permanganate solution
- Potassium thiocyanate solution
- Standard iron solution

Apparatus

- Nessler tube : 2 pc.
- Measuring cylinder : 1 pc.
- Dropper : 1 pc.

Procedure

1. Place 100 mL of the water sample in a Nessler.
2. Add 5 mL of dilute hydrochloric acid
3. Add two drops of potassium permanganate solution
4. Add 5 mL of potassium thiocyanate solution. The solution would turn brown if iron is present.
5. Compare the brown color formed with the standard prepared as follows:
    a. Add 100 mL of distilled water in a Nessler tube
    b. Add 5 mL of the dilute hydrochloric acid.
    c. Add two drops of potassium permanganate solution.
    d. Add 5 mL of potassium thiocyanate solution.
    e. Add 0.2 mL at a time of the standard iron solution until the color of the standard and the sample match.

Calculation

Total iron concentration of test sample (mg/L) =

$$\frac{\text{Standard iron solution added in dilstilled water (mL) x Conc. of standard iron solution (mg/L)}}{\text{mL of sample taken}}$$

*For diluted sample multiply the obtained result with dilution factor

Experiment No. 10

Estimation of Organic Pollution Load through Determination of BOD and COD

Introduction

One of the most important water quality parameters for natural water bodies is the amount of dissolved oxygen (DO) present in the water. Biodegradable organic substances, discharged into water bodies (pond, lake, canal, khal, river, ocean) with wastewater/effluent (domestic and industrial), get oxidized in the receiving body of water (by naturally present microorganisms) reducing the amount of available DO. As DO drops, fish and other aquatic life are threatened, and in extreme cases, killed.

There are several measures to estimate pollution load from biodegradable organic substances. The most common measures are:

(A) Biochemical Oxygen Demand (BOD); and

(B) Chemical Oxygen Demand (COD).

(A) Determination of Biochemical Oxygen Demand (BOD)

When biodegradable organic matter/waste is released into a water body, microorganisms (especially bacteria) feed on the wastes, breaking it down to simpler organic and inorganic substances. When this decomposition takes place in an aerobic environment (i.e., in the presence of oxygen; Eq. 1), it produces non-objectionable, stable end products (e.g., CO_2, SO_4, PO_4, and NO_3) and in the process draws down the dissolved oxygen (DO) of water.

$$\text{Organic matter} + O_2 \xrightarrow[\text{(aerobic bacteria)}]{} CO_2 + H_2O + \text{New cells} + \text{Stable products} \quad \text{-----(1)}$$

When insufficient oxygen is available or when oxygen is exhausted by the aerobic decomposition of wastes, then different set of microorganisms carry out the decomposition under anaerobic condition (Eq. 2) producing highly objectionable products including H_2S, NH_3, and CH_4.

$$\text{Organic matter} \xrightarrow[\text{(anaerobic bacteria)}]{} CO_2 + CH_4 + \text{New cells} + \text{Unstable products} \qquad (2)$$

The amount of oxygen required by micro-organisms to oxidize organic wastes aerobically is called *biochemical oxygen demand (BOD)*; usually expressed in mg of oxygen required per liter of water/wastewater (mg/L).

According to Bangladesh Environment Conservation Rules (1997), drinking water standard for biochemical oxygen demand (BOD_5) is 0.2 mg/L (at 20^{0}C) and standards for BOD_5 in industrial wastewater/effluent discharge is as followings:

BOD_5 at 20^{0}C	Discharge into Inland Surface Water	Public Sewerage system connected to treatment at second stage	Discharge into Irrigated Land
	50	250	100

Modeling of BOD

It is often assumed that the rate of decomposition of organic waste is proportional to the amount of waste available. If L_t represents the amount of oxygen demand left after time *t*, then, assuming a first order reaction, we can write,

$$dL_t/dt = -k\,L_t \qquad (6)$$

Where, *k* = BOD reaction rate constant (time^{-1}).

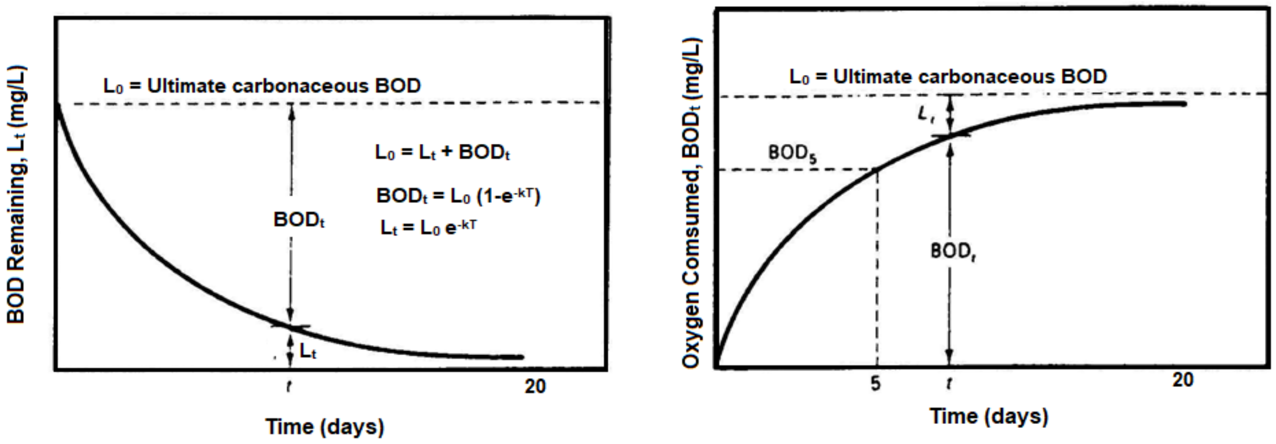
Integrating, we get, $L_t = L_o e^{-kt}$ (7)

Where, L_o= the ultimate carbonaceous oxygen demand, which is the total amount of oxygen required by microorganisms to oxidize the carbonaceous portion of the waste.

Now, $L_o = BOD_t + L_t$ (8)

After combining, we get, $BOD_t = L_o (1 - e^{-kt})$ (9)

Fig.-10.1(a) and 10.1(b) shows the graphical representations of Eq. (7) and Eq. (9), respectively.



. (a) (b)

Fig.- 10.1: Idealized carbonaceous oxygen demand: (a) the BOD remaining as a function of time, and (b) the oxygen consumed or the biochemical oxygen demand as a function of time.

Besides the oxidation of carbonaceous organic matter, a significant additional demand may be caused by the oxidation of nitrogenous compounds. To distinguish this *nitrogenous biochemical oxygen demand* from the *carbonaceous biochemical oxygen demand*, the two are sometimes referred to as NBOD and CBOD, respectively. Fig.- 2 illustrates the two demands for a typical municipal waste. Note that the NBOD does not begin to exert itself for at least 5 to 8 days, so most 5-day BOD tests are not affected by the nitrification process

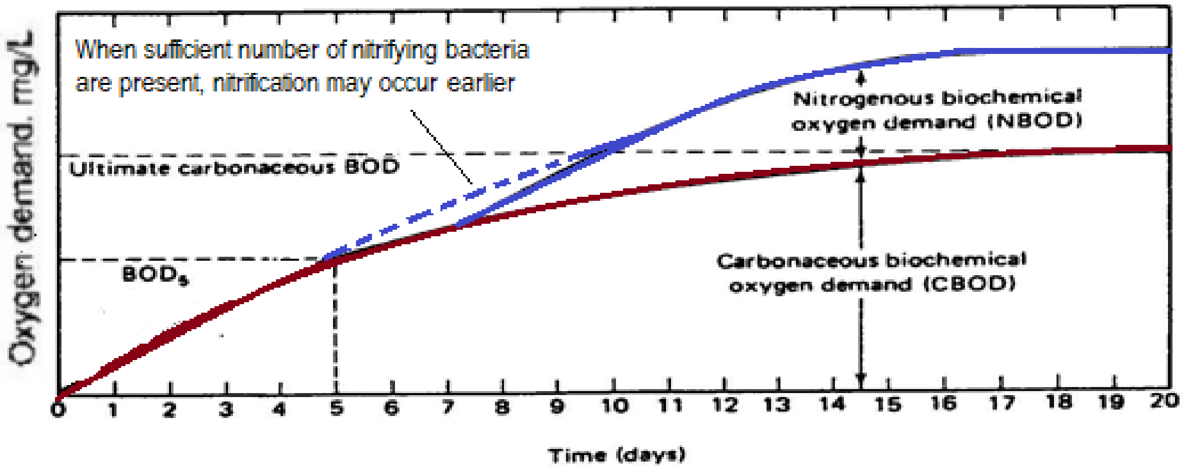


Fig.-10.2: Illustration of Carbonaceous and Nitrogenous Biochemical Oxygen Demand

Theory on Experimental Method

The oxygen demand for decomposition of biodegradable organic matter is a function of time. It is a standard practice to measure and report the oxygen demand over a 5-day period, realizing that the ultimate oxygen demand (for complete degradation of organic matter) is considerably higher and would take a much longer time to determine in the laboratory.

The 5-day BOD or BOD_5 is the amount of oxygen consumed by microorganisms during the first 5 days of biodegradation. The test is usually carried out at 20°C. In its simplest form, a BOD_5 test would involve putting a sample of waste/wastewater into a bottle, measuring the dissolved oxygen(DO) of the sample at the beginning of the test and again at the end of five days. The difference in DO would be the BOD_5 of the water /wastewater. Light must be kept out of the bottle to keep algae from adding oxygen by photosynthesis and the stopper is used to keep air from replenishing DO from outside.

Dilution of wastewater:

Since the oxygen demand of typical wastewater/effluent is several hundred mg/L, and since the saturated value of DO for water at 20^oC is only 9.1 mg/L, it is usually necessary to dilute the sample to keep final DO above zero. If during the five days of experiment, the DO drops to zero, then the test is invalid since more oxygen would have been removed had more been available.

The five-day BOD of a diluted sample is given by

$$BOD_5 = [DO_i - DO_f] \times D.F. \qquad (3)$$

where,

$$DF = \text{Dilution Factor} = \frac{\text{volume of (wastewater , } V_w + \text{dilution water, } V_d)}{\text{volume of wastewater, } V_w} = \frac{V_m}{V_w}$$

here, $V_m = V_w + V_d$

A wide variety of dilution waters have been used for BOD work. Natural surface waters have been used, but they suffer from a number of disadvantages including variable BOD, variable micro-organism population, and variable mineral content. Tap water has also been used, but it also suffers from many of the limitations of surface water plus the possibility of toxicity from chlorine residuals. Synthetic dilution water prepared by adding different salts to distilled water has been found to be the most ideal for use as dilution water.

Seeding of wastewater:

In some cases, it becomes necessary to *seed* the dilution water with microorganisms to ensure that there is an adequate bacterial population to carry out the biodegradation. In such cases, two sets of BOD bottles must be prepared, one for just the seeded dilution water (called the "blank") and the other for the mixture of wastewater and dilution water. The changes in DO in both bottles are measured. The oxygen demand of the wastewater (BOD_w) is then determined from the following relationship:

$$BOD_mV_m = BOD_wV_w + BOD_dV_d \qquad (4)$$

Where, BOD_m, BOD_w, & BOD_d represent the BOD of the mixture of wastewater and dilution water, BOD of the wastewater &BOD of the dilution water alone respectively.

Rearranging and simplifying equation-4, BOD_w can be obtained –

$$BOD_w = BOD_m \times DF - BOD_d \times (DF-1) \qquad (5)$$

Different types of microorganisms have been used for "seeding" purpose. Experience has shown that domestic wastewater, particularly from combined sewer systems provides a well-balanced population of mixed micro-organisms. Usually 2 mL of wastewater per liter of diluted water is sufficient. Some river waters are also satisfactory, but care must be taken to avoid using waters that contain algae or nitrifying bacteria in significant amount. The dilution water should always be "seeded" to ensure a uniform population of micro-organisms.

Aeration of wastewater:

Aeration is a treatment method to increase dissolved oxygen concentration in water /wastewater. For organic waste loading analysis aeration is used serve the following two purposes:

1. *For determination of BOD:*

Polluted water and wastewater often contain very little DO. For determination of BOD, the initial DO of the sample needs to be raised, so that at the end of 5-day incubation period, some DO remains in water (to be measured). This is usually carried out through "aeration" (i.e. flow of air) of the dilution water, which increases DO of the dilution water by allowing oxygen present in air to dissolve into it.

2. *Aeration as a means to reduce BOD:*

Aeration is a common technique in wastewater treatment for reduction of BOD. Through aeration (i.e., supply of excess air into the wastewater), the decomposition/degradation of organic matter present in wastewater (by bacteria) is accelerated, thereby reducing its BOD. Aeration ensures activity of aerobic bacteria, which has been identified to metabolize organic waste load in the water faster than the anaerobic bacteria. In this class, the possible effect of simple aeration on BOD would be assessed.

Theory of DO Determination

Dissolved oxygen (DO) determination is done by *Winkler Method*. The reactions involved in the various steps are presented below:

Manganous sulfate reacts with potassium hydroxide in the alkaline potassium iodide solution to produce a white precipitate of manganous hydroxide.

$$MnSO_4 + 2KOH = \underset{\text{white ppt}}{Mn(OH)_2\downarrow} + K_2SO_4 \qquad (10)$$

If the white precipitate is obtained, there was no dissolved oxygen in the sample and there is no need to proceed further. A brown precipitate shows that oxygen is present and reacted with the manganous hydroxide and forms manganic basic oxide.

$$2Mn(OH)_2 + O_2 = \underset{\text{brown ppt}}{2MnO(OH)_2\downarrow} \qquad (11)$$

Upon the addition of (sulfuric) acid, this precipitate is dissolved, forming manganic sulfate:

$$MnO(OH)_2 + 2H_2SO_4 = Mn(SO_4)_2 + 3H_2O \qquad (12)$$

This compound immediately reacts with potassium iodide, liberating iodine and resulting in the typical iodine (blue) coloration of the water.

$$Mn(SO_4) + 2KI = MnSO_4 + K_2SO_4 + I_2 \qquad (13)$$

The quantity of iodine liberated by these reactions is equivalent to the quantity of oxygen present in the sample. The quantity of iodine is determined by titrating a portion of the solution with a standard solution of sodium thiosulfate solution. The end point of titration is indicated by the disappearance of blue color produced by the reaction between iodine and starch (which is added as an indicator in the titration).

$$2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI \qquad (14)$$

Reagents

- Manganous sulfate solution
- Alkaline potassium iodide solution
- 0.025N sodium thiosulfate
- Starch solution (indicator)
- Concentrated sulfuric acid.

Apparatus

- BOD bottle : 6 nos.
- Beaker (250 ml) : 1 no.
- Measuring cylinder : 1 no.
- Dropper : 1 no.
- Stirrer : 1 no.

Procedure: *(Determination of DO)*

BOD_5 is to be determined for two types of sample –

i) Wastewater being aerated for a specified period of time and
ii) Non-aerated wastewater. Both samples may be diluted, if needed.

For each type of wastewater (aerated and non-aerated) fill two BOD bottles with sample (or diluted sample); the bottles should be completely filled. Determine initial DO (DO_i) in one bottle for both type of wastewater immediately after filling with sample (or diluted sample). Keep the other set of bottles in dark at 20^oC and after particular days (usually 5-days) determine DO (DO_f) in the samples (or diluted samples). Dissolved oxygen (DO) is determined according to the following procedure:

1. Add 1 mL of manganous sulfate solution to the BOD bottle by means of pipette, dipping in end of the pipette just below the surface of the water.
2. Add 1 mL of alkaline potassium iodide solution to the BOD bottle in a similar manner.
3. Insert the stopper and mix by inverting the bottle several times.
4. Allow the “precipitates” to settle halfway and mix again.
5. Again allow the “precipitates” to settle halfway.
6. Add 1 mL of concentrated sulfuric acid. Immediately insert the stopper and mix as before.
7. Allow the solution to stand at least 5 minutes.
8. Withdraw 100 mL of solution into an Erlenmeyer flask and immediately add 0.025N sodium thiosulfate drop by drop from a burette until the yellow color almost disappears.
9. Add about 1 mL of starch solution and continue the addition of the thiosulfate solution until the blue color just disappears. Record the ml. of thiosulfate solution used (disregard any return of the blue color).

Calculation

Dissolved oxygen, DO (mg/L) = mL of 0.025N sodium thiosulfate added x MF

$$\text{Where, MF} = \frac{\text{normality of } N_2S_2O_3 \text{ x equivalent wt. of oxygen x } 1000}{\text{mL of sample taken}}$$

Calculate:

(a) BOD_5 of the non-aerated wastewater sample (domestic sewage, as received), according to Eq. 3, 4 or 5.
(b) BOD_5 of the aerated wastewater according to Eq. 3, 4 or 5.
(c) If this wastewater is discharged into a river at a rate of 1.15 cfs over a period of 16 hrs per day, estimate the daily BOD_5 load (in kg/day) discharged into the river.

(B) Determination of Chemical Oxygen Demand (COD)

All organic substances are not degradable by microorganism (biologically inert substances). If waste/ wastewater having considerable amount of biologically inert organic matter is tested for BOD, the result would not represent the actual organic loading of the waste/ wastewater. Hence, the chemical oxygen demand (COD) test allows measurement of oxygen demand of the waste in terms of the total quantity of oxygen required for oxidation of the waste to carbon dioxide and water. The test is based on the fact that all organic compounds, with a few exceptions, can be oxidized by the action of strong oxidizing agents under acid conditions.

$$\text{Organic matter} + \text{Chemical Oxidizing agent} = CO_2 + H_2O \qquad (1)$$

The reaction in eq.-1 involves conversion of organic matter to carbon dioxide and water regardless of the biological assimilability of the substance. For example, glucose and lignin (biologically inert substance) are both oxidized completely by the chemical oxidant. As a result, COD values are greater than BOD values, especially when biologically resistant organic forms are present.

Thus one of the chief limitations of COD test is its inability to differentiate between biodegradable and non-biodegradable organic matter. In addition, it does not provide any evidence of the rate at which the biologically active material would be stabilized under conditions that exist in nature.

The major advantage of COD test is the short time required for evaluation. The determination can be made in about 3 hours rather than the 5-days required for the measurement of BOD. For this reason, it is used as a substitute for the BOD test in many instances.

Environmental Significance

COD is often measured as a rapid indicator of organic pollutant in water. It is normally measured in both municipal and industrial wastewater treatment plants and gives an indication of the efficiency of the treatment process. COD has further applications in power plant operations, chemical manufacturing, commercial laundries, pulp & paper mills, environmental studies and general education.

According to Bangladesh Environment Conservation Rules (1997), drinking water standard for COD is 4.0 mg/L. For wastewater effluent discharges, allowable concentration of COD varies from 200- 400 mg/L depending on discharge point of the effluent e.g., inland water, irrigation land, public sewer respectively (see appendix).

Theory on Experimental Method

Potassium dichromate or potassium permanganate is usually used as the oxidizing agent in the determination of COD. In this class potassium permanganate would be used in the determination of COD. Potassium permanganate is selective in the reaction and attacks the carbonaceous and not the nitrogenous matter.

In any method of measuring COD, an excess of oxidizing agent must be present to ensure that all organic matter is oxidized as completely as possible within the power of the reagent. This requires that a reasonable excess be present in all samples. It is necessary, therefore, to measure the excess in some manner so that the actual amount can be determined. For this purpose, a solution of a reducing agent (e.g., ammonium oxalate) is usually used.

Reagents

- Diluted sulphuric acid solution
- Standard potassium permanganate solution
- Standard Ammonium Oxalate solution
- COD reagent vial

Apparatus

- Beaker (250 mL) : 1 no
- Dropper : 1 no
- burette : 1 no
- Stirrer : 1 no
- Water bath : 1 no
- Burner : 1 no
- COD reactor : 1 no

Procedure (COD measurement using $KMnO_4$)

1. Pipette 100 mL of the sample into a 250 mL Erlenmeyer flask.
2. Add 10 mL of diluted sulfuric acid and 10 mL of standard $KMnO_4$ solution.
3. Heat the flask in a boiling water bath for exactly 30 minutes, keeping the water in the bath above the level of the solution in the flask. The heating enhances the rate of oxidation reaction in the flask.
4. If the solution becomes faintly colored, it means that most of the potassium permanganate has been utilized in the oxidation of organic matter. In such a case, repeat the above using a smaller sample diluted to 100 mL with distilled water.
5. After 30 minutes in the water bath, add 10 mL of standard ammonium oxalate $[(NH_4)_2C_2O_4]$ solution into the flask. This 10 mL ammonium oxalate, which is a reducing agent, is just equivalent to the 10 mL potassium permanganate (oxidizing agent) added earlier. The excess of reducing agent $[(NH_4)_2C_2O_4]$ now remaining in the flask is just equivalent to the amount of the oxidizing agent ($KMnO_4$) used in the oxidation of organic matter.
6. The quantity of ammonium oxalate remaining in the flask is now determined by titration with standard potassium permanganate. Titrate the content of the flask while hot with standard potassium permanganate to the first pink coloration. Record the mL of potassium permanganate used.

Determination of COD using Potassium dichromate in COD reactor

In this method COD is measured by oxidizing organic wastes using $K_2Cr_2O_7$ as oxidizing agent. COD reagent vials contain premeasured solutions of sulfuric acids and potassium dichromate. The measurement procedure is as follows:

i) Turn on the COD reactor and set to 150°C.
ii) Shake the sample and add 2 mL of sample into COD vial (high range or low range) and replace the cap.
iii) Once the reactor is at the proper temperature, place the vial containing sample in the reactor and set the timer for two hours.
iv) After two hours remove the vial from the reactor and allow cooling to room temperature.
v) Set the spectrophotometer to the appropriate wavelength (620 ηm) and read the absorbance.

Calculations

COD (Using $KMnO_4$)

COD (mg/L) = mL of $KMnO_4$ added in Step-6 x MF

$$\text{Where, MF} = \frac{\text{normality of } KMnO_4 \text{ x equivalent wt. of oxygen} \times 1000}{\text{mL of sample used}}$$

(Normality of $KMnO_4$ used in the lab is 0.0125 N)

Calculate: for both $KMnO_4$ & $K_2Cr_2O_7$ oxidizing agent

(a) COD of the wastewater sample (domestic sewage, as received).

(b) COD of the same wastewater, but which has been aerated for a specified period of time

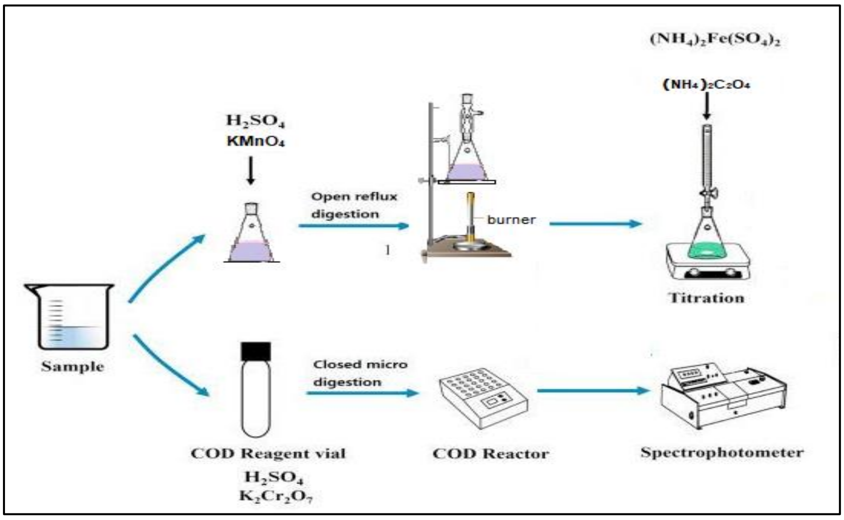


Fig-10.3: COD measurement using $KMnO_4$ & $K_2Cr_2O_7$ as oxidizing agent

Experiment No. 11

Chemical Coagulation of Water: Alum Coagulation

Introduction

All waters, especially surface waters contain dissolved, suspended particles and/or inorganic matter, as well as several biological organisms, such as bacteria, algae or viruses. This material must be removed because it can affect the water quality that can cause turbidity and color. In general, suspended particles are simply removed by conventional physical treatment like sedimentation and filtration. Dissolved molecules cannot be removed by conventional physical treatment. But colloidal elements are difficult to separate from water because the particles will not settle by gravity and are so small that they pass through the pores of most common filtration media. Table-1 shows estimated time (using Stoke's law) required to settle 1 m through water for different types of particles. Thus, the removal of colloids is the main objective and the most difficult aspect in conventional water treatment.

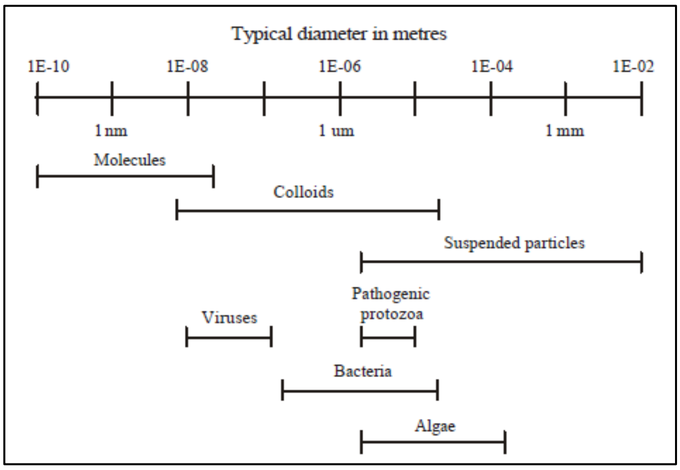


Fig-11.1: Size range of particles of concern in water treatment

Table-1: Time required for settling 1 m through water according to particle size

Particle diameter (cm)	Settling speed (cm/sec)	Time required to settle 1 m
0.05 (Sand)	10.4	10 Sec
0.01 (Fine Sand)	0.42	4 Minutes
0.001 (Silt, Clay)	0.0042	7 Hours
0.0001 (Bacteria)	0.000042	28 Days

There are two types of colloids: hydrophilic colloids and hydrophobic colloids. Hydrophobic colloids, including clay and non-hydrated metal oxides, are unstable. The colloids are easily destabilized. Hydrophilic colloids like soap- are stable. When these colloids are mixed with water, they form colloidal solutions that are not easily destabilized. Most suspended solids smaller than 0.1 mm found in waters carry negative electrostatic charges. Since the particles have similar negative electrical charges and electrical forces to keep the individual particles separate, the colloids stay in suspension as small particles.

To remove colloids, small particles have to be destabilized first and then they will form larger and heavier flocs which can be removed by conventional physical treatment. Chemical agents (coagulants) are induced in water and the positive electric charge of the coagulant reduces the negative charge of the colloids. Thus the process of destroying the stabilizing forces in colloidal particles is referred as chemical coagulation and the aggregation of colloids into larger unit is defined as flocculation. Chemical coagulation-flocculation is a treatment method widely used for removal of small sized and colloidal impurities from water.

Selection of correct chemical dose is very important for this treatment process. At higher coagulant doses, "charge reversal" is possible which may result in re-suspension of the colloids. Hence optimum coagulant doses are determined through laboratory model tests where the water to be treated are subjected to a range of doses of a coagulant and the removal efficiencies are observed.

Environmental Significance

Besides efficient removal of turbidity and color, coagulation with alum and ferric chloride or ferric sulphate is also widely used for removal of heavy metal ions (e.g., lead, arsenic) from water. In this process heavy metal ions are primarily removed by adsorption (and subsequent precipitation) onto coagulated flocs of metal (either aluminum or iron) hydroxides. Coagulation with alum and ferric chloride/sulphate are being successfully used for removal of arsenic from water.

Mechanism of Coagulation Process

The chemical used for this purpose is called are called *coagulants*. The most common *coagulants* used in water and wastewater treatment are aluminum and ferric salts such as alum, ferric chloride and ferric sulfate. Alum (aluminum sulfate) is a good coagulant for water containing appreciable organic matter. Once dissolved in water, aluminum forms hydroxo-complexes and solids [e.g., $Al(OH)_3(s)$, $Al(OH)^{2+}$, $Al(OH)_2^+$, $Al(OH)_4^-$; [eqs.1-5] and as a result pH of water is lowered, especially if alkalinity of water is low. Theoretically, each mg/L of alum consumes approximately 0.50 mg/L (as $CaCO_3$) of alkalinity. For water with low alkalinity, this may result in significant reduction in pH that may interfere with formation of aluminum hydroxide flocs. If the alkalinity is insufficient, coagulant aids such as lime [$Ca(OH)_2$], soda ash (Na_2CO_3), activated silica and poly-electrolytes are used to provide the necessary alkalinity.

Iron coagulants (e.g., Ferric Chloride) can be operated over a wider pH range and are generally effective in removing turbidity and color from water. However, they are usually more costly.

$$Al_2(SO_4)_3 . 14H_2O \text{ (alum)} = 2\,Al^{3+} + 3\,SO_4^{2-} \quad (1)$$
$$Al^{3+} + 3\,H_2O = Al(OH)_3\,(s) + 3\,H^+ \quad (2)$$
$$Al^{3+} + H_2O = Al(OH)^{2+} + H^+ \quad (3)$$
$$Al^{3+} + H_2O = Al(OH)_2^{+} + 2H^+ \quad (4)$$
$$Al^{3+} + H_2O = Al(OH)_4^{-} + H^+ \quad (5)$$

Stability of Colloids

The magnitude of the zeta potential (Zp) is usually used to indicate colloidal particle stability. The higher the zeta potential, the greater are the repulsion forces between the colloidal particles and, therefore, the more stable is the colloidal suspension. Low Zp indicates relatively unstable systems, i.e. particles tend to aggregate.

In stable condition, negatively charged colloid is surrounded by a bound layer of water in which ions of opposite charge (counter-ion, i,e.+ve) drawn from the bulk solution with greater concentration near surface of the colloid. This arrangement produces a net charge that is strongest at the bound layer and decreases exponentially with distance from the colloid. When two colloids come in close proximity there are two forces acting on them.

(i)Electrostatic Repulsion:

An electrostatic potential is created by the counter ions surrounding each colloid. As two similarly charged particles approach each other, their diffuse counter-ion atmospheres begin to interfere and cause the particles to be repulsed.

(ii) The Vander Waals' forces of Attraction:

The second force, an attractive force called the Vander Waals force, support contact. This force is inversely proportional to the sixth power of the distance between the particles and also decays exponentially with distance. It decreases more rapidly than electrostatic potential, but is a stronger force at close distances.

The sum of the two forces as they relate to one colloid in close proximity to another is repulsive at greater distances and becomes attractive only after passing through a maximum net repulsive force, called the 'energy barrier' at some distance between colloids. Once the force becomes attractive, contact between the particles takes place.

Destabilization of Colloidal Dispersion

Particle destabilization can be achieved by four mechanisms:

(a) Change characteristics of medium- Compression of double layer.

The ionization of alum in water produces sulfate (SO_4^{2-}) and aluminium (Al^{3+}) ions. Aluminium ions reacts immediately with water to form a variety of hydroxo-complexes (see Eq. 1-5). These ions become part of the ionic cloud surrounding the colloids. A high concentration these multivalent cations reduces the depth of “ionic layer” surrounding each colloid by compressing the layer toward the surface of the colloid. If the ionic layer is sufficiently compressed, then Vander Waals’ attractive forces become predominant and the net force becomes attractive (see Fig.11.2).

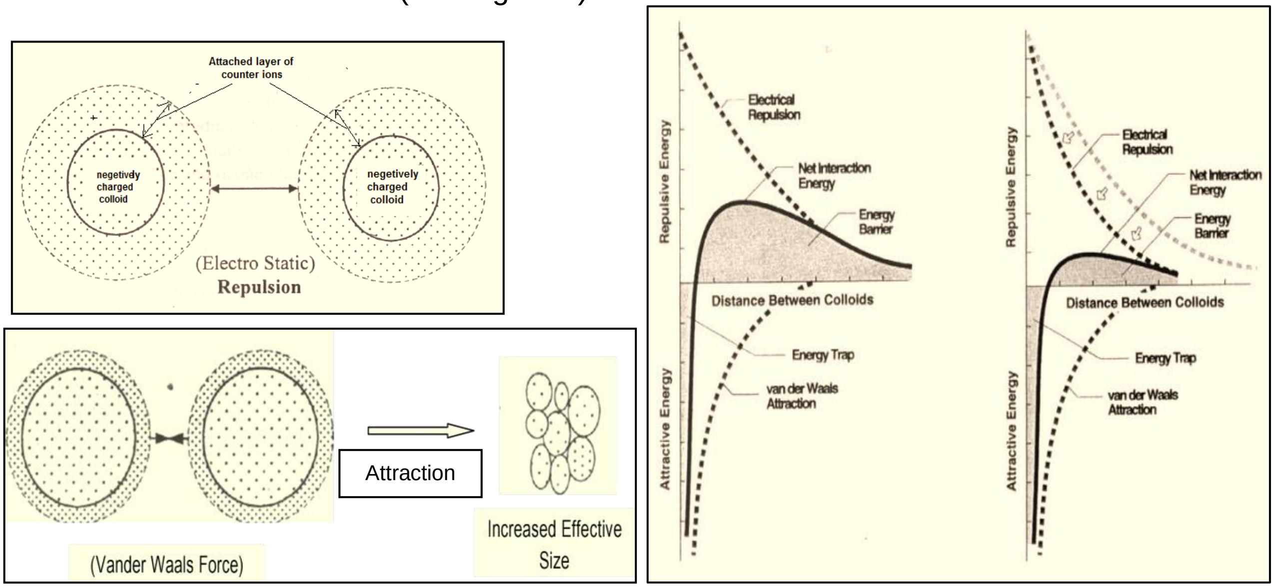


Fig- 11.2: Double layer compression of colloidal particles

(b) Change characteristics of colloid particles-Adsorption and charge neutralization.

Some chemical species are capable of being adsorbed at the surface of colloidal particles. Once the surface charge is neutralized, the ionic cloud dissipates, contact among particles occurs freely. Reduction of surface charge by adsorption is a much different mechanism than reduction by double layer compression. At higher coagulant doses, “charge reversal” (i.e., particles becomes positively charged due to adsorption of positively charged ions, and thus repulsing each other) is possible which may result in re-suspension of the colloids.

(c) Enmeshment in a precipitate: Sweep Floc

If certain metal salts are added to water or wastewater in sufficient amounts, rapid formation of precipitates will occur. Colloids may serve as condensation nuclei for these precipitates or may become enmeshed as the precipitates settle. Removal of colloids in this manner is frequently referred to as sweep-floc coagulation. At low colloid concentrations a large excess of coagulant is required to produce a large amount of precipitate that will enmesh the relatively few colloidal particles as it settles. At high colloid concentrations, coagulation will occur at a lower chemical dosage because the colloids serve as nuclei to enhance precipitate formation.

(d) Adsorption and inter-particle bridging

Natural organic polymers (e.g., starch, cellulose) and some synthetic organic polymeric compounds have large molecular size. They may be anionic, cationic or non-ionic. Polymer molecule gets attached to a colloidal particle at one or more sites due to columbic attraction (if the polymer and particle are opposite charge) or due to ion- exchange, Vander waals forces (if the polymer and particle are similar charge). The ‘’tail’’ of the adsorbed polymer will extend out into the bulk solution and can become attached to vacant site on the surface of another particle to form a chemical bridge The bridging action results in the formation of a floc particle having favorable settling characteristics.

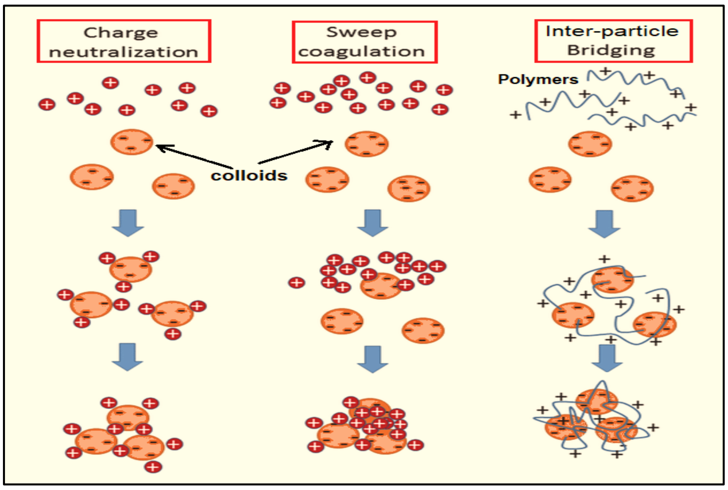


Fig- 11.3: Charge neutralization, Sweep floc & Inter-particle bridging

Experimental Setup for Determination of Coagulation Dose

In the class "jar test" will be used to determine optimum coagulant dose for removal of suspended solids from water.

Reagents

- Standard Alum solution.

Apparatus

- Jar test apparatus
- pH meter
- Turbidity meter
- Glass beakers (1000 mL- 6 nos)

Procedure

1. Determine pH and turbidity of the water to be treated.
2. Fill six 1-litre capacity beakers each with 500 mL of water to be treated.
3. Add required (as instructed in the Lab) coagulant dose (standard alum solution) to each beaker.
4. Stir the beakers with glass stirrer for one minute to mix the coagulant thoroughly in water.
5. Place six beakers on a "jar test" apparatus (Fig. 11.4). Subject the samples to one minute of rapid mixing (about 40 rpm) followed by 14 minutes of slow mixing (about 25 rpm).

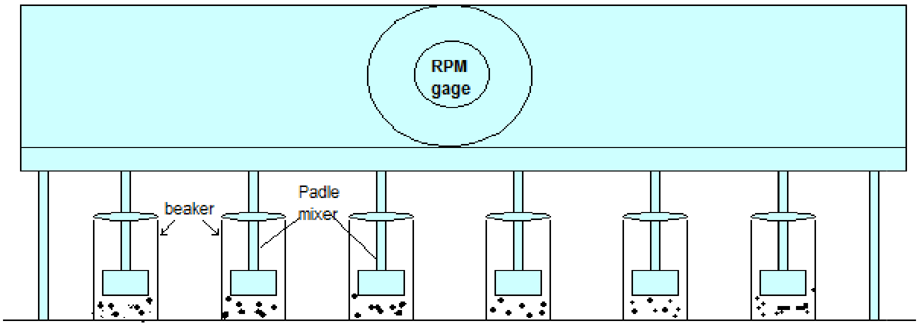


Fig- 11.4: Jar test apparatus

6. Allow the flocs to settle down for about 15 minutes. Observe the characteristics of the flocs and the settling rates. Also observe which of the 6 beakers is most clear.
7. Collect the supernatant from each beaker and measure pH and turbidity of each sample.
8. Plot pH versus alum dose in a graph paper and observe effect of alum dose on pH.
9. Plot turbidity (NTU) versus the coagulant (alum) dose (mg/L) in a graph paper. Determine optimum dose of alum from this plot.

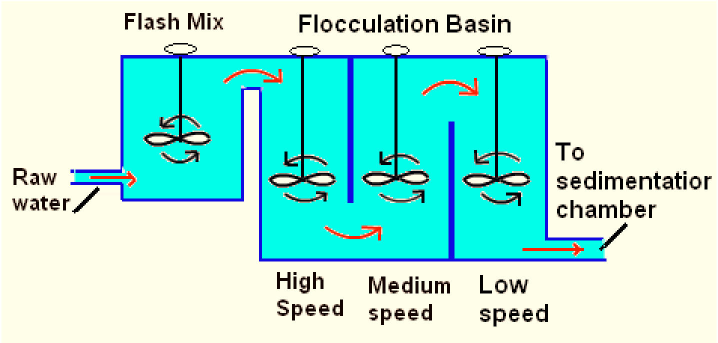


Fig- 11.5: Typical arrangement in a water treatment plant for removal of suspended solids (including colloidal particles) from water by coagulation-flocculation and subsequent sedimentation process

Experiment No. 12

Determination of Residual Chlorine and Chlorine Demand: Break Point Chlorination

Introduction

Chlorination of public water supplies and polluted waters serves primarily to destroy or de-activate disease-producing microorganisms. Disinfection with chlorine is widely practised. Chlorination may produce some adverse effects including taste and odor problem. In recent years, chlorination has been found to produce trihalomethanes (THMs) and other organics of health concern (THMs are suspected human carcinogens). Thus, use of alternative disinfectants, such as chlorine dioxide and ozone that do not cause this particular problem, is increasing.

Theory of Chlorination

Disinfectant capabilities of chlorine depend on its chemical form in water, which in turn is dependent on pH, temperature, organic content of water, and other water quality factors. Chlorine is used in the form of free chlorine [e.g., chlorine gas] or as hypochlorites [e.g., NaOCl and $Ca(OCl)_2$]. Chlorine applied to water either as free chlorine or hypochlorite initially undergoes hydrolysis to form free chlorine consisting of aqueous molecular chlorine, hypochlorous acid and hypochlorite ion.

$Cl_2 + H_2O = HOCl + H^+ + Cl^-$ (1)
$NaOCl = OCl^- + Na^+$ (2)
$Ca(OCl)_2 = 2OCl^- + Ca^{2+}$ (3)
$HOCl = H^+ + OCl^-$ (4)

The relative proportion of these “free chlorine” forms is pH and temperature-dependent. Fig.-12.1 shows a distribution diagram for the various chlorine species (Cl_2, HOCl, OCl^-) over a

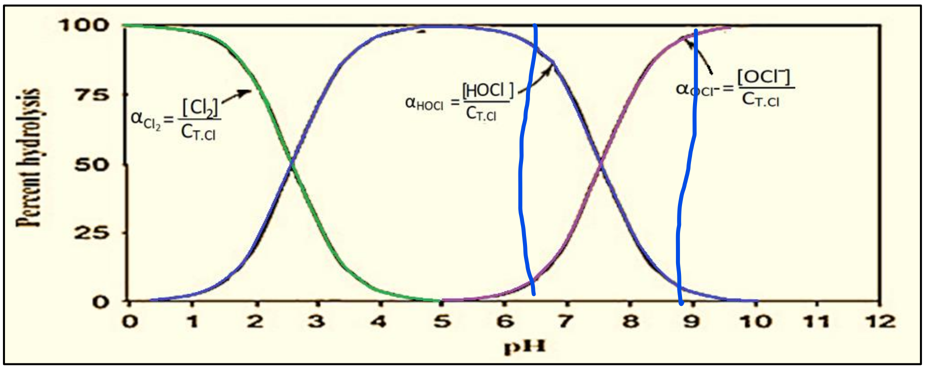


Fig.- 12.1. Distribution diagram for chlorine species at 25°C (Total Chlorine=10^{-3} M)

Temperature ↑,Alkalinity↑,pH ↑ HOCl ↓

The diagram above shows that Cl_2 can be significant only at low pH values (below pH 2); while HOCl is dominant between pH 3 and 6. Between pH 6 and 9, the relative fraction of HOCl decreases, while the corresponding fraction of OCl^- increases. The dissociation of HOCl is also temperature dependent. The effect of temperature is such that at a given pH, the fraction of HOCl will be lower at higher temperatures. Generally, the disinfectant capabilities of HOCl are greater than that of OCl^-, especially at short contact times (Montgomery, 1985).

Reactions of Chlorine with Impurities in Water

Reactions with Ammonia:

Free chlorine reacts readily with ammonia and certain nitrogenous compounds to form what are collectively known as "combined chlorine". The inorganic chloramines consist of three species: monochloramine (NH_2Cl), dichloramine ($NHCl_2$) and trichloramine or nitrogen trichloride (NCl_3). The presence and concentrations of these combined forms depend on a number of factors including the ratio of chlorine to ammonia-nitrogen, chlorine dose, temperature, pH and alkalinity.

$$NH_3 + HOCl = NH_2Cl + H_2O \quad (5)$$
$$NH_2Cl + HOCl = NHCl_2 + H_2O \quad (6)$$
$$NHCl_2 + HOCl = NCl_3 + H_2O \quad (7)$$

In addition to chlorinating ammonia, chlorine also reacts to oxidize ammonia to chlorine-free products (e.g., nitrogen gas and nitrate) as shown below.

$$3\,Cl_2 + 2\,NH_3 = N_2(g) + 6\,H^+ + 6\,Cl^- \quad (8)$$
$$4\,Cl_2 + NH_3 + 3\,H_2O = 8\,Cl^- + NO_3^- + 9\,H^+ \quad (9)$$

The mono- and dichloramines have significant disinfecting power and are therefore of interest in the measurement of chlorine residuals. Combined chlorine in water supplies may be formed in the treatment of raw waters containing ammonia; chlorinated wastewater effluents, as well as certain chlorinated industrial effluents normally contain only combined chlorine.

Reactions with Other Impurities:

Chlorine combines with various reducing agents and organic compounds thus increasing the chlorine demand which must be satisfied before chlorine is available to accomplish disinfection.

Fe^{2+}, Mn^{2+}, NO_2^-, and H_2S are examples of inorganic reducing agents present in water supplies that will react with chlorine. Chlorine can react with phenols to produce mono-, di-, or trichlorophenols, which can impart tastes and odor to water. Chlorine also reacts with humic substances present in water to form trihalomethanes (THMs, e.g., chloroform, bromoform, etc.) which are suspected human carcinogens (Note: According to USEPA, maximum allowable level of THMs in drinking water is 100 µg/L).

Break Point Chlorination

If chlorine is added to water containing reducing agents and ammonia (either naturally present or added to water to produce combined chlorine), a hump-shaped breakpoint curve is produced as shown in Fig. – 12.2. The different segment of the curve is described below:

a. If the water is free of ammonia and other compounds that may react with chlorine, the application of chlorine will yield free available chlorine residual of same concentration. This is denoted by the "no demand line" or the "zero demand line".
b. Chlorine first reacts with reducing agents such as H_2S, Fe^{2+}, Mn^{2+} and develops no measurable residual as shown by the portion of the curve from Origin up to point A.
c. Addition of chlorine beyond point A results in forming mainly mono- and di-chloramines. With mole ratios of chlorine to ammonia up to 1:1 [i.e., Cl_2:NH_3-N = 1:1], both mono and di-chloramines are formed. Chloramines thus formed are effective disinfectants and are shown as combined available chlorine residual in Fig. – 12.2 (from A to B).
d. Further increase in the mole ratio of chlorine to ammonia result in formation of some trichloramine and oxidation of part of ammonia to N_2 and NO_3^- (see Eq. 8, 9). These reactions are essentially complete when 1.5 mole of chlorine has been added for each mole of ammonia nitrogen originally present in water [i.e., Cl_2:NH_3-N = 1.5:1]. This is represented by the portion of the curve from B to C (see Fig.- 2).

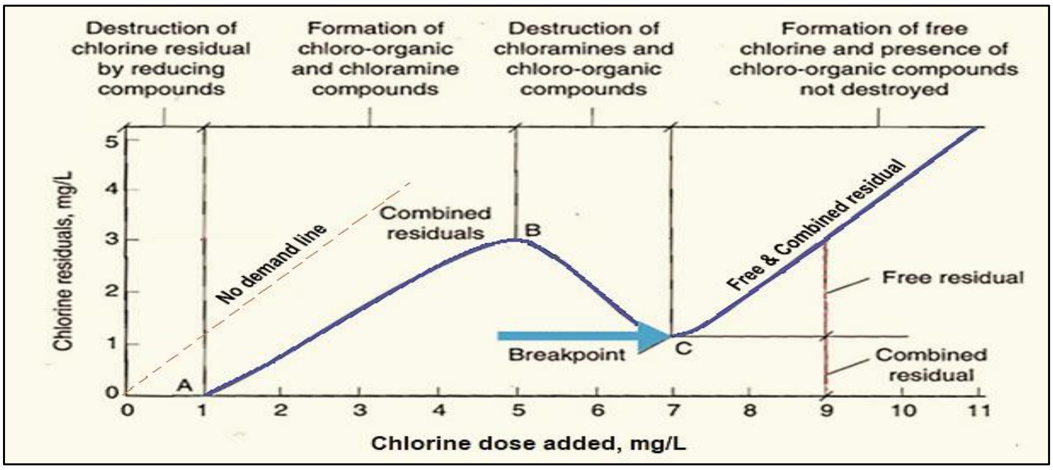


Fig – 12.2: Generalized curve obtained during breakpoint chlorination of a water sample containing ammonia

e. Addition of chlorine beyond point C would produce free chlorine residuals and is referred to as "*breakpoint chlorination*". In other words, chlorination of water to the extent that all ammonia is converted to N_2 or higher oxidation state is referred to as "*breakpoint chlorination*".

f. The difference between the amount of chlorine added to the water and the amount of residual chlorine (i.e., free and combined available chlorine remaining) at the end of a specified contact period is termed as "*chlorine demand*".

Environmental Significance

Chlorine residuals determination is used to control chlorination of domestic and industrial wastewaters. Active chlorine (free and combined) should be determined at each stage in the treatment process of drinking water and in the water mains in order to guarantee bacteriologically impeccable water. While free chlorine residuals have good disinfecting powers, they are usually dissipated quickly in the distribution system. For this reason, final treatment with ammonia is often practiced to convert free chlorine residuals to longer-lasting combined chlorine residuals.

Chlorine determination is important to avoid bad odour and change in the taste of water. It is determined in the swimming pools to avoid ill effects due to excess chlorination. Determination of chlorine residual in water distribution is useful to find the source of contamination or leakage points, so as to supply wholesome water to the consumer.

Reagents

- Starch Indicator
- Standard 0.025 N Sodium thiosulfate
- Potassium Iodine crystal
- Concentrated Acetic Acid
- Chlorine water

Apparatus

- Erlenmeyer flask (250 mL): 6 no.
- Measuring cylinder : 1 no.
- Dropper : 1 no.
- Stirrer : 1 no.

Procedure

1. Place 200-mL portion of the water to be chlorinated in each of six 250-mL flasks.
2. Add required quantity (as instructed by your teacher) of "chlorine water" (stock solution of bleaching powder in water) in each of the flasks. The chlorine content of the "chlorine

water" (determined earlier in the laboratory) would be provided to you by your teacher. Calculate the chlorine dose for each of the six flasks.

3. Shake each flask gently and allow standing for 30 minutes.
4. Determine residual chlorine of water from each flask by the starch-iodine method as described below:

Determination of Residual Chlorine by Starch-Iodine Titration Method:

The starch-iodine method is based on the oxidizing power of free and combined chlorine residuals to convert iodide ion into free iodine at pH 8 or less, as shown below.

$$Cl_2 + 2I^- = I_2 + 2\,Cl^-$$

In the starch-iodine method, the quantity of chlorine residuals is determined by measuring the amount of iodine in water by titration with a reducing agent - sodium thiosulfate ($Na_2S_2O_3$). The end point of titration is indicated by the disappearance of blue color, produced by the reaction between iodine and starch (which is added as indicator during the titration).

$$I_2 + 2\,Na_2S_2O_3 = Na_2S_4O_6 + 2\,NaI$$

or,

$$I_2 + 2\,S_2O_3^{2-} = S_4O_6^{2-} + 2I^-$$

$$I_2 + \text{starch} \rightarrow \text{blue color}$$

(Qualitative test for the presence of iodine/chlorine)

The titration is carried out at pH 3 to 4, because the reaction with thiosulfate is not stoichiometric at neutral pH due to partial oxidation of the thiosulfate to sulfate.

Determination of Iodine (equivalent to residual chlorine) in water

i) Place 200 mL of the sample in an Erlenmeyer flask.
ii) Add about 2 mL of concentrated Acetic acid to the water.
iii) Add 1g of potassium iodide (estimated on a spatula) and dissolve it by thoroughly mixing it with a stirring rod.
iv) Add 0.025 N sodium thiosulfate drop by drop from a burette until the yellow color of liberated iodine almost disappears. Perform the titration quickly, since iodine liberate faster
v) Add 1 mL of starch solution to the water.
vi) Continue addition of standard sodium thiosulfate solution until the blue color just disappears. (In many cases residual chlorine is very low and starch needs to be added before starting up the titration.)
vii) Record the quantity (in mL) of sodium thiosulfate solution used.

Calculation:

Residual chlorine (mg/L)= mL of 0.025N sodium thiosulfate used x M.F.

Where, M.F. = $$\frac{\text{normality of } Na_2S_2O_3 \text{ x equivalent wt.of } Cl_2 \text{ x} 1000}{\text{mL of sample taken}}$$

Experiment No. 13

Determination of Arsenic in Water

Introduction

Arsenic, the 20th most abundant element in the earth's crust and the 12th most abundant element in the biosphere, is a common trace inorganic contaminant in drinking water and is identified as a significant health risk. Arsenic is known for its high toxicity and its ability to induce cancer after long-term ingestion. Presence of elevated levels of arsenic in groundwater (especially from shallow aquifer) has become a major concern in Bangladesh. Arsenic pollution of groundwater is particularly challenging in Bangladesh since tubewell water extracted from shallow aquifers is the major source of drinking water for most of its population. The rural water supply is almost entirely based on groundwater supply through use of hand pump tubewells; the urban water supply is also heavily dependent on groundwater.

Awareness about the presence of arsenic in groundwater has been growing since late 1993 when arsenic was first detected in the district of Chapai Nawabgonj bordering the West-Bengal district of India. A detail survey carried out by the DPHE and BGS (British Geological Survey) in 1999 identified the presence of arsenic in 268 Upazilas out of 464 in the country (BGS and DPHE, 2001). In 2005, the number of affected Upazilas increased to 270. In 2002 and 2003, 4.7 million tube wells in Bangladesh have been screened for arsenic. Of those, 1.4 million tube wells were found to contain arsenic above the Bangladesh drinking water standard of 50 microgram per liter. According to the Multiple Indicator Cluster Survey (MICS) carried out by GoB and the Unicef in 2012-13, about 24.8 per cent of the population of Bangladesh are drinking water in the household with arsenic above the WHO provisional guideline value of 10 ppb, and 12.4 per cent of the population are exposed to water with arsenic exceeding the Bangladesh standard of 50 ppb (GoB and Unicef, 2015).

In Bangladesh, the arsenic in groundwater is of geologic origin and is probably only apparent now because it is only the last 25 – 35 years that groundwater has been extensively used for drinking in rural areas. Weathering of arsenic-rich base metal sulfides in the upstream of the Ganges basin appears to be a major source of arsenic-rich iron oxyhydroxides in the sediments of Bangladesh. Arsenic-rich iron oxyhydroxides appear to be the major source of arsenic, from which arsenic is released as a result of dissolution and desorption. Reducing environment in the alluvial aquifer, primarily due to the presence of organic matter, promotes dissolution of iron oxyhydroxides and release of arsenic. Use of phosphate fertilizer can potentially enhance release of arsenic as a result of replacement of arsenic by phosphate ions on the adsorption sites of iron oxyhydroxides. Natural and anthropogenic processes that may lead to release/mobilization of arsenic in the subsurface are being investigated. According to ECR 1997, drinking water standard for arsenic in Bangladesh is 50 μg/L (or 0.05 mg/L). The WHO guideline value for arsenic in drinking water is 10 μg/L and the USEPA is also planning to revise its standard from 50 μg/L to 10 μg/L.

Arsenic Chemistry

Arsenic occurs in water in several different forms, depending upon the pH and the redox potential, E_h. Some of the most important compounds and species are shown in Table-13.1. In groundwater, arsenic primarily exists as inorganic arsenic. Inorganic trivalent arsenic, [As(III)] or arsenite is the dominant form in reducing environment; while inorganic pentavalent arsenic [As(V)] or arsenate is the dominant form in oxidizing or aerobic environment.

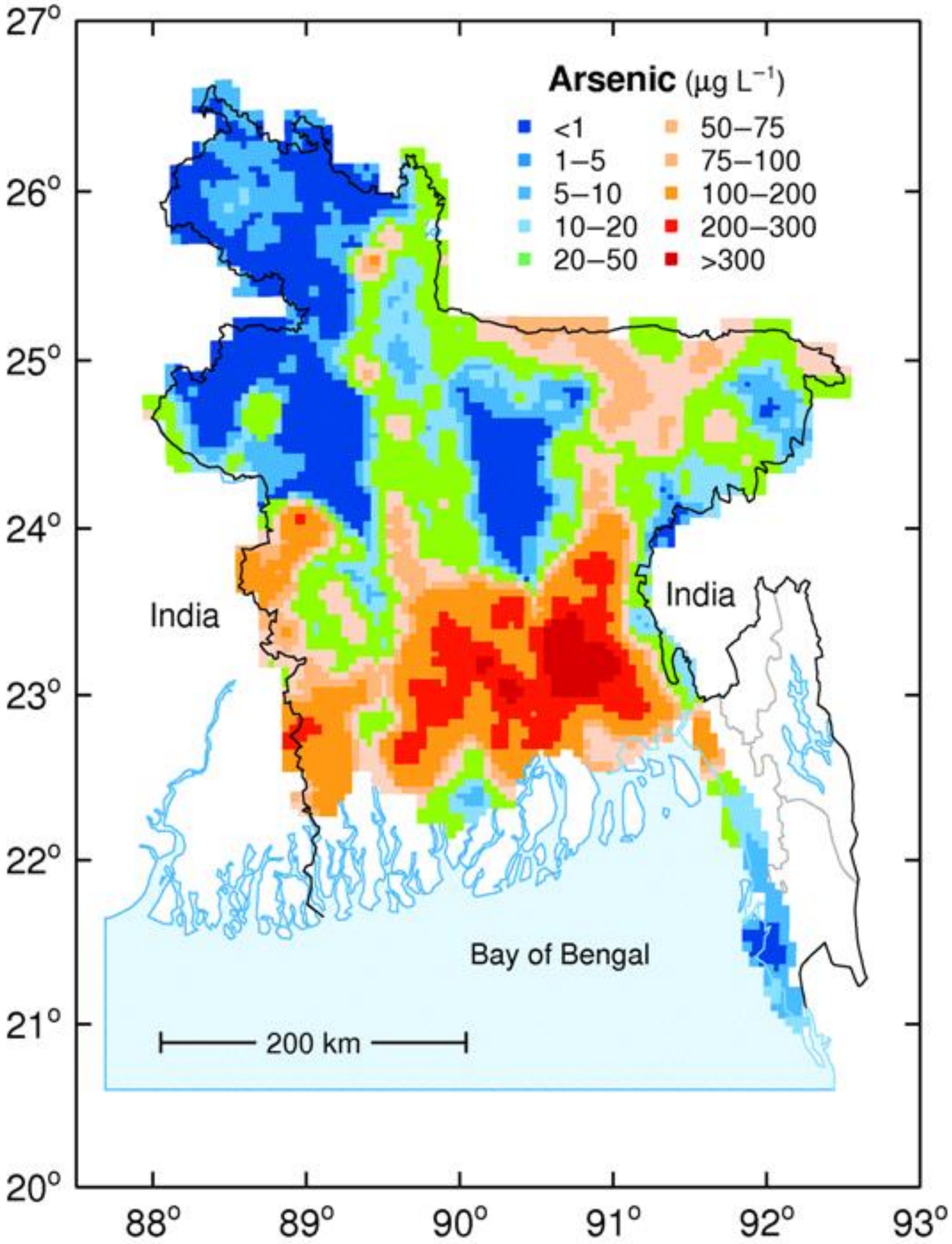


Figure 1: Distribution of Arsenic in Groundwater of Bangladesh

In groundwater environment where the conditions are mostly reducing, a significant part of the arsenic exists as As(III). As shown in Table 1, in the pH range of most groundwater (i.e., pH 6 – 9), dominant chemical form of As(III) is H_3AsO_3; while dominant chemical forms of As(V) in this pH range are: $H_2AsO_4^-$, $HAsO_4^{2-}$.

Table -13.1: Arsenic compounds and species and their environmental and toxicological importance in water

Compounds	Example	Environmental Significance/ Dominant pH region	Toxicity
Arsine	As^{3-}	Minor importance	Most toxic As species
Elemental Arsenic	As	Minor importance	Least toxic As species
Trivalent Arsenic	As(III)	Anaerobic	10 times more toxic than As(V)
Arsenite, Inorganic	H_3AsO_3, $H_2AsO_3^{1-}$, $HAsO_3^{2-}$, AsO_3^{3-}	pH = 0-9 pH = 10-12 pH = 13 pH = 14	
Methylated As(III) Organo-As(III)		Minor importance	Less toxic than Inorganic As(III)
Pentavalent arsenic	As(V)	Aerobic	10 times less toxic than As(III)
Arsenate, Inorganic	H_3AsO_4, $H_2AsO_4^{1-}$, $HAsO_4^{2-}$, AsO_4^{3-}	pH = 0-2 pH = 3-6 pH = 7-11 pH = 12-14	
Methylated As(V) Organo-As(V)		Minor importance	Less than Inorganic As(V)

Health Effects of Arsenic

In a population drinking arsenic contaminated water, a great variety of specific as well as non-specific symptoms may be observed. Table 2 lists some of the effects of arsenic reported to be due to exposure through drinking water.

In a recent study by the National Institute of Preventive and Social Medicine (NIPSOM), arsenic related diseases (arsenicosis) have been identified in 37 districts (Ahmad et al., 1998). A total of 6000 cases were identified in 162 villages in the 37 districts, mostly in the rural areas. Three stages of manifestations of chronic arsenicosis were observed in the study (Ahmad et al., 1998), but most of the patients were found in the first and second stages.

The most common presentations were melanosis, keratosis, hyperkeratosis and depigmentation. Cancers were found among 0.8% and actinic keratosis and Bowen's diseases were observed among 3.1% of the cases. It is important to note that the study found that the cases at initial and second stages of toxicity showed improvement when patients stopped taking arsenic contaminated water and increased intake of protein rich food, vitamin A, E and C.

Table- 13. 2: Toxicological effects due exposure to high arsenic concentration in drinking water (WHO, 1996, Khan, 1997)

Effect	Symptoms	Remarks
Blackfoot Disease Arsenical dermatosis	Dermal lesion, Peripheral neuropathy Keratosis, Hyperkeratosis, Hyperpigmentaion	May necessitate operation
None specific	Nausea, Abdominal Pain, Diarrhoea, Vomiting, Conjunctivitis, Oedema.	Mainly due to acute intoxication
Pregnancy disorders	Spontaneous abortions, miscarriages	-
Heart Disease	Coarctation of aorta, Cardiovascular disturb.	Among children
Cancer	Bladder, Kidney, Skin & Lungs, Liver & Colon	-
Mortality	-	Mainly due to cancer

Analytical Methods for Measuring Arsenic

The most commonly used methods for detection of arsenic in water include:

1. Inductively coupled plasma (ICP) method
2. Hydride generation atomic absorption spectrophotometric method (HG-AAS)
3. Graphite furnace atomic absorption spectrophotometric method (GF-AAS)
4. Hydride generation-scraper-spectrophotometric (SDDC) method
5. Hydride generation-scraper-indicator paper-field kit

The first three methods involve high-cost equipment and provide more accuracy and lower detection limit (minimum detection limit, MDL = 1 µg/L). The last two methods are relatively low cost methods but accuracy of determination is less. In our laboratory we have two atomic absorption spectrophotometers and can measure arsenic concentration following methods (2) and (3). In this lab determination of arsenic in water is carried out by the method -3. Basic mechanism of the method is described below.

Graphite furnace atomic absorption spectrophotometric method (GF-AAS)

Also known as Electro thermal atomic absorption spectrophotometric method (ETAAS). This method is based on the same principle as direct flame atomization but an electrically heated atomizer or graphite furnace replaces the standard burner head. Typically, determinations are made by heating the sample in three or more stages. First, a low current heats the tube to dry the sample. The second, or charring, stage destroys organic matter and volatilizes other matrix components at and intermediate temperature. Finally, a high current heat the tube to incandescence and, in an inert atmosphere, atomizes the element being determined. A photoelectric detector measures the intensity of transmitted radiation. The inverse of the transmittance is related logarithmically to the absorbance, which is directly proportional to the number density of vaporized ground-state atoms.

To understand the basic difference of ETAAS method and Hydride generation AAS method the later method is also discussed here:

Hydride generation atomic absorption spectrophotometric method

This method is also known as Flame Atomic Absorption Spectrophotometric (FAAS) method. In this method arsenic is reduced to gaseous arsine in a reaction vessel. The method is two types: i) manual hydride generation and ii) continuous hydride generation. In manual method zinc is added to speed the reaction whereas continuous in continuous hydride generation no zinc is needed. In continues measurement hydride generator a peristaltic pump is used to meter and mix reagents and a gas–liquid separator unit uses flow of argon to strip out hydrogen and arsine gas.

Preparation of Sample for Arsenic Determination:

***Preparation of Sample*:**

Arsenic is often present in very small concentration in water. Therefore, water samples are often *pre-concentrated* in order to increase the quantity of Arsenic in a given volume. Pre-concentration is usually done by acidifying 200 to 500 ml water sample and then reducing the volume to about 35 ml in a water bath.

***Precaution*:**

Arsenic is often found in groundwater with high concentration of iron (Fe). After extraction, Iron present in groundwater in ferrous (Fe^{2+}) form is converted to ferric (Fe^{3+}) form in contact with atmospheric oxygen and forms ferric hydroxide $Fe(OH)_3$ (s) flocs. Arsenic, especially As(V), is strongly adsorbed onto these ferric hydroxide flocs. Therefore, care should be taken while taking water sample from a bottle in which red coloured ferric hydroxide flocs are visible. It is better to acidify the bottle first (thus dissolving the ferric hydroxide flocs) and then take water sample for pre-concentration.

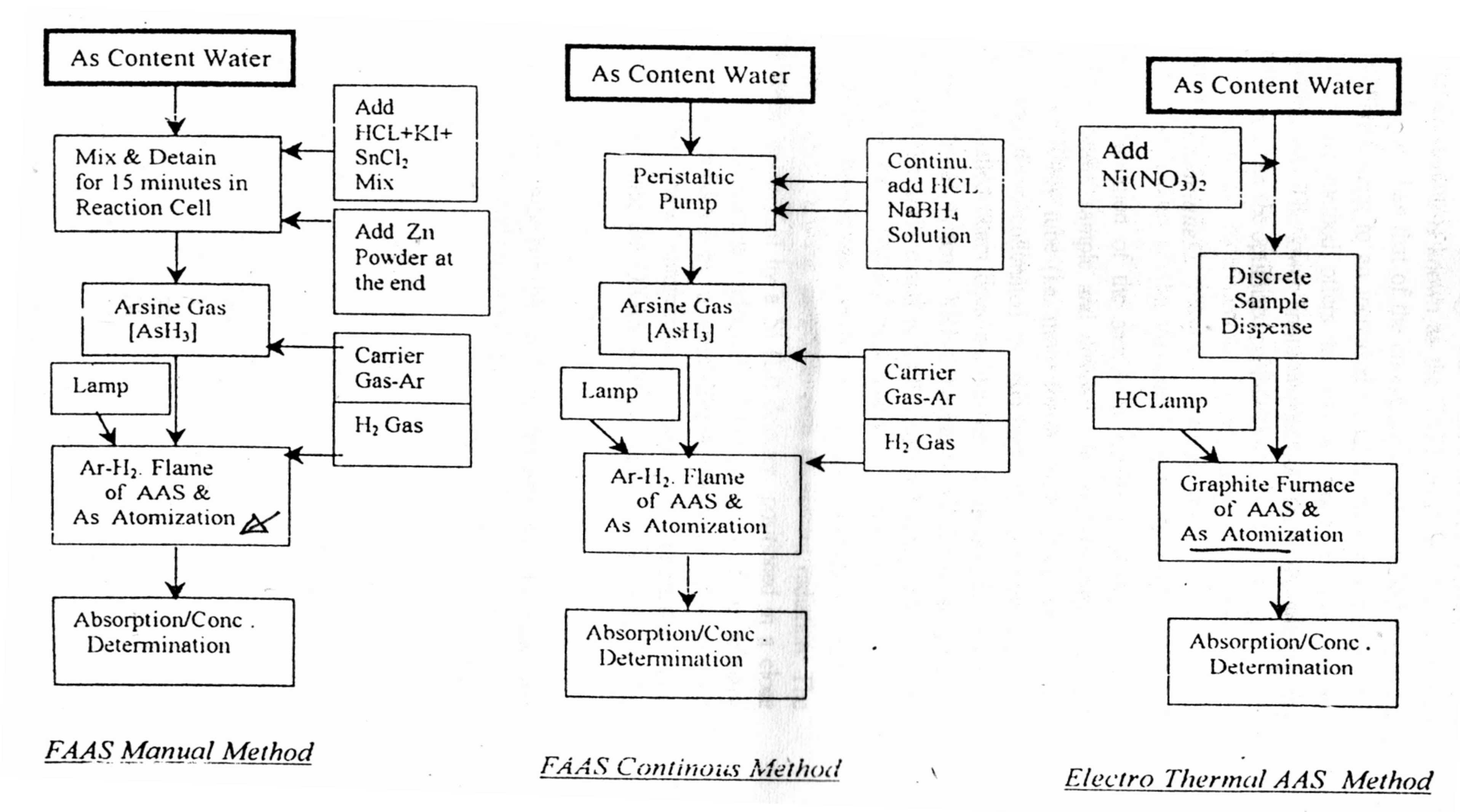


Fig. – 1: Different types of Atomic Absorption Spectrophotometric determination of arsenic

Apparatus

- Atomic Absorption Spectrophotometer (AAS)
- Glass tubes, Micro pipette etc.

Reagent

- Nickel nitrate (as matrix modifier)

Procedure

1. Take 9 mL sample and 1 mL nickel nitrate in a container.
2. Draw 20 µL using micro pipette and insert into the hole of graphite tube.
3. A hollow cathode lamp of 193.7 ηm is placed inside the spectrophotometer and switch on the machine. The temperature is raised up to 3000^0C to and As atomic cloud is formed (details will be shown in the class).
4. Repeat the procedure and take the average of three measurements.

Calculation

Calculate arsenic concentration using the calibration curve (will be supplied in the class).

Actual As concentration (ppb) = As conc.(ppb) obtained from calibration curve of AAS x D.F.

Where, D.F. = Dilution Factor = $\dfrac{\text{volume of sample} + \text{volume of NiNO}_3}{\text{volume of sample}\left(\text{mL}\right)}$

Experiment No. 14

Microbiological Quality of Water: Determination of Total Coliform and Fecal Coliform

---

Introduction

A variety of different microorganisms are found in untreated water. Most of these organisms do not pose a health hazard to humans. Certain organisms, referred to as pathogens, cause disease to humans which include species of bacteria, viruses and protozoa. These organisms are not native to aquatic systems and usually require an animal host for growth and reproduction. Pathogens are likely to gain entrance sporadically, and they do not survive for very long period of time; consequently they could be missed in a sample submitted to the laboratory. Although it is possible to detect the presence of various pathogens in water, the isolation and identification of many of these is often extremely complicated, time-consuming and expensive proposition. Hence in most cases (except when presence of any particular microorganism is suspected) the microbiological quality of water is checked using some *indicator organisms*.

An indicator organism is one whose presence presumes that contamination has occurred and suggests the nature and extent of the contaminants. An indicator organism should be a microorganism whose presence is evidence of fecal contamination of warm blooded animals. Indicators may be accompanied by pathogens, but typically do not cause disease themselves. It is considered that the ideal indicator organisms should have the following characteristics:

- always be present when pathogens are present
- always be absent where pathogens arc absent
- numbers should correlate the degree of pollution
- be present in greater number than pathogens
- there should be no after-growth or re-growth in water
- there should be greater or equal survival time than pathogens
- be easily and quickly detected by simple laboratory tests
- should have constant biochemical and identifying characteristics
- harmless to human

No organisms or group of organisms meet all of these criteria; but the *coliform* bacteria fulfill most of them, and this group is most common indicator used in microbial examination of water.

Total coliforms are grouped into two categories - i) Fecal coliform (thermo-tolerant coliform) and ii) Non-fecal coliform. Total coliforms are defined as gram negative bacteria which ferment lactose at 35 or 37°C, with the production of acid, gas and aldehyde within 24 or 48 hours. Fecal coliforms are a subgroup of total coliforms, which live in the warm blooded animals and have the same properties as the total coliforms but tolerate and grow at the higher selective temperature range of 44 to 44.5°C. In addition, they form indole from tryptophan. And these combined properties, when positive, are regarded as presumptive *Escherichia coli* (presumptive E. coli). Some coliform species are frequently associated with plant debris or may be common inhabitants in soil or surface waters which are called non-fecal coliforms.

Total coliform (TC) = Fecal coliform (FC) + Non-fecal coliform.

Thus, the total coliform group should not be regarded as an indicator of organisms exclusively of fecal origin. The use of total coliform as an indicator may therefore be of little value in assessing the fecal contamination of surface water, unprotected shallow wells etc. where contamination by coliform of non fecal origin also may occur. However, measurement of total coliform is of particular relevance for treated and/or chlorinated water supplies; in this case the absence of total coliform would normally indicate that the water has been sufficiently treated/ disinfected to destroy various pathogens. Measurement of fecal coliform is a better indicator of general contamination by material of fecal origin. The predominant species of fecal coliform group is *Escherichia coli* (E. coli), which is exclusively of fecal origin, but strains of *Klebsella pneumoniae* and *Enterobacter* species may also be present in contaminated water.

Using coliform as indicators of the presence and absence of pathogens sometimes may cause the following drawbacks:

- False positive result can be obtained from the bacterial *genus aeromonas*- a gram-negative bacteria which resemble coliform group bacteria in their fermentative ability.
- False negative result can be obtained when coliforms are present along with high population of other bacteria. The latter bacteria can act to suppress coliform activity.
- A number of pathogens have been shown to survive longer in natural waters and/ or through various treatment processes than coliform.

But the use of coliform was established first and there does not appear to be any distinct advantages to warrant shifting to other indicator organisms. Since bacteria are used as indicator organisms, the microbiological examination of water is commonly called bacteriological examination.

Methods of Bacteriological Examination Of Water

Basically there two methods of bacteriological analysis of water: (a) Multiple Tube or Most Probable Number (MPN) method, and (b) Membrane Filter (MF) method.

The most probable number (MPN) is the number of organisms that are most likely to have produced laboratory results in a particular test. The MPN method is used to quantify the concentration of the viable microorganisms in a sample and involves inoculating decimal dilutions into tubes of a broth medium, observing results and using a standard MPN table. This method is extensively used in food microbiology. In our lab we will perform membrane filter (MF) method which is discussed below.

Membrane Filter Method:

The membrane filter (MF) method gives a direct count of total coliforms and fecal coliforms present in a given sample of water. The method is based on the filtration of a known volume of water through a membrane filter consisting of a cellulose compound with a uniform pore diameter of 0.45µm; the bacteria are retained on the surface of the membrane filter. When the membrane containing the bacteria is incubated in a sterile container at an appropriate temperature with a selective differential culture medium, characteristic colonies of coliforms and fecal coliforms develop, which can be counted directly. This technique is popular with environmental engineers.

This method is not suitable for turbid waters, but otherwise it has several advantages. Its particular advantages and limitations are as follows:

Advantages:

- results are obtained more quickly as the number of coliforms can be assessed in less than 24 hours, whereas the multiple tube technique requires 48 hours both for a negative or a presumptive positive test;
- saving in work, certain supplies and glassware;
- method gives direct results;
- easy to use in laboratories, or even in the field if portable equipment is used.

Disadvantages:

- high turbidity caused by clay, algae, etc. prevents the filtration of a sufficient volume of water for analysis and it may also produce a deposit on the membrane which could interfere with bacterial growth;
- presence of a relatively high non-coliform count may interfere with the determination of coliforms;

- water containing particular toxic substances which may be absorbed by the membranes, can affect the growth of the coliforms.

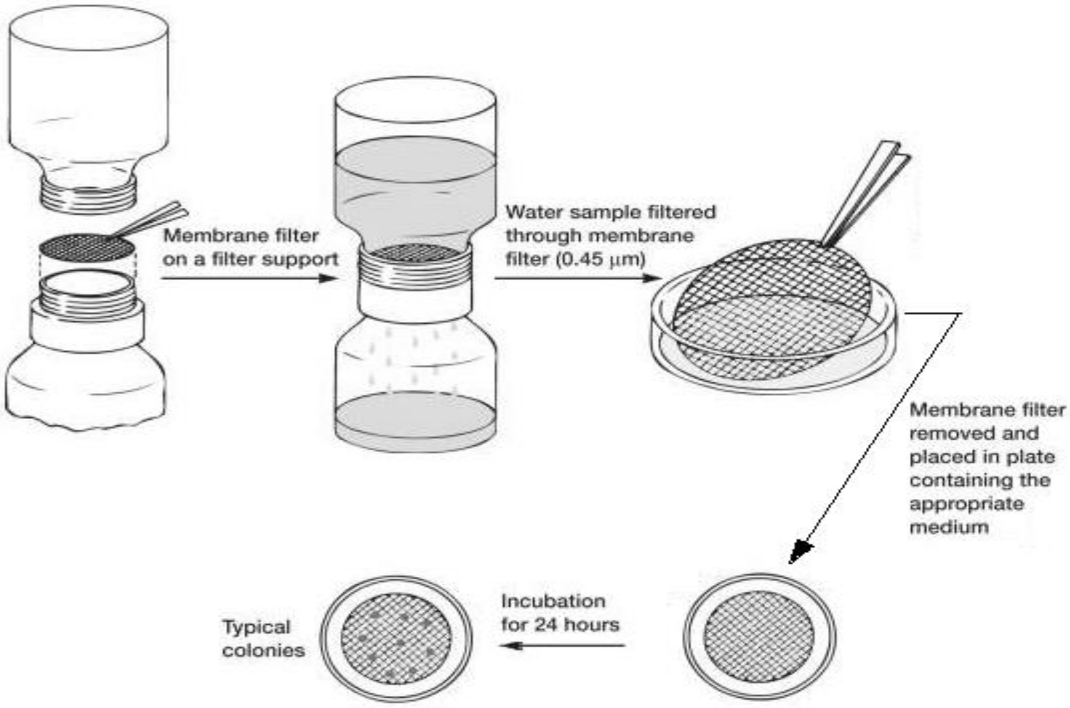


Fig-1 : Membrane Filter method

Reagents

- Distilled water
- Appropriate culture mediums (broth)
- Silver nitrate solution (0.0141 N)

Apparatus

- Erlenmeyer flask : 1 pc.
- Vacuum pump : 1 set
- Petri Dish : 2 pcs.
- Membrane filter paper (0.45 μm) : 2 pcs.
- Incubator : 2
- Measuring cylinder, beaker, dropper etc.

Test Procedure

This section describes the general procedures. It should be noted that different types of filtration units and equipment are available in the market for performing the tests.

Determination of Total Coliforms (TC):

1. Connect the Erlenmeyer (side-arm) flask to the vacuum source (turned off) and place the porous support in position. If an electric pump is used, it is advisable to put a second flask between the Erlenmeyer and the vacuum source; this second flask acts as a water trap and thus protects the electric pump.
2. Open a Petri-dish and place a pad in it. With a sterile pipette add 2 mL of selective broth (culture) medium to saturate the pad.
3. Assemble the filtration unit by placing sterile membrane filter on the porous support, using forceps sterilized earlier by flaming.
4. Place the upper container in position and secure it with the special clamps. The type of clamping to be used will depend on the type of equipment.
5. Pour the volume of sample chosen as optimal, in accordance with the type of water, into the upper container. If the test sample is less than 10 mL, at least 20 mL of sterile

dilution water should be added to the top container before filtration applying the vacuum.

6. After the sample has passed through the filter, disconnect the vacuum and rinse the container with 20-30 mL of sterile dilution water. Repeat the rinsing after all the water from the first rinse has passed through the filter.
7. Take the filtration unit apart and using the forceps, place the membrane filter in the Petri-dish on the pad with the grid side up. Make sure that no air bubbles are trapped between the pad and the filter.
8. Invert the Petri-dish for incubation. Incubate at 35°C or 37°C for 18-24 hours with 100% humidity (to ensure this, place a piece of wet cotton wool in the incubator). If ointment containers or plastic dishes with tight-fitting lids are used, humidification is not necessary.

Bacterial Colony observation:

Colonies of coliform bacteria are a medium red or dark red color, with a greenish gold or metallic surface sheen. This sheen may cover the entire colony or appear only in the centre of the colony. Colonies of other types should not be counted. The colonies can be counted with the aid of a lens. The number of total coliforms per 100 ml is then given by:

Determination of Fecal Coliforms (FC):

The procedure for fecal coliforms is similar to that used for determining total coliforms. Filter the sample as described, and place the membrane filler on the pad saturated with appropriate culture medium.

1. Place the dishes in an incubator at 44±0.5 °C for 24 hours at 100% humidity. Alternatively, tight-fitting or sealed Petri-dishes may be placed in water-proof plastic bags for incubation.
2. Submerge the bags in a water-bath maintained at 44±0.5°C for 24 hours. The plastic bags must be below the surface of the water throughout the incubation period. They can be held down by means of a suitable weight, e.g., a metal rack.

Bacterial Colony observation:

Colonies of fecal coliform bacteria are blue in color. This color may cover the entire colony, or appear only in the center of the colony. Colonies of other types should not be counted. The colonies can be counted with the aid of a lens. The number of fecal coliforms per 100 ml is then given by:

Calculation

$$\text{Total coliform (nos./ 100 mL)} = \frac{\text{no. of coliform colonies counted} \times 100}{\text{mL of sample filtered}}$$

$$\text{Fecal coliform (nos./ 100 mL)} = \frac{\text{no. of coliform colonies counted} \times 100}{\text{mL of sample filtered}}$$

ASSESSMENT PATTERN

CIE- Continuous Internal Evaluation (30 Marks)

SEE- Semester End Examination (20 Marks)

SEE- Semester End Examination (40 Marks) (should be converted in actual marks (20))

Bloom’s Category	**Tests**
Remember	**05**
Understand	**07**
Apply	**08**
Analyze	**07**
Evaluate	**08**
Create	**05**

CIE- Continuous Internal Evaluation (100 Marks) (should be converted in actual marks (30))

Bloom’s Category Marks (out of 100)	Lab Final (30)	Lab Report (10)	**Continuous lab performance** (30)	Presentation & Viva (10)	External Participation in Curricular/**Final Project Exhibition** (10)
Remember/**Imitation**	05		05	02	Attendance 10
Understand/**manipulation**	05	05	05	03	
Apply/**Precision**	05		05		
Analyze/**Articulation**	05		05		
Evaluate/**Naturalisation**	05	05	05		
Create	05		05	05	