UNITED STATES DEPARTMENT OF AGRICULTURE

DEPARTMENT BULLETIN No. 1216

Washington, D. C. ▼ May, 1924

TENTATIVE STANDARD METHODS OF SAMPLING AND TESTING HIGHWAY MATERIALS

Adopted by the

AMERICAN ASSOCIATION OF STATE HIGHWAY OFFICIALS

And Approved by the

SECRETARY OF AGRICULTURE

For Use in Connection With Federal-Aid Road Construction

CONTENTS

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CONTENTS.

INTRODUCTION.

The tentative standard methods of sampling and testing highway materials set forth in this bulletin are those recommended by the committee on tests and investigations of the American Association of State Highway Officials.[1] They were adopted by the association at its annual meeting at Kansas City, Mo., December 4 to 7, 1922. They have since been approved by the Secretary of Agriculture for use in connection with the administration of Federal appropriations for construction of the Federal-aid highway system, superseding, for this purpose, the methods described in Department Bulletin 949, Standard and Tentative Methods of Sampling and Testing Highway Materials.

[1] The membership of the committee on tests and investigations of the American Association of State Highway Officials follows: H. S. Mattimore, State Highway Department, Pennsylvania, *Chairman;* B. A. Anderton, United States Bureau of Public Roads, Washington, D. C.; J. H. Bateman, Department of Public Works, Massachusetts; J. B. Bragg, State Highway Commission, New Jersey; A. V. Bratt, Department of Public Works, Massachusetts; Shreve Clark, State Highway Commission, Virginia; H. F. Clemmer, Department of Public Works and Buildings, Illinois; W. F. Cooper, State Highway Commission, Louisiana; R. W. Crum, State Highway Commission, Iowa; R. B. Dayton, State Road Commission, West Virginia; R. B. Gage, State Highway Commission, New Jersey; C. A. Hall, State Highway Commission, Oregon; H. A. Hall, State Road Department, Florida; G. W. Hutchinson, State Highway Commission, North Carolina; F. H. Jackson, United States Bureau of Public Roads, Washington, D. C.; A. N. Johnson, State Roads Commission, Maryland; F. C. Lang, State Highway Department, Minnesota; E. T. Maddock, Department of Public Works, California; J. H. Milburn, United States Bureau of Public Roads, Washington, D. C.; J. E. Myers, Commission of Highways, New York; W. A. Norris, State Highway Department, Wyoming; R. L. Oberholser, State Highway Commission, North Carolina; W. F. Purrington, State Highway Department, New Hampshire; A. S. Rea, Department of Highways and Public Works, Ohio; C. H. Scholer, State Highway Commission, Kansas; C. M. Strahan, State Highway Department, Georgia; D. V. Terrill, Department of State Roads and Highways, Kentucky; M. H. Ulman, State Highway Department, Pennsylvania; C. G. Wickline, State Highway Department, Texas; N. C. Withey, State Highway Commission, Wisconsin.

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METHODS OF SAMPLING AND TESTING NON-BITUMINOUS HIGHWAY MATERIALS.

METHODS OF SAMPLING STONE, SLAG, GRAVEL, SAND, STONE BLOCK, AND PAVING BRICK FOR USE AS HIGHWAY MATERIALS, INCLUDING SOME MATERIAL SURVEY METHODS.

(A. S. T. M. standard method, serial designation D 75–22, modified.)

1. Samples of all materials to be tested as a basis for acceptance or rejection of the supply shall be taken by the engineer or his authorized representative. Samples for inspection or preliminary test may be submitted by the producer or owner of the supply.

I. SAMPLING OF STONE.

A. Sampling of Stone from Local Ledges and Noncommercial Quarries for Quality.

Method of sampling.

2. The ledge or quarry face shall be inspected closely to determine any variations in different layers. Any difference in color or structure shall be observed, and if necessary to secure a representative sample unweathered specimens shall be broken from different layers.

Size and weight of sample for stone tests.

3. (*a*) For the standard stone tests, separate samples, at least 30 pounds of fresh unweathered specimens in each, shall be taken from all layers that appear to vary in color or structure. When more than one piece is taken, the minimum size shall be 2 inches, except that there shall be one piece of each sample to be used in the toughness or compression test which shall have a minimum size of 4 by 5 by 3 inches on which the bedding plane shall be marked and which shall be free from seams or fractures.

Samples for concrete aggregate.

(*b*) The character of samples to be tested for concrete aggregate will depend upon the kind of tests to be made and the number of specimens necessary.

Information with sample.

4. In addition to the general information to be furnished with all samples, the following information shall be furnished with all samples from local ledges which are not commercial sources: Name of owner; approximate quantity available (if quantity is very large this may be recorded as practically unlimited); amount and character of overburden or stripping; haul to nearest point on road where the material is to be used; character of haul (kind of road and grade); also some detailed record of the extent and location of the material represented by each sample. For this purpose a sketch, plan and elevation showing the thickness and location of the different layers is recommended.

B. Sampling of Stone from Commercial Quarries for Quality and Size.

Method of sampling.

5. When practicable, samples from commercial quarries shall be taken from the ledge or quarry face and the same procedure shall be followed as when sampling local ledges.

6. The following factors affect the size of broken stone: Size and shape of screen openings; length of screen sections; the angle of screen with the horizontal; the speed of screen rotation; and the rate at which the screen is fed. A general inspection and record of these conditions shall be made. The sample shall be taken preferably from cars or boats while loading from stock piles or bins. In order that the sample may be representative, it is recommended that separate samples be taken at different times while the material is being loaded. If the sample must be taken from the bin or stock pile, several samples shall be taken from different parts of the stock pile and from the top of the bin and the loading chute. These separate samples

shall be well mixed to form a composite sample and the sample for testing shall be obtained by the quartering method.

7. (*a*) If it is not practicable to visit the plant, samples for both quality and size shall be taken from different parts of the car or boat during unloading. It is recommended that separate samples be taken from the top, middle, and bottom of the car or boat. These separate samples shall be well mixed to form a composite sample and the sample for testing shall be obtained by the quartering method. The results of tests of crushed stone for quality are not considered comparable with results from samples specially broken for test in the laboratory, but the general quality of the stone can be ascertained and check tests on various shipments should indicate any change in quality. In this case, tests for both quality and size can be made on the same sample.

(*b*) If the stone is to be tested for size only, it is recommended that a small set of screens and a pair of scales or some receptacle for measuring volume be used for field testing in order to avoid delaying the decision on the use of the material. Occasional check tests can be obtained from the laboratory to assure a fair degree of accuracy in the field testing.

Weight of sample.

8. (*a*) The sample of crushed stone for mechanical analysis shall weigh at least 50 times the weight of the largest piece therein.

(*b*) The character of samples to be tested for concrete aggregate will depend upon the kind of tests to be made and the number of specimens necessary.

C. Sampling of Field Stone and Bowlders.

Method of sampling.

9. A detailed inspection of the deposits of field stone and bowlders shall be made in the area from which the supply is to be obtained. The different kinds of stone and their state of preservation in the various deposits shall be recorded.

10. Separate samples shall be taken of all stone of different classes which, as determined by visual examination of their state of preservation and degree of lamination, should be considered for use in construction.

Information with samples.

11. Records accompanying samples of field stone and bowlders, in addition to general information, shall include the following information:

(*a*) Location of supply.

NOTE.—The plotting of the field stone and bowlder area on a United States topographic or a similar map is recommended for this purpose.

(*b*) Approximate quantity available.

NOTE.—A fairly accurate estimate of the amount of stone in fences can be made by measuring a cross section and pacing the length. It is more difficult to estimate the amount of stone scattered on the ground, and ordinarily it does not pay to work such deposits unless the accumulation is heavy.

(*c*) Information in regard to the relative amounts of the different classes of stone sampled and the materials rejected by visual examination which have to be handled and spoiled.

NOTE.—The percentages of different kinds of material can only be estimated and the degree of accuracy attained will depend almost entirely upon the experience and skill of the individual.

II. SAMPLING OF BLAST-FURNACE SLAG.

12. It is recommended that blast-furnace slag be sampled for size and quality by the method specified for broken stone.

III. SAMPLING OF SAND AND GRAVEL.

A. Sampling of Noncommercial Deposits.

13. Noncommercial as used here includes all undeveloped sand and gravel deposits and all developed deposits where the material is not washed or screened.

Method of sampling.

14. (*a*) The investigator should realize that few, if any, natural sand and gravel deposits are uniform, and when a sample is taken the quantity of material in the deposit similar to the sample shall be ascertained as nearly as possible.

(*b*) If possible, samples of sand shall be taken when it is in a damp condition.

(*c*) If the deposit is worked as a bank or pit and has an open face, the sample shall be taken by channeling the open face so as to represent all material suitable for use as determined by a visual examination. Care shall be taken to eliminate any overburden or stripping at the top and any material that has fallen along the face from the top. It is necessary, especially in small deposits, to excavate test pits some distance back of and in a line parallel to the face to determine the extent of the supply. The number and depth of these pits will depend upon the quantity of material that is to be taken from the deposit. Separate samples shall be taken from the face of the bank and from the test pits. These shall be well mixed to form a composite sample and the sample for testing shall be obtained by the quartering method.

(*d*) Deposits that have no open face shall be sampled by means of test pits. The number and depth of these pits will depend upon local conditions and the amount of material to be used from the source. A separate sample shall be taken from each pit and where visual examination indicates there is no radical difference in size of grain, color, etc., the several samples shall be well mixed to form a composite sample and the sample for testing shall be obtained by the quartering method. Where visual examination indicates there is a distinct difference in material from different pits, separate samples shall be taken for testing.

(*e*) It is suggested that the colorimetric test be used for determining the percentage of organic material.[2]

15. In addition to the general information accompanying all samples from sand and gravel deposits which are not commercial sources, detailed information shall be supplied similar to that specified to be furnished with samples of stone taken from local ledges.

B. Sampling of Sand and Gravel from Commercial Plants.

Method of sampling.

16. Samples of both sand and gravel from commercial screening or washing plants shall be taken from the bins or storage piles preferably while the material is being loaded from these supplies. The sand and gravel shall be sampled as separate units and shipped as such. It is difficult to secure a representative sample from stock piles and bins and, if conditions require sampling from these sources, the following is recommended: Separate samples shall be taken from different parts of the stock pile, care being taken to avoid sampling a segregated area of coarse-grained material which is likely to exist at the base of the pile. In sampling from a bin, separate samples shall be taken from the top and the loading chute. At the latter place at least one-half cubic yard of material shall be run off and representative samples taken from it. These separate samples shall be well mixed to form a composite sample and the sample for testing shall be obtained by the quartering method.

17. The size of sand and gravel will be affected by the same factors as broken stone and the same inspection is necessary. In addition, the relative amount of water used in screening must be taken into consideration. The same procedure for taking a sample of broken stone for size is recommended for sampling sand and gravel for size, both at plant and at delivery.

18. Where it is not practicable to visit the plant, samples for both quality and size shall be taken from different parts of the

[2] For a description of this test, see the Standard Method of Test for Organic Impurities in Sands for Concrete, serial designation, C 40–22, of the American Society for Testing Materials.

car or boat during unloading. It is recommended that separate samples be taken from the top, middle, and bottom of car or boat. These separate materials shall be well mixed in a composite sample and the sample for test obtained by the quartering method.

C. Quantity of Sand and Gravel Samples.

Size of samples.

19. Samples of run-of-bank (material in which the sand and gravel are combined) shall consist of at least 100 pounds of material when the gravel content is 50 per cent or more of the whole. If the material contains less than 50 per cent of gravel, the sample shall be increased in proportion. For example, when the gravel percentage is 25 per cent of the whole the sample should weigh 200 pounds.

20. Samples of sand shall contain at least 20 pounds of material.

21. Samples of gravel shall contain at least 50 pounds of material.

22. The quantity of samples to be tested for concrete aggregate will depend upon the kind of tests to be made and the number of specimens necessary.

IV. SAMPLING OF MISCELLANEOUS MATERIALS.

23. Samples of slag sand, screenings, mine tailings, and all other materials used as substitute for sand and gravel or broken stone, shall be inspected and sampled in the same way as the materials for which they are substituted.

V. SAMPLING OF STONE BLOCK.

24. Samples shall be taken either at the quarry or from cars or boats as directed by the engineer. They shall be representative of the block which it is proposed to use and no sample shall include block that would be rejected by visual examination.

25. The sample shall consist of at least six blocks and the bedding plane shall be marked on at least two of these.

VI. SAMPLING OF PAVING BRICK.

Place of sampling.

26. *Place of sampling.*—Where a standard rattler has been installed and is kept in an approved condition, and acceptable facilities for testing are furnished by the manufacturer, samples will be taken and tested at the plant by an inspector while cars are being loaded, provided daily shipments are sufficient to warrant it. Under all other conditions samples will be taken from the cars upon arrival at their destination.

Method of sampling.

27. *Method of selecting samples.*—A single sample will be taken to represent each 10,000 to 15,000 brick according to conditions described later. No brick are to be included in the sample which are cracked or so deformed as to be unfit for laying under the specifications.

Samples from kilns.

28. (*a*) *Samples from kilns.*—When samples are taken directly from the kiln, the first set will usually be chosen as soon as enough brick have been removed from the front of the kiln to permit of obtaining a sample representative of both the full width and height of the kiln. Later samples are to be taken with the idea of representing three degrees of burning in the kiln, one sample to be selected from localities (usually the top and sides) to represent the hardest burned brick, one sample usually selected from the more central portion of any cross section of the kiln to represent medium burned brick, and one sample usually from the bottom layers and away from the sides to represent the least burned brick. Each of these samples should represent approximately 15,000 brick.

Samples from piles.

(*b*) *Samples taken from piles.*—When the appearance of the pile of brick shows clearly that there is a considerable range in the degree of burning, samples to represent each of three degrees

should be selected as in the case of samples taken from kilns, each sample to represent about 10,000 brick. When the surface appearance of the brick does not furnish any indication of their uniformity, samples are to be taken at random representing approximately 10,000 brick.

Samples from cars.

(c) *Samples from cars.*—When a sample is taken from a carload it shall be selected in the same manner as previously described to represent three approximate degrees of burning.

Shipment of samples.

29. *Shipment of samples.*—Samples which must be transported long distances by freight or express shall be carefully put up in packages holding not more than 12 brick each. When more than 6 brick are shipped in one package, it shall be so arranged as to carry two parallel rows of brick side by side, and these rows shall be separated by a partition. In event of some of the brick being cracked or broken in transit, the sample shall be disqualified if there are not remaining 10 sound, undamaged brick.

Storage of samples.

30. *Storage and care of samples.*—Samples shall be carefully handled to avoid breakage or injury. They shall be kept in the dry so far as practicable. If wet when received, or known to have been immersed or subjected to recent prolonged wetting, they shall be dried for at least six hours in a temperature of 100° F. before testing.

VII. GENERAL DIRECTIONS FOR SHIPPING AND MARKING SAMPLES.

31. Samples of ledge stone, crushed stone, and slag shall be shipped in a secure box or bag.

32. Samples of stone block shall be securely crated.

Shipment of sample.

33. Samples of run-of-bank gravel, sand screenings, and other fine material shall be shipped in a tight box or closely woven bag so there shall be no loss of the finer particles.

Information with sample.

34. Each sample or separate container shall be accompanied by a card or regular form, preferably in the container, giving the following information: By whom taken; official title or rank of the sampler; by whom submitted; source of supply; proposed use of the material; and in case of commercial supplies, daily production; geographic location; shipping facilities (name of railroad, canal or river, or other common carrier); and price of the material.

ABRASION TEST FOR BROKEN STONE.

(A. S. T. M. standard method, serial designation D 2–08, slightly modified.)

The machine.

(1) The machine shall consist of one or more hollow iron cylinders, closed at one end and furnished with a tightly fitting iron cover at the other; the cylinders to be 20 centimeters in diameter and 34 centimeters in depth inside. These cylinders are to be mounted on a shaft at an angle of 30° with the axis of rotation of the shaft.

Test sample.

(2) The rock to be tested shall be broken from large pieces to as nearly uniform size and shape as possible, and as near to 50 pieces as possible shall constitute a test sample. No pieces with edges or faces that have been rounded by wear shall be included. The total weight of rock in a test shall be within 10 grams of 5 kilograms. All test pieces shall be washed and thoroughly dried before weighing. Ten thousand revolutions, at the rate of between 30 and 33 per minute, shall constitute a test. Only the percentage of material worn off which will pass through a 0.16 centimeter (one-sixteenth inch) mesh sieve shall be considered in determining the amount of wear. This shall be expressed as the percentage of the 5 kilograms used in the test.

(3) When the material has a specific gravity below 2.20 the quantity used for the test shall be adjusted on a weight basis, retaining the specified number of pieces. For such materials a weight of 4,000 grams of the broken stone or broken slag shall be used.

(4) The results shall be reported as per cent of wear.

ABRASION TEST FOR GRAVEL.

Test sample.

(1) The aggregate shall first be screened through screens having circular openings 2 inches, 1½ inches, 1 inch, three-quarters inch, and one-half inch in diameter. The material of these sizes shall be washed and dried. The following weights of the dried stone shall then be taken: 1,250 grams of the size passing the 2-inch and retained on the 1½-inch screen, 1,250 grams of the size passing the 1½-inch and retained on the 1-inch screen, 1,250 grams passing the 1-inch and retained on the three-quarters inch screen, 1,250 grams passing the three-quarters inch and retained on the one-half inch screen. This material shall be placed in the cast-iron cylinder of the Deval machine as specified for the standard abrasion test on stone. Six cast-iron spheres 1.875 inches in diameter and weighing approximately 0.95 pound (0.45 kilogram) each shall be placed in the cylinder as an abrasive charge. These spheres are the same as those used in the standard rattler test for paving brick.

Duration of test.

(2) The duration of the test and the rate of rotation shall be the same as specified for the standard test for stone, namely, 10,000 revolutions at a rate of 30 to 33 revolutions per minute. At the completion of the test the material shall be taken out and screened over a one-sixteenth inch mesh sieve. The material retained upon the sieve shall be washed and dried and the percentage loss by abrasion of the material passing the one-sixteenth inch mesh sieve calculated.

(3) When the material has a specific gravity below 2.20 a total weight of 4,000 grams made up of the four groups of sizes described above, instead of 5,000 grams, shall be used in the abrasion test.

TEST FOR TOUGHNESS OF ROCK.

(A. S. T. M. standard method, serial designation D 3–18, slightly modified.)

1. The term "toughness," as applied to rock, is defined as the resistance offered to fracture under impact, expressed as the fall in centimeters of a standard hammer which, after a series of blows from increasing heights, finally causes fracture of a cylindrical test specimen of given dimensions.

Size of sample.

2. Quarry samples of rock from which test specimens are to be prepared shall measure at least 6 inches on a side and at least 4 inches in thickness, and when possible shall have the plane of structural weakness of the rock plainly marked thereon. Samples shall be taken from freshly quarried material, and only from pieces which show no evidences of incipient fracture due to blasting or other causes. The samples shall preferably be split from large pieces by the use of plugs and feathers and not by sledging. Commercial stone-block samples from which test specimens are to be prepared shall measure at least 3 inches on each edge.

Preparation of specimen.

3. Specimens for testing shall be cylinders prepared as described in section 4, 25 millimeters in height and from 24 to 25 millimeters in diameter. Three test specimens shall constitute a test set. The ends of the specimen shall be plane surfaces at right angles to the axis of the cylinder.

4. One set of specimens shall be drilled perpendicular and another parallel to the plane of structural weakness of the rock, if such plane is apparent. If a plane of structural weakness is not apparent, one set of specimens shall be drilled at random. Specimens shall be drilled in a manner which will not subject the material to undue stresses and which will insure the specified

dimensions.[3] The ends of the cylinders may be sawed by means of a band or diamond saw,[4] or in any other way which will not induce incipient fracture, but shall not be chipped or broken off with a hammer. After sawing, the ends of the specimens shall be ground plane with water and carborundum or emery on a cast-iron lap until the cylinders are 25 millimeters in length.

Impact machine.

5. Any form of impact machine which will comply with the following essentials may be used in making the test:

(*a*) A cast-iron anvil weighing not less than 50 kilograms, firmly fixed upon a solid foundation;

(*b*) A hammer weighing 2 kilograms, arranged so as to fall freely between suitable guides;

(*c*) A plunger made of hardened steel and weighing 1 kilogram, arranged to slide freely in a vertical direction in a sleeve, the lower end of the plunger being spherical in shape with a radius of 1 centimeter;

(*d*) Means for raising the hammer and for dropping it upon the plunger from any specified height from 1 to not less than 75 centimeters, and means for determining the height of fall to approximately 1 millimeter;

(*e*) Means for holding the cylindrical test specimen securely on the anvil without rigid lateral support, and under the plunger in such a way that the center of its upper surface shall be tangent to the spherical end of the plunger at its lowest point throughout the test.

6. The test shall consist of a 1-centimeter fall of the hammer for the first blow, a 2-centimeter fall for the second blow, and falls increasing by 1 centimeter for each succeeding blow until failure of the test specimen occurs.

7. The height of fall in centimeters at failure shall be taken as the toughness of the test specimen. The individual and the average toughness of three test specimens shall be reported when no plane of structural weakness is apparent. When a plane of structural weakness is apparent, the individual and average toughness of the three specimens in each set shall be reported and identified. Any peculiar condition of a test specimen which might affect the result, such as the presence of seams, fissures, etc., shall be noted and recorded with the test result.

TEST FOR SOUNDNESS OF COARSE AGGREGATE.

Immerse 10 small pieces (total weight about 1,000 grams) of the rock in a saturated solution at 70° F. of sodium sulphate (Na_2SO_4) for 20 hours, after which place them for 4 hours in a drying oven maintained at 100° C. Repeat the treatment five times. Note the condition of the rock as to soundness at the end of the test.

Samples which exhibit marked checking, cracking or disintegration shall be considered to have failed in this test.

LITHOLOGICAL COUNT OF GRAVEL PARTICLES.

From 100 to 200 pieces of gravel stones, selected at random, ranging in size from one-half to 2 inches shall be separated into the various rock types and the percentage composition noted.

NOTE.—This is a simple test which, especially in dealing with the northern glacial gravels, will obviate considerable laboratory work. There is no single laboratory test for this type of material which will give such conclusive results as to quality of the material as this when the adjacent bedrock and direction of the glaciation are known.

[3] The form of diamond drill described in Bulletin No. 347, U. S. Department of Agriculture, pp. 6–7, is recommended, and should prove satisfactory if the instructions are strictly followed.

[4] A satisfactory form of diamond saw is described in Bulletin No. 347, U. S. Department of Agriculture, pp. 7–9.

IMPACT TEST FOR GRAVEL AGGREGATE.

The material to be tested is accurately quartered until a sample is obtained which contains at least 50 pieces ranging in size from three-quarters inch to 2 inches in diameter. Care should be taken to see that the various sizes appear in about the same proportion as in the original sample. The pieces thus obtained are then tested individually by first determining the average diameter across the shortest dimension and then subjecting each to a single blow of the hammer using the machine described in a paper printed in the Proceedings of the American Society for Testing Materials for 1922 entitled "An Impact Test for Gravel," by F. H. Jackson, Bureau of Public Roads.

NOTE.—It is suggested that the height of fall shall be that indicated in the following table:

Diameter of particle.	Height of fall.	
	Sedimentary origin.	Igneous or metamorphic origin.
Inches.	*Inches.*	*Inches.*
¾	2	3
1	3	4
1–¼	4	5
1–½	5	6

TEST FOR PERCENTAGE OF SHALE IN GRAVEL.

It is suggested that for the separation of shale and other pieces having low specific gravity from concrete aggregate, a solution of zinc chloride ($ZnCl_2$) or some other satisfactory liquid having a specific gravity of approximately 1.95 be used. A sample of the pebbles should be first dried to constant weight at not over 110° C., then placed in a container partially filled with the solution. Agitate for five minutes, skim off the lighter materials, and then pour the solution through a sieve which will retain the pebbles. Repeat the operation until the entire sample has been separated. Dry to constant weight, measure the volume of retained material, and compute the percentage of volume of shale or other soft material.

TEST FOR QUANTITY OF CLAY AND SILT IN GRAVEL FOR HIGHWAY CONSTRUCTION.

(A. S. T. M. standard method, serial designation D 72–21, slightly modified.)

1. This is a test for the determination of the quantity of clay and silt in natural gravel to be used in highway construction.

2. The sample as received shall be moistened and thoroughly mixed, then dried to constant weight at a temperature between 100° and 110° C. (212° and 230° F.). *Method of test.*

3. The pan or vessel to be used in the determination shall be 12 inches (30.5 centimeters in diameter by not less than 4 inches (10.2 centimeters) deep, as nearly as may be obtained.

4. (*a*) A representative portion of the dry material weighing not less than 50 times the weight of the largest stone in the sample shall be selected from the sample and placed in the pan which has been dried and accurately weighed.

(*b*) Sufficient water shall be poured into the pan to cover the gravel. The gravel shall be agitated vigorously for 15 seconds with a trowel or stirring rod. After it has settled for 15 seconds the water shall be poured off into a tared evaporating dish,

care being taken not to pour off any sand. This process shall be repeated until the wash water is clear, a glass rod being used to stir the material for the last few washings. The pan and washed sand shall be dried to constant weight in an oven at a temperature between 100° and 110° C. (212° and 230° F.), weighed, and the net weight of sand determined.

Calculation of percentage. 5. The percentage of clay and silt shall be calculated from the formula:

$$\text{Percentage of clay and silt} = \frac{\text{Original weight} - \text{Weight after washing}}{\text{Original weight}} \times 100$$

6. For a check on the results, the wash water shall be evaporated to dryness and the residue weighed:

$$\text{Percentage of clay and silt} = \frac{\text{Weight of residue}}{\text{Original weight}} \times 100$$

TEST FOR QUANTITY OF CLAY AND SILT IN SAND FOR HIGHWAY CONSTRUCTION.

(A. S. T. M. standard method, serial designation D 74–21, slightly modified.)

1. This is a test for the determination of the quantity of clay and silt in natural sand to be used in highway construction.

Method of test. 2. The sample as received shall be moistened and thoroughly mixed, then dried to constant weight at a temperature between 100° and 110° C. (212° and 230° F.).

3. The pan or vessel to be used in the determination shall be substantially 9 inches (22.9 centimeters) in diameter by not less than 4 inches (10.2 centimeters) deep. It shall have vertical sides and shall be provided with a pouring lip.

4. A representative portion of the dry material weighing 500 grams shall be selected from the sample and placed in the pan which has been dried and accurately weighed. Sufficient water shall be poured into the pan to cover the sand (about 225 cubic centimeters) and agitated vigorously for 15 seconds. After it has settled for 15 seconds the water shall be poured off into a tared evaporating dish, care being taken not to pour off any sand. This process shall be repeated until the wash water is clear, a glass rod being used to stir the material for the last few washings. The pan and washed sand shall be dried to constant weight in an oven at a temperature between 100° and 110° C. (212° and 230° F.), weighed, and the net weight of sand determined.

Calculation of percentage. 5. The percentage of clay and silt shall be calculated from the formula:

$$\text{Percentage of clay and silt} = \frac{\text{Original weight} - \text{Weight after washing}}{\text{Original weight}} \times 100$$

6. For a check on the results, the wash water shall be evaporated to dryness and the residue weighed.

$$\text{Percentage of clay and silt} = \frac{\text{Weight of residue}}{\text{Original weight}} \times 100$$

TESTS FOR APPARENT SPECIFIC GRAVITY AND ABSORPTION OF STONE OR OTHER COARSE MATERIALS.

(1) The apparent specific gravity shall be obtained by weighing the water displaced by a sample of the material weighing approximately 1,000 grams, broken into pieces about 1½ inches in diameter. The vessel to be used consists of a galvanized-iron cylinder closed at one end and measuring 5 inches in diameter by 8 inches high. A brass spout one-half inch in diameter is soldered into the side of the cylinder 6 inches from the bottom. The spout is inclined at an angle of 2° with the horizontal and

is 2½ inches long. A notch is filed across its lower end, to stop the drip from the displaced water. To determine the specific gravity and absorption, the dried and cooled sample shall be weighed to the nearest 0.5 gram and immersed in water for 24 hours. The pieces shall then be surface-dried individually with a towel, the sample reweighed and immediately placed in the cylinder, which has been previously filled to overflowing with water at room temperature.

(2) The weight of water displaced by the sample shall be used to calculate its apparent specific gravity. The difference between the original weight of the sample and its weight after 24 hours shall be used to determine the absorption.

TEST FOR MECHANICAL ANALYSIS OF AGGREGATES FOR CONCRETE.

(A. S. T. M. standard method, serial designation C 41–22, slightly modified.)

Selection of sample.

1. A representative test sample of the aggregate shall be selected, by quartering or by use of a sampler, which after drying will give not less than the following:

(*a*) Fine aggregate, 500 grams.

(*b*) Coarse aggregate or a mixture of fine and coarse aggregates, weight in grams, 3,000 times size of largest sieve required, measured in inches.

TABLE 1.

Sieve number or size in inches.[1]	Sieve opening.		Wire diameter.		Tolerance.		
					Average opening.	Wire diameter.	Maximum opening.
	Inches.	*Mm.*	*Inches.*	*Mm.*	*Per cent.*	*Per cent.*	*Per cent.*
No. 100	0.0059	0.149	0.0040	0.102	6	20	40
No. 50	.0117	.297	.0074	.188	6	20	40
No. 30	.0232	.59	.0130	.33	4	10	25
No. 16	.0469	1.19	.0213	.54	3	10	10
No. 8	.0937	2.38	.0331	.84	3	10	10
No. 4	.187	4.76	.050	1.27	3	10	10
⅜ inch	.375	9.5	.092	2.33	3	10	10
¾ inch	.75	19.0	.135	3.42	3	10	10
1 inch	1.00	25.4	.162	4.12	3	10	10
1½ inches	1.50	38.0	.177	4.50	3	10	10
2 inches	2.00	50.8	.192	4.88	3	10	10
3 inches	3.00	76.0	.25	6.3	3	10	10

[1] Sieves No. 100 to No. 4 are based on "Table of Fundamental Data on Standard Specifications for Sieves," issued by the U. S. Bureau of Standards, 1920. The liberal tolerances will permit the use of certain sieves which do not exactly correspond to the numbers given in the table.

Method of test.

2. The sample shall be dried at not over 110° C. (230° F.) to constant weight.

3. (*a*) The sieves shall be of square-mesh wire cloth and shall be mounted on substantial frames constructed in a manner that will prevent loss of material during sifting.

(*b*) The size of wire and sieve openings shall be as given in Table 1.

4. (*a*) The sample shall be separated into a series of sizes by means of the sieves specified in section 3. Sifting shall be continued until not more than 1 per cent by weight of the sample passes any sieve during one minute.

(*b*) Each size shall be weighed on a balance or scale which is sensitive to one one-thousandth of the weight of the test sample.

(*c*) The percentage by weight of the total sample which is finer than each of the sieves shall be computed.

5. (*a*) The percentages in sieve analysis shall be reported to the nearest whole number.

(*b*) If more than 15 per cent of a fine aggregate is coarser than the No. 4 sieve, or more than 15 per cent of a coarse aggregate is finer than the No. 4 sieve, the sieve analysis of the portions finer and coarser than this sieve shall be reported separately.

NOTE.—It is recommended that the one-quarter-inch sieve be used rather than the No. 4 sieve and such additional U. S. Bureau of Standard sieves as may be decided as test limits. The Tyler standard screens 100, 48, 28, 14, 8, 4 and three-eighth-inch come within the tolerance allowed for the Bureau of Standard screens 100, 50, 30, 16, 8, 4 and three-eighth-inch.

TEST FOR APPARENT SPECIFIC GRAVITY OF SAND, STONE, AND SLAG SCREENINGS, AND OTHER FINE NONBITUMINOUS HIGHWAY MATERIALS.

(A. S. T. M. standard method, serial designation D 55–19, slightly modified.)

1. The following tests, "Le Chatelier" and "Jackson," are equally suited for use in determining the apparent specific gravity of sand, stone, and slag screenings and other fine nonbituminous highway materials, and may be considered as alternates.

LE CHATELIER TEST.

Le Chatelier test.

2. The determination of specific gravity shall be made with a standardized Le Chatelier apparatus. This apparatus is standardized by the United States Bureau of Standards. Kerosene, free from water, or benzene, not lighter than 62° Baumé, shall be used in making this determination.

3. (*a*) The flask shall be filled with either of these liquids to a point on the stem between zero and 1 cubic centimeter and 64 grams of sand or other fine nonbituminous highway material of the same temperature as the liquid shall be slowly introduced, taking care that the material does not adhere to the inside of the flask above the liquid and to free the material from air by rolling the flask in an inclined position. After all material is introduced the level of the liquid will rise to some division of the graduated neck; the difference between readings is the volume displaced by 64 grams of the material. The specific gravity shall then be calculated from the formula:

$$\text{Specific gravity} = \frac{\text{Weight of material (grams.)}}{\text{Displaced volume (centimeters.)}}$$

(*b*) The flask, during the operation, shall be kept immersed in water, in order to avoid variations in the temperature of the liquid in the flask, and any variation that occurs shall not exceed 0.5° C. The results of repeated tests should agree within 0.01.

JACKSON TEST.

Jackson test.

4. The determination shall be made with a Jackson specific-gravity apparatus which shall consist of a burette, with graduations reading to 0.01 in specific gravity, about 23 centimeters (9 inches) long and with an inside diameter of about 0.6 centimeter (0.25 inch), which shall be connected with a glass bulb approximately 13 centimeters (5.1 inches) long and 4.5 centimeters (1.75 inches) in diameter, the glass bulb being of such size that from a mark on the neck at the top to a mark on the burette just below the bulb, the capacity is exactly 180 centimeters (6.09 liquid ounces); and an Erlenmeyer flask, which shall contain a hollow ground-glass stopper having a neck of the same bore as the burette and a capacity of exactly 200 centimeters (6.76 ounces) up to the graduation on the neck of the stopper.

5. The method is as follows:

(1) Dry at not more than 110° C. (230° F.) to constant weight a sample weighing about 55 grams; (2) weigh 50 grams of the dry sample to 0.1 gram and pour it into the unstoppered Erlenmeyer flask, which shall be cleaned and dried before each determination; (3) fill the bulb and burette with kerosene, leaving just space enough to take the temperature by introducing a thermometer through the neck; (4) remove the thermometer and add sufficient kerosene to fill exactly to the mark on the neck, drawing off any excess with the burette; (5) run into the flask about one-half of the kerosene in the bulb to remove air bubbles and then run in more kerosene, removing any material adhering to the neck of the flask, until the kerosene is just below the ground glass; (6) place the hollow ground-glass stopper in position and turn it to fit tightly, and then run in kerosene exactly to the 200 cubic centimeter (6.76 ounces) graduation on the neck, care being taken to remove all air bubbles in the flask; (7) read the specific gravity from the graduation on the burette, and the temperature of the oil in the flask, noting the difference between the temperature of the oil in the bulb before the determination and that of the oil in the flask after the determination; (8) make a temperature correction to the reading of the specific gravity in accordance with the table furnished by the manufacturer of the apparatus, adding the correction if the temperature of the kerosene has increased and subtracting it if the temperature of the kerosene has decreased.

TEST FOR UNIT WEIGHT OF AGGREGATE FOR CONCRETE AND PERCENTAGE OF VOIDS.

(A. S. T. M. standard method, serial designation C 29–21, slightly modified.)

1. The unit weight of fine, coarse, or mixed aggregates for concrete shall be determined by the following method:

2. (*a*) The apparatus required consists of a cylindrical metal measure, a tamping rod, and a scale or balance, sensitive to 0.5 per cent of the weight of the sample to be weighed. *Apparatus.*

(*b*) *Measures.*—The measure shall be of metal, preferably machined to accurate dimensions on the inside, cylindrical in form, water-tight, and of sufficient rigidity to retain its form under rough usage, with top and bottom true and even, and preferably provided with handles.

The measure shall be one-tenth, one-half, or 1 cubic foot capacity, depending on the maximum diameter of the coarsest particles in the aggregate, and shall be of the following dimensions:

Capacity.	Inside diameter.	Inside height.	Diameter of largest particles of aggregate.
	Inches.	*Inches.*	*Inches.*
$\frac{1}{10}$ cubic foot	6.00	6.10	Under $\frac{1}{2}$
$\frac{1}{2}$ cubic foot	10.00	11.00	Under $1\frac{1}{2}$
1 cubic foot	14.00	11.23	Over $1\frac{1}{2}$

(*c*) *Tamping rod.*—The tamping rod shall be a straight metal rod three-fourths inch in diameter and 18 inches long, with one end tapered for a distance of 1 inch to a blunt, bullet-shaped point.

3. The measure shall be calibrated by accurately determining the weight of water at 16.7° C. (62° F.) required to fill it. The factor for any unit shall be obtained by dividing the unit weight of water at 16.7° C. (62° F.)[5] by the weight of water at 16.7° C. (62° F.) required to fill the measure.

[5] The unit weight of water at 16.7° C. (62° F.) is 62.355 pounds per cubic foot.

Method of test. 4. The sample of aggregate shall be room-dry and thoroughly mixed.

5. (*a*) The measure shall be filled one-third full and the top leveled off with the fingers. The mass shall be tamped with the pointed end of the tamping rod 25 times, evenly distributed over the surface. The measure shall then be filled two-thirds full and again tamped 25 times as before. The measure shall then be filled to overflowing, tamped 25 times, and the surplus aggregate struck off, using the tamping rod as a straight edge.

In tamping the first layer the rod should not be permitted forcibly to strike the bottom of the measure. In tamping the second and final layers, only enough force to cause the tamping rod to penetrate the last layer of aggregate placed in the measure should be used. No effort should be made to fill holes left by the rod when the aggregate is damp.

(*b*) The net weight of the aggregate in the measure shall be determined. The unit weight of the aggregate shall then be obtained by multiplying the net weight of the aggregate by the factor found as described in section 3.

6. Results with the same sample should check within 1 per cent.

7. The percentage of voids in the aggregate may be determined from the weight per cubic foot and the specific gravity in the usual manner.

TEST FOR ORGANIC IMPURITIES IN SANDS FOR CONCRETE.

(A. S. T. M. standard method, serial designation C 40–22, slightly modified.)

1. The test herein specified is an approximate test for the presence of injurious organic compounds in natural sands for cement mortar or concrete. The principal value of the test is in furnishing a warning that further tests of the sand are necessary before they are used in concrete. Sands which produce a color in the sodium hydroxide solution darker than the standard color should be subjected to strength tests in mortar or concrete before use.

Method of test. 2. (*a*) A representative test sample of sand weighing about 1 pound shall be obtained by quartering or by the use of a sampler.

(*b*) A 12-ounce graduated glass prescription bottle shall be filled to the 4½-ounce mark with the sand to be tested.

(*c*) A 3 per cent solution of sodium hydroxide (NaOH) in water shall be added until the volume of sand and liquid after shaking gives a total volume of 7 liquid ounces.

(*d*) The bottle shall be stoppered and shaken thoroughly and then allowed to stand for 24 hours.

(*e*) A standard color solution shall be prepared by adding 2.5 cubic centimeters of a 2 per cent solution of tannic acid in 10 per cent alcohol to 22.5 cubic centimeters of a 3 per cent sodium hydroxide solution. This shall be placed in a 12-ounce prescription bottle, stoppered, and allowed to stand for 24 hours, then 25 cubic centimeters of water added.

(*f*) The color of the clear liquid above the sand shall be compared with the standard color solution prepared as in paragraph (*e*) or with a glass of color similar to the standard solution.

3. Solutions darker in color than the standard color have a "color value" higher than 250 parts per million in terms of tannic acid.

NOTE.—Special attention is called to the fact that this test is a warning that additional tests of the sand may be necessary.

COMPRESSION AND TENSION TESTS OF FINE AGGREGATE FOR CONCRETE.

1. Fine aggregate shall be tested for strength either by tension or compression in a mortar proportioned either by volume or by weight, using a 1:3 mix.

2. Mortars made of natural sand shall be compared to mortars made with the same cement and standard Ottawa sand mixed in the same proportion and of the same consistency.

I. TENSION TEST.

Tension test specimens.

3. The briquets for the tension test shall be molded in the standard mold as used for testing Portland cement. Single or gang molds may be used. They shall be wiped with an oily cloth before using.

4. Immediately after mixing, the mortar shall be placed in the molds, pressed in firmly with the thumbs, and smoothed off with a trowel without ramming. Additional mortar shall be heaped above the mold and smoothed off with a trowel; the trowel shall be drawn over the mold in such a manner as to exert a moderate pressure on the material. The mold shall then be turned over and the operation of heaping, thumbing, and smoothing off repeated.

Method of test.

5. Tests shall be made with any standard machine. The briquets shall be tested as soon as they are removed from the water. The bearing surfaces of the clips and briquets shall be free from grains of sand or dirt. The briquets shall be carefully centered and the load applied continuously at the rate of 600 pounds per minute.

6. Testing machines shall be calibrated frequently in order to determine their accuracy.

7. Briquets that are manifestly faulty, or which give strengths differing more than 15 per cent from the average value of all test pieces made from the sample and broken at the same period, shall not be considered in determining the tensile strength.

II. COMPRESSION TEST.

Compression test specimens.

8. A cylindrical test piece 2 inches in diameter and 4 inches in length shall be used in making the compression test. The molds (single or gang molds) shall be made of noncorroding metal. The ends of the mold shall be parallel. They shall be oiled before using. During the molding of the test piece, the mold shall rest on a clean, plane surface (preferably a piece of plate glass which is allowed to remain in place until the mold is removed).

9. The mortar shall be placed in the molds in four layers about 1 inch in thickness, each layer being thoroughly compacted, but not rammed by a steel tamper. The steel tamper shall have a total length of about 6½ inches. It shall be 1 inch in diameter for 1¼ inches with the handle 5¼ inches long and five-eighths inch in diameter. It shall weigh approximately three-quarters of a pound. The surface of each layer shall be roughened before the addition of the next layer. In compacting the test pieces no mixing water shall be forced out of the mortar. In finishing the test piece, mortar shall be heaped above the mold and smoothed off with a trowel. As soon as the test pieces from one sample are molded, the top of each mold shall be covered with a piece of glass which shall be brought to a firm bearing on the fresh mortar. The cover glasses shall remain in place until the molds are removed.

Method of test.

10. Tests of mortar cylinders shall be made in any testing machine which is adapted to meet the specified requirements. The test pieces shall be broken as soon as they are removed from the water. The ends of the test cylinders shall be smooth, plane surfaces. The metal bearing plates of the testing machine shall be placed in direct contact with the ends of the test piece. During the test a spherical bearing block shall be used on top of the cylinder. In order to secure a uniform distribution of the load over the test cylinder, the spherical block must be accurately centered. The diameter of the spherical bearing block should be only a little greater than that of the test piece. The test piece

shall be loaded continuously to failure. The moving head of the testing machine shall travel at the rate of not less than 0.05 or more than 0.10 inch per minute.

11. Testing machines should be frequently calibrated in order to determine their accuracy.

12. Cylinders that are manifestly faulty, or which give strengths differing more than 15 per cent from the average value of all test pieces tested at the same period and made from the same sample, shall not be considered in determining the compressive strength.

III. STORAGE OF TEST PIECES.

Storage.

13. The moist closet may consist of a soapstone, slate, or concrete box, or a wooden box lined with metal. If a wooden box is used, the interior should be covered with felt or broad wicking kept wet. The bottom of the moist closet should be covered with water. The interior of the closet should be provided with nonabsorbent shelves on which to place the test pieces, the shelves being so arranged that they may be withdrawn readily.

14. Unless otherwise specified, all test pieces, immediately after molding, shall be placed in the moist closet for from 20 to 24 hours.

15. Cylinders or briquets shall be kept in molds on glass plates in the moist closet for at least 20 hours. After 24 hours in moist air the briquets or cylinders shall be immersed in clean water in storage tanks of noncorroding material.

16. The air and water shall be maintained as nearly as practicable at a temperature of 21° C. (70° F.)

NOTE.—It is recommended that the laboratory method of proportioning fine aggregate viz, by weight or by volume, for either the compression or tension test shall be the same as the method used in proportioning concrete in the field.

When the mortar is proportioned by volume the required quantity of fine aggregate and cement shall be determined from the unit weight of the material.

Example:

Weight of sand, 100 pounds per cubic foot.

Weight of cement, 94.0 pounds per cubic foot.

Required, 75 cubic inches of mortar.

$$\text{Quantity of sand, } \frac{75}{1728} \times 100 \times 453.6 = 1{,}969 \text{ grams.}$$

$$\text{Quantity of cement, } \frac{25}{1728} \times 94.0 \times 453.6 = 617 \text{ grams.}$$

METHOD OF SAMPLING AND TESTING SEMIGRAVEL, TOPSOIL, AND SAND-CLAY AGGREGATE FOR ROAD SURFACING.

I. DEFINITION.

1. The terms, clay, silt, sand, and coarse material, used herein, are defined as follows:

NOTE.—The definitions here given are intended to apply to these terms only when used in connection with roads of these types.

Clay.—Material separated by subsidence through water and possessing plastic or adhesive properties, generally less than 0.02 millimeter in diameter.

Silt.—Fine material, other than clay, which passes a 200-mesh sieve, generally from 0.07 to 0.02 millimeter in diameter.

Sand.—Hard material, usually siliceous, which passes a 10-mesh sieve and is retained on a 200-mesh sieve, generally from 1.85 to 0.07 millimeter in diameter.

Coarse material.—Hard material of gravelly nature, retained on a 10-mesh sieve, i. e., more than 1.85 millimeters in diameter.

II. METHOD OF SAMPLING.

2. Samples of materials of this class shall be of two kinds:

Class I. Samples of the raw material taken from the natural deposits.

Class II. Samples of the loose material after being mixed in place on the roadbed and before consolidation.

3. Class I samples shall be used for preliminary tests as to the suitability of the aggregate subject to admixture of one or more ingredients to adjust the composition to the limits set forth in the specifications.

4. The final acceptance of the material as satisfying the specifications shall be based on Class II samples.

5. *Standard containers.*—(1) A three-compartment box of pasteboard, wood, or metal, outside dimensions 5 by 10 by 10 inches. (2) Close-woven bags or sacks of material which do not allow sifting out of fine particles, dimensions 6 inches wide by 12 inches long.

Labeling samples.

6. *Labeling.*—Each compartment in the box container must contain a label showing at what depth the contents were taken. The whole sample shall be accompanied by a card, securely attached thereto, stating date, by whom taken, by whom submitted, source of supply, exact location where sample was taken, position within the deposit where taken, owner, quantity available, amount and character of stripping, if any, whether material from same source has been previously used, where, and with what results, haul to nearest point on road, average haul to job, character of haul, initial cost of material.

7. When bag containers are used, one complete sample shall comprise three bags, each bag labeled as to depth from which the material was taken.

8. Each bag, or, if preferred, a larger receptacle containing the three bags, is to be labeled with the information detailed above.

Class I samples.

9. *Method of taking Class I samples.*—For each acre or less of area, two samples must be taken, one a local sample and the other a composite sample.

10. The local sample is to be taken near the center of the area, and is intended to represent the vertical average of the material at the point. It shall be taken in three layers, each layer 4 inches thick, according to the method described as follows:

(*a*) The material is to be loosened over a 3 by 3 foot area to a specified depth, usually 4 inches. The loose material is to be intermixed with a shovel and the sample for one compartment of the box container or one of the bags is to be taken therefrom.

(*b*) The remaining loose material is to be shoveled out and discarded. The second layer is to be loosened to equal depth, usually 4 inches, to be intermixed as before, and a second compartment or bag is to filled. The same procedure shall apply to the third layer and the filling of the third compartment or bag.

(*c*) In exceptionally thick deposits the depth of each layer or the number of layers may be increased to cover the entire thickness of the deposit.

11. The composite sample is to taken as follows:

(*a*) Roughly divide the area to be represented by the sample into squares not exceeding 50 feet in size. At the corners of all squares loosen a 3 by 3 foot area to a depth of —[6] inches. Thoroughly mix the loose material. Carry an equal amount of the material from each such point to a central point and intimately mix the various samples. Not less than 200 pounds of material must be so mixed. From the center of the pile of mixed material fill a container and label for shipment.

(*b*) Where the material occurs as a substratum, sink no less than four 3 by 3 foot pits per acre, or smaller area, to intersect

[6] Depth of 8 inches is suggested.

material. Remove the covering and sample the exposed bed as for a local sample described above.

Class II samples.

12. *Method of taking Class II samples.*—These are the most important samples, and should be taken by the engineer or competent inspector while work is in progress.

(*a*) When the materials have been spread and intimately mixed in accordance with properly drawn clauses covering methods of construction, the engineer should fill a container at intervals of —[7] feet, along the road, and also at such other points as his judgment may dictate, where evidence of unsatisfactory mixing is apparent.

(*b*) Very prompt examination of these samples should be made in order that defects of composition may be remedied by the builder before consolidation has progressed.

III. METHOD OF TEST.

Analysis.

13. Dry 500 grams of the material at a temperature of 212° F. (100° C.) to constant weight. Gently pulverize to break down soft clods or masses, but not grind or break hard material. Pass through a 10-mesh sieve, weigh the material retained and record as "coarse material." Use the material passing the 10-mesh sieve as the starting point of a percentage analysis as follows:

(*a*) Weigh out two samples of 50 grams of this material for duplicate analysis. Place each in a tared wide-mouth bottle (5 to 6 centimeters diameter and about 12 to 15 centimeters high.)

(*b*) Mix with distilled water (about 200 cubic centimeters) and heat to simmering temperature for one hour. Allow subsidence for eight minutes and siphon to a depth of 8 centimeters from the surface of the liquid. (The depth of the liquid should be sufficient to leave about 4 centimeters below the point of siphoning.) Add distilled water and 5 cubic centimeters of diluted ammonia (1 : 500), brush the material with a stiff brush, stir thoroughly, allow to settle for eight minutes, and siphon to a depth of eight centimeters from the surface. Continue this process until the supernatant liquid is clear. Be careful to wash the stopper and neck of the bottle free from coarse material before decanting. The washings may be drawn off, collected and evaporated to dryness for direct recovery of the fine sediment classed as "Total clay." Ordinarily the sediment loss or "total clay" is computed as a difference.

NOTE.—The "total clay" may be separated further by the centrifuge into "clay" and "suspension clay" when desired. See methods used by the Bureau of Public Roads in its examination of subgrade materials.

14. Wash the contents of the bottle cleanly into a porcelain evaporating dish and carry to dryness on a water bath. The dried residue should be carefully scraped from the dish and passed through a nest of 20, 60, and 200 mesh sieves. The residue retained on each sieve is weighed and recorded as sand of the respective sizes. Their sum constitutes the total "sand." The residue passing the 200-mesh sieve and caught in the pan is weighed and recorded as "silt." Duplicate samples should check within 1 per cent.

Character of material.

(*a*) The coarse material should be examined for hardness and with the magnifying glass to identify its character as quartz, hard iron compounds, feldspar, schistose material, or indurated clay. Hard quartz gravels are valuable in themselves and as indicating the quality of the finer aggregate. Feldspar, mica, and clay nodules are worthless, and indicate that the accompanying soil is poor for road building.

(*b*) The sands should be examined with the magnifying glass for identification, as quartz, and for the presence of mica scales

[7] Intervals of 500 feet are suggested.

or feldspar. If mica or feldspar is present in appreciable amounts, the sample should be rejected.

(*c*) When the clay is recovered by evaporation, it can be examined for tenacity by cementing together two glass plates, each 1 inch wide set at right angles, with a layer of clay whose thickness is fixed by a fine bent wire laid between the plates. The moist clay covers the wire on one plate and the other plate is squeezed down tightly on the wire. After drying, the one plate being held firmly against cleats, wire slings are run symmetrically from the ends of the upper plate to one arm of a beam balance and the tension necessary to separate the plates is given by shot or weights in the other pan of the balance.

NOTE.—This test is tedious and is of service chiefly on low-grade samples which are of doubtful efficiency, but which represent the only available material for local construction.

Tenacity of clay.

(*d*) Approximate tests for tenacity of mixture can be made as follows:

(1) Make cylinders from the material passing the 10-mesh sieve, 25 by 25 millimeters. The material is worked into a stiff mud, and molded under 132 kilograms per square centimeter pressure. Dry thoroughly at 100° C. (212° F.) and break by means of the small Page impact machine using a 1 kilogram hammer and 1 centimeter drop. Record the number of strokes as the relative measure of tenacity.

(2) Mix 50 grams of the material passing the 10-mesh sieve with —[8] grams of water and knead with the hands into a spherical ball. Measure the diameter. Let this ball drop from a height of —[8] centimeters on a flat slab. Measure and record the reduction in diameter and examine the surface for cracks.

(3) Usually the plastic character and adhesiveness of a good road soil can be judged by the feeling of the mud made from this material, its adherence to the hands, and its stretch under light pulling.

METHOD OF PROPORTIONING NATURAL SAND AND GRAVEL DEPOSITS FOR CONCRETE CONSTRUCTION.

For determining the amount of cement to make concrete equivalent to any base mixture, when the percentage of sand in the total aggregate varies from the base mixture—

Rule.—Decrease or increase the ratio by weight of total aggregate to cement by 0.05 times the increase, or decrease in the percentage of sand in the total aggregates by weight. This involves the following steps in the process:

1. If the base mixture is stated as a relation by volume, it will be necessary to transfer the proportion to a statement of relation by weight. This requires that the weights per unit of loose volume of the various materials be known or assumed.

2. Determine the percentage of fine aggregate in the total aggregate in the base mixture and in the new mixture to be used, using the weight proportions.

3. Multiply the difference between these two percentages by 0.05 and subtract the result from the sum of the parts of fine and coarse aggregates in the weight proportion for the base mixture if the percentage of fine aggregate is greater in the new mixture. If the percentage of fine aggregate is less in the new mixture, add instead of subtracting.

4. If screened materials are to be used, divide the parts of total aggregate determined above into fine and coarse aggregate according to the percentage of fine aggregate in the new mixture.

5. Transfer the resulting weight proportion into a statement of relation by loose volumes.

[8] No definite weight of water or height of fall is recommended. See Bulletin of University of Georgia, June, 1922, Volume XXII, No. 5a.

Example:

Assume—Base mix, 1 : 2 : 3½ by volume.

Unit weights—

Fine aggregate, 95 pounds per cubic foot.
Coarse aggregate, 110 pounds per cubic foot.
Cement, 94 pounds per cubic foot.

Percentage of fine aggregate in new mixture=60.

1. 1 : 2 : 3½=94 : 2 times 95 : 3½ times 110.
   =94 : 190 : 385 proportion by weight.
   =1 : 2.02 : 4.10=1 : 6.12.
2. Percentage of sand in base mixture aggregate=

$$\frac{190}{190+385}\text{ times }100=33$$

Percentage of sand in new mixture=60.

3. 60—33=27.
   27 times 0.05=1.35
   6.12—1.35=4.77, therefore the new proportion by weight is 1 : 4.77.
4. Since 60 per cent of the total aggregate is to be fine aggregate,
   1 : 4.77=1 : 2.86 : 1.91
5. 1 : 2.86 : 1.91=94 pounds : 269 pounds : 180 pounds.
   Dividing by the unit weights of materials.

$$\frac{94}{94}:\frac{269}{95}:\frac{180}{110}=1\text{ cubic foot}:2.83\text{ cubic feet}:1.64\text{ cubic feet.}$$

Or if unscreened gravel is to be used, determine the unit weight of the gravel. Assume for this case 115 pounds per cubic foot.

Then 1 : 4.77=94 pounds to 449 pounds.

Dividing by unit weights,

$$\frac{94}{94}:\frac{449}{115}=1\text{ cubic foot}:3.90\text{ cubic feet.}$$

This method applies to either screened or unscreened mixtures of sand and gravel in which the specific gravities of the fine and coarse aggregate are approximately equal, and to percentages of fine aggregate greater than 33.

NOTE.—The method can be applied to mixtures of materials of different specific gravities, such as sand and crushed limestone, by reducing the proportions to relations by absolute volume and making the adjustment upon that basis.

Recommended practice in the use of unscreened gravel.

1. The fine aggregate portion must pass the user's specification for fine aggregate.
2. The coarse aggregate portion must pass the user's specification for coarse aggregate.
3. Unscreened gravel should not be used unless in the opinion of the engineer in charge, the amount of sand in the aggregate will not vary more than 10 per cent.
4. Unscreened gravel should only be used from stock piles containing the largest practicable amount of material.

Reference.—For a theoretical discussion of the method of proportioning see Bulletin No. 60, Engineering Experiment Station, Iowa State College. For verification see paper by R. W. Crum, Proceedings American Society for Testing Materials, 1922.

COMPRESSION TEST OF CONCRETE.

(A. S. T. M. tentative method, serial designation C 39–21 T, modified.)

1. These methods are intended to cover compression tests of concrete made in a laboratory where accurate control of quantities of materials and test conditions is possible. They are designed to apply primarily to hand-mixed concrete. These methods

may be used with slight modification in making tests for concrete for wearing resistance, bond between concrete and steel, impermeability, etc. The investigation of machine-mixed concrete will require certain obvious changes in the method.

2. Materials shall be brought to room temperature (65° to 70° F.) before beginning tests. Cement shall be stored in a dry place, preferably in covered metal cans. The cement shall be thoroughly mixed in advance, in order that the sample may be uniform throughout the tests. It shall be sifted through a No. 16 sieve and all lumps rejected. Aggregates shall be in a room-dry condition when used in concrete tests.

Tests of materials.

3. Representative samples of all concrete materials shall be secured for preliminary tests prior to the proportioning and mixing of the concrete. Cement test samples may be made up of a small quantity from each sack used in the concrete tests. Test samples of aggregates may be taken from larger lots by quartering.

4. Cement shall be subjected to test, using the methods described in the standard specifications and tests for Portland cement.

5. Fine aggregates shall be subjected, when required, to the following tests:

(*a*) Sieve analysis test made in accordance with the test for mechanical analysis of aggregates for concrete.

(*b*) Test for organic impurities (natural sand only) made in accordance with the test for organic impurities in sands for concrete.

(*c*) Test for quality of silt, clay, or dust made in accordance with the test for quantity of clay and silt in sand for highway construction.

(*d*) Test for unit weight made in accordance with the test for unit weight of aggregate for concrete and percentage of voids.

(*e*) Strength test of 1:3 mortar at 7 and 28 days in comparison with standard sand mortar, in accordance with the compression and tension tests of fine aggregate for concrete.

6. Coarse aggregates shall be subjected, when required, to the following tests:

(*a*) Sieve-analysis test as specified under section 5 (*a*).

(*b*) Test for quantity of silt, clay, or dust, in accordance with the test for quantity of clay and silt in gravel for highway construction.

(*c*) Test for unit weight as specified under section 5 (*d*).

7. The unit weight of mixed fine and coarse aggregates as used in concrete tests shall be determined in accordance with the method specified in section 5 (*d*).

8. The quantities of each size of aggregate to be used in each batch shall be determined on the basis of the sieve analysis and the unit weight of the mixed aggregate. The exact quantities of cement and of each size of aggregate for each batch shall be determined by weight. The quantity of water for each batch shall be accurately measured. The quantities of materials may be expressed as (*a*) 1 volume of cement to – volumes of total aggregate mixed as used, or (*b*) 1 volume of cement, – volumes fine aggregate, and – volumes of coarse aggregate.

Preparation of test specimen.

9. Compression tests of concrete shall be made on cylinders of a diameter equal to one-half the length. The standard shall be 6 by 12 inch cylinders where the coarse aggregate does not exceed 2 inches in size; for aggregates larger than 2 inches, 8 by 16 inch cylinders shall be used; 2 by 4 inch cylinders may be used for mixtures without coarse aggregate.

10. (*a*) Concrete shall be mixed by hand in batches of such size as to leave a small quantity of concrete after molding the test pieces. The batch shall be mixed as follows:

(*b*) The cement and fine aggregate shall be mixed dry until the mixture is homogeneous in color.

(*c*) The coarse aggregate shall be added and mixed dry.

(*d*) Sufficient water shall be added to produce concrete of the required workability.

NOTE.—Concrete tests should be made on plastic mixtures. It is of the utmost importance that a uniform degree of workability be secured in tests involving comparisons of different materials and methods.

(*e*) The whole shall be mixed thoroughly until the entire mass is homogeneous in appearance.

11. The workability or plasticity of each batch of concrete shall be measured immediately after mixing by one of the following methods:

Slump test. (*a*) Slump test, made in accordance with the test for consistency of Portland cement concrete (A. S. T. M. tentative method, serial designation D 138–22 T).

Flow test. (*b*) Flow test, made by placing a metal form in the shape of a frustum of a cone $6\frac{3}{4}$ inches in top diameter, 10 inches in bottom diameter, and 5 inches deep, on the table of the flow apparatus. The fresh concrete shall be placed in the mold in two layers. Each layer shall be puddled and finished as described in section 13. Immediately after molding, the form shall be removed by a steady upward pull; the specimen raised one-half inch and dropped 15 times in about six seconds by means of a suitable cam and crank. The spread of the fresh concrete due to this treatment as compared with the original bottom diameter of the cone, expressed as a percentage, is the "flow."

Forms. 12. The forms shall preferably be of metal. Each form shall be provided with a machined metal base plate, and shall be oiled with a heavy mineral oil before using. Particular care shall be taken to obtain tight forms so that the mixing water will not escape during molding.

NOTE:—Satisfactory forms can be made from lengths of cold-drawn steel tubing, split along one element and closed by means of a circumferential band and bolt, lengths of steel water pipe machined on the inside, from rolled metal plates, from galvanized steel, machined iron or steel castings. Paraffined cardboard molds will give good results under expert supervision.

13. Concrete test pieces shall be molded by placing the fresh concrete in the form in layers 3 to 4 inches in thickness. Each layer shall be puddled with 25 strokes of a five-eighths inch round steel bar of a length 9 inches greater than the length of the mold, pointed at the lower end. After the top layer has been puddled, the surplus concrete shall be cleaned off with a trowel, and the mold covered with a machined metal plate or a piece of plate glass at least one-quarter inch thick, which will be used later in capping the test piece.

Capping. 14. Two to four hours after molding, the test pieces shall be capped with a thin layer of stiff, neat, cement paste in order that the cylinders may present a smooth end for loading. The cap shall be formed by means of a machined metal plate or a piece of plate glass of suitable size, at least one-quarter inch thick, worked down on the fresh cement paste until it rests on the top of the cylinder form. The cement for capping shall be mixed to a stiff paste before beginning to mix the concrete; in this way the tendency of the cap to shrink will be largely eliminated. The adhesion of the concrete to the metal base plate and the glass can be largely eliminated by oiling the cover plate or by inserting a sheet of paraffined tissue paper.

15. Concrete test pieces shall be removed from the forms 20 to 48 hours after molding, marked, weighed, and stored in damp sand, under damp cloths, or in a moist chamber until the date of test. The temperature of the curing room should not fall outside the range of 60° to 75° F.

16. Tests shall be made at the age of 7 and 28 days; ages of 3 months and 1 year are recommended, if longer time tests are required.

17. Three to five test pieces should be made in investigations in which accurate comparisons are desired.

Method of test.

18. Compression tests shall be made immediately upon removal of the concrete test pieces from the curing room; that is, the test pieces shall be loaded in a damp condition. The length and average diameter of the test piece shall be measured in inches and hundredths; two diameters shall be measured at right angles near the mid length. The test piece shall be weighed immediately before testing.

19. In general, only the ultimate compression strength of the cylinders need be observed. The metal bearing plates of the testing machine shall be placed in contact with the ends of the test piece; cushioning materials shall not be used. An adjustable bearing block shall be used to transmit the load to the test piece. The bearing block shall be placed on top of the test piece in vertical testing machines. The diameter of the bearing block shall be approximately the same as that of the test piece. The upper section of the bearing block shall be kept in motion as the head of the testing machine is brought to a bearing on the test piece.

20. The load shall be applied uniformly and without shock. The moving head of the testing machine should travel at the rate of about 0.1 inch per minute when the machine is running idle.

21. The total load indicated by the testing machine at failure of the test piece shall be recorded and the unit compressive strength calculated in pounds per square inch, the area computed from the average diameter of the cylinder being used. The type of failure and appearance of the concrete shall be noted.

Weight.

22. The weight of the concrete in pounds per cubic foot shall be determined from the weight of the specimens and their dimensions.

Density.

23. Density and yield of concrete when required shall be calculated from the unit volumes of the constituent materials and the volume of the concrete. Density is here understood to be the ratio of solids in the concrete to the total volume of the mass. Yield is the volume of concrete resulting from one volume of aggregate mixed as used.

Report of test.

24. The report of tests shall include the following:

(*a*) The kind and origin of concrete materials;

(*b*) Complete data on all tests of cement and aggregates;

(*c*) A description of methods of making and testing the concrete;

(*d*) The quantities of cement, aggregates, and water in each batch;

(*e*) The method of measuring workability or plasticity with "slump" or "flow" of concrete;

(*f*) The quantity of water expressed as a ratio to volume of cement;

(*g*) The age at test;

(*h*) The size of test pieces;

(*i*) The date of molding and testing each cylinder;

(*j*) The compression strength in pounds per square inch of each test piece and average of tests in a set;

(*k*) A description of the failure and appearance of the concrete in each test piece;

(*l*) The unit weight, density and yield of the concrete.

METHOD OF MAKING AND STORING SPECIMENS OF CONCRETE IN THE FIELD.

(A. S. T. M. standard method, serial designation, C 31–21, slightly modified.)

1. The methods herein specified apply to molding and storing of test specimens made from samples of concrete being used in construction.

2. The test specimens shall be cylindical in form with the length twice the diameter. In general, a mold whose diameter is not less than four times the diameter of the largest size aggregate shall

be used. (The sizes most commonly used are 6 by 12 inches and 8 by 16 inches.)

Molds.

3. (*a*) The molds shall be cylindrical in form, made of nonabsorbent material, and shall be substantial enough to hold their form during the molding of the test specimens. They shall not vary in diameter more than one-sixteenth inch in any direction, nor shall they vary in height more than one-sixteenth inch from the height required. They shall be substantially water-tight so that there will be no leakage of water from the test specimen during molding.

(*b*) Each mold shall be provided with a base plate having a plane surface and made of nonabsorbent material. This plate shall be large enough in diameter to support the form properly without leakage. Plate glass or planed metal are satisfactory for this purpose. A similar plate should be provided for covering the top surface of the test specimen after it is molded.

Sampling.

4. (*a*) Concrete for the test specimens shall be taken immediately after it has been placed in the work. All the concrete for each sample shall be taken from one place. A sufficient number of samples—each large enough to make one test specimen—shall be taken at different points so that the test specimens made from them will give a fair average of the concrete placed in that portion of the structure selected for tests. The location from which each sample is taken shall be noted clearly for future reference.

(*b*) In securing samples, the concrete shall be taken from the mass by a shovel or similar implement and placed in a large pail or other receptacle for transporting to the point of molding. Care shall be taken to see that each test specimen represents the total mixture of the concrete at that place. Different samples shall not be mixed together but each sample shall make one specimen.

5. (*a*) The pails or other receptacles containing the samples of concrete shall be taken as quickly as possible to the place selected for molding test specimens. To offset segregation of the concrete during transportation, each sample shall be dumped into a nonabsorbent, water-tight receptacle and, after slight stirring, immediately placed in the mold.

Preparation of specimen.

(*b*) The test specimens shall be molded by placing the concrete in the form in layers approximately 4 inches in thickness. Each layer shall be puddled with from 25 to 30 strokes of a five-eighths to three-quarter-inch bar about 2 feet long, tapered slightly at the lower end. After puddling the top layer, the surface concrete shall be struck off with a trowel and covered with the top cover plate which will later be used in capping the test specimens.

Capping.

6. Two to four hours after molding, the test specimens shall be capped with a thin layer of stiff, neat, cement paste in order that the cylinder may present a smooth end for testing. The cap can best be formed by means of a piece of plate glass one-quarter inch thick and of a diameter 2 or 3 inches larger than that of the mold. This plate is worked on the fresh cement paste until it rests on top of the form. The cement for capping should be mixed to a stiff paste some time before it is to be used, in order to avoid the tendency of the cap to shrink. Adhesion of the concrete to the top and bottom plates can be avoided by oiling the plates or by inserting a sheet of paraffined tissue paper.

Curing and storing.

7. At the end of 48 hours the test specimens shall be removed from the molds and buried in damp sand, unless cardboard molds are used, in which case test specimens may be buried in damp sand without removal of the mold, thus permitting shipping of the test specimens in the molds.

8. (*a*) The test specimens shall remain buried in damp sand until 10 days prior to the date of test. They shall then be well packed in damp sand or wet shavings and shipped to the testing laboratory, where they shall be stored either in a moist room or in damp sand until the date of test.

(*b*) Should a 7-day test be required, the test specimens shall remain at the works as long as possible to harden and then shall be shipped so as to arrive at the laboratory in time to make the test on the required date.

METHOD OF SECURING SPECIMENS OF HARDENED CONCRETE FROM THE STRUCTURE.

(A. S. T. M tentative method, serial designation, C 42–21 T.)

1. The methods and precautions herein specified apply to securing test specimens from hardened concrete in the structure.

2. A specimen from hardened concrete to be tested for strength shall not be taken until the concrete has become hard enough so that the cutting of the specimen will not disturb the bond between the mortar content and the coarse aggregate of the specimen.

Securing samples.

3. The specimen from the structure can best be secured by use of a core drill. For specimens taken perpendicular to a horizontal surface a drill using chilled shot may be used, but when taken perpendicular to a vertical surface a diamond drill should be used.

4. (*a*) The core specimen taken shall be as nearly as possible a cylinder whose length is twice the diameter.

(*b*) In securing a specimen perpendicular to a horizontal surface care shall be taken to secure, if possible, a specimen whose beds shall be parallel to the horizontal bed of the concrete as originally placed.

(*c*) In securing a specimen perpendicular to a vertical surface, or to a surface with a batter, care shall be taken as to the point of securing the specimen. The lower portion of any one unit of depositing in mass concrete is more dense than is the upper portion of the unit, therefore a specimen shall be taken from near the middle of such unit of deposit.

5. A specimen to be taken from hardened concrete which has been removed from a structure shall be cut out by a drill or shall be cut into a symmetrical test piece by tooling or by sawing. The method of sawing can seldom be used to advantage in the field. In selecting this test specimen care shall be used to see that the concrete selected has not been injured or shattered by the method of its removal from the structure.

6. The specimen secured shall have ends, or beds, as nearly as possible perpendicular to its axis, and if the specimen has ends with uneven surfaces, these ends shall be made parallel plane surfaces with a mortar richer than the mortar of the specimen or with a mixture of cement and calcined gypsum (plaster of Paris).

ABSORPTION TEST OF CONCRETE.

1. Specimens for the absorption test shall be apparently sound, solid pieces of concrete and shall not show cracks or fissures. The specimens shall be so marked as to permit the identity of each one to be ascertained at any stage of the test.

Method of test.

2. Preparatory to the absorption test all specimens shall first be weighed and then dried in a drier or oven at a temperature of not less than 110° C. (230° F.) for not less than three hours. After removal from the drier, the specimens shall be allowed to cool to a temperature of 20° to 25° C. (68° to 77° F.) and reweighed. If the specimens were apparently dry when taken, and the second weight closely checks the first, the specimens shall be considered dry. If the specimens were known to be wet when taken, they shall be placed in the drier for a further drying treatment of two hours, and reweighed. If the third weight checks the second, the specimens shall be considered dry. In case of any doubt, the specimens must be redried for 2-hour periods until check weights are obtained.

3. The balance used shall be sensitive to 0.05 gram when loaded with 1 kilogram and weighings shall be read at least to the nearest gram. When other than metric weights are used, the same order of accuracy must be obtained. In reweighing after immersion, the specimens shall be removed from the water, not allowed to drain more than one minute, the superficial water removed by towel or blotting paper, and the specimens at once put upon the balance.

4. Specimens after weighing shall be placed in a suitable receptacle, covered with distilled water or rain water, raised to the boiling point and boiled for five hours, and then cooled in water to a final temperature of 10° to 15° C. (50° to 59° F.).

5. The test results shall be calculated as percentages of the initial dry weight carried to the nearest first decimal place. The results shall be reported separately for each individual specimen, together with the mean of the specimens comprising the sample.

TRANSVERSE TEST OF CONCRETE.

Specimens for the transverse test shall be 12 inches wide, 8 inches deep, and 30 inches long. They shall be tested by a center load over a 24-inch span.

The methods of proportioning, mixing, molding, and curing of the test pieces shall be the same as for making specimens of concrete for the compression test.

The modulus of rupture S_r may be found from the following formula:

$$S_r = \frac{36P}{bd^2}, \text{ where}$$

P=center load in pounds.
b=breadth of the slab in inches.
d=depth of the slab in inches.

TESTS FOR QUALITY OF WATER TO BE USED IN CONCRETE.

Acidity and alkalinity.

1. *Acidity and alkalinity.*—The acidity or alkalinity shall be determined with standard solutions of N/10 alkali or acid respectiveiy, using not less than 200 cubic centimeters of the water under examination. Phenolphthalein or methyl orange should be used as an indicator. Excessive acidity or alkalinity indicates the necessity for further tests.

Total solids.

2. *Total solids and inorganic matter.*—Five hundred cubic centimeters of the water shall be evaporated to dryness in a weighed dish. For this purpose a platinum dish of 100 to 200 cubic centimeters capacity is found most convenient. The dish shall be nearly filled with the water and placed on a water bath, additional portions of the sample of water being added from time to time until 500 cubic centimeters have been used. The contents of the dish shall be evaporated to dryness and the dish and contents cooled in a desiccator and weighed. The weight of the residue in grams divided by 5 is the percentage of total solids in the water.

Organic matter.

3. The total solids obtained as described may consist of organic matter, of inorganic matter, or of combinations of organic and inorganic matter. The platinum dish shall be ignited at low red heat, and the darkening of the residue during the early stage of the ignition usually indicates the presence of organic matter. The per cent loss on ignition at low red heat will usually be an indicator of the amount of organic matter, but it should be noted that some mineral salts tend to volatilize or partly decompose on heating.

4. The determination of the composition of the mineral matter in the water usually requires a complete chemical analysis of the total solids obtained by the evaporation of 500 cubic centimeters or more, of the water, and is not generally undertaken

except when the percentage of total solids is large, or the water appears to give abnormal tests in other respects.

5. A comparison of the given water with a water of known satisfactory quality can be obtained by making standard soundness, time-of-setting, and 1 : 3 mortar-strength tests with standard sand, using the same cement of standard quality with each water. (Suggested limits for the last-named test are as follows: Any indication of unsoundness, marked change in time of setting, or a variation of more than 10 per cent in strength from results obtained with mixtures containing the water of satisfactory quality, shall be sufficient cause for rejection of the water under test.)

SPECIFICATIONS AND TESTS FOR PORTLAND CEMENT.

(A. S. T. M. standard specifications, serial designation C 9–21, slightly modified.)

SPECIFICATIONS.

1. Portland cement is the product obtained by finely pulverizing clinker produced by calcining to incipient fusion an intimate and properly proportioned mixture of argillaceous and calcareous materials, with no additions subsequent to calcination excepting water and calcined or uncalcined gypsum.

I. CHEMICAL PROPERTIES.

Chemical properties.

2. The following limits shall not be exceeded:

	Per cent.
Loss on ignition	4.00
Insoluble residue	.85
Sulphuric anhydride (SO_3)	2.00
Magnesia (MgO)	5.00

II. PHYSICAL PROPERTIES.

Physical properties.

3. The specific gravity of cement shall be not less than 3.10 (3.07 for white Portland cement). Should the test of cement as received fall below this requirement, a second test may be made upon an ignited sample. The specific gravity test will not be made unless specifically ordered.

4. The residue on a standard No. 200 sieve shall not exceed 22 per cent by weight.

5. A pat of neat cement shall remain firm and hard, and show no signs of distortion, cracking, checking, or disintegration in the steam test for soundness.

6. The cement shall not develop initial set in less than 45 minutes when the Vicat needle is used or 60 minutes when the Gillmore needle is used. Final set shall be attained within 10 hours.

7. The average tensile strength in pounds per square inch of not less than three standard mortar briquets (see section 50) composed of 1 part cement and 3 parts standard sand, by weight, shall be equal to or higher than the following:

Age at test.	Storage of briquets.	Tensile strength per square inch.
Days.		*Pounds.*
7	1 day in moist air, 6 days in water	200
28	1 day in moist air, 27 days in water	300

8. The average tensile strength of standard mortar at 28 days shall be higher than the strength at 7 days.

III. PACKAGES, MARKING AND STORAGE.

Packages, marking, and storing.

9. The cement shall be delivered in suitable bags or barrels with the brand and name of the manufacturer plainly marked thereon, unless shipped in bulk. A bag shall contain 94 pounds net. A barrel shall contain 376 pounds net.

10. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building which will protect the cement from dampness.

IV. INSPECTION.

Inspection.

11. Every facility shall be provided the purchaser for careful sampling and inspection at either the mill or at the site of the work, as may be specified by the purchaser. At least 10 days from the time of sampling shall be allowed for the completion of the 7-day test, and at least 31 days shall be allowed for the completion of the 28-day test. The cement shall be tested in accordance with the methods hereinafter prescribed. The 28-day test shall be waived only when specifically so ordered.

V. REJECTION.

Rejection.

12. (*a*) The cement may be rejected if it fails to meet any of the requirements of these specifications.

(*b*) Samples of each lot shall be required to show practically uniform results in tests. Marked deviation from the uniform result may be considered cause for rejection, even though the test requirements may be otherwise fulfilled.

13. Cement shall not be rejected on account of failure to meet the fineness requirement if upon retest after drying at 100° C. for one hour it meets this requirement.

14. In all cases the user, purchaser, or engineer in control of any construction where Portland cement is used reserves the option to take check samples for the purpose of making tests to determine the stability of the product and such check tests will be the basis for acceptance or rejection regardless of previous decisions.

15. Packages varying more than 5 per cent from the specified weight may be rejected; and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified, the entire shipment may be rejected.

TESTS.

VI. SAMPLING.

Sampling.

16. Tests may be made on individual or composite samples as may be ordered. Each test sample should weigh at least 8 pounds.

17. (*a*) *Individual sample.*—If sampled in cars, one test sample shall be taken from each 50 barrels or fraction thereof. If sampled in bins, one sample shall be taken from each 100 barrels.

(*b*) *Composite sample.*—If sampled in cars, one sample shall be taken from 1 sack in each 40 sacks (or 1 barrel in each 10 barrels) and combined to form one test sample. If sampled in bins or warehouses, one test sample shall represent not more than 200 barrels.

18. Cement may be sampled at the mill by any of the following methods that may be practicable, as ordered:

(*a*) *From the conveyor delivering to the bin.*—At least 8 pounds of cement shall be taken from approximately each 100 barrels passing over the conveyor.

(*b*) *From filled bins by means of proper sampling tubes.*—Tubes inserted vertically may be used for sampling cement to a maximum depth of 10 feet. Tubes inserted horizontally may be

used where the construction of the bin permits. Samples shall be taken from points well distributed over the face of the bin.

(*c*) *From filled bins at points of discharge.*—Sufficient cement shall be drawn from the discharge openings to obtain samples representative of the cement contained in the bin, as determined by the appearance at the discharge openings of indicators placed on the surface of the cement directly above these openings before drawing of the cement is started.

19. Samples preferably shall be shipped and stored in air-tight containers. Samples shall be passed through a sieve having 20 meshes per linear inch in order to mix the sample thoroughly, break up lumps, and remove foreign materials.

VII. CHEMICAL ANALYSIS.

Loss on Ignition.

Loss on ignition.

20. One gram of cement shall be heated in a weighed covered platinum crucible, of 20 to 25 cubic centimeters capacity, as follows, using either method (*a*) or (*b*) as ordered:

(*a*) The crucible shall be placed in a hole in an asbestos board, clamped horizontally so that about three-fifths of the crucible projects below, and blasted at a full red heat for 15 minutes with an inclined flame; the loss in weight shall be checked by a second blasting for 5 minutes. Care shall be taken to wipe off particles of asbestos that may adhere to the crucible when withdrawn from the hole in the board. Greater neatness and shortening of the time of heating are secured by making a hole to fit the crucible in a circular disk of sheet platinum and placing this disk over a somewhat larger hole in an asbestos board.

(*b*) The crucible shall be placed in a muffle at any temperature between 900° and 1,000° C. for 15 minutes and the loss in weight shall be checked by a second heating for 5 minutes.

21. A permissible variation of 0.25 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 4 per cent.

Insoluble Residue.

Insoluble residue.

22. To a 1-gram sample of cement shall be added 10 cubic centimeters of water and 5 cubic centimeters of concentrated hydrochloric acid; the liquid shall be warmed until effervescence ceases. The solution shall be diluted to 50 cubic centimeters and digested on a steam bath or hot plate until it is evident that decomposition of the cement is complete. The residue shall be filtered, washed with cold water, and the filter paper and contents digested in about 30 cubic centimeters of a 5 per cent solution of sodium carbonate, the liquid being held at a temperature just short of boiling for 15 minutes. The remaining residue shall be filtered, washed with cold water, then with a few drops of hot hydrochloric acid, 1:9, and finally with hot water, and then ignited at a red heat and weighed as the insoluble residue.

23. A permissible variation of 0.15 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 0.85 per cent.

Sulphuric Anhydride.

Sulphuric anhydride.

24. One gram of the cement shall be dissolved in 5 cubic centimeters of concentrated hydrochloric acid diluted with 5 cubic centimeters of water, with gentle warming; when solution is complete, 40 cubic centimeters of water shall be added, the solution filtered, and the residue washed thoroughly with water. The solution shall be diluted to 250 cubic centimeters, heated to boiling, and 10 cubic centimeters of a hot 10 per cent solution of barium chloride shall be added slowly, drop by drop, from a

pipette and the boiling continued until the precipitate is well formed. The solution shall be digested on the steam bath until the precipitate has settled. The precipitate shall be filtered, washed, and the paper and contents placed in a weighed platinum crucible and the paper slowly charred and consumed without flaming. The barium sulphate shall then be ignited and weighed. The weight obtained multiplied by 34.3 gives the percentage of sulphuric anhydride. The acid filtrate obtained in the determination of the insoluble residue may be used for the estimation of sulphuric anhydride instead of using a separate sample.

25. A permissible variation of 0.10 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 2.00 per cent.

Magnesia.

Magnesia.

26. To 0.5 gram of the cement in an evaporating dish shall be added 10 cubic centimeters of water to prevent lumping and then 10 cubic centimeters of concentrated hydrochloric acid. The liquid shall be gently heated and agitated until attack is complete. The solution shall then be evaporated to complete dryness on a steam or water bath. To hasten dehydration the residue may be heated to 150° or even 200° C. for one-half to one hour. The residue shall be treated with 10 cubic centimeters of concentrated hydrochloric acid diluted with an equal amount of water. The dish shall be covered and the solution digested for ten minutes on a steam bath or water bath. The diluted solution shall be filtered and the separated silica washed thoroughly with water.[9]

Five cubic centimeters of concentrated hydrochloric acid and sufficient bromine water to precipitate any manganese which may be present shall be added to the filtrate (about 250 cubic centimeters). This shall be made alkaline with ammonium hydroxide, boiled until there is but a faint odor of ammonia, and the precipitated iron and aluminum hydroxides, after settling, shall be washed with hot water, once by decantation and slightly on the filter. Setting aside the filtrate, the precipitate shall be transferred by a jet of hot water to the precipitating vessel and dissolved in 10 cubic centimeters of hot hydrochloric acid. The paper shall be extracted with acid, the solution and washings being added to the main solution. The aluminum and iron shall then be reprecipitated at boiling heat by ammonium hydroxide and bromine water in a volume of about 100 cubic centimeters, and the second precipitate shall be collected and washed on the filter used in the first instance if this is still intact. To the combined filtrates from the hydroxides of iron and aluminum, reduced in volume if need be, 1 cubic centimeter of ammonium hydroxide shall be added, the solution brought to boiling, 25 cubic centimeters of a saturated solution of boiling ammonium oxalate added and the boiling continued until the precipitated calcium oxalate has assumed a well-defined granular form. The precipitate after one hour shall be filtered and washed, then with the filter shall be placed wet in a platinum crucible, and the paper burned off over a small flame of a Bunsen burner; after ignition it shall be redissolved in hydrochloric acid and the solution diluted to 100 cubic centimeters. Ammonia shall be added in slight excess, and the liquid boiled. The lime shall then be reprecipitated by ammonium oxalate, allowed to stand until settled, filtered and washed. The combined filtrates from the calcium precipitates shall be acidified with hydrochloric acid, concentrated on the steam bath to about 150 cubic centimeters, and made slightly alkaline with ammonium hydroxide, boiled

[9] Since this procedure does not involve the determination of silica, a second evaporation is unnecessary.

and filtered (to remove a little aluminum and iron and perhaps calcium). When cool, 10 cubic centimeters of a saturated solution of sodium-ammonium-hydrogen phosphate shall be added with constant stirring. When the crystalline ammonium-magnesium orthophosphate has formed, ammonia shall be added in moderate excess. The solution shall be set aside for several hours in a cool place, filtered and washed with water containing 2.5 per cent of NH_3. The precipitate shall be dissolved in a small quantity of hydrochloric acid, the solution diluted to about 100 cubic centimeters, 1 cubic centimeter of a saturated solution of sodium-ammonium-hydrogen phosphate added, and ammonia drop by drop, with constant stirring, until the precipitate is again formed as described and the ammonia is in moderate excess. The precipitate shall then be allowed to stand about two hours, filtered and washed as before. The paper and contents shall be placed in a weighed platinum crucible, the paper slowly charred, and the resulting carbon carefully burned off. The precipitate shall then be ignited to constant weight over a Meker burner, or a blast not strong enough to soften or melt the pyrophosphate. The weight of magnesium pyrophosphate obtained multiplied by 72.5 gives the percentage of magnesia. The precipitate so obtained always contains some calcium and usually small quantities of iron, aluminum, and manganese as phosphates.

27. A permissible variation of 0.4 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 5.00 per cent.

VIII. DETERMINATION OF SPECIFIC GRAVITY.

Specific gravity.

28. The determination of specific gravity shall be made with a standardized Le Chatelier apparatus. This apparatus is standardized by the United States Bureau of Standards. Kerosene free from water, or benzene not lighter than 62° Baumé, shall be used in making this determination.

29. The flask shall be filled with either of these liquids to a point on the stem between zero and 1 cubic centimeter, and 64 grams of cement, of the same temperature as the liquid, shall be slowly introduced, taking care that the cement does not adhere to the inside of the flask above the liquid and to free the cement from air by rolling the flask in an inclined position. After all the cement is introduced, the level of the liquid will rise to some division of the graduated neck; the difference between readings is the volume displaced by 64 grams of the cement. The specific gravity shall then be obtained from the formula

$$\text{Specific gravity} = \frac{\text{Weight of cement (grams)}}{\text{Displaced volume (cubic centimeters)}}.$$

30. The flask, during the operation, shall be kept immersed in water, in order to avoid variations in the temperature of the liquid in the flask, and any variation that occurs shall not exceed 0.5° C. The results of repeated tests should agree within 0.01.

31. The determination of specific gravity shall be made on the cement as received; if it falls below 3.10, a second determination shall be made after igniting the sample as described in section 20.

IX. DETERMINATION OF FINENESS.

Fineness.

32. Wire cloth for standard sieves for cement shall be woven (not twilled) from brass, bronze, or other suitable wire, and mounted without distortion on frames not less than $1\frac{1}{2}$ inches below the top of the frame. The sieve frames shall be circular, approximately 8 inches in diameter, and may be provided with a pan and cover.

33. A standard No. 200 sieve is one having nominally a 0.0029-inch opening and 200 wires per inch, standardized by the United States Bureau of Standards, and conforming to the following requirements:

The No. 200 sieve should have 200 wires per inch, and the number of wires in any whole inch shall not be outside the limits of 192 to 208. No opening between adjacent parallel wires shall be more than 0.0050 inch in width. The diameter of the wire should be 0.0021 inch and the average diameter shall not be outside the limits 0.0019 to 0.0023 inch. The value of the sieve as determined by sieving tests made in conformity with the standard specification for these tests on a standardized cement which gives a residue of 25 to 20 per cent on the No. 200 sieve, or on other similarly graded material, shall not show a variation of more than 1.5 per cent above or below the standards maintained at the Bureau of Standards.

34. The test shall be made with 50 grams of cement. The sieve shall be thoroughly clean and dry. The cement shall be placed on the No. 200 sieve, with pan and cover attached, if desired, and shall be held in one hand in a slightly inclined position so that the sample will be well distributed over the sieve, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the upstroke. The sieve shall be turned every 25 strokes about one-sixth of a revolution in the same direction. The operation shall continue until not more than 0.05 gram passes through in one minute of continuous sieving. The fineness shall be determined from the weight of the residue on the sieve expressed as a percentage of the weight of the original sample.

35. Mechanical sieving devices may be used, but the cement shall not be rejected if it meets the fineness requirement when tested by the hand method described in section 34.

X. MIXING CEMENT PASTES AND MORTARS.

Pastes and mortars.

36. The quantity of dry material to be mixed at one time shall not exceed 1,000 grams nor be less than 500 grams. The proportions of cement or cement and sand shall be stated by weight in grams of the dry materials; the quantity of water shall be expressed in cubic centimeters (1 cubic centimeter of water=1 gram). The dry materials shall be weighed, placed upon a non-absorbent surface, thoroughly mixed dry if sand is used, and a crater formed in the center, into which the proper percentage of clean water shall be poured; the material on the outer edge shall be turned into the crater by the aid of a trowel. After an interval of one-half minute for the absorption of water the operation shall be completed by continuous, vigorous mixing, squeezing and kneading with the hands for at least one minute.[10] During the operation of mixing, the hands should be protected by rubber gloves.

37. The temperature of the room and the mixing water shall be maintained as nearly as practicable at 21° C. (70° F.).

Normal consistency.

XI. NORMAL CONSISTENCY.

38. The Vicat apparatus consists of a frame bearing a movable rod, weighing 300 grams, one end being 1 centimeter in diameter for a distance of 6 centimeters, the other having a removable needle 1 millimeter in diameter, 6 centimeters long. The rod is reversible, and can be held in any desired position by a screw, and has midway between the ends a mark which moves under a scale (graduated to millimeters) attached to the frame. The paste is held in a conical, hard-rubber ring 7 centimeters in diameter at

[10] In order to secure uniformity in the results of tests for the time of setting and tensile strength the manner of mixing above described should be carefully followed. At least one minute is necessary to obtain the desired plasticity which is not appreciably affected by continuing the mixing for several minutes. The exact time necessary is dependent upon the personal equation of the operator. The error in mixing should be on the side of overmixing.

the base, 4 centimeters high, resting on a glass plate about 10 centimeters square.

39. In making the determination, 500 grams of cement, with a measured quantity of water, shall be kneaded into the paste, as described in section 36, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained about 6 inches apart; the ball resting in the palm of one hand shall be pressed into the larger end of the rubber ring held in the other hand, completely filling the ring with paste; the excess at the larger end shall then be removed by a single movement of the palm of the hand; the ring shall then be placed on its larger end on a glass plate and the excess paste at the smaller end sliced off at the top of the ring by a single oblique stroke of a trowel held at a slight angle with the top of the ring. During these operations care shall be taken not to compress the paste. The paste confined in the ring, resting on the plate, shall be placed under the rod, the larger end of which shall be brought in contact with the surface of the paste; the scale shall then be read, and the rod quickly released. The paste shall be of normal consistency when the rod settles to a point 10 millimeters below the original surface in one-half minute after being released. The apparatus shall be free from all vibrations during the test. Trial pastes shall be made with varying percentages of water until the normal consistency is obtained. The amount of water required shall be expressed in percentage by weight of the dry cement.

40. The consistency of standard mortar shall depend on the amount of water required to produce a paste of normal consistency from the same sample of cement. Having determined the normal consistency of the sample, the consistency of standard mortar made from the sample shall be as indicated in Table 2, the values being in percentage of the combined dry weights of the cement and standard sand.

TABLE 2.—*Percentage of water for standard mortars.*

For neat cement paste of normal consistency.	For one cement, three standard Ottawa sand.	For neat cement paste of normal consistency.	For one cement, three standard Ottawa sand.
15	9.0	23	10.3
16	9.2	24	10.5
17	9.3	25	10.7
18	9.5	26	10.8
19	9.7	27	11.0
20	9.8	28	11.2
21	10.0	29	11.3
22	10.2	30	11.5

XII. DETERMINATION OF SOUNDNESS.[11]

Soundness.

41. A steam appartus which can be maintained at a temperature between 98° and 100° C. is recommended. The capacity of this appartus may be increased by using a rack for holding the pats in a vertical or inclined position.

42. A pat from cement paste of normal consistency about 3 inches in diameter, one-half inch thick at the center, and tapering to a thin edge, shall be made on clean glass plates about 4 inches square, and stored in moist air for 24 hours. In molding the pat,

[11] Unsoundness is usually manifested by change in volume which causes distortion, cracking, checking or disintegration. Pats improperly made or exposed to drying may develop what are known as shrinkage cracks within the first 24 hours; these are not an indication of unsoundness. These conditions are illustrated in Plate I. The failure of the pats to remain on the glass or the cracking of the glass to which the pats are attached does not necessarily indicate unsoundness.

the cement paste shall first be flattened on the glass and the pat then formed by drawing the trowel from the outer edge toward the center.

43. The pat shall then be placed in an atmosphere of steam at a temperature between 98° and 100° C. upon a suitable support 1 inch above boiling water for 5 hours.

44. Should the pat leave the plate, distortion may be detected best with a straight edge applied to the surface which was in contact with the plate. (See Plate I.)

XIII. DETERMINATION OF TIME OF SETTING.

Time of setting.

45. The following are alternate methods, either of which may be used as ordered:

46. The time of setting shall be determined with the Vicat apparatus described in section 38.

47. A paste of normal consistency shall be molded in the hard-rubber ring as described in section 39, and placed under the rod, the smaller end of which shall then be carefully brought in contact with the surface of the paste, and the rod quickly released. The initial set shall be said to have occurred when the needle ceases to pass a point 5 millimeters above the glass in one-half minute after being released; and the final set, when the needle does not sink visibly into the paste. The test pieces shall be kept in moist air during the test. This may be accomplished by placing them on a rack over water contained in a pan and covered by a damp cloth, kept from contact with them by means of a wire screen; or they may be stored in a moist closet. Care shall be taken to keep the needle clean, as the collection of cement on the sides of the needle retards the penetration, while cement on the point may increase the penetration. The time of setting is affected not only by the percentage and temperature of the water used and the amount of kneading the paste receives but by the temperature and humidity of the air, and its determination is therefore only approximate.

48. The time of setting shall be determined by the Gillmore needles.

49. The time of setting shall be determined as follows: A pat of neat cement paste about 3 inches in diameter and one-half inch in thickness with a flat top, mixed to a normal consistency, shall be kept in moist air at a temperature maintained as nearly as practicable at 21° C. (70° F.). The cement shall be considered to have acquired its initial set when the pat will bear, without appreciable indentation, the Gillmore needle one-twelfth inch in diameter, loaded to weigh one-quarter pound. The final set has been acquired when the pat will bear without appreciable indentation, the Gillmore needle one twenty-fourth inch in diameter, loaded to weigh 1 pound. In making the test the needles shall be held in a vertical position and applied lightly to the surface of the pat.

XIV. TENSION TESTS.

Tension tests.

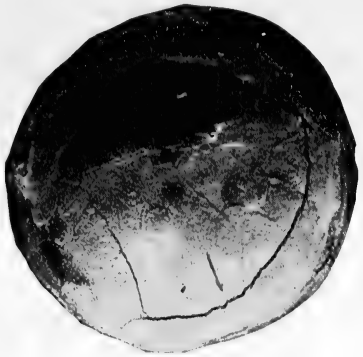
50. The standard form of test piece shall be used. The molds shall be made of noncorroding metal and have sufficient material in the sides to prevent spreading during molding. Gang molds may be used. Molds shall be wiped with an oily cloth before using.

51. The sand to be used shall be natural sand from Ottawa, Ill., screened to pass a No. 20 sieve and retained on a No. 30 sieve. This sand may be obtained from the Ottawa Silica Co., at a cost of 3 cents per pound, f. o. b. cars, Ottawa, Ill.

52. This sand, having passed the No. 20 sieve, shall be considered standard when not more than 5 grams pass the No. 30 sieve after one minute continuous sieving of a 500-gram sample.

53. The sieve shall conform to the following specifications:

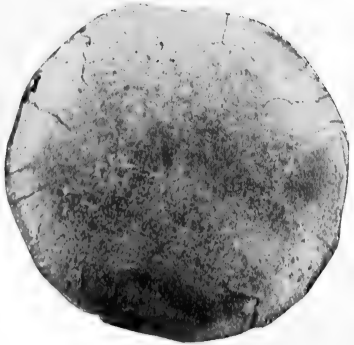
The No. 20 sieve shall have between 19.5 and 20.5 wires per whole inch of the warp wires and between 19 and 21 wires per



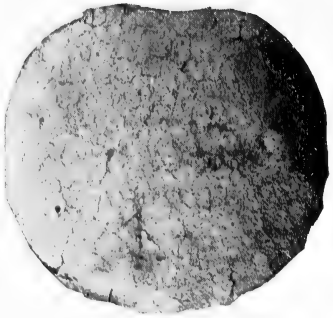
Shrinkage.

Shrinkage.

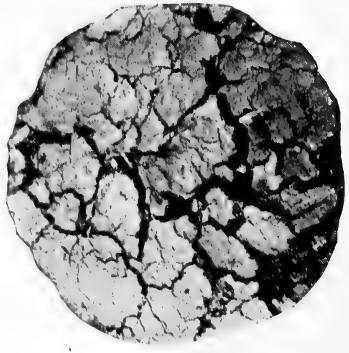
Distortion.



Cracking.



Checking.



Disintegration.

TYPICAL FAILURES IN SOUNDNESS TEST.

PLATE I.

whole inch of the shoot wires. The diameter of the wire should be 0.0165 inch and the average diameter shall not be outside the limits of 0.0160 and 0.0170 inch.

The No. 30 sieve shall have between 29.5 and 30.5 wires per whole inch of the warp wires and between 28.5 and 31.5 wires per whole inch of the shoot wires. The diameter of the wire should be 0.0110 inch and the average diameter shall not be outside the limits 0.0105 to 0.0115 inch.

54. Immediately after mixing, the standard mortar shall be placed in the molds, pressed in firmly with the thumbs, and smoothed off with a trowel without ramming. Additional mortar shall be heaped above the mold and smoothed off with a trowel; the trowel shall be drawn over the mold in such a manner as to exert a moderate pressure on the material. The mold shall then be turned over and the operation of heaping, thumbing, and smoothing off repeated.

55. Tests shall be made with any standard machine. The briquets shall be tested as soon as they are removed from the water. The bearing surfaces of the clips and briquets shall be free from grains of sand or dirt. The briquets shall be carefully centered and the load applied continuously at the rate of 600 pounds per minute.

56. Testing machines should be frequently calibrated in order to determine their accuracy.

57. Briquets that are manifestly faulty, or which give strengths differing more than 15 per cent from the average value of all test pieces made from the same sample and broken at the same period, shall not be considered in determining the tensile strength.

XV. STORAGE OF TEST PIECES.

Storage.

58. The moist closet may consist of a soapstone, slate, or concrete box, or a wooden box lined with metal. If a wooden box is used, the interior should be covered with felt or broad wicking kept wet. The bottom of the moist closet should be covered with water. The interior of the closet should be provided with nonabsorbent shelves on which to place the test pieces, the shelves being so arranged that they may be withdrawn readily.

59. Unless otherwise specified all test pieces, immediately after molding, shall be placed in the moist closet for from 20 to 24 hours.

60. The briquets shall be kept in molds on glass plates in the moist closet for at least 20 hours. After 24 hours in moist air the briquets shall be immersed in clean water in storage tanks of noncorroding material.

61. The air and water shall be maintained as nearly as practicable at a temperature of 21° C. (70° F.).

RATTLER TEST FOR PAVING BRICK.

(A. S. T. M. standard method, serial designation, C 7–15, slightly modified.)

THE CONSTRUCTION OF THE RATTLER.

The rattler.

1. *General design.*—The machine shall be of good mechanical construction, self-contained, and shall conform to the following details of material and dimensions, and shall consist of barrel, frame, and driving mechanism as herein described.

2. *The barrel.*—The barrel of the machine shall be made up of the heads, head liners, staves, and stave liners.

The heads may be cast in one piece with the trunnions, which shall be 2½ inches in diameter, and shall have a bearing 6 inches in length, or they may be cast with heavy hubs, which shall be bored out for $2\frac{7}{16}$-inch shafts, and shall be keyseated for two keys, each one-half by three-eighths inch and spaced 90° apart. The shaft shall be a snug fit and when keyed shall be entirely

free from lost motion. The distance from the end of the shaft or trunnion to the inside face of the head shall be 15⅜ inches in the head for the driving end of the rattler, and 11⅜ inches for the other head, and the distance from the face of the hubs to the inside face of the heads shall be 5⅛ inches.

The heads shall be not less than three-fourths inch thick, nor more than seven-eighths inch thick. In outline, each head shall be a regular 14-sided polygon inscribed in a circle 28⅜ inches in diameter. Each head shall be provided with flanges not less three-fourths inch thick and extending outward 2½ inches from the inside face of the head to afford a means of fastening the staves. The surface of the flanges of the head shall be smooth and give a true and uniform bearing for the staves. To secure the desired true and uniform bearing, the surfaces of the flanges of the head shall be either ground or machined. The flanges shall be slotted on the outer edge, so as to provide for two three-fourth-inch bolts at each end of each stave, the slots to be thirteen-sixteenths inch wide and 2¾ inches center to center. Each slot shall be provided with a recess for the bolthead, which shall act to prevent the turning of the same. Between each two slots there shall be a brace three-eighths inch thick, extending down the outward side of the head not less than 2 inches.

There shall be for each head a cast-iron head liner 1 inch in thickness and conforming to the outline of the head, but inscribed in a circle 28⅛ inches in diameter. This head liner shall be fastened to the head by seven five-eighths-inch cap screws, through the head from the outside. Whenever these head liners become worn down one-half inch below their initial surface level at any point of their surface they shall be replaced with new ones. The metal of these head liners shall be hard machinery iron and should contain not less than 1 per cent of combined carbon.

The staves shall be made of 6-inch medium-steel structural channels, 27¼ inches long and weighing 15.5 pounds per lineal foot. The staves shall have two holes thirteen-sixteenths inch in diameter, drilled in each end, the center line of the holes being 1 inch from the end and 1⅜ inches each way from the longitudinal center line. The spaces between the staves shall be as uniform as practicable, but shall not exceed five-sixteenths inch.

The interior or flat side of each stave shall be protected by a liner three-eighths inch thick by 5½ inches wide and 19¾ inches long. The liner shall consist of medium-steel plate, and shall be riveted to the channel by three one-half-inch rivets, one of which shall be on the center line both ways and the other two on the longitudinal center line and spaced 7 inches from the center each way. The rivet holes shall be countersunk on the face of the liner and the rivets shall be driven hot and chipped off flush with the surface of the liners. These liners shall be inspected from time to time, and if found loose shall be at once reriveted.

Any test at the expiration of which a stave liner is found detached from the stave or seriously out of position shall be rejected. When a new rattler, in which a complete set of new staves is furnished, is first put into operation, it shall be charged with 400 pounds of shot of the same sizes, and in the same proportions as provided in section 4, and shall then be run for 18,000 revolutions at the usual prescribed rate of speed. The shot shall then be removed and a standard shot charge inserted, after which the rattler may be charged with brick for a test.

No stave shall be used for more than 70 consecutive tests without renewing its lining. Two of the 14 staves shall be removed and relined at a time in such a way that of each pair, one falls upon one side of the barrel and the other upon the opposite side, and also so that the staves changed shall be consecutive but not contiguous; for example, 1 and 8, 3 and 10, 5 and 12, 7 and 14,

2 and 9, 4 and 11, 6 and 13, etc., to the end that the interior of the barrel at all times shall present the same relative condition of repair. The changes in the staves should be made at the time when the shot charges are being corrected, and the record must show the number of charges run since the last pair of new lined staves was placed in position.

The staves when bolted to the heads shall form a barrel 20 inches long, inside measurement, between head liners. The liners of the staves shall be so placed as to drop between the head liners. The staves shall be bolted tightly to the heads by four three-quarter-inch bolts, and each bolt shall be provided with a lock nut, and shall be inspected at not less frequent intervals than every fifth test and all nuts kept tight. A record shall be made after each inspection showing in what condition the bolts were found.

3. *The frame and driving mechanism.*—The barrel shall be mounted on a cast-iron frame of sufficient strength and rigidity to support it without undue vibration. It shall rest on a rigid foundation with or without the interposition of wooden plates, and shall be fastened thereto by bolts at not less than four points.

It shall be driven by gearing whose ratio of driver to driven is not less than 1 to 4. The countershaft upon which the driving pinion is mounted shall be not less than $1\frac{15}{16}$ inches in diameter, with bearing not less than 6 inches in length. If a belt is used, the pulley shall not be less than 18 inches in diameter and $6\frac{1}{2}$ inches in face. A belt at least 6 inches in width, properly adjusted to avoid unnecessary slipping, should be used.

4. *The abrasive charge.*—The abrasive charge shall consist of cast-iron spheres of two sizes. When new, the larger spheres shall be 3.75 inches in diameter and shall weigh approximately 7.5 pounds (3.40 kilograms) each. Ten spheres of this size shall be used. ***Abrasive charge.***

These shall be weighed separately after each 10 tests, and if the weight of any large sphere falls to 7 pounds (3.175 kilograms) it shall be discarded and a new one substituted; provided, however, that all of the large spheres shall not be discarded and new ones substituted at any single time, and that so far as possible the large spheres shall compose a graduated series in various stages of wear.

When new, the smaller spheres shall be 1.875 inches in diameter and shall weigh approximately 0.95 pounds (0.43 kilogram) each. In general, the number of small spheres in a charge shall not fall below 245 nor exceed 260. The collective weight of the large and small spheres shall be as near 300 pounds as possible. No small sphere shall be retained in use after it has been worn down so that it will pass a circular hole 1.75 inches in diameter, drilled in an iron plate one-quarter inch in thickness, or weigh less than 0.75 pound (0.34 kilogram). Further, the small spheres shall be tested by passing them over the above plate or by weighing, after every 10 tests, and any which pass through or fall below the specified weight, shall be replaced by new spheres; provided, further, that all of the small spheres shall not be rejected and replaced by new ones at any one time, and that so far as possible the small spheres shall compose a graduated series in various stages of wear. At any time that any sphere is found to be broken or defective it shall at once be replaced.

The iron composing these spheres shall have a chemical composition within the following limits:

	Per cent.
Combined carbon	Not under 2.50
Graphitic carbon	Not over 0.25
Silicon	Not over 1.00
Manganese	Not over 0.50
Phosphorus	Not over 0.25
Sulphur	Not over 0.08

For each new batch of spheres used the chemical analysis shall be furnished by the maker or shall be obtained by the user before introducing into the charge, and unless the analysis meets the above specifications the batch of spheres shall be rejected.

THE OPERATION OF THE TEST.

Brick charge.

5. *The brick charge.*—The number of brick per test shall be 10 for all brick of so-called "block size," whose dimensions fall between 8 and 9 inches in length, 3 and 3¾ inches in breadth, and 3¾ and 4¼ inches in thickness.[12] No brick should be selected as part of a regular test that would be rejected by any other requirements of the specifications under which the purchase is made.

Speed and duration of revolution.

6. *Speed and duration of revolution.*—The rattler shall be rotated at a uniform rate of not less than 29.5 nor more than 30.5 revolutions per minute, and 1,800 revolutions shall constitute the test. A counting machine shall be attached to the rattler for counting the revolutions. A margin not to exceed 10 revolutions will be allowed for stopping. Only one start and stop per test is generally acceptable. If, from accidental causes, the rattler is stopped and started more than once during a test, and the loss exceeds the maximum permissible under the specifications, the test shall be disqualified and another made.

7. *The scales.*—The scales must have a capacity of not less than 300 pounds, and must be sensitive to 0.5 ounce, and must be tested by a standard test weight at intervals of not less than every 10 tests.

8. *The results.*—The loss shall be calculated in percentage of the initial weight of the brick composing the charge. In weighing the rattled brick, any piece weighing less than 1 pound shall be rejected.

Record form.

9. *The records.*—A complete and continuous record shall be kept of the operation of all rattlers working under these specifications. This record shall contain the following data concerning each test made:

(1) The name of the person, firm, or corporation furnishing each sample tested.

(2) The name of the maker of the brick represented in each sample tested.

(3) The name of the street, or contract, which the sample represented.

(4) The brands or marks upon the brick by which they were identified.

(5) The number of brick furnished.

(6) The date on which they were received for test.

(7) The date on which they were tested.

(8) The drying treatment given before testing, if any.

(9) The length, breadth, and thickness of the brick.

(10) The collective weight of the ten large spherical shot used in making the test at the time of their last standardization.

(11) The number and collective weight of the small spherical shot used in making the test, at the time of their last standardization.

(12) The total weight of the shot charge, after its last standardization.

(13) Certificate of the operator that he examined the condition of the machine as to staves, liners, and any other parts affecting the barrel, and found them right at the beginning of the test.

(14) Certificate of the operator of the number of charges tested since the last standardization of shot charge and last renewals of stave liners.

(15) The time of the beginning and ending of each test, and the number of revolutions made by the barrel during the test, as shown by the indicator.

[12] Where brick of larger or smaller sizes than the dimensions given above for block are to be tested, the same number of brick per charge should be used, but allowance for the difference in size should be made in setting the limits for average and maximum rattler loss.

(16) Certificate of the operator as to number of stops and starts made in each test.

(17) The initial collective weight of the 10 brick composing the charge and their collective weight after rattling.

(18) The loss calculated in percentage of the initial weight, and the calculation itself.

(19) The number of broken brick and remarks upon the portions which were included in the final weighing.

(20) General remarks upon the test and any irregularities occurring in its execution.

(21) The location and name of the owner of the rattler with which the test was made.

(22) The certificate of the operator that the test was made under the specifications of the American Society for Testing Materials and that the record is a true record.

(23) The signature of the operator or person responsible for the test.

(24) The serial number of the test.

In the event of more than one copy of the record of any test being required, they may be furnished on separate sheets, and marked duplicates, but the original record shall always be preserved intact and complete.

Serial No------

REPORT OF STANDARD RATTLER TEST FOR PAVING BRICKS.

IDENTIFICATION DATA.

Name of the firm furnishing sample----------
Name of the firm manufacturing sample----------
Street or job which sample represents----------
Brands or marks on the bricks----------
Quantity furnished---------- Drying treatment----------
Date received---------- Date tested----------
Length---------- Breadth---------- Thickness----------

STANDARDIZATION DATA.

	Weight of charge (after standardization).	Condition of lock nuts on staves.	Condition of scales.	Number and position of fresh stave liners.	Repairs. (Note any repairs affecting the condition of the barrel.)
10 large spheres					
Small spheres					
Total					

RUNNING DATA.

	Time readings.			Revolution counter readings.	Running notes, stops, etc.
	Hours.	Minutes.	Seconds.		
Beginning of test					
Final reading					

WEIGHTS AND CALCULATIONS.

		Percentage loss. (Note: The calculation must appear.)
Initial weight of 10 bricks		
Final weight of same		
Loss of weight		

Number of broken bricks and remarks on same----------

I certify that the foregoing test was made under the specifications of the American Society for Testing Materials, and is a true record.

(Signature of tester)----------

Date ----------

Location of laboratory----------

METHOD OF SAMPLING AND TESTING BUILDING BRICKS FOR MASONRY CONSTRUCTION.

I. SAMPLING.

1. For the purpose of testing, bricks shall be selected by an experienced person so as to represent the commercial product. All bricks shall be carefully examined and their condition noted before being subjected to any kind of test. For the purpose of the testing, 10 bricks will be required; they shall be thoroughly dried to constant weight in a suitable oven at a temperature of from 225° F. (107° C.) to 250° F. (121° C.).

II. PHYSICAL TESTS.

Absorption.

2. (*a*) At least five dry bricks shall be weighed and completely submerged in water at a temperature between 60° and 80° F. (15° and 27° C.). The water shall be heated to boiling within one hour, boiled continuously for five hours and then allowed to cool to a temperature between 60° and 80° F. (15° and 27° C.). The bricks shall then be removed, the surface water wiped off with a damp cloth, and the bricks quickly weighed.

(*b*) The percentage of absorption shall be computed on the dry weight, according to the relation:

$$\text{Percentage of absorption} = \frac{100\,(B-A)}{A}$$

where A=weight of dry bricks and B=weight of saturated bricks.

Compression test.

3. (*a*) Compression tests shall be made on at least five half bricks, previously dried, each taken from a different brick. The half bricks shall be prepared either by sawing or cutting upon a yielding bed with a sharp mason's chisel, which shall be the full width of the brick. The specimens shall be tested on edge. To secure a uniform bearing in the testing machine, the edge surfaces shall be bedded in a thin coat of calcined gypsum (plaster of Paris) spread upon plate glass previously coated with a film of oil. Before applying the calcined gypsum (plaster of Paris), the bearing surface of the bricks shall receive a coating of shellac. The bricks shall be pressed firmly upon the surface, making the layer as thin as possible, and shall be permitted to remain undisturbed until set. The depression of recessed or paneled bricks shall be filled with neat Portland cement mortar, which shall stand at least 24 hours before testing.

(*b*) The machine used for the compression tests shall be equipped with a spherical bearing block kept thoroughly lubricated to insure accurate adjustment, which should be made by hand under a small initial load. During the test the beam of the testing machine shall be kept constantly in a floating position.

(*c*) The breaking load shall be divided by the area in compression and the results reported in pounds per square inch.

4. (*a*) At least five brick, previously dried, shall be tested, laid flatwise, with a span of 7 inches, and with the load applied at mid span.

(*b*) The modulus of rupture shall be computed in pounds per square inch by the following formula:

$$R = \frac{3\,WI}{2\,bd^2}$$

in which I=the distance between supports in inches, b=the breadth and d=depth of the brick in inches, and W=the load in pounds at which the brick failed.

Record form.

5. In recording the results of the tests the type of brick shall be defined, whether stiff-mud, soft-mud, dry-pressed, repressed, sand-lime or other types. It is recommended that the data obtained be recorded as indicated on the accompanying "Laboratory record."

LABORATORY RECORD.

Brick received from_____
Address _____
Sampled by_____
Type_____
Appearance of brick_____
Date_____
Class_____

ABSORPTION TEST.

Sample No.	Weight.			Absorption, per cent $\frac{B-A}{A} \times 100$	Remarks.
	Dry (A).	Saturated (B).	Difference ($B-A$).		
1					
2					
3					
4					
5					
Sum					
Average					

COMPRESSION TESTS.

(Half bricks tested on edge.)

Sample No.	Dimensions of half brick.			Load.		Compressive strength per square inch $\frac{(L)}{(a)}$.	Remarks.
	Depth (d).	Length (l).	Area (a) $a=dl$.	At first crack.	At failure (L).		
	Inches.	*Inches.*	*Square Inches.*	*Pounds.*	*Pounds.*	*Pounds.*	
1							
2							
3							
4							
5							
Sum							
Average							

TRANSVERSE TEST.

Sample No.	Dimensions.			Load (w).	Modulus of rupture $R=\frac{3\,Wl}{2\,bd^2}$	Remarks.
	Width (b).	Depth (d).	Span (l).			
	Inches.	*Inches.*	*Inches.*	*Pound.*		
1						
2						
3						
4						
5						
Sum						
Average						

SUMMARY.

Average absorption, per cent.	Average compressive strength, pounds per square inch.	Average modulus of rupture.

Observers:

_____
_____

_____
In charge of tests.

METHOD OF SAMPLING AND TESTS OF WOOD BLOCK.

1. *Sampling.*—Ten blocks shall be taken as a sample.

2. *Measurement and weight.*—Each block is measured for length, breadth, and thickness, and the total volume in cubic inches of the 10 blocks is computed. The whole number of blocks are weighed to the nearest gram or one-eighth ounce and the weight in pounds per cubic foot of the block is calculated.

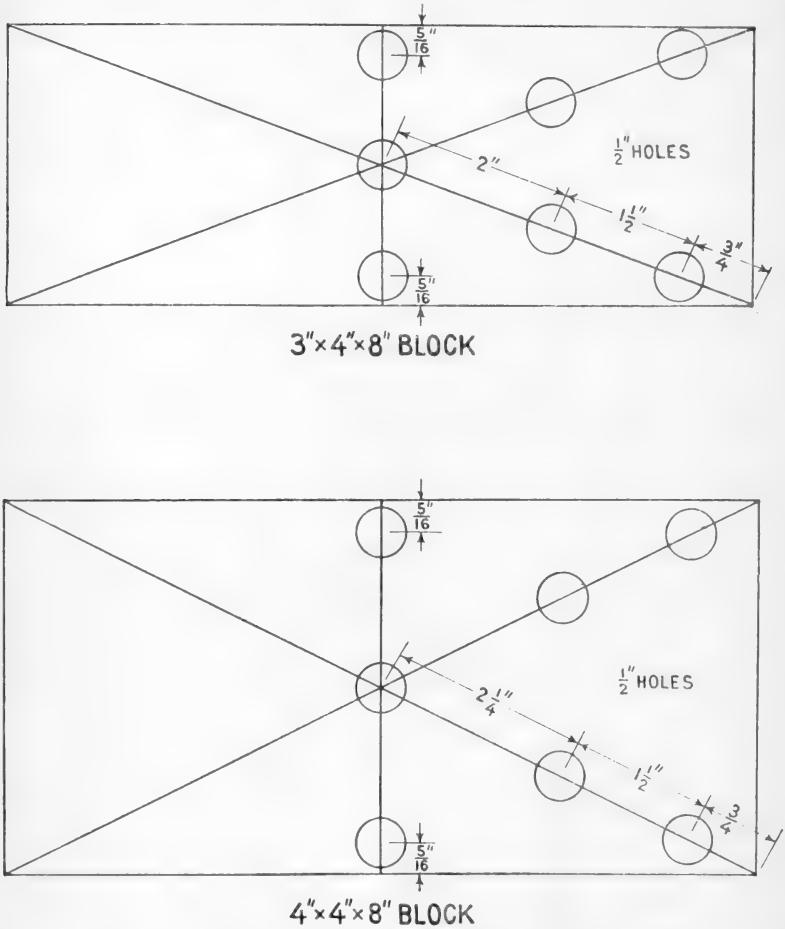


FIG. 1.—Method of boring wood blocks for sample. Borings made in end grain with ½-inch bit or drill. Three borings on center line made half way through block; other borings through block.

ANALYSES.

3. The sample for analyses shall be obtained by boring into the end grain of all the blocks, according to Figure 1.

Moisture.

4. *Moisture.*—Take 25 grams of borings and place in a 250 cubic centimeter Erlenmeyer flask and pour on about 75 to 100 cubic centimeters of water-saturated xylol. Connect the flask to a condenser with a short tube and distill off the xylol rapidly, continuing the distillation to near dryness, collecting the distillate

in a cylinder graduated to tenths of a cubic centimeter. The distillate will consist of xylol and water. The water will separate and collect in the bottom of the cylinder where the amount may be read off and calculated in percentage.

Oil. 5. *Oil.*—Take a C. S. & S. capsule or a folded hard paper (C. S. & S. No. 575) which has been previously extracted with benzol and dried. Weigh into this about 5 to 10 grams of borings, making the weighing in a weighing bottle. The capsule and borings are then extracted in some suitable extraction apparatus using benzol as the solvent until the washings run through practically colorless. Chloroform is then substituted for benzol as the solvent until the washings again run through practically colorless. The extraction removes from the borings water, rosin, and oil.

Rosin. 6. *Rosin.*—The benzol and chloroform washings are united and shaken out in a separatory funnel with a 3 per cent solution of sodium carbonate or preferably sodium hydrate, using about three 100 cubic centimeter portions. The alkali washings are drawn off and united, acidulated with sulphuric acid, and then shaken out with petroleum ether. The ether washings are united and filtered into a tared flask, evaporated on a hot plate, and the residue dried in the oven. This residue represents mainly rosin, although there is a possibility that it may contain some tar acids from the oil. The amount of oil with which the block is impregnated is obtained by taking the percentage of extract as determined under "Oil" and subtracting from it the percentage of water plus rosin.

7. All these results shall be expressed in percentage of the original weight of the block, and also in pounds per cubic foot.

METHODS OF SAMPLING AND TESTING BITUMINOUS MATERIALS.

(A. S. T. M. tentative method, serial designation, D 140–22 T, slightly modified.)

1. Samples may be taken for either of two purposes:

(*a*) To represent as nearly as possible an average of the bulk of the material sampled.

(*b*) To ascertain the maximum variation in characteristics which the material may possess. In either case they shall be obtained by methods hereinafter described.

2. (*a*) Care shall be taken that the samples are not contaminated with dirt or any other extraneous matter and that the sample containers are perfectly clean and dry before filling.

(*b*) Immediately after filling, the sample containers shall be tightly closed and properly marked for identification on the container itself or on a linen tag attached to the container.

3. (*a*) Whenever practicable, bituminous material shall be sampled at the point of manufacture, and at such time as to allow the tests controlling acceptance or rejection to be made in advance of shipment.

(*b*) When impracticable to take samples at the point of manufacture, they should be taken from the shipment immediately upon delivery.

4. For routine laboratory examination to determine the acceptability of a given lot, not less than 1 quart of material should be submitted which should be representative of the average sample collected as hereinafter described.

Containers. 5. Containers for liquid bituminous materials shall be small-mouth cans with cork-lined screw caps. Containers for semisolid and solid materials shall be friction top cans.

I. SAMPLING AT PLACE OF MANUFACTURE.

6. The inlet and outlet to the storage tank shall be sealed and a 1-gallon sample drawn from the top, middle, and bottom contents. The sample may be taken from drain cocks on the side of the tank, if such are available. Enough material should be dis-

carded to insure a representative sample. Otherwise, samples may be taken by lowering weighted bottles or cans into the material. The bottle or can should be fitted with a stopper which can be removed by a string or wire attached to it after it has been lowered to the proper depth. The three samples from bulk storage shall be tested separately for consistency in order to detect stratification. They may then be combined and thoroughly mixed for other tests that may be required.

7. Where tank cars, distributors, or barrels are being filled, samples may conveniently be taken from the pipe line through which the material is flowing, as hereinafter described.

(A) When Material is Pumped Under Pressure.

Material pumped.

8. The sampling pipe shall be inserted into a rising section of the pipe line on the discharge side of the pump. The sampling pipe shall be not more than one-eighth the diameter of the line pipe, and its inner open end shall be turned at an angle of 90° facing the flow of the liquid. This pipe shall be provided with a plug cock and shall discharge into a receiving drum of 50 gallons capacity. The plug cock shall be so adjusted that there is a steady continuous flow of bituminous material through it and shall be so regulated that the receiver will fill in the same time that is required to make the entire pumping. In the case of semisolid materials, the receiver shall be provided with a steam coil which shall keep the contents at a temperature just above the liquefying point. At completion of the pumping, the receiver shall be thoroughly agitated, and a 1-quart sample taken therefrom. The sampling shall be so regulated that for each 1,000 gallons of material pumped at least 1 gallon of sample is taken, but 40 gallons shall be the limit required for any one cargo.

Care should be taken that the drip cock, after once having been set, allows a constant flow during the pumping.

This method is also applicable to gravity flows where the pipe line is completely filled by the outflowing liquid and has a rising section.

(B) When Material Flows by Gravity.

Material flowing by gravity.

9. Material flowing by gravity through pipes which are not completely filled may be sampled by taking dipperfuls at the outlet at frequent and regular intervals. These samples shall be combined and the total sample shall be not less than 0.1 per cent of the whole material. The samples shall be collected in a receiver and resampled as described in section 8.

II. SAMPLING AT POINT OF DELIVERY.

10. Samples may be taken by means of a weighted bottle or can, as described in section 6, or from the unloading pipe line as described in section 7.

Liquid bituminous materials shall be sampled before heating. Semisolid or solid bituminous materials shall be rendered fluid by heating. Sampling should be so conducted as to eliminate the possibility of adventitious water resulting from leaky steam heating coils, rain, or snow.

11. Samples may be taken from distributors by means of a weighted bottle or can, as described in section 6. One sample shall be sufficient.

Liquid material in barrels.

12. (*a*) Samples of liquid bituminous materials from barrels or drums may be secured by taking 1 quart of material from 1 out of every 10 packages, combining and thoroughly mixing these samples, and then removing an average sample from the combined material.

Solids and semisolids in barrels.

(*b*) Sampling of solid or semisolid materials from barrels is more difficult to accomplish properly. It is recommended that samples be taken from each car lot, but if this sample fails to meet

the specification requirements, then a sample shall be taken from each batch represented or from 1 out of each 20 barrels, samples to be taken from at least 3 inches below the surface and at least 3 inches from the side of the barrel. A clean hatchet may be used if the material is hard enough to shatter and a broad, stiff putty knife if the material is soft. The samples so taken shall be melted and thoroughly mixed and an average sample taken from the combined material for examination.

METHODS OF SAMPLING BITUMINOUS MIXTURES.

A daily sample shall be taken by any of the following methods:

(*a*) Samples shall be cut from the finished pavement about 6 to 8 inches square and extending full depth of pavement before application of any sealing coat which may be used.

(*b*) Samples shall be taken during preparation of the paving mixture from trucks or wagons at the asphalt plant and combined into composite samples of not less than 1 pound each.

(*c*) Samples shall be taken either at the road or asphalt plant of any bituminous mixture which appears defective.

METHOD OF SAMPLING MINERAL FILLER.

Mineral filler shall be sampled according to the method for sampling Portland cement received in carload lots.

METHOD OF SAMPLING PREMOLDED JOINT FILLERS.

One section at least 1 foot long and the full width of the joint shall be submitted from each consignment.

DETERMINATION OF SPECIFIC GRAVITY OF BITUMINOUS MATERIALS.

1. The specific gravity of bituminous materials shall be expressed as the ratio of the weight of a given volume of the material at 25° C. (77° 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° F.).

A. Hydrometer Method (Used for Thin Fluid Bituminous).

Hydrometer method.

2. The specific gravity of thin fluid bituminous road materials may be determined with the above-mentioned apparatus by first pouring a sufficient quantity of the material into a tin cup which is then placed in a large dish containing cold or warm water, as occasion may require. The material in the cup should be stirred with the thermometer until it is brought to a temperature of 25° C., after which it should be immediately poured into the hydrometer jar and its gravity determined by means of the proper hydrometer. In case the hydrometer sinks slowly, owing to the viscosity of the material, it should be given sufficient time to come to a definite resting point, and this point should be checked by raising the hydrometer and allowing it to sink a second time. The hydrometer should never be pushed below the point at which it naturally comes to rest until the last reading has been made. It may then be pushed below the reading for a distance of three or four of the small divisions on the scale, whereupon it should immediately begin to rise. If it fails to do so, the material is too viscous for the hydrometer method, and the pycnometer method should be employed.

3. The direct specific gravity reading obtained by the foregoing method is based upon water at 15.5° C. taken as unity. For all practical purposes this reading may be corrected to water at 25° C., considered as unity, by multiplying it by 1.002. Thus: Specific gravity 25° C./25° C.=specific gravity 25°/15.5° C.×1.002.

B. PYCNOMETER METHOD (USED FOR VISCOUS FLUID AND SEMI-SOLID BITUMENS AND EMULSIONS).

Pycnometer method.

4. The determination of specific gravity shall be made with a Hubbard-Carmick pycnometer or weighing bottle, which shall consist of a conical or Erlenmeyer-shaped flask approximately 45 millimeters high, 40 millimeters in diameter at the bottom, and 25 millimeters in diameter at the mouth, carefully ground to receive an accurately fitting solid glass stopper with a hole of about 1 millimeter bore in place of the usual capillary opening. The lower surface of the stopper is made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shaped depression shall be about 4.8 millimeters at the center. The stoppered flask has a capacity of about 25 cubic centimeters and when empty weighs about 25 grams.

5. Before making a determination, the pycnometer with stopper shall first be calibrated by weighing it clean and dry upon an analytical balance. This weight is called *a*. It shall then be filled with freshly boiled distilled water at a temperature of 25° C. (77° F.), the stopper firmly inserted, all surplus moisture wiped from the surface with a clean, dry cloth, and again weighed. This weight is called *b*.

6. When determining the specific gravity of road oils or road tars which flow readily, the material shall be brought to a temperature of 25° C. (77° F.) and poured into the pycnometer until it is full, with care to prevent the inclusion of air bubbles. The stopper is then firmly inserted and all excess of material forced through the opening is carefully removed with a clean dry cloth. The pycnometer and contents are then weighed and this weight is called *c*. The specific gravity of the material shall be calculated from the formula:

$$\text{Specific gravity} = \frac{c-a}{b-a}$$

7. When determining the specific gravity of tar and asphalt products which are too viscous for the method described in section 4, a small amount of the material shall be brought to a fluid condition by the gentle application of heat, care being exercised to prevent loss by evaporation. When sufficiently fluid, enough is poured into the clean, dry pycnometer to about half fill it. Precautions shall be taken to keep the material from touching the sides of the tube above the final level and to prevent the inclusion of air bubbles. The tube should be slightly warmed before filling. The pycnometer and contents are then cooled to room temperature and weighed with the stopper. This weight is called *c*. The pycnometer is next removed from the balance, filled with freshly boiled distilled water, and the stopper firmly inserted. It is then completely immersed for not less than 30 minutes in a beaker of distilled water maintained at 25° C. (77° F.), after which it is removed, and all surplus water is wiped off with a clean cloth. It is immediately weighed. This weight is called *d*. The specific gravity of the material shall be calculated from the formula:

$$\text{Specific gravity} = \frac{c-a}{(b-a)-(d-c)}$$

8. When making the specific gravity determination it is important that:

(*a*) Only freshly boiled distilled water shall be used.

(*b*) When weighing the pycnometer completely filled, the temperature of its contents shall be within 1° C. (1.8° F.) of 25° C. (77° F.).

(*c*) Precautions shall be taken to prevent expansion and overflow of the contents from the heat of the hand when wiping the surface of the pycnometer.

(*d*) The presence of all air bubbles shall be eliminated in filling the pycnometer and inserting the stopper.

(*e*) Weighings shall be made quickly after filling the pycnometer and shall be accurate to 1 milligram. A number of trial fillings and catchweights may be necessary to obtain the desired degree of accuracy.

(*f*) To prevent breakage of the pycnometer when cleaning it out after a determination has been made upon a very viscous or semisolid material, it will be found advisable to warm it in an oven at not over 100° C. until most of the material may be poured out and then to swab it with a piece of soft cloth or cotton waste. When cool, it may be finally rinsed with carbon disulphide, benzol, or other solvent and wiped clean.

9. The limit of accuracy of the test is ±0.005 specific gravity.

C. Displacement Method (Used for Hard, Solid Bitumens).

Displacement method for solids.

10. For materials which are hard enough to be broken and handled in fragments at room temperature the following method will prove convenient: A small fragment of the bitumen (about 1 cubic centimeter) is suspended by means of a silk thread from the hook on one of the pan supports, about 1½ inches above the pan, and weighed. This weight is called *a*. It is then weighed immersed in water at 25° C., and this weight is called *b*. The specific gravity may then be calculated by means of the following formula:

$$\text{Specific gravity} = \frac{a}{a-b}$$

D. Displacement Method (Used for Semisolids).

Displacement method for semisolids.

11. The specific gravity of semisolid bituminous materials may be determined by the displacement method. Weigh a silica crucible suspended from the beam of the balance in air and call the weight *a*, and in water and call the weight *b*. Fill the crucible approximately two-thirds full with the material under examination. Free from bubbles by heating on a hot plate, cool and weigh, calling this weight *c*. Immerse the filled crucible in water at 25° C. for one-half hour, then suspend by a wire from the beam of the balance and weigh it immersed in water at 25° C.; call this weight *d*. The specific gravity is then calculated by means of the following formula:

$$\text{Specific gravity} = \frac{c-a}{d-b}$$

DETERMINATION OF BITUMEN SOLUBLE IN CARBON DISULPHIDE.

1. This test consists in dissolving the bitumen in carbon disulphide and recovering any insoluble matter by filtering the solution through an asbestos felt. The form of Gooch crucible best adapted for the determination is 4.4 centimeters wide at the top, tapering to 3.6 centimeters at the bottom, and is 2.5 centimeters deep.

Method of test.

2. For preparing the felt, the asbestos is cut with scissors into pieces not exceeding 1 centimeter in length, after which it is shaken up with just sufficient water to pour easily. The Gooch crucible is filled with the suspended asbestos, which is allowed to settle for a few moments. A light suction is then applied to draw off all the water and leave a firm mat of asbestos in the crucible. More of the suspended material is added, and the operation is repeated until the felt is so dense that it scarcely transmits light when held so that the bottom of the crucible is between the eye and the source of light. The felt should then be washed several times with water and drawn firmly against the bottom of the crucible by an increased suction. The crucible is removed to a drying oven for a few minutes, after which it is ignited at red heat over a Bunsen burner, cooled in a desiccator, and weighed.

3. From 1 to 2 grams of bitumen or about 10 grams of an asphalt topping or rock asphalt are now placed in the Erlenmeyer flask, which has been previously weighed, and the accurate weight of the sample is obtained. One hundred cubic centimeters of chemically pure carbon disulphide are poured into the flask in small portions, with continual agitation, until all lumps disappear and nothing adheres to the bottom. The flask is then corked and set aside for 15 minutes.

4. After being weighed, the Gooch crucible containing the felt is set up over the dry-suction flask, and the solution of bitumen in carbon disulphide is decanted through the felt with light suction or without suction by gradually tilting the flask, with care not to stir up any precipitate that may have settled out. At the first sign of any sediment coming out, the decantation is stopped and the filter allowed to drain. A small amount of carbon disulphide is then washed down the sides of the flask, after which the precipitate is brought upon the felt and the flask scrubbed, if necessary, with a feather or "policeman" to remove all adhering material. The contents of the crucible are washed with carbon disulphide, until the washings run colorless. Suction is then applied until there is practically no odor of carbon disulphide in the crucible, after which the outside of the crucible is cleaned with a cloth moistened with a small amount of solvent. The crucible and contents are dried in the hot-air oven at 100° C. for about 20 minutes, cooled in a desiccator, and weighed. If any appreciable amount of insoluble matter adheres to the flask, it should also be dried and weighed, and any increase over the original weight of the flask should be added to the weight of insoluble matter in the crucible. The total weight of insoluble material may include both organic and mineral matter. The former, if present, is burned off by ignition at a red heat until no incandescent particles remain, thus leaving the mineral matter or ash, which can be weighed on cooling. The difference between the total weight of material insoluble in carbon disulphide and the weight of substance taken equals the total bitumen, and the percentage weights are calculated and reported as total bitumen, and organic and inorganic matter insoluble, on the basis of the weight of material taken for analysis.

5. This method is quite satisfactory for straight oil and tar products, but where certain natural asphalts are present it will be found practically impossible to retain all of the finely divided mineral matter on an asbestos felt. It is therefore generally more accurate to obtain the results for total mineral matter by direct ignition of a 1-gram sample in a platinum crucible. The total bitumen is then determined by deducting from 100 per cent the sum of the percentage of total mineral matter and organic matter insoluble. If the presence of a carbonate mineral is suspected, the percentage of mineral matter may be most accurately obtained by treating the ash with a few drops of ammonium carbonate solution, drying at 100° C., then heating for a few minutes at a dull red heat, cooling, and weighing again.

6. When difficulty in filtering is experienced—for instance, when Trinidad asphalt is present in any amount—a period of longer subsidence than 15 minutes is necessary, and the following method proposed by the committee on standard tests for road materials of the American Society for Testing Materials is recommended:

7. From 2 to 15 grams (depending on the richness in bitumen of the substance) are weighed into a 150 cubic centimeter Erlenmeyer flask, the tare of which has been previously ascertained, and treated with 100 cubic centimeters of carbon disulphide. The flask is then loosely corked and shaken from time to time until practically all large particles of the material have been broken up, when it is set aside and not disturbed for 48 hours. The solution is then decanted off into a similar flask that has been previously weighed, as much of the solvent being poured off as possible without disturbing the residue. The first flask is again

treated with fresh carbon disulphide and shaken as before, when it is put away with the second flask and not disturbed for 48 hours.

8. At the end of this time the contents of the two flasks are carefully decanted off upon a weighed Gooch crucible fitted with an asbestos filter, the contents of the second flask being passed through the filter first. The asbestos filter shall be made of ignited long-fiber amphibole, packed in the bottom of a Gooch crucible to the depth of not over one-eighth of an inch. After passing the contents of both flasks through the filter, the two residues are shaken with more fresh carbon disulphide and set aside for 24 hours without disturbing, or until it is seen that a good subsidation has taken place, when the solvent is again decanted off upon the filter. This washing is continued until the filtrates or washings are practically colorless.

9. The crucible and both flasks are then dried at 125° C. and weighed. The filtrate containing the bitumen is evaporated, the bituminous residue burned, and the weight of the ash thus obtained added to that of the residue in the two flasks and the crucible. The sum of these weights deducted from the weight of substance taken gives the weight of bitumen extracted.

NOTE.—This method for recovery of mineral material should also be used in case of asphalt topping and rock asphalt if analyzed as previously described.

DETERMINATION OF BITUMEN INSOLUBLE IN CARBON TETRACHLORIDE.

1. This determination is conducted in exactly the same manner as described under "Determination of bitumen soluble in carbon disulphide," using 100 cubic centimeters of chemically pure carbon tetrachloride as a solvent in place of carbon disulphide.

2. The percentage of bitumen insoluble is reported upon the basis of total bitumen taken as 100, as described under "Determination of bitumen insoluble in paraffin naphtha."

DETERMINATION OF BITUMEN INSOLUBLE IN PARAFFIN NAPHTHA.

1. This determination is made in the same general manner as the total bitumen determination, except that 100 cubic centimeters of 86° to 88° Baumé paraffin naphtha, at least 85 per cent distilling between 35° and 65° C., is employed as a solvent instead of carbon disulphide. Considerable difficulty is sometimes experienced in breaking up some of the heavy semisolid bitumens; the surface of the material is attacked, but it is necessary to remove some of the insoluble matter in order to expose fresh material to the action of the solvent. It is, therefore, advisable to heat the sample after it is weighed, allowing it to cool in a thin layer around the lower part of the flask. If difficulty is still experienced in dissolving the material, a rounded glass rod will be found convenient for breaking up the undissolved particles. Not more than one-half of the total amount of naphtha required should be used until the sample is entirely broken up. The balance of the 100 cubic centimeters is then added, and the flask is twirled a moment in order to mix the contents thoroughly, after which it is corked and set aside for 30 minutes.

2. In making the filtration the utmost care should be exercised to avoid stirring up any of the precipitate, in order that the filter may not be clogged and that the first decantation may be as complete as possible. The sides of the flask should then be quickly washed down with naphtha and, when the crucible has drained, the bulk of insoluble matter is brought upon the felt. Suction may be applied when the filtration by gravity almost ceases, but should be used sparingly, as it tends to clog the filter by packing

the precipitate too tightly. The material on the felt should never be allowed to run entirely dry until the washing is completed, as shown by the colorless filtrate. When considerable insoluble matter adheres to the flask no attempt should be made to remove it completely. In such cases the adhering material is merely washed until free from soluble matter and the flask is dried with the crucible at 100° C. for about one hour, after which it is cooled and weighed. The percentage of bitumen insoluble is reported upon the basis of total bitumen taken as 100.

3. The difference between the material insoluble in carbon disulphide and in the naphtha is the bitumen insoluble in the latter. Thus, if in a certain instance it is found that the material insoluble in carbon disulphide amounts to 1 per cent and that 10.9 per cent is insoluble in naphtha, the percentage of bitumen insoluble would be calculated as follows:

$$\frac{\text{Bitumen insoluble in naphtha}}{\text{Total bitumen}} = \frac{10.9-1}{100-1} = \frac{9.9}{99} = 10 \text{ per cent.}$$

TEST FOR LOSS ON HEATING OF OIL AND ASPHALTIC COMPOUNDS.

(A. S. T. M. standard method, serial designation D 6–20, slightly modified.)

1. This test is used to determine the loss in weight (exclusive of water) of oil and asphaltic compounds when heated as hereinafter prescribed. The material under examination must therefore first be tested for water, and if water is found to be present it must be removed by suitable methods of dehydration before the material is subjected to the loss-on-heating test, or another sample obtained which is free from water.

I. APPARATUS.

Oven.

2. The oven may be either circular or rectangular in form and may be heated by either gas or electricity. Its interior dimensions shall be approximately as follows: Height, not less than 40.64 centimeters (16 inches); width and depth or diameter, at least 5.08 centimeters (2 inches) greater than the diameter of the revolving shelf.

It shall be well ventilated and shall be fitted with a window in the upper half of the door, so placed and of sufficient size to permit the accurate reading of the thermometer without opening the door. It shall also be provided with a perforated circular shelf, preferably of approximately 24.8 centimeters (9.75 inches) in diameter. This shelf shall be placed in the center of the oven and shall be suspended by a vertical shaft and provided with mechanical means for rotating it at the rate of five or six revolutions per minute. It shall be provided with recesses equidistant from the central shaft in which the tins containing the samples are to be placed.

Thermometer.

3. The thermometer shall be between 12.7 centimetres (5 inches) and 15.24 centimeters (6 inches) in length, and the mercury bulb shall be from 10 to 15 millimeters (0.39 to 0.59 inch) in length. The scale shall be engraved on the stem, shall be clear cut and distinct, and shall run from 150° to 175° C. (302° to 347° F.) in 1° C. divisions and shall commence substantially 3.81 centimeters (1½ inches) above the top of the bulb. Every fifth graduation shall be larger than the intermediate ones and shall be numbered. The degrees shall be substantially 3.17 millimeters (one-eighth inch) apart. The thermometer shall be furnished with an expansion chamber at the top and have a ring for attaching tags. It shall be made of a suitable quality of glass and shall be so annealed as not to change its readings under conditions of use. It shall be correct to 0.25° C. (0.45° F., as determined by comparison at full immersion with a similar thermom-

eter calibrated at full immersion by the United States Bureau of Standards.

Container.

4. The container in which the sample is to be tested shall be of tin, cylindrical in shape, and shall have a flat bottom. Its inside dimensions shall be substantially as follows: Diameter, 55 millimeters (2.17 inches); depth, 35 millimeters (1.38 inches). (A 3-ounce gill style ointment box, deep pattern, fulfills these requirements.) For cold-application road oils the container shall be a 2-ounce can approximately 60 millimeters in diameter and 20 millimeters deep.

II. PREPARATION OF SAMPLE.

5. The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

III. PROCEDURE.

Method of test.

6. Weigh 50 grams of the water-free material to be tested into a tared container conforming to the requirements of section 4. For cold-application road oils use 20 grams of material. Bring the oven to a temperature of 163° C. (325° F.) and place the tin box containing the sample in one of the recesses of the revolving shelf. The thermometer shall be immersed for the depth of its bulb in a separate 50-gram sample of the material under test, placed in a similar container, and shall be conveniently suspended from the vertical shaft. This sample shall rest in one of the recesses upon the same shelf and revolve with the sample or samples under test. Then close the oven and rotate the shelf five to six revolutions per minute during the entire test. Maintain the temperature at 163° C. (325° F.) for five hours, then remove the sample from the oven, cool, and weigh, and calculate the loss due to volatilization.

7. During the five-hour period the temperature shall not vary more than 1° C. All tests showing a greater variation in temperature shall be rejected.

8. Under ordinary circumstances a number of samples having about the same degree of volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. Where 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. Such duplicates shall check within the limits of accuracy given below. Results obtained on samples showing evidence of foaming during the test shall be rejected.

IV. ACCURACY.

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

Volatilization loss.	Numerical correction.	True volatilization loss.
Per cent.		*Per cent.*
5.0	±0.50	4.50 to 5.50
5.5	± .51	4.91 to 6.01
6.0	± .52	5.48 to 6.52
10.0	± .60	9.40 to 10.60
15.0	± .70	14.30 to 15.70
25.0	± .90	24.10 to 25.90
40.0	±1.20	38.80 to 41.20

NOTE.—If additional periods of heating are desired, it is recommended that they be made in successive increments of five hours each. When other tests of the sample after heating are required, melt the residue in the container at the lowest possible temperature and thoroughly mix by stirring, taking care to avoid incorporating air bubbles in the mass.

TEST FOR FLASH AND FIRE POINTS BY MEANS OF OPEN CUP.

(A. S. T. M. tentative method, serial designation D 92–21 T, slightly modified.

1. (*a*) The open-cup flash shall be determined in the Cleveland open cup, or by method 2 (*b*).

(*b*) The method used shall be stated in the report of the test.

APPARATUS.

Flash cup.

2. (*a*) The flash cup proper shall be made of brass and shall have the following dimensions:

	Dimensions.	Tolerances.	Dimensions.	Tolerances.
	Inches.	*Inches.*	*Centimeters.*	*Centimeters.*
Inside diameter	2½	±1/32	6.350	±0.079
Outside diameter	2⅛	±1/32	6.826	± .079
Inside height	1⅜	±1/32	3.334	± .079
Thickness of bottom	⅛	±1/64	.318	± .040
Depth of filling mark below top of cup	3/8	±1/64	.953	± .040

The cup shall be heated by contact with a metal plate one-quarter inch (0.635 centimeter) thick and 6 inches (15.24 centimeter) wide. (The plate may be of any suitable metal and may be either circular or square.) In the center of the plate there shall be a plane depression one-thirty-second inch (0.079 centimeter) deep and of diameter just sufficient to fit the cup. The plate shall be covered with a sheet of hard asbestos board one-quarter inch (0.635 centimeter) thick and of the same shape as the metal plate. There shall be cut in the center of the asbestos board a circular hole just fitting the cup. The metal plate may be heated in any convenient manner. The use of a gas burner, electric heater, or alcohol lamp is permitted. If a flame heater is used, it may be protected from drafts or excessive radiation by any suitable type of shield that does not project above the level of the upper surface of the asbestos board.

(*b*) The cup proper shall be a 3-ounce gill type can 55 millimeters in diameter and 35 millimeters in depth. The can shall be heated in a sand bath of a diameter approximately twice that of the can. The sand shall be about one-quarter inch deep beneath the can and an additional amount of sand shall be spread around the can, so as to surround it to a depth of one-half inch.

Thermometer.

3. The thermometer shall conform to the following specifications:

Type: Etched-stem glass.

Total length: 305 millimeters.

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 millimeters.

Bulb: Corning Normal, Jena 16 III, or equally suitable thermometric glass. Length, 13 millimeters maximum. Diameter, not greater than stem.

Actuating liquid: Mercury.

Range: 20° to 760° F.

Immersion: 1 inch. The words "1 in. immersion" shall be etched on the stem and also a line around the stem to indicate the depth of immersion.

Distance to 20° line from bottom of bulb: 40 to 50 millimeters.

Distance to 760° line from top of stem: 30 to 45 millimeters.

Filled: Nitrogen gas.

Top finish: Red glass ring.

Graduation: All lines, figures, and letters clear-cut and distinct. Scale graduated in 5° divisions. Scale numbered every 20°, the first and each succeeding 10° F. line to be longer than the others.

Special marking: "A. S. T. M. open flash," serial number, and manufacturers name or trade-mark etched on stem.

Accuracy: Error at any point on scale shall not exceed one-half of the smallest scale division up to 700° F.

Test for permanency of range: After being subjected to a temperature of 700° F. for 24 hours the accuracy shall be within the limit specified.

Points to be tested for certification: 32°, 212°, 400°, 700° F.

PROCEDURE.

Method of test.

4. (*a*) The thermometer shall be suspended or held in a vertical position by any suitable device. The bottom of the bulb shall be one-quarter inch (0.635 centimeter)[13] from the bottom of the cup and above a point halfway between the center and back of the cup.

(*b*) The cup shall be filled with the oil to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature. The surface of the oil shall be free from bubbles. There shall be no oil above the filling line or on the outside of the apparatus.

(*c*) The test flame shall be approximately five thirty-seconds inch (0.397 centimeter) in diameter.

NOTE.—For purposes of comparison it is recommended that a bead of suitable light-colored material be mounted in a convenient position so that the size of the test flame can be determined. The device for applying the flame may be of any suitable type, but it is suggested that the tip be approximately one-sixteenth inch (0.159 centimeter) in diameter at the end and that the orifice be one thirty-second inch (0.079 centimeter) in diameter. If the device for operating the test flame be mounted in such a manner as to permit automatic duplication of the sweep of the test flame, the radius of swing shall be not less than 6 inches.

(*d*) The test flame shall be applied as the temperature read on the thermometer reaches each successive 5° F. mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 6 inches) across the center of the cup and at right angles to the diameter passing through the thermometer. The test flame shall, while passing across the surface of the oil, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approximately 1 second.

(*e*) The rate of heating of the oil shall be such that the temperature read on the thermometer increases not less than 9° nor more than 11° F. per minute.

5. The flash point shall be taken as the temperature read on the thermometer when a flash appears at any point on the surface of the oil. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame.

6. After determining the flash point the heating shall be continued at the specified rate, and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn for a period of at least 5 seconds. The temperature read when this occurs shall be taken as the fire point.

7. The flash-point and fire-point tests shall be made in a room or compartment free from air drafts. The operator shall avoid breathing over the surface of the oil. It is desirable that the room or compartment be darkened sufficiently so that the flash may be readily discernible.

TEST FOR PENETRATION OF BITUMINOUS MATERIALS.

(A. S. T. M. standard method, serial designation D 5–21, slightly modified.)

I. DEFINITION.

1. Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically

[13] The immersion line engraved on the thermometer stem will be one-sixteenth inch (0.159 centimeter) below the level of the rim of the cup.

penetrates a sample of the material under known conditions of loading, time, and temperature. Where the conditions of test are not specifically mentioned, the load, time, and temperature are understood to be 100 grams, 5 seconds, 20° C. (77° F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

II. APPARATUS.

Container. 2. The container for holding the material to be tested shall be a flat-bottom, cylindrical dish, 55 millimeters ($2\frac{3}{16}$ inches) in diameter and 35 millimeters ($1\frac{3}{8}$ inches) deep.[14]

Needle. 3. The needle for this test shall be a cylindrical steel rod 50.8 millimeters (2 inches) long, having a diameter of 1.01 to 1.02 millimeters and having a taper of 6.34 to 6.36 millimeters measured on the axis. After tapering, the point shall be "blunted" by grinding off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 millimeters in diameter. A Roberts No. 2 parabola needle checked against the standard needle may be used.

Water bath. 4. The water bath shall be maintained at a temperature not varying more than 0.1° C. from 25° C. (77° F.). The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 centimeters (4 inches) and shall be supported on a perforated shelf not less than 5 centimeters (2 inches) from the bottom of the bath.

Penetration machine. 5. Any apparatus which will allow the needle to penetrate without appreciable friction, and which is accurately calibrated to yield results in accordance with the definition of penetration, will be acceptable.

Transfer dish. 6. The transfer dish for the container shall be a small dish or tray of such capacity as will insure complete immersion of the container during the test. It shall be provided with some means which will insure a firm bearing and prevent rocking of the container.

III. PREPARATION OF SAMPLE.

Preparation of sample. 7. The sample shall be completely melted at the lowest possible temperature and stirred thoroughly until it is homogenous and free from air bubbles. It shall then be poured into the sample container to a depth of less than 15 millimeters (five-eighths inches). The sample shall be protected from dust and allowed to cool in an atmosphere not lower than 18° C. (65° F.) for one hour. It shall then be placed in the water bath along with the transfer dish and allowed to remain one hour.

IV. PROCEDURE.

Method of test. 8. (*a*) In making the test the sample shall be placed in the transfer dish filled with water from the water bath of sufficient depth completely to cover the container. The transfer dish containing the sample shall be placed upon the stand of the penetration machine. The needle loaded with specified weight, shall be adjusted to make contact with the surface of the sample. This may be accomplished 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. Either the reading of the dial shall then be noted or the needle brought to zero. The needle is then released for the specified period of time, after which the penetration machine is adjusted to measure the distance penetrated.

At least three tests shall be made at points on the surface of the sample not less than 1 centimeter (three-eighths inch) from the side of the container and not less than 1 centimeter (three-

[14] This requirement is fulfilled by the American Can Co.'s gill style ointment box, deep pattern, 3-ounce capacity.

eighths inch) apart. After each test the sample and transfer dish shall be returned to the water bath and the needle shall be carefully wiped toward its point with a clean, dry cloth to remove all adhering bitumen. The reported penetration shall be the average of at least three tests whose values shall not differ more than four points between maximum and minimum.

(*b*) When desirable to vary the temperature, time, and weight, and, in order to provide for a uniform method of reporting results when variations are made, the samples shall be melted and cooled in air as above directed. They shall then be immersed in water or brine, as the case may require, for one hour at the temperature desired. The following combinations are suggested:

At 0° C. (32° F.) 200 grams weight, 60 seconds.
At 46.1° C. (115° F.) 50 grams weight, 5 seconds.

FLOAT TEST FOR BITUMINOUS ROAD MATERIALS.

(A. S. T. M. tentative method, serial designation, D 139–22 T, slightly modified.)

I. APPARATUS.

1. The float shall be made of aluminum alloy and shall be in accordance with the following requirements: *Float.*

	Minimum.	Normal.	Maximum.
Weight of float.....grams..	37.80	37.95	38.10
Total height of float.....millimeters..	34.0	35.0	36.0
Height of rim above lower side of shoulder .....millimeters..	26.5	27.0	27.5
Thickness of shoulder.....do....	1.2	1.4	1.6
Diameter of opening.....do....	10.7	11.1	11.5
Height of rim above water with load of 5.5 grams.....millimeters..	7.0	8.5	10.0

2. The collar shall be made of brass and shall be in accordance with the following requirements: *Collar.*

	Minimum.	Normal.	Maximum.
Weight of collar.....grams..	9.9	9.95	10.0
Over-all height of collar.....millimeters..	22.3	22.5	22.7
Inside diameter at bottom.....do....	12.78	12.80	12.82
Inside diameter at top.....do....	9.66	9.68	9.70

3. The thermometer shall conform to the following specifications: *Thermometer.*

Total length: 370 to 400 millimeters (14.57 to 15.75 inches).
Diameter: 6.5 to 7.5 millimeters (0.256 to 0.295 inch).
Bulb length: Not over 14 millimeters (not over 0.55 inch).
Bulb diameter: 4.5 to 5.5 millimeters (0.177 to 0.217 inch).

The scale shall be engraved upon the stem of the thermometer, shall be clear-cut and distinct, and shall run from 0° to 80° C. (32° to 176° F.) in 0.2° C. divisions. It shall commence not less than 75 millimeters (2.95 inches) above the bottom of the bulb. The thermometer shall be furnished with an expansion chamber at the top and have a ring for attaching tags. It shall be made of a suitable quality of glass and shall be so annealed as not to change its readings under conditions of use. It shall be correct to 0.25° C. (0.45° F.) as determined by comparison at full immersion with a similar thermometer calibrated at full immersion by the United States Bureau of Standards.

4. The minimum diameter of the bath shall be twice the diameter of the float; the minimum depth of water shall be equal to the diameter of the bath.

II. PREPARATION OF SAMPLE.

Preparation of sample.

5. The sample shall be completely melted at the lowest possible temperature, heating only sufficiently to bring the product into a fluid condition, but not above 350° F., and stirred thoroughly until it is homogeneous and free from air bubbles.

The brass collar shall be placed with the smaller end on a brass plate which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate and then with mercury. The sample shall be

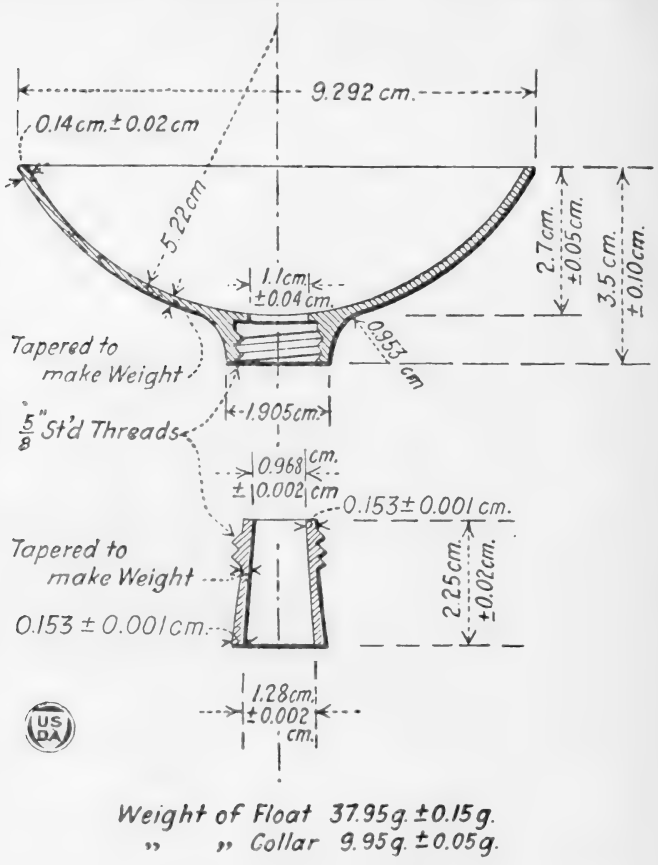


FIG. 2.—Aluminum float for float test.

poured into the collar in any convenient way until slightly more than level with the top. It shall be cooled to room temperature, placed in water maintained at 5° C. for 5 minutes, after which the surplus material shall be removed by means of a spatula, or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., ±1° C., and left in this bath for at least 15 minutes.

III. PROCEDURE.

Method of test.

6. (*a*) The bath shall be filled with water and the water heated to the temperature at which the test is to be made. This temperature shall be accurately maintained and shall at no time

throughout the test be allowed to vary more than 0.5° C. from the temperature specified.

(*b*) After the material to be tested has been kept in the ice water for not less than 15 minutes nor more than 20 minutes, the collar with its contents shall be removed from the plate and screwed into the aluminum float and immersed in water at 5° C. for 1 minute. Any water shall then be removed from the inside of the float and the latter immediately floated in the warm bath. As the plug of material becomes warm and fluid, it is forced upward and out of the collar until the water gains entrance into the saucer and causes it to sink.

(*c*) The time in seconds between the placing of the apparatus on the water and the instant when the water breaks through the material shall be determined by means of a stop watch, and shall be taken as a measure of the consistency of the material under examination.

NOTE.—Special precaution should be taken to insure the collar fitting tightly into the float and to see that there is no seepage of water between the collar and float during the test.

TEST FOR SOFTENING POINT OF BITUMINOUS MATERIALS (RING-AND-BALL METHOD).

(A. S. T. M. standard method, serial designation D 36–21, slightly modified.)

1. The softening of bituminous materials generally takes place at no definite moment or temperature. As the temperature rises, they gradually and imperceptibly change from a brittle or exceedingly thick and slow-flowing material to a softer and less viscous liquid. For this reason the determination of the softening point must be made by a fixed, arbitrary, and closely defined method if the results obtained are to be comparable.

I. APPARATUS.

2. The apparatus shall consist of the following:

Brass ring.

(*a*) A brass ring 15.875 millimeters (five-eight inch) in inside diameter and 6.35 millimeters (one-quarter inch) deep; thickness of wall, 2.38 millimeters (three thirty-second inch); permissible variation of inside diameter and thickness of ring, 0.25 millimeters (0.01 inch). This ring shall be attached in a convenient manner to a No. 13 B. & S. gauge brass wire (diameter 1.83 millimeters=0.0720 inch). (See fig. 3.)

Steel ball.

(*b*) A steel ball 9.53 millimeters (three-eighth inch) in diameter weighing between 3.45 and 3.55 grams.

Vessel.

(*c*) A glass vessel, capable of being heated, not less than 8.5 centimeters (3.34 inches) in diameter and measuring 10.5 centimeters (4.13 inches) in depth from the bottom of the flare. (A 600 cubic centimeter beaker, low form, meets this requirement.)

Thermometer.

(*d*) A thermometer which shall conform to the following specifications:

Total length: 370 to 400 milimeters (14.57 to 15.75 inches).
Diameter: 6.5 to 7.5 millimeters (0.0256 to 0.295 inch).
Bulb length: Not over 14 millimeters (not over 0.55 inch).
Bulb diameter: 4.5 to 5.5 millimeters (0.177 to 0.217 inch).

The scale shall be engraved upon the stem of the thermometer, shall be clear-cut and distinct, and shall run from 0° to 80° C. (32° to 176° F.) in 0.2° C. divisions. It shall commence not less than 7.5 centimeters (2.95 inches) above the bottom of the bulb. The thermometer shall be furnished with an expansion chamber at the top and have a ring for attaching tags. It shall be made of a suitable quality of glass and shall be so annealed as not to change its readings under conditions of use. It shall be correct to 0.25° C. (0.45° F.) as determined by comparison at full immersion with a similar thermometer calibrated at full immersion by the United States Bureau of Standards.

II. PREPARATION OF SAMPLE.

Preparation of sample.

3. The sample shall be melted and stirred thoroughly, avoiding incorporating air bubbles in the mass, and then poured into the ring so as to leave an excess on cooling. The ring, while being filled, should rest on a brass plate which has been amalgamated to prevent the bituminous material from adhering to it. After cooling, the excess material shall be cut off cleanly with a slightly heated knife.

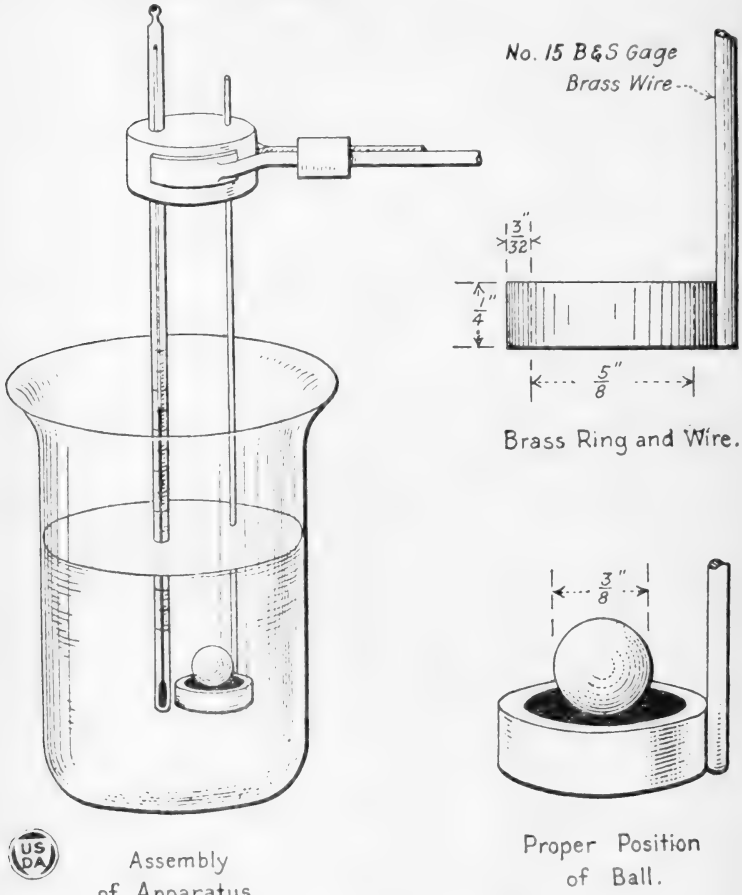


FIG. 3.—Apparatus for ring-and-ball method.

III. PROCEDURE.

Method of test—materials of low softening points.

(A) BITUMINOUS MATERIAL HAVING SOFTENING POINTS 80° C. (176° F.) OR BELOW.

4. Assemble the apparatus as shown in Figure 3. Fill the glass vessel to a depth of substantially 8.25 centimeters (3.25 inches) with freshly boiled, distilled water 5° C. (41° F.). Place the ball in the center of the upper surface of the bitumen in the ring and suspend it in the water so that the lower surface of the filled ring is exactly 2.54 centimeters (1 inch) above the bottom of the glass vessel and its upper surface is 5.08 cen-

timeters (2 inches) below the surface of the water. Allow it to remain in the water for 15 minutes before applying heat Suspend the thermometer so that the bottom of the bulb is level with the bottom of the ring and within 0.635 centimeters (one-quarter inch) but not touching the ring.

NOTE.—More than one ring may be used at one time.

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

6. The temperature recorded by the thermometer at the instant the bituminous material touches the bottom of the glass vessel shall be reported as the softening point.

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

(A) BITUMINOUS MATERIALS HAVING SOFTENING POINTS ABOVE 80° C. (176° F.).

Method of test—materials of high softening points.

8. To use the same method as given under (A), except that glycerin shall be used instead of water, and that the thermometer shall conform to the following specifications:

Total length: 370 to 400 millimeters (14.57 to 15.75 inches).
Diameter: 6.5 to 7.5 millimeters (0.256 to 0.295 inch).
Bulb length: Not over 14 millimeters (not over 0.55 inch).
Bulb diameter: 4.5 to 5.5 millimeters (0.177 to 0.217 inch).

The graduations shall be from 30° to 160° C. in 0.5°C. and shall be clear-cut and distinct. The 30° mark shall be at least 75 millimeters above the bottom of the bulb. The length between the 30° mark and the 160° mark shall be between 230 and 275 millimeters.

The thermometer shall be furnished with an expansion chamber at the top and have a ring for attaching tags. It shall be made of a suitable quality of glass and shall be so annealed as not to change its readings under conditions of use. It shall be correct to 0.25° C. (0.45° F.) as determined by comparison at full immerson with a similar thermometer calibrated at full immersion by the United States Bureau of Standards.

IV. PRECAUTIONS.

9. 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.

V. ACCURACY.

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

METHOD FOR DETERMINATION OF SPECIFIC VISCOSITY.

Engler viscosimeter.

The viscosity of fluid bituminous road materials may be determined at any suitable temperature by means of the Engler viscosimeter. This apparatus is shown in Figure 4, and may be described as follows: *a* is a brass vessel for holding the material to be tested, and may be closed by the cover *b*. To the conical bottom of *a* is fitted a conical outflow tube, *c*, exactly 20 millimeters long, with a diameter at the top of 2.9 millimeters and at the bottom of 2.8 millimeters. This tube can be closed and opened

by the pointed hardwood stopper *d*. Pointed metal projections are placed on the inside of *a* at equal distances from the bottom and serve for measuring the charge of material, which is 240 cubic centimeters. The thermometer *e* is used to ascertain the temperature of the material to be tested. The vessel *a* is surrounded by a brass jacket, *f*, which holds the material used as a heating bath, either water or cottonseed oil, according to the temperature at which the test is to be made. A tripod, *g*, serves as a support for the apparatus and also carries a ring burner, *h*, by means of which the bath is directly heated. The measuring cylinder of 50 cubic centimeter capacity, which is sufficiently accurate for work with road materials, is placed directly under the outflow tube.

Method of test. As all viscosity determinations should be compared with that of water at 25° C., the apparatus should be previously calibrated as follows: The cup and outlet tube should first be scrupulously

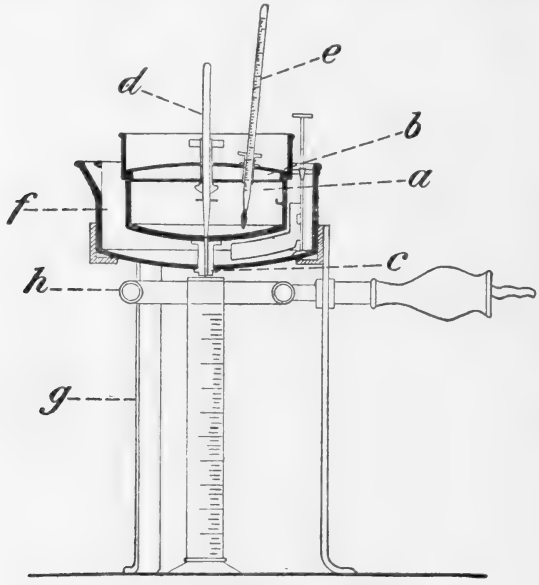


FIG. 4.—Engler viscosimeter.

cleaned. A piece of soft tissue paper is convenient for cleaning the latter. The stopper is then inserted in the tube and the cup filled with water at 25° C. to the top of the projections. The measuring cylinder should be placed directly under the outflow tube so that the material, upon flowing out, will not touch the sides, and the stopper may then be removed. The time required for 50 cubic centimeters to run out should be ascertained by means of a stop watch, and the results so obtained should be checked a number of times. The time required for 50 cubic centimeters of water should be about 11 seconds.

Bituminous road materials are tested in the same manner as water, and the temperature at which the test is made is controlled by the bath. The material should be brought to the desired temperature and maintained there for at least three minutes before making the test. The results are expressed as specific viscosity compared with water at 25° C., as follows:

$$\text{Specific viscosity at } A^\circ \text{ C.} = \frac{\text{seconds for passage of 50 cubic centimeters at } A^\circ \text{ C.}}{\text{seconds for passage of 50 cubic centimeters of water at } 25^\circ \text{ C.}}$$

TEST FOR DUCTILITY OF BITUMINOUS MATERIALS.

(A. S. T. M. tentative method, serial designation D 113–21 T., slightly modified.)

1. The ductility of an asphalt cement or semisolid bitumen is the distance to which it will elongate before breaking when two ends of a briquet of the material are pulled apart at a specified speed and at a specified temperature. When the conditions of the test are not specifically mentioned, the rate and the temperature are understood to be 5 centimeters per minute at 25° C. (77° F.).

APPARATUS.

Mold.

2. The mold shall be as shown in Figure 5. It shall be made of brass, the ends, *B* and *B'*, being known as clips, and the parts *A* and *A'*, as sides of the mold. The dimensions of the mold shall be as follows:

Total length (internal): 7.45 to 7.55 centimeters.
Distance between clips: 2.97 to 3.06 centimeters.
Width of clips at mouth: 1.98 to 2.02 centimeters.
Width of briquet at minimum cross section (halfway between clips): 0.99 to 1.01 centimeters.
Thickness of briquet throughout: 0.99 to 1.01 centimeters.

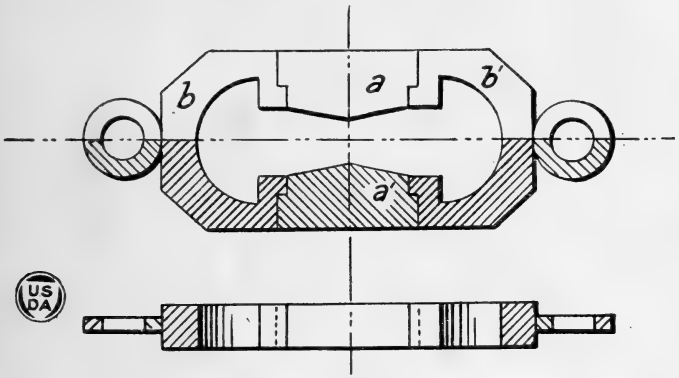


FIG. 5.—Ductility mold.

Water bath.

3. The water bath shall be maintained at a temperature not varying more than 0.1° C. from 25° C. (77° F.). The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 centimeters and shall be supported on a perforated shelf not less than 5 centimeters from the bottom of the bath.

Ductility apparatus.

4. Any apparatus may be used for pulling the briquet of bitumen apart that is so constructed that the briquet will be continuously immersed in water and the two clips pulled apart at a uniform speed of 5 centimeters per minute.

PROCEDURE.

Method of test.

5. The asphalt cement or bituminous material to be tested shall be completely melted at such a temperature that it will be thoroughly fluid—with ordinary paving asphalt cement this is about 160° C. (320° F.). It shall then be strained through a 50-mesh sieve and, after a thorough stirring, poured into the mold. The mold shall be assembled on a brass plate and, so as to prevent the material under test from sticking, the surface of the plate and the interior surfaces of the side pieces *A A'* of the mold shall be thoroughly amalgamated. In filling the mold, care shall be taken not to disarrange the parts and thus distort the briquet.

In filling, the material shall be poured in a thin stream back and forth from end to end of the mold until it is more than level full. It shall then be left to cool in the air at a temperature not lower than 15.5° C. (60° F.), for 30 minutes, after which the excess of bitumen shall be cut off by means of a hot putty knife or spatula so that the mold shall be just level full.

6. The brass plate and mold, with briquet, shall then be placed in the water bath and kept at a temperature of 25° C. (77° F.) for at least 1½ hours, when the briquet shall be removed from the plate and the side pieces detached.

7. The rings on the clips shall then be attached to the pins or hooks in the ductility machine and the two clips pulled apart at a uniform speed of 5 centimeters per minute (±5 per cent). While the test is being made the water in the tank of the ductility machine shall cover the sample by at least 25 centimeters and shall be kept continuously at a temperature within 0.5° C. of 25° C. (77° F.). When the specimen of bituminous material breaks, the distance from the original position of the clip before pulling to its present position shall be measured, and shall be known as the ductility. The average of three tests shall be taken, excepting that any abnormal result shall be rejected.

TEST FOR DISTILLATION OF BITUMINOUS MATERIALS SUITABLE FOR ROAD TREATMENT.

(A. S. T. M. standard method, serial designation D 20–18, slightly modified.)

1. The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

2. The distillation test shall be made on the sample as received, unless the sample shows in the distillation test water in excess of 3 per cent. In that case the sample shall be previously dehydrated in accordance with the following method: About 500 cubic centimeters of the material are placed in an 800 cubic centimeter copper still provided with a distilling head connected with a water-cooled condenser. A ring burner is used, starting with a small flame at the top of the still and gradually lowering it, if necessary, until all the water has been driven off. The distillate is collected in a 200 cubic centimeter separatory funnel, with the tube cut off close to the stopcock. When all the water has been driven over and the distillate has settled out, the water is drawn off and the oils returned to the residue in the still. The contents of the still shall have cooled to below 100° C. before the oils are returned, and they shall be well stirred and mixed with the residue.

3. The apparatus shall consist of the following standard parts:

Flask. (*a*) *Flask.*—The distillation flask shall be a 250 cubic centimeter Engler distilling flask, having the following dimensions:

Diameter of bulb: 8 centimeters.
Length of neck: 15 centimeters.
Diameter of neck: 1.7 centimeters.
Surface of material to lower side of tubulature: 11 centimeters.
Length of tubulature: 15 centimeters.
Diameter of tubulature: 0.9 centimeter.
Angle of tubulature: 75°.

A variation of 3 per cent from the above measurements will be allowed.

Thermometer. (*b*) *Thermometer*—The thermometer shall conform to the following requirements:

It shall be made of thermometric glass of a quality equivalent to suitable grades of Jena or Corning make. It shall be thoroughly annealed. It shall be filled above the mercury with inert gas which will not act chemically on or contaminate the mercury.

The pressure of the gas shall be sufficient to prevent separation of the mercury column at all temperatures of the scale. There shall be a reservoir above the final graduation large enough so that the pressure will not become excessive at the highest temperature. The thermometer shall be finished at the top with a small glass ring or button suitable for attaching a tag. Each thermometer shall have for identification the maker's name, a serial number, and the letters "A. S. T. M. Distillation."

The thermometer shall be graduated from 0° to 400° C. at intervals of 1° C. Every fifth graduation shall be longer than the intermediate ones, and every tenth graduation beginning at zero shall be numbered. The graduation marks and numbers shall be clear-cut and distinct.

The thermometer shall conform to the following dimensions:

Total length, maximum: 385 millimeters.
Diameter of stem: 7 millimeters; permissible variation, 0.5 millimeters.
Diameter of bulb, minimum, 5 millimeters, and shall not exceed diameter of stem.
Length of bulb: 12.5 millimeters; permissible variation, 2.5 millimeters.
Distance from 0° to bottom of bulb: 30 millimeters; permissible variation, 5.0 millimeters.
Distance from 0° to 400°: 295 millimeters; permissible variation, 10.0 millimeters.

The accuracy of the thermometer when delivered to the purchaser shall be such that when tested at full immersion the maximum error shall not exceed the following:

From 0° to 200° C: 0.5° C.
From 200° to 300° C.: 1.0° C.
From 300° to 375° C.: 1.5° C.

The sensitiveness of the thermometer shall be such that when cooled to a temperature of 74° C. below the boiling point of water at the barometric pressure, at the time of test, and plunged into free flow of stream, the meniscus shall pass the point 10° C. below the boiling point of water in not more than six seconds.

The thermometer shall be set up as for the distillation test, using water, naphthalene, and benzophenone as distilling liquids. The correctness of the thermometer shall be checked at 0° and 100° C. after each third distillation until seasoned.

(*c*) *Condenser.*—The condenser tube shall have the following dimensions: **Condenser.**

Adapter: 70 millimeters.
Length of straight tube: 185 millimeters.
Width of tube: 12 to 15 millimeters.
Width of adapter end of tube: 20 to 25 millimeters.

(*d*) *Stand.*—One iron stand with a universal clamp for holding the condenser, and a light grip arm with a cork-lined clamp for holding the flask, shall be provided.

(*e*) *Burner and shield.*—A Bunsen burner shall be provided, with a tin shield 20 centimeters long by 9 centimeters in diameter. The shield shall have a small hole for observing the flame. **Burner.**

(*f*) *Cylinders.*—The cylinders used in collecting the distillate shall have a capacity of 25 cubic centimeters and shall be graduated in 0.1 centimeter. **Cylinders.**

4. The apparatus shall be set up as shown in Figure 6, the thermometer being placed so that the top of the bulb is opposite the middle of the tubulature. All connections should be tight.

5. One hundred cubic centimeters of the dehydrated material to be tested shall be placed in a tared flask and weighed. After adjusting the thermometer, shield, condenser, etc., the distillation is commenced, the rate being so regular that 1 cubic centimeter passes over every minute. The receiver is changed as the mercury column just passes the fractionating point.

To determine the amount of residue, the flask is weighed again when distillation is complete. During the distillation the condenser tube shall be warmed when necessary to prevent the deposition of any sublimate. The percentages of fractions shall be reported by weight.

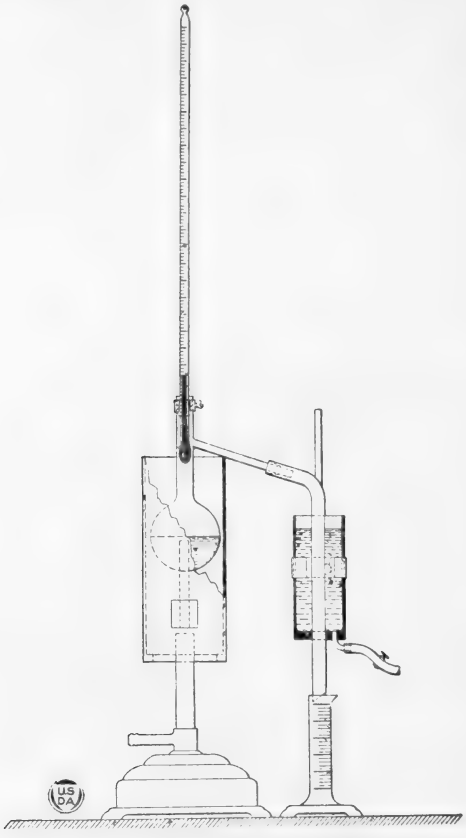


FIG. 6.—Distillation apparatus.

DETERMINATION OF PERCENTAGE OF RESIDUE OF DESIRED PENETRATION.

Fifty grams of the oil are placed in a 3-ounce deep, seamless tin box; the box is placed in a sand bath and heated over a Bunsen burner. A thermometer is suspended in the oil, the bulb not touching the bottom of the box. The temperature of the oil is kept at from 249° to 260° C. (480° to 500° F.) and the oil is stirred from time to time with the thermometer to prevent overheating in any part. Depending upon the nature of the oil, as usually indicated by its flash, consistency at 25° C. (77° F.), and specific gravity, the operator can with experience tell about what percentage it will be necessary to evaporate before cooling and taking a penetration of the residue. It is sometimes necessary to make several trials before the desired result is obtained. When the required penetration is reached, the residue left from evaporation is weighed and its per cent of the original sample taken is computed.

METHOD FOR EXAMINATION OF BITUMINOUS MIXTURES.

A. Centrifugal Method.

Centrifugal method.

The aggregate is prepared for analysis by heating it in an enamel-ware pan on the hot plate until it is sufficiently soft to be thoroughly disintegrated by means of a large spoon. Care must be taken, however, that the individual particles are not crushed. If a section of pavement is under examination, a piece weighing somewhat over 1 kilogram may be cut off with hammer and chisel. The disintegrated aggregate is then allowed to cool. Not less than 500 grams of aggregate containing particles larger than three-quarters inch in diameter or 200 grams of aggregate with all particles smaller than three-quarters inch are placed in the bowl of the centrifuge extractor, and a ring three-quarters of an inch wide, cut from felt paper, is fitted on the rim, after which the cover plate is placed in position and drawn down tightly by means of the milled nut. If the bitumen is to be recovered and examined, the felt ring should be treated previously in the empty extractor with a couple of charges of carbon disulphide in order to remove any small amount of grease or resin that may be present, although a proper grade of felt should be practically free from such products. The bowl is then placed on the motor shaft of the extractor and the slot and pin are carefully locked. An empty bottle is placed under the spout and 150 cubic centimeters of carbon disulphide (carbon tetrachloride, benzol, or chloroform may also be used as solvents) is poured into the bowl through the small holes. After allowing the material to digest for a few minutes, the motor is started, slowly at first in order to permit the aggregate to distribute uniformly. The speed should then be increased sufficiently by means of the regulator to cause the dissolved bitumen to flow from the spout in a thin stream. When the first charge has drained, the motor is stopped and a fresh portion of disulphide is added. This operation is repeated from four to six times with 150 cubic centimeters of disulphide. With a little experience the operator can soon gauge exactly what treatment is necessary for any given material. When the last addition of solvent has drained off, the bowl is removed and placed with the cover plate uppermost on a sheet of manila paper. The cover plate and felt ring are carefully laid aside on the paper and, when the aggregate is thoroughly dry, it can be brushed on a pan of the rough balance and weighed. The difference between this weight and the original weight taken shows the amount of bitumen extracted. The aggregate may then be tested as occasion requires.

When it is desired to examine the bitumen, or recover the solvent, a suitable distillation apparatus may be used, taking necessary precautions when handling inflammable solvents.

The solution of bitumen should be allowed to stand overnight in order to permit the settling of any fine mineral matter that is sometimes carried through the felt ring in the extractor. The solution is then decanted into the flask, and the solvent is driven off by means of heat from an incandescent lamp until the residue is of a thick sirupy consistency. Meanwhile the solvent is condensed and recovered in the flask. The residue is poured into an 11-centimeter porcelain evaporating dish and evaporated on a steam bath. The most scrupulous care must be taken at all times that no flames are in its immediate vicinity. Evaporation is carried on at a gentle heat, with continual stirring, until foaming practically ceases. It is advisable to have a large watch glass at hand to smother the flames quickly should the material ignite. As the foaming subsides, the heat of the steam bath may be gradually raised, and evaporation continued until the bubbles beaten or stirred to the surface of the bitumen fail to give a blue flame or odor of sulphur dioxide when ignited by

a small gas jet. The dish of bitumen should then be set in a hot-air oven maintained at 105° C. for about an hour, after which it should be allowed to cool. Its general character is noted and any tests for bitumens that are necessary are then made upon it.

The difference between the final aggregate and the orignal amount taken gives the amount of bitumen extracted, which is subject to correction, dependent on the amount of ash determined from the washings.

Ash correction shall be made in the following manner: The total solution of bitumen, well stirred, is rapidly measured and an aliquot portion taken, usually 100 cubic centimeters, and poured into a previously weighed suitable flat-bottom dish, preferably quartz. The solvent is evaporated over a very low flame and the residual coke is then ignited with a burner capable of furnishing high temperature, such as a Meker. (Caution: When an inflammable solvent is used evaporation should be conducted on a steam bath and care should be taken that no flames are in the immediate vicinity.) The dish and contents are then cooled in a desiccator and the percentage of ash calculated.

B. Hot Extraction Method.

Hot extraction method.

The New York Testing Laboratory extractor consists of a large brass cylinder, through the bottom of which projects a 16-candlepower incandescent carbon-filament bulb to supply heat to the extraction apparatus proper, which is held in the upper portion of the cylinder. This apparatus is composed of a cylindrical brass vessel for holding the solvent, a cylindrical wire basket made of 80-mesh wire cloth, suspended in the cylinder, and an inverted conical condenser which serves as a top.

The aggregate is prepared for analysis by heating it in a tin dish on the hot plate until it is sufficiently soft to be disintegrated by means of a large spoon. The disintegrated aggregate is then allowed to cool. Not less than 500 grams of aggregates containing particles larger than three-quarters inch in diameter or 200 grams of aggregates with all particles smaller than three-quarters inch are then closely packed in the wire basket or suitably sized extraction thimbles and covered with a disk or wad of absorbent cotton or felt. From 175 to 200 cubic centimeters of carbon disulphide (carbon tetrachloride, benzol, or chloroform may be used) are next placed in the inside vessel, in which the wire basket should be suspended. The top is then placed in position and cooling water circulated through it. Heat is applied by means of the electric-light bulb. The solvent is boiled in the lower part of the extractor and condenses on the undersurface of the top, from which it drips upon the wad of absorbent cotton and then percolates through the sample. A complete extraction may be made in three hours. At the end of this time the apparatus is allowed to cool and the basket containing the extracted aggregate carefully removed. After thoroughly drying, the aggregate is placed upon a pan of the rough balance and weighed. The difference between this weight and the original weight taken shows the amount of bitumen extracted, which is calculated upon a percentage basis of the original. This figure should be corrected for fine mineral matter which passes through the meshes of the wire basket, as follows: The solution of extracted bitumen is thoroughly agitated and measured in a glass graduate. Five or ten cubic centimeters are then poured into a weighed platinum crucible or dish, burned, and ignited to ash. The amount of mineral matter in the entire solution may then be calculated from the amount of ash produced from that portion ignited. The total percentage of such ash is then deducted from the percentage of bitumen already calculated in order to obtain the true percentage of bitumen. The amount of this correction will ordinarily vary from 0.1 per cent in uniformly coarse aggregates to 1 or 2 per cent in the analysis of aggregates containing a considerable amount of very fine mineral matter.

SUGGESTED METHOD FOR EXAMINATION OF BITUMINOUS MORTARS.

Bituminous mortars.

Bituminous mortars may be extracted by the use of bronze tubes which are capable of being whirled in the type of centrifuge similar to the Babcock milk tests. This method is based on decantation of the supernatant solvent. The difference between the amount of final aggregate and the original amount taken gives the amount of bitumen extracted, which is subject to correction by deducting the amount of ash determined from the washings. The ash correction to be made as given under A.

MECHANICAL ANALYSIS OF EXTRACTED AGGREGATES.

(A. S. T. M. standard method, serial designation D 19–16, slightly modified.)

The mineral aggregate left after extraction of the bituminous material shall be dried at not over 110° C. (230° F.) to a constant weight. It shall be separated by the use of a screen having openings one-quarter inch in diameter. The portion retained on the screen shall be examined in accordance with the method for mechanical analysis of broken stone, etc. The portion passing this screen shall be examined in accordance with the method for the mechanical analysis of sand or other fine aggregate, except that the percentage of 200-mesh material shall be determined as follows: A 10-mesh sieve shall be superimposed on a 200-mesh sieve and the aggregate placed on the 10-mesh sieve. The material is then immersed and gently oscillated in a bath of gasoline until all fine material has been washed off. The washed material is then dried and weighed. The weight of material passing the 200-mesh sieve may be obtained as follows:

Weight of total mineral aggregate (including weight of mineral material recovered from extract) minus weight of material retained on sieve gives weight of mineral matter passing 200-mesh sieve.

The mechanical analysis shall be recorded in the following manner:

	Per cent.
Passing 200-mesh sieve	_____
Passing 80-mesh sieve and retained on a 200-mesh sieve	_____
Passing 40-mesh sieve and retained on a 80-mesh sieve	_____
Passing 10-mesh sieve and retained on a 40-mesh sieve	_____
Passing one-quarter-inch screen and retained on a 10-mesh sieve	_____
Passing one-half-inch screen and retained on a one-quarter-inch screen	_____
Passing three-quarter-inch screen and retained on a one-half-inch screen	_____
	100.00

In the sieve analysis of the sand fraction, the following sizes of sieves shall be used: 10-mesh, 40-mesh, 80-mesh, and 200-mesh.

MECHANICAL ANALYSIS OF BROKEN STONE OR BROKEN SLAG, EXCEPT AGGREGATES USED IN CEMENT CONCRETE.

(A. S. T. M. standard method, serial designation D 18–16, slightly modified.)

The method shall consist of (1) drying at not over 110° C. (230° F.) to constant weight a sample weighing in pounds six times the diameter in inches of the largest holes required; (2)

passing the sample through such of the following sizes of screens having circular openings as are required or called for by the specifications, screens to be used in order named: 3½ inch (8.89 centimeters), 3 inch (7.62 centimeters), 2½ inch (6.35 centimeters), 2 inch (5.08 centimenters), 1½ inch (3.81 centimeters), 1¼ inch (3.18 centimeters), 1 inch (2.54 centimeters), three-quarter inch (1.09 centimeters), one-half inch (1.27 centimeters), one-quarter inch (0.64 centimeters); (3) determining the percentage by weight retained on each screen; and (4) recording the mechanical anaylsis in the following manner:

	Per cent.
Passing one-quarter-inch screen	
Passing one-half-inch screen and retained on a one-quarter-inch screen	
Passing three-quarter-inch screen and retained on a one-half-inch screen	
Passing 1-inch screen and retained on a three-quarter-inch screen	
Passing 1½-inch screen and retained on a 1-inch screen	
	100.00

MECHANICAL ANALYSIS OF SAND OR OTHER FINE HIGHWAY MATERIAL, EXCEPT FINE AGGREGATES USED IN CEMENT CONCRETE.

(A. S. T. M. standard method, serial designation D 7–18, slightly modified.)

The method shall consist of (1) drying at not over 110° C. (230 F.) to constant weight a sample weighing 50 grams; (2) passing the sample through each of the mesh sieves (American Society for Testing Materials standard sieves) specified in Table 3; [15](3) determining the percentage by weight retained on each sieve, the sifting being continued on each sieve until less than 1 per cent of the weight retained on each sieve shall pass through the sieve during the last minute of sifting; and (4) recording the mechanical analysis in the following manner:

	Per cent.
Passing 200-mesh sieve	
Passing 80-mesh sieve and retained on 200-mesh sieve	
Passing 40-mesh sieve and retained on 80-mesh sieve	
Passing 10-mesh sieve and retained on 40-mesh sieve	
	100.00

[15] The order in which the sieves are to be used in the process of sifting is immaterial and shall be left optional; but in reporting results the order in which the sieves have been used shall be stated.

TABLE 3.—*A. S. T. M. standard sieves.*

Mesh designation.	Unit of measure.	Actual mesh.	Opening.	Wire diameter.	Permissible variations.	
					Mesh.	Diameter.
10	Centimeter	3.9	0.200	0.056	±0.04	±0.005
	Inch	9.9	.079	.022	±0.1	±.002
20	Centimeter	8	.085	.040	±0.2	±.0015
	Inch	20.3	.0335	.0157	±0.5	±.0006
30	Centimeter	12	.050	.033	±0.4	±.0012
	Inch	30.5	.0197	.0130	±1.0	±.0005
40	Centimeter	16	.036	.026	±0.6	±.0010
	Inch	40.6	.0142	.0102	±1.5	±.0004
50	Centimeter	20	.029	.021	±0.8	±.0010
	Inch	50.8	.0114	.0083	±2	±.0004
80	Centimeter	31	.017	.015	±1	±.0008
	Inch	78.7	.0067	.0059	±3	±.0003
100	Centimeter	39	.014	.0116	±1	±.0008
	Inch	99.1	.0055	.0046	±3	±.0003
200	Centimeter	79	.0074	.0053	±3	±.0005
	Inch	200.7	.0029	.0021	±8	±.0002

NOTE—It is recommended that the 10, 40, 80, and 200 mesh sieve be used.

METHOD FOR DETERMINATION OF FINENESS OF MINERAL FILLER.

1. The fineness test of mineral filler shall be made on a 200-mesh sieve which meets the requirements of the 200-mesh sieve specified for Portland cement.

2. The test shall be made with 50 grams of filler. The sieve shall be thoroughly clean and dry. The filler shall be placed on the No. 200 sieve, with a pan and cover attached, if desired, and shall be held in one hand in a slightly inclined position so that the sample will be well distributed over the sieve, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the upstroke. The sieve shall be turned every 25 strokes about one-sixth of a revolution in the same direction. The operation shall continue until not more than 0.05 gram passes through in one minute of continuous sieving. The fineness shall be determined from the weight of the residue on the sieve expressed as a percentage of the weight of the original sample.

3. Mechanical sieving devices may be used, but the filler shall not be rejected if it meets the fineness requirement when tested by the hand method described in section 2.

TEST FOR WATER IN EMULSIONS.

The percentage of water may be obtained by the method for determination of water in bituminous material, without the addition of naphtha or benzol, or by the calcium chloride method.

CALCIUM CHLORIDE METHOD FOR DETERMINATION OF PER CENT WATER IN BITUMINOUS EMULSIONS.

Approximately 10 grams of the emulsion is accurately weighed in an Erlenmeyer flask and exactly 25 cubic centimeters of a 10 per cent solution of calcum chloride added and thoroughly agitated by shaking. The liquid content of the flask is then poured into a graduated cylinder. Carbon disulphide is then added to the Erlenmeyer flask and the separated asphalt dissolved and washed with carbon disulphide into the graduated cylinder above referred to containing the decanted liquid. The total contents of

the cylinder are then thoroughly shaken and after standing the quantity of separated supernatant water read.

$$\text{Calculations:} \quad \text{Per cent water} = \frac{A - B}{C} \times 200$$

A = volume of supernatant water.
B = volume of 10 per cent calcium chloride solution originally used.
C = weight of emulsion taken for water determination.

TEST FOR WATER IN BITUMINOUS MATERIAL.

(A. S. T. M. tentative method, serial designation D 114–21 T.)

1. The apparatus shall consist of a copper still, 6 inches by 3½ inches inside diameter, with an adjustable ring burner to fit the still. The still shall be provided with a connecting tube, a condenser trough, a condenser tube, and a separatory funnel. A thermometer, 0° to 250° C., shall be provided. The apparatus shall be set up as shown in Figure 7.

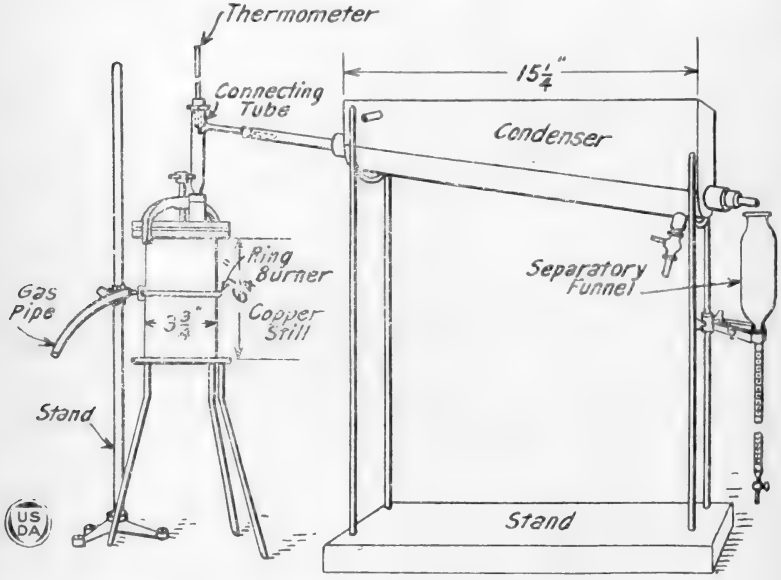


FIG. 7.—Apparatus for dehydration test.

2. Fifty cubic centimeters of coal-tar naphtha or light oil shall be *measured into* a 250 cubic centimeter graduated cylinder, and 200 cubic centimeters of the material to be tested shall be added. In the case of petroleum products, petroleum naphtha may be substituted for the coal-tar naphtha. The contents shall be transferred to the copper still and the cylinder shall be washed with 100 to 150 cubic centimeters more of naphtha, and the washings added to the contents of the still. The lid and clamp shall be attached, using a paper gasket. The condenser trough shall be filled with water. Heat shall be applied by means of the ring burner, and the distillation continued until the vapor temperature has reached 205° C. (401° F.). The distillate shall be collected in the separatory funnel, in which 15 to 20 cubic centimeters of benzol or naphtha have been previously placed in order to effect a clean separation of the water and oil. The reading shall be

made after twirling the funnel and allowing the contents to settle for a few minutes. The percentage shall be computed by volume. The naphtha or light oil used shall be tested to determine freedom from water.

3. The accuracy obtainable by this method is within 0.1 per cent.

4. Every precaution must be taken to insure a representative sample. Bituminous materials containing separated water are especially difficult to sample.

METHODS OF SAMPLING AND ANALYZING CREOSOTE OIL.

(A. S. T. M. standard methods, serial designation D 38–18 slightly modified.)

SAMPLING.

Sampling.

1. Wherever the oil is being loaded or discharged by means of a pump the following method shall be used:

A one-half inch sampling pipe shall be inserted in the line through which the oil is being pumped, on the discharge side of

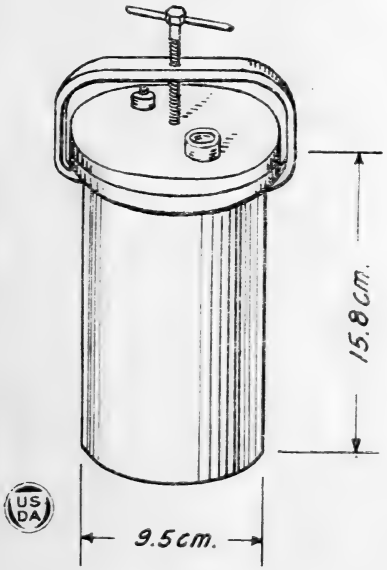


FIG. 8.—Copper still.

the pump, preferably in a rising section of the pipe line. This sampling pipe shall extend one-half way to the center of the main pipe and with the inner open end of the sampling pipe turned at an angle of 90° and facing the flow of the liquid. This pipe shall be provided with a plug cock and shall discharge into a receiver of 50 to 100 gallons capacity. The plug cock shall be so adjusted that, with a steady continuous flow of the oil, the receiver shall be filled in the time required to pump the entire shipment. The receiver shall be provided with a steam coil sufficient to keep the contents at a temperature not exceeding 120° F. Immediately upon completion of the pumping, the contents of the receiver shall be very thoroughly agitated and a duplicate 1-quart sample taken immediately for the test. The amount of the drip sample collected shall be not less than 1 gallon for each 1,000 gallons of oil handled, except in the case of large boat shipments, where a maximum of 100 gallons is sufficient.

ANALYSIS.

Water Test.

Still. 2. (*a*) *Still.*—A vertical, cylindrical copper still, with removable flanged top and yoke, of the form and approximate dimensions shown in Figure 8 shall be used.

Thermometer. (*b*) *Thermometer.*—The standard distillation thermometer, as specified under "Distillation," section 9 (*e*), shall be used.

Condenser. (*c*) *Condenser.*—A copper trough condenser shall be used, with straight-walled glass tube, having approximately the form and dimensions shown in Figure 9.

Separatory funnel. (*d*) *Separatory funnel.*—A separatory funnel of the form shown in Figure 9 shall be used, having a total capacity of 120 cubic centimeters, and the outlet graduated in fifths of a cubic centimeter.

3. The apparatus shall be set up as shown in Figure 9.

4. When any measurable amount of water is present in the distillate below 210° C. on testing in accordance with part D on

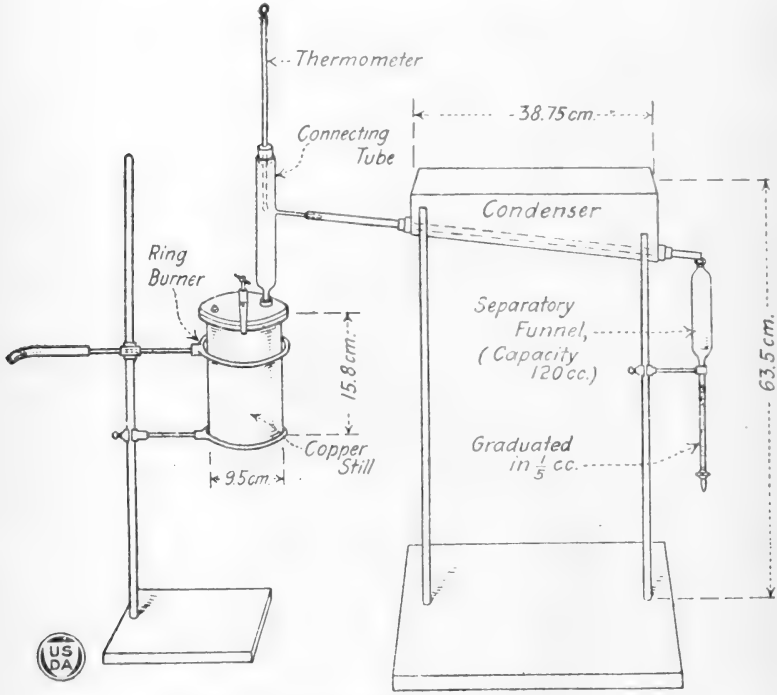


FIG. 9.—Assembled apparatus for water test.

"Distillation," the oil and water in this fraction shall be separated, if possible, and measured separately. If more than 2 per cent of water is present, or if the water is apparently present to an extent in excess of 2 per cent, but an accurate separation is impossible, the percentage of water present shall be determined by the following method, and the water-free oil so obtained shall be used in the distillation test:

Method of test. Measure 200 cubic centimeters of oil in a graduated cylinder and pour into copper still, allowing the cylinder to drain into the still for several minutes. Attach lid and clamp, using a paper gasket slightly wet with oil around the flange of the still. Apply heat by means of the ring burner, which shall be placed just above

the level of the oil in the still at the beginning of the test and gradually lowered when most of the water has distilled over. Continue the distillation until the vapor temperature, indicated by the thermometer with the bulb opposite the offtake of the connecting tube, reaches 205° C. Collect distillate in separatory funnel. When the distillation is completed and a clear separation of water and oil in the funnel has taken place, read the water by volume and draw off; and return any light oil distilled over with the water to the oil in the still. The dehydrated oil from the still shall be used for the distillation test.

Matter Insoluble in Benzol.

Extractor.

5. (*a*) *Extractor.*—The extractor may be of any form in which the oil is subjected to direct washing by the boiling vapors of the solvent.

Filtering medium.

(*b*) *Filtering medium.*—The filtering medium may be either two thicknesses of S. & S. No. 575 hardened filter paper, 15 centimeters in diameter, arranged in cup shape by folding symmetrically, or alundum thimbles, flat bottom, 30×80 RA 98. If filter papers are used, prior to using they shall be soaked in benzol to remove any grease, dried in a steam oven and kept in a desiccator until ready to be used. The filter-paper cup may be suspended in the extractor flask by a wire basket hung from two small hooks on the undersurface of the metal cover of the flask. If the alundum thimble is used, it may be supported by making two perforations in the top of the thimble and suspending from the cover by German silver or platinum wires.

Method of test.

6. Weigh 10 grams of dry oil in a 100 cubic centimeter beaker. Add about 50 cubic centimeters of pure benzol and transfer at once to the filter cup. The filter cup or thimble is previously weighed, and the paper cup shall always be kept in a weighing bottle until ready for use. Wash out the beaker with benzol, passing all washings through the filter cup, and place latter at once in the extraction apparatus. Extractor shall contain a suitable quantity of pure benzol. Sufficient heat to boil the solvent shall be provided by means of an electric heater or a steam bath. Continue the extraction until the descendng solvent is practically colorless, and remove the filter cup and dry in steam oven until all solvent is driven off; cool in desiccator and weigh. The balance used for this purpose should be accurate to 0.5 milligram.

Specific Gravity.

Specific gravity.

7. (*a*) *Hydrometer.*—The hydrometer shall have the following dimensions:

Length of stem: 125 millimeters; permissible variation, 12.5 millimeters.

Length of bulb: 105 millimeters; permissible variation, 10.5 millimeters.

Length of scale: 80 millimeters; permissible variation, 8.0 millimeters.

Diameter of stem: 6 millimeters; permissible variation, 0.5 millimeters.

Diameter of bulb: 22 millimeters; permissible variation, 2.0 millimeters.

A set of two hydrometers with ranges 1.00 to 1.08 and 1.07 to 1.15 will suffice.

(*b*) *Cylinder.*—The cylinder shall have the following dimensions:

Length: 300 millimeters; permissible variation, 30 millimeters.

Diameter: 32 millimeters; permissible variation, 3 millimeters.

8. The oil shall be brought to a temperature of 38° C. (100° F.), and the determination shall be made at that temperature unless the oil is not entirely liquid at 38° C. In case the oil

requires to be brought to a higher temperature than 38° C. in order to render it completely fluid, it shall be tested at the lowest temperature at which it is completely fluid and a correction made by adding 0.0008 to the observed specific gravity for each degree centigrade above 38° C. at which the test is made. This correction factor does not apply with equal accuracy to all oils, but serious error due to its use will be avoided if the foregoing precaution is observed with respect to avoiding unnecessarily high temperature. Before taking the specific gravity the oil in the cylinder should be stirred thoroughly with a glass rod, and this rod when withdrawn from the liquid should show no solid particles at the instant of withdrawal. Care should be taken that the hydrometer does not touch the sides or bottom of the cylinder when the reading is taken and that the oil surface is free from froth and bubbles.

Distillation.

Distillation retort.

9. (*a*) *Retort.*—This shall be a tubulated glass retort of the form and approximate dimensions shown in Figure 10 with a capacity of 250 to 290 cubic centimeters. The capacity shall be measured by placing the retort with the bottom of the bulb and the end of the offtake in the same horizontal plane, and pouring water into the bulb through the tubulature until it overflows the offtake. The amount remaining in the bulb shall be considered its capacity.

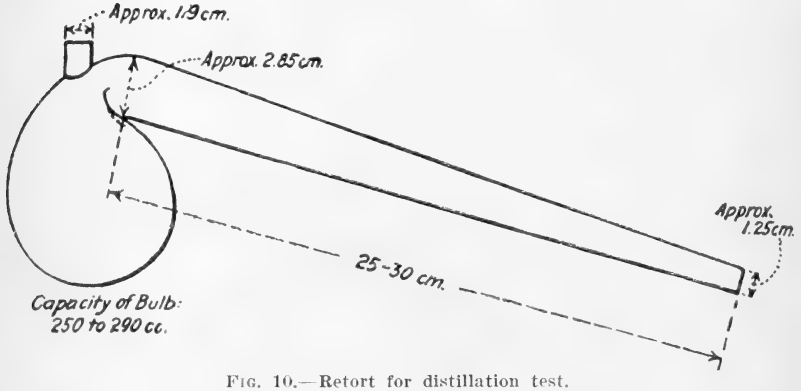


Fig. 10.—Retort for distillation test.

Condenser tube.

(*b*) *Condenser tube.*—The condenser tube shall be a suitable form of tapered glass tubing of the following dimensions:

Diameter of small end: 12.5 millimeters; permissible variation, 1.5 millimeters.

Diameter of large end: 28.5 millimeters; permissible variation 3.0 millimeters.

Length: 360.0 millimeters; permissible variation, 4.0 millimeters.

Shield.

(*c*) *Shield.*—An asbestos shield of the form and approximate dimensions shown in Figure 11 shall be used to protect the retort from air currents and to prevent radiation. This may be covered with galvanized iron, as such an arrangement is more convenient and more durable.

Receivers.

(*d*) *Receivers.*—Erlenmeyer flasks of 50 to 100 cubic centimeter capacity are the most convenient form.

Thermometer.

(*e*) *Thermometer.*—The thermometer shall conform to the following requirements:

The thermometer shall be made of thermometric glass of a quality equivalent to suitable grades of Jena or Corning make. It shall be thoroughly annealed. It shall be filled above the mercury with inert gas which will not act chemically on or

contaminate the mercury. The pressure of the gas shall be sufficient to prevent separation of the mercury column at all temperatures of the scale. There shall be a reservoir above the final graduation large enough so that the pressure will not become excessive at the highest temperature. The thermometer shall be finished at the top with a small glass ring or button suitable for attaching a tag. Each thermometer shall have for identification the maker's name, a serial number, and the letters "A. S. T. M. Distillation."

The thermometer shall be graduated from 0° to 400° C. at intervals of 1° C. Every fifth graduation shall be longer than the intermediate ones, and every tenth graduation beginning at

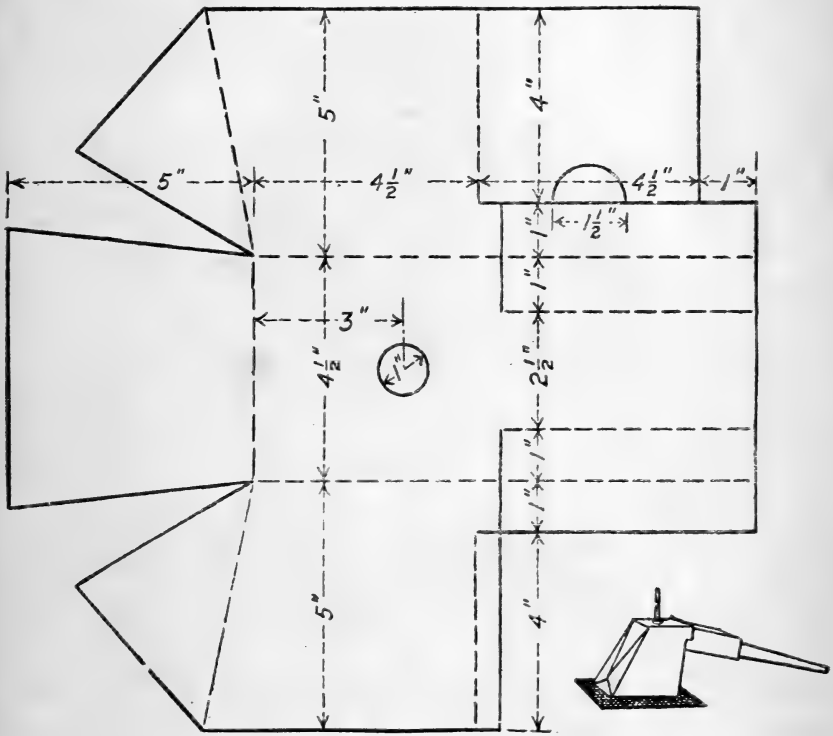


FIG. 11.—Asbestos shield.

zero shall be numbered. The graduation marks and numbers shall be clear-cut and distinct.

The thermometer shall conform to the following dimensions:

Total length, maximum: 385 millimeters.

Diameter of stem: 7 millimeters; permissible variation, 0.5 millimeter.

Diameter of bulb, minimum: 5 millimeters; and shall not exceed diameter of stem.

Length of bulb: 12.5 millimeters; permissible variation 2.5 millimeters.

Distance, 0° to bottom of bulb: 30 millimeters; permissible variation, 5 millimeters.

Distance, 0° to 400°: 295 millimeters; permissible variation, 10 millimeters.

The accuracy of the thermometer when delivered to the purchaser shall be such that when tested at full immersion the maximum error shall not exceed the following:

From 0° to 200° C.: 0.5° C.
From 200° to 300° C.: 1.0° C.
300° to 375° C.: 1.5° C.

The sensitiveness of the thermometer shall be such that when cooled to a temperature of 74° C. below the boiling point of water at the barometric pressure at the time of test and plunged into free flow of steam the meniscus shall pass the point 10° C. below the boiling point of water in not more than six seconds.

10. The retort shall be supported on a tripod or rings over two sheets of 20-mesh gauze, 6 inches square, as shown in Figure 12. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the tubulature with the bottom of the bulb one-half inch from the surface of the oil in the retort. The exact location of the thermometer bulb shall be determined by placing a vertical rule graduated in divisions not exceeding one-sixteenth inch back of the retort when the latter is in position for the test, and sighting the level of the liquid and the point for the bottom of the thermometer bulb. The distance from the bulb of the thermometer to the outlet end of the condenser tube shall be not more than 24 nor less than 20 inches. The burner should be protected from drafts by a suitable shield or chimney (see fig. 12).

Method of test. 11. Exactly 100 grams of oil shall be weighed into the retort, the apparatus assembled, and heat applied. The distillation shall be conducted at the rate of at least 1 drop and not more than 2 drops per second, and the distillation collected in weighed receivers. The condenser tube shall be warmed whenever necessary to prevent accumulation of solid distillates. Fractions shall be collected at the following points: 210°, 235°, 270°, 315°, and 355° C. The receivers shall be changed as the mercury passes the dividing temperature for each fraction. When the temperature reaches 355°, the flame shall be removed from the retort, and any oil which has condensed in the offtake shall be drained in the 355° fraction.

The residue shall remain in the retort with the cork and the thermometer in position until no vapors are visible; it shall then be weighed. If the residue is to be further tested, it shall then be poured directly into the brass collar used in the float test[16] or into a tin box and covered and allowed to cool to air temperature. If the residue becomes so cool that it can not be poured readily from the retort, it shall be reheated by holding the bulb of the retort in hot water or steam, and not by the application of flame.

For weighing the receivers and fractions, a balance accurate to at least 0.05 gram shall be used.

During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer.

When any measurable amount of water is present in the distillate it shall be separated as nearly as possible and reported separately, all results being calculated on a basis of dry oil. When more than 2 per cent of water is present, water-free oil shall be obtained by separately distilling a larger quantity of oil, returning to the oil any oil carried over with the water, and using dried oil for the final distillation. (See "Water test.")

Specific Gravity of Fractions.

12. As specific gravity is an absolute physical determination, any recognized method which can be applied to the quantity and quality of material at hand to be tested must be considered satisfactory. The following methods are convenient and accurate means for the relatively small amounts of oil available in determining specific gravity of fractions to be tested.

[16] See "Float test of residue," p. 78.

(A) LIQUID FRACTIONS.

13. The Westphal balance may be used.

Specific gravity liquid fractions.

14. If the fraction to be tested is liquid at a temperature not exceeding 60° C., the Westphal balance can be used with convenience and rapidity. A special type of Westphal balance is obtainable, designed for testing very small quantities. However, the ordinary type of Westphal balance can be adapted to testing small fractions by the use of a special plummet. When using the ordinary balance with the special plummet, extra care is needed that the adjustment of the balance be accurately made. The plummet can readily be made in the laboratory from a piece of ordinary glass tubing 7 millimeters outside diameter, sealed at the end, and melting into the glass where sealed a short platinum wire. After cooling, place 9 to 10 grams of mercury in the tube, making a column 35 to 40 millimeters high. Seal off the tube within 20 millimeters of the top of the mercury column with a blowpipe flame. The plummet shall have a length of about 55 to 60 millimeters over all, and shall weigh between 10 and 12 grams.

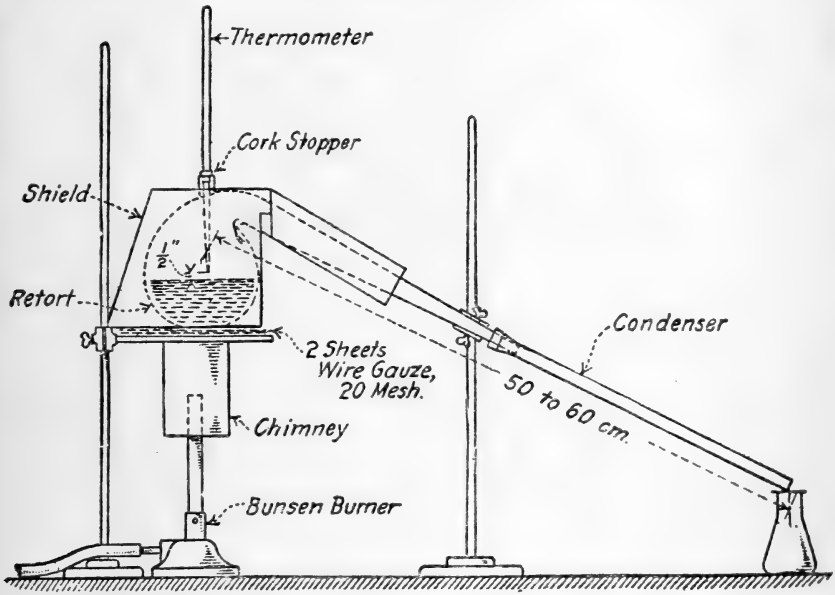


FIG. 12.—Assembled apparatus for distillation test.

(B) SOLID AND SEMISOLID FRACTIONS.

Specific gravity solid and semisolid fractions.

15. A pan having the following approximate dimensions may be used.

Diameter of base: 20 millimeters.
Diameter of top: 25 millimeters.
Depth: 12 millimeters.
Diameter of wire: 1 millimeter.
Total weight: 7 grams.

The pan and wires are made of platinum or nickel.

16. Solid or semisolid fractions of oil which can not be readily liquefied can be rapidly and accurately tested in this apparatus by the usual method of weighing in air and in water. The usual precautions of igniting the pan before use, and avoiding the inclosure of air or water in the sample, should be observed.

NOTE.—The method for liquid fractions is usually applicable to the fractions 235° to 315° C., and the method for solid and semisolid fractions to the fraction 315° to 355° C.

FLOAT TEST OF RESIDUE.

17. The float test of the residue shall be made in accordance with the "Float test for bituminous road materials."

METHODS OF SAMPLING AND TESTING DRAIN TILE AND CULVERT PIPE.

METHODS FOR TEST OF DRAIN TILE, CLAY CULVERT PIPE, AND CEMENT-CONCRETE CULVERT PIPE, PLAIN OR REINFORCED.

(Methods from A. S. T. M. standard specifications, serial designations C 14–20 and C 4–21, slightly modified.)

1. The physical tests of tile and pipes shall include: Crushing test, absorption test, and freezing and thawing tests, at option of the purchaser.

2. The specimens to be tested shall be selected by the purchaser or his representative at the point or points designated by him when placing the order. The manufacturer or seller shall furnish specimens for testing, without separate charge.

3. Failure of the specimens to meet the requirements of any of the tests imposed shall result in rejection of all the pipe in the shipment or delivery corresponding to the sizes thus failing to comply, except that in the event the specimens in any size fail to meet the requirements the manufacturer or seller may, with the consent of the consumer or purchaser, furnish for testing additional specimens from the same shipment without charge.

4. The specimens of pipes shall be sound, full-sized pipe. They shall first be freed from all visible moisture. When dry each specimen shall be weighed, measured, and inspected. The results of these observations shall be recorded and preserved.

5. Specimens which, when placed in a vertical position, do not give a metallic ring when struck with a hammer, or are observed to have cracks or other defects in form or dimensions in excess of the limits permitted in the specifications, shall be discarded and replaced with additional specimens from the shipment.

(A) CRUSHING TEST.

Crushing test.

6. (*a*) Any prime mover or hand power which will apply the load at a uniform rate of about 2,000 pounds per minute, or in increments of not more than 100 pounds at the same rate, may be used in making the test.

(*b*) The pipe shall not be allowed to stand under load longer than is required to apply the load and to observe and record it.

(*c*) The testing machine shall be substantial and rigid throughout, so that the distribution of the load will not be affected appreciably by the deformation or yielding of any part.

(*d*) The bearings and the specimen shall be accurately centered so as to secure a symmetrical distribution of the loading on each side of the center of the pipe in every direction.

(*e*) The load shall be applied until the pipe yields by cracks passing through the shell.

7. Except as otherwise hereinafter specified, the pipe to be tested shall be supported by a metallic knife bearing 1 inch wide and extending from a point just back of the socket to the spigot end of the pipe. Before the pipe is placed, a fillet of plaster of Paris and sand 1 inch wide, and thick enough to compensate for all the inequalities of the pipe barrel, shall be cast on the surface of the knife bearing. The pipe shall be placed upon the fillet while the plaster of Paris is still somewhat plastic. The load shall be applied through an upper knife bearing of the same size and length as the lower bearing. A plaster of Paris fillet 1 inch wide shall be cast along the length of the crown of

the pipe to equalize the lower bearing before the upper one is brought into contact.

Both of the bearings shall be sufficiently rigid to transmit and receive uniform loads throughout their lengths without deflection, and shall be so attached to the machine as to transmit and receive the maximum stresses produced by the tests without lost motion, vibration or sudden shock.

At the option of the consumer or purchaser the crushing test may be applied with sand bearings or with two or three edge bearings.

The crushing strength shall be calculated by dividing the total load required to break each pipe by the net inside length of the barrel of the pipe, measuring from the bottom of the socket to the end of the spigot, and by then multiplying the quotient by the following factors:

For knife or two-edge bearings	$\frac{1.0}{7}$
For three-edge bearings	$\frac{1.0}{7}$
For sand bearings	1.00

8. When three-edge bearings are used, the ends of each specimen of pipe shall be accurately marked in halves of the circumference prior to the test.

The two lower bearings shall consist of two wooden strips with vertical sides, each strip having its interior top corner rounded to a radius of approximately one-half inch. They shall be straight and shall be securely fastened to a rigid block with their interior vertical sides 1 inch apart.

The upper bearing shall be a wooden block, straight and true from end to end.

The test load shall be applied through the upper bearing block in such a way as to leave the bearing free to move in a vertical plane passing midway between the lower bearings.

In testing a pipe which is "out of sight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair bearings.

9. When sand bearings are used, the ends of each specimen of pipe shall be accurately marked prior to the test in quarters of the circumference. Specimens shall be carefully bedded, above and below, in sand, for one-fourth the circumference of the pipe measured on the middle line of the barrel. The depth of bedding above and below the pipe at the thinnest points shall be one-half the radius of the middle line of the barrel.

The sand used shall be clean, and shall be such as will pass a No. 4 screen.

The top bearing frame shall not be allowed to come into contact with the pipe or with the top bearing plate. The upper surface of the sand in the top bearing shall be struck level with a straightedge, and shall be covered with a rigid top bearing plate, with lower surface a true plane, made of heavy timbers or other rigid material, capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate in such a manner as to permit free motion of the plate in all directions. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used. The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate, provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the pipe and that the platform shall not be allowed to touch the top bearing frame.

The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of sand. The interior surfaces of the frames shall be

dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may, if desired, be attached to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

10. Pieces of the crushed pipe may be used as specimens in making the absorption test and freezing and thawing test.

(B) Absorption Test.

Absorption test.

11. The specimens shall be sound pieces with all edges broken, and may be from pipes broken in the crushing or other tests. They shall be from 12 to 20 square inches in area and shall be as nearly square as they can be readily prepared. They shall be free from observable cracks, fissures, laminations, or shattered edges.

12. Preparatory to the absorption test the specimen shall first be weighed and then dried in a drier or oven at a temperature of not less than 110° C. (230° F.) for not less than three hours. After removal from the drier the specimen shall be allowed to cool in dry air to a temperature of 20° to 25° C. (68° to 77° F.) and then reweighed.

If the specimen is comparatively dry when taken and the second weight closely agrees with the first, it shall be considered dry. If the specimen is wet when taken, it shall be placed in the drier for a drying treatment of two hours and reweighed. If the first weight checks the second, the specimen shall be considered dry. In case of doubt the specimen shall be redried for two-hour periods until check weights are obtained.

13. The balance used shall be sensitive to 0.5 gram when loaded with 1 kilogram, and weighings shall be read to the nearest gram. When other than metric weights are used, the same degree of accuracy shall be obtained.

14. The specimen after final drying, cooling, and weighing shall be placed with other similar specimens in a suitable wire receptacle, packed tightly enough to prevent jostling, covered with distilled water or rain water, raised to the boiling point and boiled for five hours, and then cooled in water to a final temperature of 10° to 15° C. (50 to 59° F.).

15. The specimen shall be allowed to drain for one minute and, the superficial moisture having been removed by towel or blotting paper, placed upon the balance.

16. The test result shall be calculated as percentage of the initial dry weight.

17. Three specimens shall be tested of each pipe broken in the crushing test.

18. The results shall be reported separately for each individual specimen, together with the mean for all the specimens from the same shipment of pipe.

19. Each specimen shall be marked so that it may be identified with the pipe used in the crushing test from which the specimen was taken. The marking shall be applied so that the pigment used shall not cover more than 1 per cent of the total superficial area of the specimen.

(C) Freezing and Thawing Tests.

Freezing and thawing tests.

20. The test specimens employed in making the freezing and thawing test shall be another set of three selected as specified in section 11.

21. The specimens shall be dried as specified in section 12.

22. The same scales and weights as are specified in section 13 for the absorption test or others of equivalent sensitiveness and accuracy shall be employed for the weighings required in the freezing and thawing test. The same procedure in weighing and reweighing as specified in section 13 shall be used.

23. The specimens shall be immersed for 72 hours in water having a temperature of 18° to 24° C. (65° to 75° F.) and then weighed as specified in section 12.

24. When the specimens (either from the absorption test or from a specially prepared series) have been weighed after saturation with water, they shall be returned to the water and kept immersed till the freezing is begun. For freezing they shall be placed with their concave faces upward in water-tight metal trays, suitably mounted in a rigid metal crate, and immersed in ice water until the specimens have attained substantially the temperature of the water, after which the water shall be drawn down to a depth of one-half inch in each tray. The crate shall then be lifted as a whole, without disturbing the specimens, and placed in the freezing apparatus.

Freezing shall be performed in a quiet atmosphere, free from perceptible natural or artificial currents. If artificial freezing apparatus is employed, the apparatus shall have sufficient heat-absorbent capacity to enable the temperature of the freezing chamber to be brought to —10° C. (+14° F.) or below within 30 minutes after the introduction of the specimens. The temperature in the freezing apparatus shall not fall lower than —20° C. (—4° F.). The freezing shall be continued until the water in the trays is frozen solid. Exposure to freezing conditions in excess of this requirement shall be considered as without significance.

At the conclusion of freezing under the specified conditions, the crate of specimens shall be withdrawn and at once immersed in water at temperature of 18° to 24° C. (65° to 75° F.) in a special receptacle of proper size. Heating shall be continued so that the water will regain the required temperature as soon as practicable after the specimens are immersed. A temperature of 18° to 24° C. (65° to 75° F.) shall then be maintained for not less than two hours. At the conclusion of the thawing treatment, the crate of specimens shall be inspected and the condition of each sample after each thawing shall be noted in the records.

25. Failure under the freezing and thawing treatment shall be considered to be reached when:

(*a*) The specimens show superficial disintegration or spalling with loss of weight of more than 5 per cent of the initial dry weight; or

(*b*) The specimens are badly cracked in other than lamination planes; or

(*c*) The specimens show evident serious loss of structural strength.

NOTE.—Routine tests for control of quality and for basis of acceptance of pipe shall preferably be made at the manufacturer's plant. In addition to these tests the plants should be inspected at frequent intervals by a competent engineer who should note all the details of manufacture and take samples for test of all materials being used.

TEST FOR THE AMOUNT OF SPELTER COATING ON CULVERT METAL.

1. The amount of spelter coating shall be determined by one of the following methods:

2. (*a*) *Antimony hydrochloric acid method.*—Use three 2¼ by 2¼-inch pieces, weighed together to three decimal places. They are then immersed separately for one-half minute in hydrochloric acid of specific gravity 1.20 to which has been added 5 cubic centimeters of antimony chloride solution prepared by dissolving 20 grams of antimony trioxide in 1,000 cubic centimeters of hydrochloric acid of specific gravity 1.20. The pieces are scrubbed with a brush under running water, dried, and again weighed. About 100 cubic centimeters of the hydrochloric acid will usually be suffi-

Antimony hydrochloric acid method.

cient for immersing the test pieces if a 200 cubic centimeter beaker is used. The same portion of hydrochloric acid may be used for at least five test pieces. Five cubic centimeters of the antimony chloride solution, however, should be added for each sample on account of the antimony being removed from the solution by the iron. The difference in weight in grams, divided by 3 (number of pieces taken) is equal to the coating in ounces per square foot.

Cushman's method.

3. (*b*) *Cushman's method.*—The determination of the weight of spelter coating by this method is based upon the action of hydrochloric acid upon the galvanized coating, collecting and measuring the hydrogen gas evolved.[17] The weight of coating may be determined upon flat sheets, corrugated sheets, and formed culverts, by the use of differently shaped rings provided with the apparatus. The coating upon wire can be determined by placing a definite length of wire under the flat ring on a glass plate.

4. The metallic rings are made of nickel, tinned iron, or other acid-resisting metal, and are fitted with No. 12, three-hole rubber stoppers. Through one hole passes the filling tube provided with glass stopcock. Through the other holes pass the exit tubes, the short tube to a position even with the bottom of the stopper, the long tube extending to a position even with the bottom of the ring. A measuring burette and leveling bottle are provided for collecting and measuring the hydrogen evolved.

5. The measuring burette is first filled with water, allowing a small amount of water in the leveling bottle. The proper ring is selected for the culvert to be tested, and is placed upon the culvert and sealed with "Plasteline," or other acid-resisting wax. The stopcock on the acid tube is turned so as to communicate with the short tube and is then connected with the measuring burette by means of a rubber tube. Water is now placed in the filling tube, the stopcock opened, and the ring and connecting tubes completely filled with water by lowering the leveling bottle and allowing the air to flow into the burette. By means of the three-way stopcock on the measuring burette, it is again filled with water without disturbing the water in the ring.

6. The stopcocks in the measuring burette are opened and the stopcock on the exit tube turned to connect the long tube with the burette. If there are any leaks in the apparatus, the water in the measuring burette will fall. With everything prepared and ready, about 30 cubic centimeters of concentrated hydrochloric acid are placed in the filling tube and about 5 cubic centimeters admitted to the ring. The hydrogen generated from the zinc will force out the water in the ring. As soon as gas appears in the long exit tube, the stopcock is quickly reversed to the short exit tube, 3 cubic centimeters of antimony chloride solution[18] are added to the acid in the filling tube, and the acid allowed to run into the ring.

7. When the generation of gas has ceased, the ring and connecting tube are completely filled with water through the filling tube by lowering the leveling bottle, and as soon as the liquid reaches the burette the stopcock is turned off, the water in the leveling bottle and burette brought to the same level, and the volume of hydrogen recorded.

8. The burette stopcock is now turned to communicate with the waste beaker and enough water passed through the ring by means of the filling tube to remove all acid. By turning the stopcock to the long exit tube the ring can be completely drained. In case the ring is lower than the burette stopcock, it is necessary to blow out the water with a rubber tube and stopper inserted in the filling tube. The ring can then be removed and the spot on the culvert cleaned with gasoline. The spot may then be coated with a paste of zinc powder and zinc chloride (50 per cent solution)

[17] The apparatus for making this test can be obtained from the Kauffman-Lattimer Co., Columbus, Ohio.

[18] Five grams of antimony chloride dissolved in 100 cubic centimeters of concentrated hydrochloric acid, specific gravity 1.20.

and heated with a blowtorch until fused, or it may be coated with a zinc powder paint or aluminum paint.

9. The number of cubic centimeters of hydrogen measured at 20° C. (68° F.) multiplied by the factor provided with each ring will give the ounces of spelter coating per square foot of actual surface on one side of the culvert. By doubling this figure the coating in ounces per square foot of sheet surface can be obtained. The factors for each ring are given in a table accompanying the apparatus.

TEST AND INSPECTION OF CORRUGATED METAL CULVERT.

1. Corrugated metal culvert shall be subject to inspection at the factory, trench, or other point of delivery by the engineer or his authorized representative.

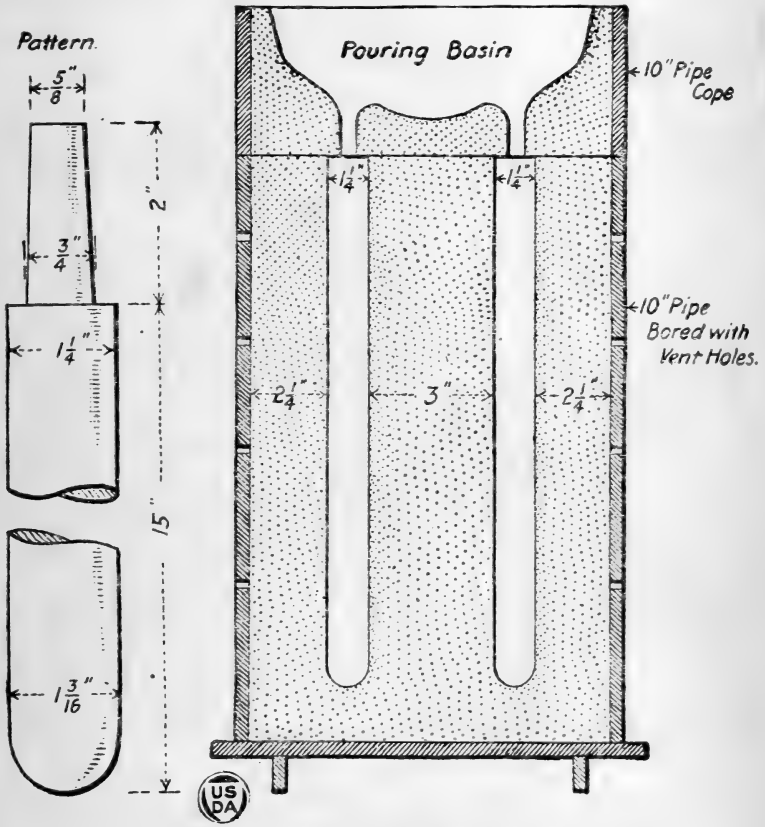


FIG. 13.—Mold for arbitration test bar.

2. The inspector shall determine the gauge of the metal, inside diameter, depth and spacing of the corrugations, width of lap, rivet spacing, and weight per linear foot of the finished pipe. He shall also take a sample of the pipe material approximately 6 inches wide and the full diameter of the pipe for measurement, chemical analysis, and determination of the amount of galvanizing or spelter coating.

3. Chemical analysis will be made of all metal culvert pipe to determine whether the requirements of the specifications as to quality of the material are met.

4. The amount of galvanizing or spelter coating will be determined in accordance with the methods for testing the amount of spelter coating on culvert metal, page 81.

TEST AND INSPECTION OF CAST-IRON CULVERT PIPE.

1. All cast-iron culvert pipe shall be made of cast iron of the quality specified and of such character as shall make the metal of the castings strong, tough, and of even grain. The size and weight of the pipe shall be as specified.

PHYSICAL PROPERTIES AND TESTS.

Test specimens.

2. The transverse test specimens (arbitration test bars) specified in section 4 shall be placed horizontally upon supports 12 inches apart and tested under a centrally applied load. The average load and deflection shall not be less than that specified.

3. The form and dimensions of the mold for the arbitration test bar shall be in accordance with Figure 13. The bottom of the bar shall be one-sixteenth inch smaller in diameter than the top to allow for draft and for the strain of pouring. The pattern shall not be rapped before withdrawing. The flash shall be rammed up with green molding sand, a little damper than usual, well mixed, and put through a No. 8 sieve, with a mixture of 1 to 12 bituminous facing. The mold shall be rammed evenly and fairly hard, thoroughly dried, and not cast until it is cold. The test bar shall not be removed from the mold until cold enough to be handled. It shall not be rumbled or otherwise treated, being simply brushed off before testing.

4. From each melt of metal not less than three test specimens (arbitration test bars) shall be poured, the first of which shall be poured within five minutes after the first ladle is tapped and the remainder at intervals not exceeding one hour throughout the melt.

5. The manufacturer shall afford the inspector representing the purchaser, free of cost, all reasonable facilities to satisfy him that the castings are being furnished in accordance with the specifications. All tests and inspections shall be so conducted as not to interfere unnecessarily with the operation of the work, and shall be made prior to shipment unless otherwise specified.

METHODS OF SAMPLING AND TESTING METALLIC MATERIALS.

(A. S. T. M. standard method, serial designation E 1–18.)

I. METHODS FOR TENSION TESTS OF METALS.

Definition of Terms.

1. *Elastic limit* is the greatest load per unit of original cross section which does not produce a permanent set.

Note.—This determination is rarely made in the commercial testing of materials.

Proportional limit is the load per unit of original cross section at which the deformations cease to be directly proportional to the loads.

Note.—This determination is rarely made in the commercial testing of materials.

Yield point is the load per unit of original cross section at which a marked increase in the deformation of the specimen occurs without increase of load. It is usually determined by the drop of the beam of the testing machine or by the use of dividers.

2. Information obtained from the various laboratories in which tension tests are made shows that in many cases the forms and dimensions of specimens as recommended by the American Society for Testing Materials are in use and that in other cases these forms and dimensions most nearly reconcile the differences that exist between the various forms employed.

3. It is therefore recommended that the selection of specimens and their forms and dimensions shall conform to the specifications for each material as are now adopted by the American Society for Testing Materials.

In the case of flats one-quarter inch or less in thickness, the dimensions shall be as follows: Width equal to five times the thickness of the specimen, except that in no case shall the width be less than three-quarters inch; gauge length equal to twenty-four times the thickness of the specimen, except that in no case shall the gauge length be less than 2 inches.

4. It is believed that the distance between the end of the gauge length and the beginning of the shoulders as prescribed in the standard specifications of the American Society for Testing Materials is ample to avoid interference with proper elongation, and no grounds are found for recommending any change.

Crosshead speed.

5. The pulling speed has a marked influence on the tensile properties shown by materials tested, an increase in speed increasing the values found for yield point and tensile strength. In testing steel and wrought iron in gauge lengths of 2 and 8 inches in accordance with the specifications of the American Society for Testing Materials the speed of the machine, by which is meant the speed of the crosshead when the machine is running idle, shall conform to the following requirements:

The crosshead speed of the testing machine shall be such that the beam of the machine can be kept balanced, but in no case shall be values given in the following table be exceeded:

Specified minimum tensile strength of material (pounds per square inch.)	Gauge length.	Maximum crosshead speed in determining—	
		Yield point.	Tensile strength.
	Inches.	*Inches per minute.*	*Inches per minute.*
80,000 or under	2	0.50	2.0
	8	2.00	6.0
Over 80,000	2	.25	1.0
	8	.50	2.0

6. In determining the elastic limit (so called) by the method prescribed in the American Society for Testing Materials specifications for forgings and cold-rolled axles (serial designation A 18, A 19, A 63, and A 22) the crosshead speed for the 2-inch gauge length shall not exceed 0.125 inch per minute.

7. In determining the proportional limit, the crosshead speed shall not exceed 0.025 inch per inch of gauge length per minute.

8. In determining the modulus of elasticity, the elastic limit, and the proportional limit, the extensometer should be attached to at least two sides of the specimen, to compensate for unequal elongation, for improper holding, or any slight bending that may exist in the specimen.

9. All authorities seem to regard it as desirable to measure the elongation on two or more sides of the test specimen, and most extensometers provide for so doing.

10. The greatest accuracy is required in determining the modulus of elasticity, since small errors in measuring elongation are of considerable consequence in the result.

11. Since the modulus is determined for points well within the elastic limit, the total elongation to be measured is much smaller than at the elastic limit.

12. The elastic limit should be determined with great care, but any inaccuracy will cause less proportionate error than in the case of the modulus. The yield point, being less well defined, can not be so closely determined, and it is believed that in most cases the use of dividers instead of an extensometer will give sufficiently accurate results.

13. It is considered undesirable in accurate determinations of the modulus of elasticity to use a shorter gauge length than 8 inches. It is evident that the greater the total elongation measured the less will be the error due to inaccuracy of the reading, and the accuracy thus appears to increase directly as the gauge length.

14. That the difference between short and long gauge lengths has a greater influence in affecting results than other factors (personal error, inaccuracy of the testing machine, etc.) is shown by the closely agreeing readings obtained with the greater lengths.

15. The effect of improper methods of holding specimens can not be established from the results of actual tests. The result of improper methods of gripping materials of low elongation, such as cast iron, is well known, and it is probable that in material of a more ductile nature the effect is largely local and does not extend to the portion of the specimen within the gauge marks.

Conditions to Insure Correct Testing Machines.

16. It is recommended that in machines on which specimen tests are made, whether the power be applied hydraulically or by means of screws and gears, the load be measured by a separate system of levers and knife-edges, or by a method similar to that employed in the Emery testing machines.

17. All knife-edges shall be kept sharp, and free from oil and dirt, and the machine shall be sensitive to a variation in load of one two-hundred-and-fiftieth of the load carried. Design and workmanship on testing machines shall be good, and they shall be calibrated at least once every six months by the following method.

Calibration of Testing Machines.

18. Test for accuracy by loading the weighing table with standard weights and compare the actual weight at each addition with the reading of the beam. If the table is uniformly loaded in this manner with the full amount of weights that it will accommodate, the proportionality of the levers and the weighing beam can be successfully established. This relation, in a properly designed machine, will remain constant for all loads, but as a further test for sensitiveness under greater loads than can be accommodated in this manner, the following procedure is recommended:

19. Place in the machine a tension bar of such cross section that the maximum capacity will not stress it to the elastic limit. Stress this bar to various extents through the full range of the machine and at each load balance the beam and place upon the weighing table standard weights of 100 pounds. A weight one two-hundred-and-fiftieth of the total load on the machine should produce a readable movement of the beam.

20. Where evidence of the accuracy of the machine over its whole range is desired, a known load may be applied by means of an extensometer and calibrated bar whose modulus of elasticity has been determined with exactness.

21. It is recommended that a device be adopted conforming to the following reqiurements, in which the extensometer and bar are permanently attached to each other:

(*a*) The bar should be of high-elastic-limit material, and of such cross section that this limit will be well above the total capacity of the machine on which it is to be used.

(*b*) This bar shall be annealed or otherwise treated so as to eliminate internal or unequal stress in the material, and to insure its elastic modulus being uniform for successive tests.

(*c*) The extensometer shall be permanently attached to the bar and shall measure the elongation on two opposite sides.

(*d*) The extensometer shall be preferably of the indicating or direct-reading type and shall indicate to ten-thousandths of an inch or less.

(*e*) The method of securing the bar in the drawheads of the machine shall be positive and without slip, and shall insure its axial location.

(*f*) The length of the bar measured by the extensometer shall be sufficient so that the smallest extensometer division—that is, 0.0001 inch—shall correspond to a difference in loading of 0.2 per cent of the capacity of the machine or less.

(*g*) The extensometer shall be protected from injury by a permanently attached case with cover removable for reading the scale.

(*h*) The apparatus shall itself be calibrated either by the United States Bureau of Standards or in a manner that will insure equally trustworthy results.

(*i*) The apparatus shall be plainly marked with the maximum load that can be applied safely without injury.

Methods of Gripping Test Specimens.

22. It is recommended that for specimens of rolled material, serrated grips, flat and V-shaped, be adopted, the former for rectangular and the latter for round specimens. Serrated grips with curved faces appear to have no advantage, and to cause crushing of the material.

23. Wedges with ball and socket do not seem to be necessary, and for commercial testing their use has been generally discontinued.

24. Specimens of turned form, with threaded ends, should be secured in such a manner that side bending stresses are avoided.

25. It is considered important for correct results that the specimen be located in the exact center of the heads, and to better secure this condition the openings in the heads should be lined up with each other by means of a plumb bob and should be tested for parellelism with a spirit level. Each pair of packing pieces and wedges that are to be used together in the same head should correspond exactly in thickness and other dimensions, and the wedges should be inserted the same distance when the specimen is in place.

Selection and Preparation of Specimen.

26. Specimens representative of steel castings may be cut from the bottom of a sink head or riser, or from a coupon attached to the casting. In either case the part from which the specimen is taken should be relatively large in proportion to the size of the casting and should be annealed with it.

27. Workmanship on specimens shall be of the most careful nature, and surfaces should be free from nicks and tool marks. All wire edges should be removed and corners generously rounded.

28. If specimens of rolled material are sheared in the rough from sections, at least one-eighth inch of the material should be removed from the sheared edges in machining.

General Requirements for the Measuring of Elongation.

29. In determining the modulus of elasticity and the elastic limit, it is recommended that when practicable the elongation be measured in a length not less than 8 inches, and that the following requirements be provided for:

(*a*) The specimen shall be round in section, finished as smooth as possible, and shall be provided with threaded ends for attachment to the drawheads of the machine.

(*b*) The specimen shall be placed in the exact center of the heads and be secured in some positive manner so that slip and side bending stresses do not occur.

(*c*) The extensometer should be of a type to measure the elongation on two or more sides of the specimen.

(*d*) It should read to 0.0001 inch or less.

(*e*) It should be of such a design that no change of zero will occur upon release of the load in determining the real elastic limit.

(*f*) The load shall be applied so slowly that simultaneous readings of elongation and load can be obtained with certainty.

(*g*) The testing machine shall have previously been calibrated for accuracy and sensitiveness, and heads lined up and made parallel.

II. METHODS FOR COMPRESSION TESTS OF METALS.

Definition of Terms.

1. *Elastic limit* is the greatest load per unit of original cross section which does not produce a permanent set.

Note:—This determination is rarely made in the commercial testing of materials.

Proportional limit is the load per unit of original cross section at which the deformations cease to be directly proportional to the loads.

Note:—This determination is rarely made in the commercial testing of materials.

Yield point is the load per unit of original cross section at which a marked increase in the deformation of the specimen occurs without increase of load. It is usually determined by the drop of the beam of the testing machine, or by the use of dividers.

2. The test specimen shall be a cylinder having plane ends truly normal to its axis.

Note—Only two replies from testing laboratories mention cubes. A cylindrical specimen will usually be cheaper to prepare than a cube. The stresses are probably less uniformly distributed over a square than over a circular section, owing to the influence of the corners, this being especially the case with the internal shearing stresses which accompany the compression.

3. The diameter of the specimen shall be not less than 1 inch nor greater than 1.13 inches. A specimen 1 inch in diameter is to be preferred.

Note—The range of diameter mentioned in the replies from testing laboratories is from 1 inch to 1.129 inches. A diameter of 1.1284 inches gives a sectional area of 1 square inch.

4. The length of the specimen should be between 2.5 and 4 diameters.

Note.—Two testing laboratories use a length of 1 diameter, one a length of from 1.5 to 2 diameters, one a length of 2.6 diameters, and one a length of 10.5 diameters. It is believed that a length less than 2.5 diameters is not sufficient for the internal shear to be properly developed, and that such short lengths give a fictitious strength owing to the friction of the bearing plates of the machine, which causes the specimen to assume a barrel-like form.

5. No bedding should be used for the ends of the specimen.

Note.—Only one reply favors bedding. It is known by general experience that bedding modifies the breaking load and that different kinds of bedding have different influences.

6. The bearing blocks which transmit the pressure from the testing machine should be truly normal to the plane ends of the specimen. To secure this, one of the blocks should be provided with a hemispherical bearing which can turn freely.

Note.—These requirements seem essential in order that the load may not be eccentrically applied to the specimen, and are generally recommended in the replies from testing laboratories.

7. The speed of compression should be slow, not exceeding 0.1 inch per minute. Near the elastic limit and yield point the load should be increased very slowly. *Speed of compression.*

NOTE.—A lower speed than that stated might be advisable if permitted by the testing machine. Evidently a higher speed may be allowed with a long specimen than with a short one.

8. For determining modulus of elasticity, the linear compression of the specimen should be observed by a precise compressometer which is attached to the specimen anad does not touch the bearing blocks of the machine. Readings of the compressometer should be taken for three loads, the first at about one-fourth, the second at about one-half, and the third at about three-fourths of the elastic limit.

NOTE.—It is believed that these measurements are sufficient for most commercial work. Nothing is said about the release of the specimen from load, since opinions differ as to its advisability.

9. To determine the elastic and also the proportional limit, several readings of the compressometer should be taken as that limit is approached for load increments of 1,000 pounds per square inch.

NOTE.—This requirement seems sufficient to determine the proportional limit, for materials in which such a limit exists. It does not seem wise to require the first permanent set to be observed for ordinary commercial work.

10. The yield point is to be noted as corresponding to that load for which the compressometer shows a linear compression without an increase in load. In the absence of a compressometer this point may be noted, for ductile materials, by the drop of the scale beam.

NOTE.—This requirement corresponds to the usual practice of testing laboratories. It is regarded as important that the term "elastic limit" should not be used to designate the yield point.

11. Measurements for the modulus of elasticity, elastic limit, proportional limit, and yield point may be made, if desired, on a specimen ranging in length from 10 to 15 diameters.

NOTE.—This clause is inserted because it may often be difficult to apply a compressometer in a length shorter than 4 inches.

12. The record of the test should mention any phenomena observed near the elastic limit, proportional limit, and yield point. The manner of final failure should also be noted when the test is carried to this limit.

NOTE.—This requirement furnishes data for comparing the behavior of brittle and ductile metals near critical points of molecular change.

III. METHOD FOR TRANSVERSE TESTS OF METALS

1. In the case of cast metals, when transverse tests are to be used to aid in determining the quality of the material, the specimen used shall be cast vertical, shall be $1\frac{1}{4}$ inches in diameter, and long enough to use a span of at least 15 times the diameter.

NOTE.—It is important that a definite and uniform standard be adopted so that the results may be comparable with each other; hence the diameter specified above (sectional area corresponding to practically 1 square inch). The determination of span is at present the subject of international tests to decide upon a definite distance to replace the present standard of 12 inches. It will probably be from 16 to 18 inches. The circular section will best secure a uniform thickness of skin, and thus avoid this irregularity when other sections are employed.

In the case of ductile materials (except in impact tests) transverse tests shall never be used to determine the quality of the material, tension tests being those suitable for the purpose.

NOTE.—In small round or square bars of ductile material both the modulus of rupture and the transverse elastic limit vary considerably with the span.

In the case of tests made for determining constants to be used for designing, the specimen shall conform as nearly as possible

with the form and size of the piece to be used. Thus, if I or T sections are to be used, the specimens shall be of I or T section. In the case of flat springs or plate glass, they shall be flat; in the case of timber, rectangular; etc.

NOTE.—It is well known that the modulus of rupture varies with the shape of the section, being very much greater in the case of round than in I sections. Hence the modulus of rupture suitable for use for one would be entirely unsuitable for the other. In rolled sections, the smaller ones are subjected to a more thorough working in the process of rolling than the larger.

2. In the case of the "arbitration bar" adopted for cast iron, the span has been fixed at 12 inches, but may be extended as above stated. The bar will serve for cast and brittle materials.

In the case of ductile materials, when the modulus of rupture is desired, the span shall generally be less than 12 or 15 times the depth. Exceptions, however, occur, as in flat springs and in some cases in full-sized pieces, when the spans and methods of supporting the ends, etc., shall conform to the conditions of service.

3. In the case of cast and brittle metals, the speed of testing shall not exceed 0.2 inch per minute. For other specimens the speed shall be correspondingly low.

Preparation of specimens.

4. The preparation of the specimen shall be such that it truly represents the material itself. The introduction of extraneous influences should be avoided as far as the knowledge of the material will permit. Thus, in cast metals no coupons shall be used; cast materials for tests shall go into dry molds standing vertical.

No specimen shall be machined before testing, except when information is specifically desired regarding the strength of such machined specimens.

5. The transverse yield point for ductile materials shall be noted approximately by the drop of the scalebeam.

6. If the transverse elastic limit is to be determined for comparison with that obtained in the tensile test, the successive increments of load in the neighborhood of the transverse elastic limit shall be comparatively small, and after each load has been applied and the corresponding deflection measured by means of the deflectometer, the load shall be removed and the deflection measured again to determine the permanent set.

In those cases where the arbitration bar is used for such cast materials as have an elastic limit, the increment of load used near the transverse elastic limit shall be 250 pounds.

NOTE.—It is well known that when the transverse elastic limit is determined of course by means of a transverse test, the extreme fiber stress at this transverse elastic limit is not the same as that at the tensile elastic limit of the material; and, moreover, that it varies with both the section and the span; hence the desirability of comparing the transverse elastic limit with the tensile elastic limit.

7. In the case of ductile materials the arrangement of the supports shall be such that longitudinal tension in the specimen due to the rigidity of the supports is avoided.

8. In the case of ductile materials special care shall be used when determining the ultimate load. For this purpose it will be necessary when approaching the ultimate (that is, the maximum) load to make the speed of testing slow enough to enable the observer to note the maximum load.

In many cases, as in I and T beams, the maximum load can be easily ascertained, while in others, such as round or flat sections with short spans, it may not be possible to determine it exactly, but it will almost always be possible to determine it with sufficient accuracy for all practical purposes.

IV. METHODS FOR BRINELL HARDNESS TESTS OF METALS.

CHEMICAL COMPOSITION AND HEAT TREATMENT OF BALLS.

1. The chemical composition, as far as carbon and chromium are concerned, should be from 1 to 1.2 per cent of carbon and from 1 to 1.5 per cent of chromium.

2. The heat treatment should be such as will result in making the balls as hard as possible consistent with the ability to resist the pressure without cracking or crushing.

NOTE.—While a long series of careful experiments would be needed to justify the specification of more exact conditions in these regards, and while some users of these tests think that the chemical composition (within limits) plays a very small part, if any, in the problem, it is believed that the above requirements will be found satisfactory for commercial work until such time as suitable experiments shall have furnished the data necessary for making the conditions more precise.

DIAMETER AND FORM OF BALLS.

Balls.

3. The standard diameter of balls should be 10 millimeters, with a permissible variation of 0.0025 millimeter (0.0001 inch), plus or minus; no ball, either new or old, showing a greater variation is to be employed. The standard diameter should always be employed except in very rare cases when some other is absolutely necessary. If at any time in testing a hardness of No. 600 be exceeded, the balls should be remicrometered.

NOTE.—Thus far there is not sufficient evidence to show that the hardness numbers will be the same when different diameters of balls are used, and some of the evidence indicates that the reverse is the case; hence the importance of adhering to one size of ball.

PRESSURE.

Pressure.

4. The standard pressures used should be 3,000 kilograms for steel and 500 kilograms for softer metals. Departure from these pressures should never be tolerated except in rare cases where it is unavoidable. The time of pressure should be at least 30 seconds.

NOTE.—The fact that, with our present light on the subject, we can only regard the results as comparative renders it important to employ as few different pressures as possible.

MEASUREMENT OF DIAMETER OR DEPTH OF INDENTATION.

Measurement.

5. Whether the diameter or the depth of the indentation is measured, apparatus should be used that will give results as accurately as a microscope mounted on, and moved by, a micrometer screw.

NOTE.—As to the choice between the two, there exists a very decided difference of opinion, some thinking one and some the other more conducive to accuracy. The source of error in either case (assuming the measuring apparatus to be accurate) is the depression or the elevation of the metal immediately surrounding the indentation.

TESTS OF WIRE ROPE (GUARDRAIL).

1. The report of the physical test of wire rope shall include the following information: Diameter of the rope; diameter of the wire; number of strands and wires; length of lay of strands and wires; cross-sectional area in square inches, breaking load in pounds, and a note as to whether the failure was in the body of the rope or at the socket.

DEFINITIONS.

2. (*a*) The "lay of the rope" is the length expressed in inches for each complete turn of a strand around the axis of the rope measured along the axis.

(*b*) The "lay of the strand" is the length expressed in inches for each complete turn of a wire around the axis of the strand measured along the axis.

(*c*) The diameter of a wire rope is the diameter of a circle inclosing it.

3. The cross-sectional area of the rope shall be determined as follows: Measure the diameter of the component wires of a

strand and obtain the mean diameter. The area calculated from the mean diameter of the wire multiplied by the number of wires in each strand and by the number of strands gives the aggregate area of the wires in the rope.

4. The test specimen for the tension test shall be free from bends and not less than 4 feet in length. Before cutting the test pieces from the coil of rope the ends must be "served" or wound about with wire for a length of about 1 inch to prevent the strands from unlaying.

PREPARATION OF SPECIMEN FOR TEST.

Preparation of specimens.

5. A socket shall be attached to each end of the rope for the tension test. In preparing the rope for socketing, the ends are first served or wound around with fine soft wire for a length of about one-half inch at the ends and also at a distance from the ends equal to the lengths of the basket of the socket. The ends of the rope are then slipped through the socket and the serving wire removed from the end only. Unlay the strands, separate the individual wires, and straighten them. Cut out the hemp center, if any. Cleanse the wire thoroughly with kerosene or gasoline and wipe dry. Tin the wires by dipping first in a mild zinc chloride solution and then in molten zinc. When removed from the latter, they should be knocked with a stick or hammer to remove the excess zinc. Repeat the tinning until all the wires are thoroughly coated. Pull the frayed ends back into the basket of the socket and spread the wires evenly in the socket and even with the top of the basket. Put fire clay or asbestos fiber around the rope at the bottom of the socket, heat the socket for a short time with a blowtorch, and pour in molten *pure zinc* (not babbitt or lead).

6. Put the sockets through the heads of the testing machine and place pins through the eyes of the sockets, using steel blocks on each side of the socket, if necessary, to secure a firm bearing (pins should be as large as possible). Apply the load slowly to give the strands and wires opportunity to properly bed upon one another during the application of the load. Continue loading until fracture of one or more strands of the rope occurs.

7. The uniformity of the galvanizing or spelter coating, if any, shall be determined in accordance with the method for uniformity of spelter coating.

TEST FOR UNIFORMITY OF GALVANIZING OR SPELTER COATING ON WIRE.

Coating.

1. This method gives in detail the test to be applied to galvanized wire.

2. (*a*) *Coating.*—The galvanizing shall consist of a continuous coating of pure zinc of uniform thickness, and so applied that it adheres firmly to the surface of the iron or steel. The finished product shall be smooth.

Cleaning.

3. (*b*) *Cleaning.*—The sample shall be cleaned before testing, first with Carbona, benzine, or turpentine, and cotton waste (not with a brush), and then thoroughly rinsed in clean water and wiped dry with clean cotton waste.

4. The samples shall be clean and dry before each immersion in the solution.

Solution.

5. (*c*) *Solution.*—The standard solution of copper sulphate shall consist of commercial copper sulphate crystals dissolved in cold water, about in the proportion of 36 parts, by weight, of crystals in 100 parts, by weight, of water. The solution shall be neutralized by the addition of an excess of chemically pure cupric oxide (CuO). The presence of an excess of cupric oxide will be shown by the sediment of this reagent at the bottom of the containing vessel.

6. The neutralized solution shall be filtered before using by passing through filter paper. The filtered solution shall have a

specific gravity of 1.186 at 65° F. (reading the scale at the level of the solution) at the beginning of each test. In case the filtered solution is high in specific gravity, clean water shall be added to reduce the specific gravity to 1.186 at 65° F. In case the filtered solution is low in specific gravity, filtered solution of a higher specific gravity shall be added to make the specific gravity 1.186 at 65° F.

7. As soon as the stronger solution is taken from the vessel containing the unfiltered neutralized stock solution, additional crystals and water must be added to the stock solution. An excess of cupric oxide shall always be kept in the unfiltered stock solution.

8. (*d*) *Quantity of solution.*—Wire samples shall be tested in a glass jar of at least 2 inches inside diameter. The jar without the wire samples shall be filled with standard solution to a depth of at least 4 inches.

9. Solution shall not be used for more than one series of four immersions.

10. (*e*) *Samples.*—Not more than seven wires shall be simultaneously immersed in the specified quantity of solution. *Samples.*

11. The samples shall not be grouped or twisted together, but shall be well separated so as to permit the action of the solution to be uniform upon all immersed portions of the samples.

12. (*f*) *Test.*—Clean and dry samples shall be immersed in the required quantity of standard solution in accordance with the cycle of immersions called for in the specifications. *Method of test.*

13. The temperature of the solution shall be maintained between 62° and 68° F. at all times during the test.

14. After each immersion the samples shall be immediately washed in clean water having a temperature between 62° and 68° F. and wiped dry with cotton waste.

15. (*g*) *Rejection.*—If after the test described in section (*f*) there should be a bright metallic copper deposit upon the samples, the lot represented by the samples shall be rejected.

16. Copper deposits on zinc or within 1 inch of the cut end shall not be considered causes for rejection.

17. In case of a failure of only one wire in a group of seven wires immersed together, or if there is a reasonable doubt as to the copper deposit, two check tests shall be made on these seven wires and the lot reported in accordance with the majority of the sets of tests.

NOTE.—The equipment necessary for the test herein outlined is as follows:

- Commercial copper sulphate crystals.
- Chemically pure cupric oxide (CuO).
- Running water.
- Warm water or ice as per needs.
- Carbona, benzine, or turpentine.
- Glass jars at least 2 inches inside diameter by at least 4½ inches high.
- Glass or earthenware jars for hardware samples.
- Vessel for washing samples.
- Tray for holding jars of stock solution.
- Jars, bottles, and porcelain basket for stock solution.
- Cotton waste.
- Hydrometer cylinder 3 inches in diameter by 15 inches high.
- Thermometer with large Fahrenheit scale correct at 62° and 68°.
- Hydrometer correct at 1.186 at 65° F.
- Filter paper.

INDEX.

ORGANIZATION OF THE UNITED STATES DEPARTMENT OF AGRICULTURE.

January 8, 1924.

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96

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