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UNITED STATES DEPARTMENT OF AGRICULTURE



DEPARTMENT BULLETIN No. 1216



Washington, D. C.

Issued May, 1924

Revised September, 1928

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

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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 including all revisions adopted prior to August 1, 1928

INTRODUCTION

Since the publication of United States Department of Agriculture Bulletin No. 1216 in May, 1924, a number of revisions in the official methods of testing of the American Association of State Highway Officials have been adopted by the association upon recommendation of the subcommittee on tests and investigations (now the committee on materials.)¹ All of these revisions have been incorporated in the methods of tests as herein given.

In the preparation of its standard methods of testing, the association, whenever possible, has adopted without change existing American Society for Testing Materials standard or tentative standard methods. Whenever this has been done the fact has been so indicated in the present bulletin by placing the reference to the American Society for Testing Materials method immediately following the title. Where the official method of the association is

¹ The personnel of the committee on materials is as follows: H. S. Mattimore, State Highway Department, Pennsylvania, *Chairman*; F. H. Jackson, U. S. Bureau of Public Roads, Washington, D. C., *Secretary*; J. H. Bateman, State Highway Commission, Louisiana; W. L. Blaum, Department of Public Works, New York; A. V. Bratt, Department of Public Works, Massachusetts; Shreve Clark, State Highway Commission, Virginia; R. W. Crum, State Highway Commission, Iowa; R. B. Dayton, State Road Commission, West Virginia; W. J. Emmons, Department of Highways, Michigan; N. M. Finkbner, State Highway Commission, Oregon; F. G. Flood, State Highway Commission, Connecticut; R. B. Gage, State Highway Commission, New Jersey; V. L. Glover, Department of Public Works, Illinois; Harvey A. Hall, State Road Department, Florida; Raymond Harsch, Department of Public Works, Idaho; C. A. Hogentogler, U. S. Bureau of Public Roads, Washington, D. C.; John V. Kelly, State Board of Public Roads, Rhode Island; E. D. Kivett, State Highway Commission, North Carolina; F. C. Lang, Department of Highways, Minnesota; E. T. Maddock, State Highway Commission, California; D. D. McGuire, Department of Highways and Public Works, Tennessee; C. L. McKesson, State Highway Commission, California; H. M. Milburn, U. S. Bureau of Public Roads, Washington, D. C.; Levi Muir, jr., State Road Commission, Utah; J. E. Myers, Department of Public Works, New York; W. A. Norris, State Highway Department, Wyoming; W. F. Purrington, State Highway Department, New Hampshire; A. S. Rea, Department of Highways and Public Works, Ohio; F. V. Reigel, State Highway Commission, Missouri; C. H. Scholer, State Highway Commission, Kansas; M. H. Ulman, State Highway Department, Pennsylvania.

the same as the American Society for Testing Materials method with the exception of slight changes in wording, such changes are shown in the text by bracketing those portions of the American Society for Testing Materials text which have been omitted by the association and italicizing additions made by the association to the American Society for Testing Materials method.^a In a few instances the association method, although based on the American Society for Testing Materials method, differ sufficiently from it as to phraseology as to make it impossible to follow the procedure above indicated. In such cases, reference to the fact that the method is based on an American Society for Testing Materials method is indicated immediately following the title.

^a This does not apply to table heads, all of which are italicized as a matter of style.

1. METHODS OF SAMPLING AND TESTING PORTLAND CEMENT

(A. S. T. M. standard method (4)² serial designation C9-26, sections 16-57, inclusive)

SAMPLING

16. Tests may be made on individual or composite samples as may be ordered. Each test sample should weigh at least 4 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 represent each 200 barrels unless otherwise specified by the purchaser.

(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 unless otherwise specified by the purchaser.

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

(a) From the conveyor delivering to the bin.—At least 4 pounds of cement shall be taken from approximately each 100 barrels passing over the conveyor. This may be secured by taking the entire test sample at a single operation, known as the grab method, or by combining several portions taken at regular intervals, known as the composite method.

(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. Sample 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. The sampling shall be done by or under the direction of a responsible representative of the purchaser.

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

CHEMICAL ANALYSIS

LOSS ON IGNITION

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

Method (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 five 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.

Method (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.

² *Italic figures in parentheses refer to literature cited, p. 129.*

22. 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

23. To a 1-gram sample of cement shall be added 25 cubic centimeters of water and 5 cubic centimeters of concentrated hydrochloric acid (specific gravity 1.19). Material shall be ground with the flattened end of a glass rod until it is evident that the decomposition of the cement is complete. The solution shall then be diluted to 50 cubic centimeters and digested on a steam bath for 15 minutes. 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 hot water, then with a few drops of hot hydrochloric acid (1:9), and finally with hot water, then ignited at a red heat and weighed as the insoluble residue.

24. 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

25. To a 1-gram sample of cement shall be added 25 cubic centimeters of water and 5 cubic centimeters of concentrated hydrochloric acid (specific gravity, 1.19). Material shall be ground with the flattened end of glass rod until it is evident that decomposition of the cement is complete. The solution shall be diluted to 50 cubic centimeters and digested on a steam bath for 15 minutes, filtered, and the residue washed thoroughly with hot 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 then be digested on the steam bath at least three hours, preferably overnight. 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.

26. 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

27. To 0.5 gram of the cement in an evaporating dish shall be added 10 cubic centimeter of water to prevent lumping and then 10 cubic centimeter of concentrated hydrochloric acid (specific gravity, 1.19). The material shall be ground with the flattened end of a glass rod 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° C. or even 200° C. for one-half to one hour. The residue shall be treated with 10 cubic centimeters of hydrochloric acid diluted with an equal amount of water. The dish shall be covered, and the solution digested for 10 minutes on a steam bath or water bath. The solution shall be diluted to 75 cubic centimeters, filtered into a beaker, and the separated silica washed thoroughly with hot water.³ Five cubic centimeters of concentrated hydrochloric acid and two drops of methyl red indicator (0.2 per cent alcoholic solution) shall be added to the filtrate⁴ (about 250 cubic centimeters).

³ Since this procedure does not involve the determination of silica, a second evaporation is unnecessary.

⁴ Manganese, if present, is distributed between the precipitates of iron and alumina, calcium, and magnesium. In the amounts usually present in Portland cement it may be neglected. If it is desired to determine the small amount of manganese that may be present in the magnesium pyrophosphate, this may be done colorimetrically as described in the U. S. Geological Survey Bulletin No. 700 (9, p. 153). If present in larger amounts, manganese should be precipitated with the iron and alumina, preferably by the persulphate method (U. S. Geological Survey Bulletin No. 700 (9, p. 112)). If this method is used more aluminum passes into solution than in the method above described.

Dilute ammonium hydroxide shall be added dropwise until the color of the solution changes to a distinct yellow. The solution shall be boiled for one or two minutes and filtered at once. The beaker and precipitate shall be washed slightly with a hot 2 per cent solution of ammonium chloride (or ammonium nitrate). 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 then be extracted with acid, the solution and washings being added to the solution of the precipitate. The aluminum and iron shall then be reprecipitated at boiling heat by ammonium hydroxide as before in a volume of about 100 cubic centimeters and the second precipitate shall be collected and washed with a hot 2 per cent solution of ammonium chloride (or ammonium nitrate) 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, and the filtrate set aside. 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 cautiously moistened with water, redissolved in hydrochloric acid, and the solution diluted to 100 cubic centimeters; ammonia shall be added in slight excess, the liquid boiled, and filtered if a precipitate appears. The lime shall then be reprecipitated by ammonium oxalate, allowed to stand till 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 and filtered (to remove a little iron and aluminum, and perhaps calcium). When cool, the solution shall be acidified with hydrochloric acid, 10 cubic centimeters of saturated solution of sodium-ammonium-hydrogen phosphate added, and ammonia drop by drop, with constant stirring. When the crystalline ammonium-magnesium orthophosphate has formed, 5 cubic centimeters excess ammonia shall be added. The solution shall be set aside for not less than four hours, preferably overnight, in a cool place, filtered and washed with water containing 2.5 per cent NH_3 . The precipitate shall be dissolved in a small quantity of hot 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.

28. 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.

DETERMINATION OF FINENESS

29. 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 about 2 inches below the top of the frame. The joint between the cloth and frame shall be smoothly filled with solder to prevent lodging of the cement. The sieve frames shall be circular, approximately 8 inches in diameter, and may be provided with a pan and cover.

30. A standard No. 200 sieve is one having nominally a 0.0029-inch opening, certified by the United States Bureau of Standards, and conforming to the specifications for this sieve in the Standard Specifications for Sieves for Testing Purposes (serial designation: E 11) of the American Society for Testing Materials (3). The correction to the sieving value of the sieve shall be determined by sieving tests made in conformity with the standard specifications

for these tests on a standardized cement which gives a residue of about 20 per cent on the No. 200 sieve.

31. 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, applying the sieve correction.

NOTES.—The essential points in the sieving operation may be summarized as follows: (1) Rotation of the sieve throughout the process. (2) Guarding against loss of material. Sieve over white paper and always tap the sieve gently. (3) Use of a balance which will give results correct within 5 milligrams and sufficiently sensitive so that the rest point will be deflected at least two divisions of the scale for an added load of 5 milligrams. (4) Washers, shot, and slugs should never be used on the sieve. (5) Excessive humidity interferes with good sieving. It tends to decrease the percentage of cement passing the sieve, and in general to produce irregular results.

A plus (+) sieve correction indicates the amount to be added to and a minus (–) sieve correction the amount to be subtracted from the percentage passing the sieve to obtain the true fineness. The percentage passing is the amount obtained by subtracting the per cent residue from 100. The corrections are used in the reverse order when applied to the residues direct.

32. 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 31.

MIXING CEMENT PASTES AND MORTARS

33. The quantities of dry materials to be mixed at one time shall be 500 grams for neat cement mixtures and 1,000 grams for mortar mixtures. 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 nonabsorbent 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 the water the operation shall be completed by continuous, vigorous mixing, squeezing, and kneading with the hands for at least one minute. During the operation of mixing, the hands shall be protected by rubber gloves.

NOTE.—In order to secure uniformity in the results of tests for the time of setting and tensile strength the manner of mixing above described shall 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 shall be on the side of overmixing.

34. The temperature of the room, the materials, the mixing water, the moist closet, and storage-tank water shall be maintained as nearly as practicable at 21° C. (70° F.) and the mixing water, moist closet, and the water in the storage tank shall not vary from this temperature more than 3° C. (5° F.).

NORMAL CONSISTENCY

35. The Vicat apparatus consists of a frame A (fig. 1) bearing a movable rod B, weighing 300 grams, one end C being 1 centimeter in diameter for a distance of 6 centimeters, the other having a removable needle D, 1 millimeter in diameter, 6 centimeters long. The rod is reversible, and can be held in any desired position by a screw E, and has midway between the ends a mark F which moves under a scale (graduated to millimeters) attached to the frame A. The paste is held in a rigid conical ring, resting on a glass plate about 10 centimeters square. The ring shall be made of a noncorroding, nonabsorbent material, and shall have an inside diameter of 7 centimeters at the base, 6 centimeters at the top, and a height of 4 centimeters.

36. In making the determination, 500 grams of cement, with a measured quantity of water, shall be kneaded into a paste, as described in section 33, 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 conical 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, and the top smoothed, if necessary, with a few light touches of the pointed end of the trowel. 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 sur-

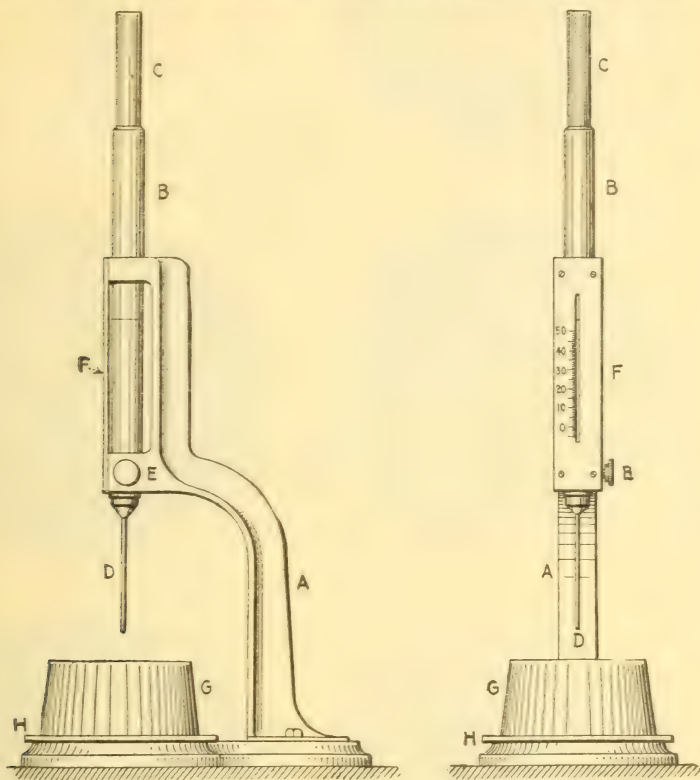


FIG. 1.—Vicat apparatus

face 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. Each trial shall be made with fresh cement. The amount of water required shall be expressed in percentage by weight of the dry cement.

37. 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 same sample shall be as indicated in Table I, the values being in percentage of the combined dry weights of the cement and standard sand.

TABLE 1.—Percentage of water for standard mortars

Percentage of water for neat cement paste of normal consistency	Percentage of water for 1 cement, 3 standard Ottawa sand	Percentage of water for neat cement paste of normal consistency	Percentage of water for 1 cement, 3 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

DETERMINATION OF SOUNDNESS⁵

38. A steam apparatus, which can be maintained at a temperature between 98° and 100° C., or one similar to that shown in Figure 2, is recommended. The capacity of this apparatus may be increased by using a rack for holding the pats in a vertical or inclined position.

39. 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 flat clean glass plates about 4 inches square and stored in moist air for 24 hours. In molding the pat, 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, then flattening the top. The pats used for the time of setting tests by the Gillmore method may be used for soundness tests.

40. 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 five hours.

NOTE.—It is important that the specimens be 24 hours old when placed in steam, since variations in their age will produce differences in the results of the steam tests. Particularly noticeable are the effects of steaming pats too soon, for many specimens steamed when only 10 or 12 hours old give apparently satisfactory results, while failure would be observed if they were not placed in steam until 24 hours old.

41. Should the pat leave the plate, distortion may be detected best with a straightedge applied to the surface which was in contact with the plate.

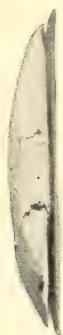
DETERMINATION OF TIME OF SETTING

42. The following are alternate methods, either of which may be used as specified:

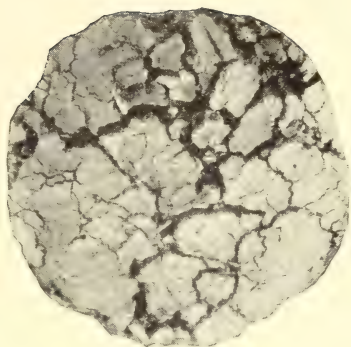
43. The time of setting shall be determined with the Vicat apparatus described in section 35. (See fig. 1.)

44. A paste of normal consistency shall be molded in the ring G as described in section 36 and placed under the rod B, 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 plate 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 should 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

⁵ 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 and are not an indication of unsoundness. These conditions are illustrated in Plate 1. 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.



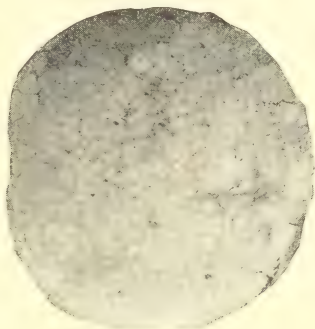
Distortion.



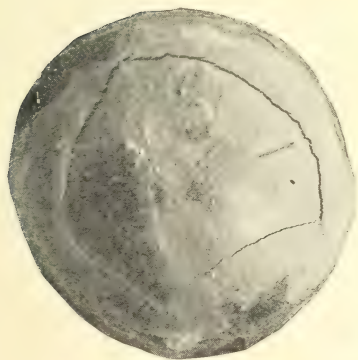
Disintegration.



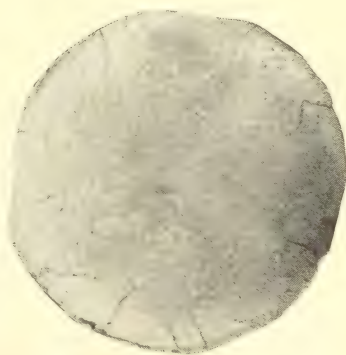
Shrinkage.



Checking.



Shrinkage.



Cracking.

Typical failures in soundness test



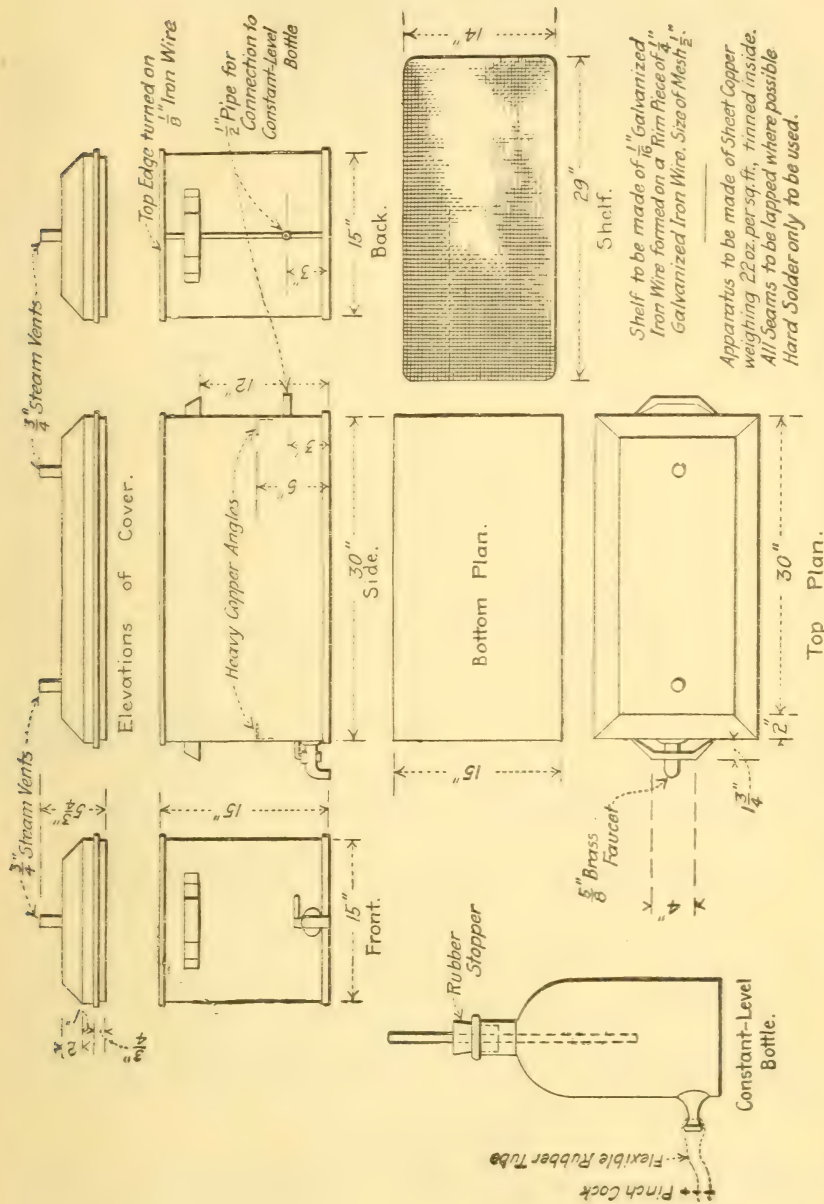


FIG. 2.—Apparatus for making soundness test of cement

water used and the amount of kneading the paste received, but by the temperature and humidity of the air, and its determination is therefore only approximate.

45. The time of setting shall be determined by the Gillmore needles. The Gillmore needles should preferably be mounted as shown in Figure 3 B, and the needle ends shall be maintained in a clean condition and shall be plane and at right angles to the axis of the rod.

46. 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 (fig. 3 A), mixed to a normal consistency, shall be kept in moist air at a tem-

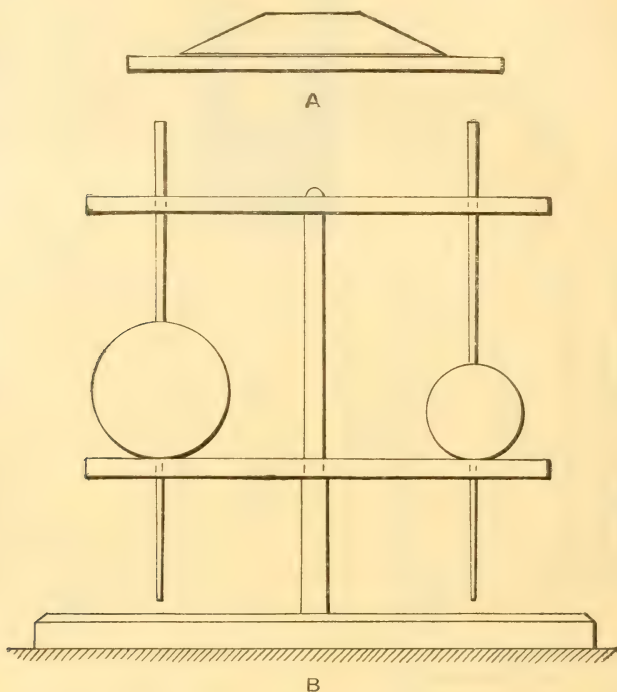


FIG. 3.—Pat and instruments for determining time of set; (A) Pat with top surface flattened for determining time of setting by Gillmore method; (B) Gillmore needles

perature 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-fourth 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.

TENSION TESTS

47. The form of test piece shown in Figure 4 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 when used shall be of the type shown in Figure 5. Molds shall be oiled with a mineral oil.

48. 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., Ottawa, Ill.

49. This sand shall be considered standard when not more than 5 grams are retained on the No. 20 sieve, and not more than 5 grams pass the No. 30 sieve, after one minute continuous sieving of a 100-gram sample, in the manner specified for sieving cement on the No. 200 sieve. (See section 31.)

50. The Nos. 20 and 30 sieves shall conform to the requirements for these sieves as given in the standard specifications for sieves for testing purposes (serial designation E 11) of the American Society for Testing Materials (3).

51. Immediately after mixing the standard mortar in accordance with the methods for mixing cement pastes and mortars the molds shall be filled heaping full without compacting. Then the mortar shall be pressed in firmly with the thumbs, applying pressure 12 times to each briquet, at points to include the entire surface. The pressure shall be such that the simultaneous application

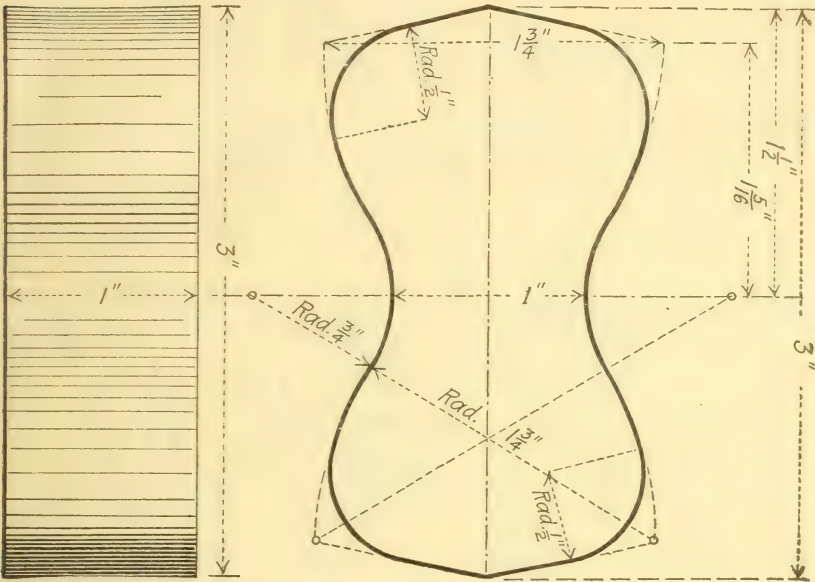


FIG. 4.—Details for briquet

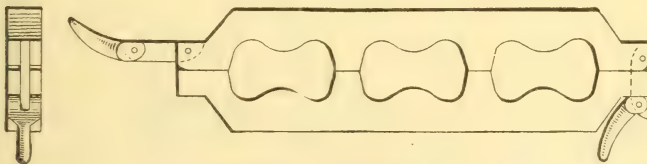


FIG. 5.—Gang mold

of both thumbs will register a pressure of between 15 and 20 pounds. Each application of the thumbs shall be maintained not longer than sufficient to attain the specified pressure. Then the 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 pressure of not more than 4 pounds. The mold shall then be turned over upon a plane plate oiled with mineral oil, and the operation of heaping, thumbing, and smoothing off repeated. No ramming or tamping shall be used, nor any troweling in excess of that required to smooth off the specimen.

52. The briquets shall be tested as soon as they are removed from the water. Tests may be made with any machine meeting the following requirements: The machine shall be capable of weighing the applied load within 0.5 per cent of the nominal value. The sensibility reciprocal (the weight required to be added to the load to move the beam from a horizontal position of equilibrium

to a position of equilibrium at the top of the trig loop) shall not exceed 1 pound at the full capacity of the machine or at any lesser load. The clips for holding the tension test specimens shall be in accordance with Figure 6. The bearing surfaces of the clips and briquets shall be free from sand or dirt,

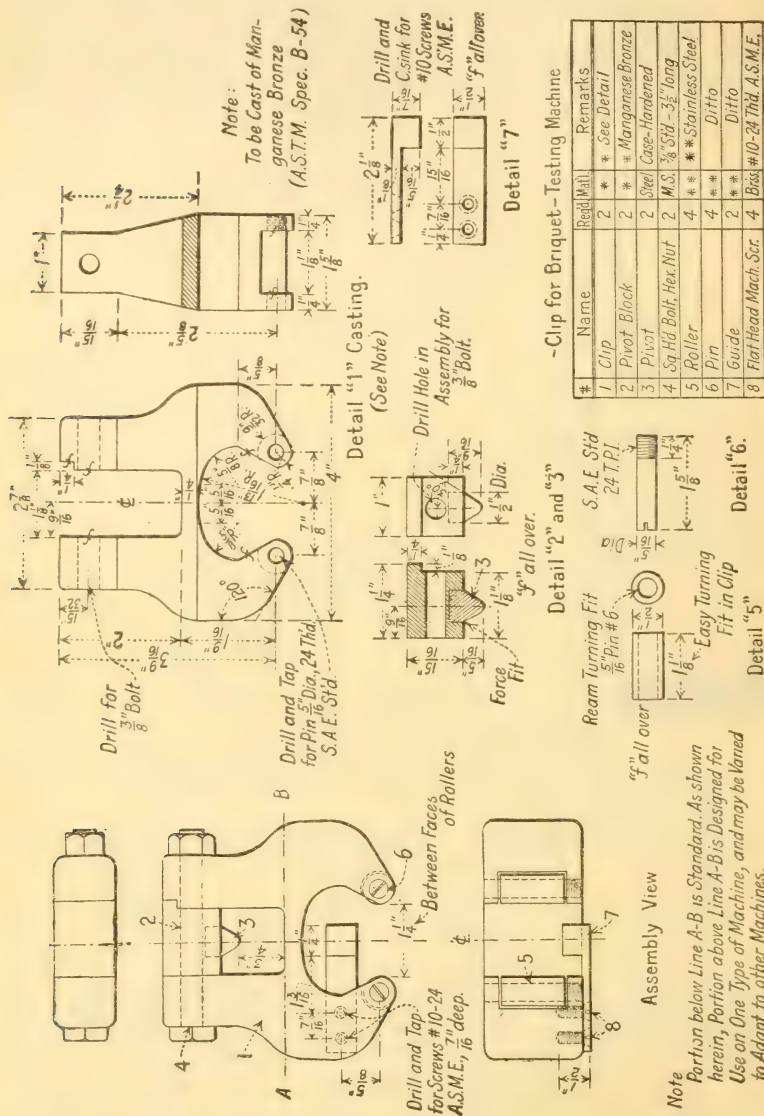


FIG. 6.—Clip for briquet-testing machine

and the roller bearings shall be well oiled and maintained so as to insure freedom of turning. The briquets shall be carefully centered in the clips and the load applied continuously at the rate of 600 pounds per minute.

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

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

STORAGE OF TEST SPECIMENS

55. The moist closet may consist of a soapstone, slate, or concrete box, or a wooden box lined with metal. The interior wall surfaces of all closets shall be covered with felt or broad wicking kept wet. The bottom of the moist closet shall be covered with water to a depth of at least 2 inches.

56. All test specimens, immediately after molding, shall be kept in the molds on plane plates in the moist closet for from 20 to 24 hours in such manner that the upper surfaces shall be exposed to the moist air.

57. The briquets shall then be removed from the molds and immersed in clean water in storage tanks constructed of noncorroding material.

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

(A. S. T. M. standard method (j) serial designation D 75-22, except as indicated. See p. 1)

1. Samples of all materials for test upon which is to be based the 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 a producer or owner of the supply.

SAMPLING OF STONE

A. SAMPLING OF STONE FROM LEDGES OR QUARRIES FOR QUALITY

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

3. (a) For standard stone test, separate samples shall be taken of at least 30 pounds each of fresh unweathered specimens 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 of a minimum size of 4 by 5 by 3 inches, on which the bedding plane is marked, and which shall be free from seams or fractures to be used in the toughness or compression test.

(b) The sample for concrete test will depend on the kind of tests to be made and the number of specimens necessary.

4. In addition to the general information accompanying all samples, samples from local ledges not commercial sources shall contain the following: Name of owner, approximate quantity available (if quantity is very large this can 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

5. Where practical, 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. At the plant, the following factors affect the size of broken stone: Size and shape of screen openings, length of screen sections, the angle of screen with 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. It is recommended, in order that the sample may be representative, that separate samples be taken at different times while the material is being loaded. If the sample has to 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 in a composite sample and the sample for test obtained by the quartering method.

7. (a) Where it is not practical 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 car or boat. These separate samples shall be well mixed in a composite sample and the sample for test obtained by the quartering method. The results of tests on 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) Where test is to be made for size only, it is recommended that a small set of screens and a pair of scales or some unit measure receptacle for measuring volume be used for field testing in order not to delay decision on the use of the material. Occasional check tests can be obtained from the laboratory to assure a fair degree of accuracy in field testing.

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 sample for concrete test will depend on the kind of tests to be made and the number of specimens necessary.

C. SAMPLING OF FIELD STONE AND BOWLERS

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

10. Separate samples shall be taken of all stone of different classes that a visual inspection indicates: from state of preservation and degrees of lamination, would be considered for use in construction.

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

(a) Location of supply.

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.

A fairly accurate estimate of 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 such deposits do not pay to work unless quite congested.

(c) Information regarding the percentage of different classes of stone which were sampled and the percentages of material which can be rejected by visual examination and may have to be handled and spoiled.

This information regarding 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.

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.

SAMPLING OF SAND AND GRAVEL

A. 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.

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) Where 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 material that visual inspection indicates could be used. Care shall be taken to eliminate any overburden or stripping at the top or any 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 parallel to the face to determine the extent of the supply. The number and depth of these pits depend on 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 test pits. These shall be well mixed in a composite sample and the sample for test 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 will depend on 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 no radical difference in size of grain, color, etc., these shall be well mixed in a composite sample and the sample for test obtained by the quartering method. Where visual inspection indicates a distinct difference in material from different pits, separate samples shall be taken for test.

(e) It is suggested that the colorimetric test be used for determining the percentage of organic material.⁶

15. In addition to the general information accompanying all samples from sand and gravel deposits not commercial sources, the same detailed information shall be supplied as with samples of stone taken from local lodges.

B. COMMERCIAL SAND AND GRAVEL PLANTS

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, or in some other satisfactory manner. 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 in a composite sample and the sample for test 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 practical 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 car or boat. These separate samples 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

19. Samples of run of bank (where the sand and gravel are combined) shall consist of at least 100 pounds of material where the gravel content is 50 per cent or more of the whole. If the gravel is less in percentage, the sample shall be increased in proportion. For example, where the gravel percentage is 25 per cent of the whole the sample should contain 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 sample for concrete test will depend on the kind of tests to be made and the number of specimens necessary.

⁶ For a description of this test see page 28.

SAMPLING OF MISCELLANEOUS MATERIALS

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

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 blocks that would be rejected by visual inspection.

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

GENERAL DIRECTIONS FOR SHIPPING AND MARKING SAMPLES

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

27. Samples of stone block shall be securely crated.

28. 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.

29. 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 for 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.

3. METHOD OF TEST FOR ABRASION OF BROKEN STONE AND BROKEN SLAG

(Based on A. S. T. M. standard method (4), serial designation D 2-26)

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 shall be mounted on a shaft at an angle of 30° with the axis of rotation of the shaft.

2. The rock to be tested shall be broken in the laboratory from pieces at least $3\frac{1}{2}$ inches in size 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 a rate of between 30 and 33 per minute, shall constitute a test. Only the percentage of material worn off which will pass through an American Society for Testing Materials standard No. 12 sieve shall be considered in determining the amount of wear. This shall be expressed as a percentage of the 5 kilograms used in the test.

NOTE.—In the case of broken stone, samples should, wherever possible, be selected from the quarry face.

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 percentage of wear.

5. The operator is cautioned that drying some varieties of stone at high temperatures has a deleterious effect upon the abrasion loss, and therefore care

should be taken that the temperature be low enough to prevent injury to the stone through heat.

4. METHOD OF TEST FOR ABRASION OF GRAVEL

A. TEST FOR GRAVEL CONTAINING NO CRUSHED PIECES

1. The sample shall consist entirely of uncrushed fragments of gravel, and the test shall be made using one of the four gradings (A, B, C, or D) given in paragraph 3. The grading most nearly representing that of the material furnished for the work shall be selected for the test.
2. The aggregate shall first be screened into the different sizes required for the test, and the material of these sizes shall be washed and dried.
3. The sample shall consist of 5,000 grams of the dry gravel, with the different sizes combined to conform to one of the following four gradings:

TABLE 2.—*Gradings of gravel*

Grading	Size of screens (circular openings)		Percentage
	Passing	Retained on	
	Inch	Inch	
A	$\frac{3}{4}$	$\frac{1}{2}$	25
	1	$\frac{3}{4}$	25
	$1\frac{1}{2}$	1	25
B	2	$1\frac{1}{2}$	25
	$\frac{3}{4}$	$\frac{1}{2}$	25
	1	$\frac{3}{4}$	50
C	$1\frac{1}{2}$	1	25
	$\frac{3}{4}$	$\frac{1}{2}$	50
	1	$\frac{3}{4}$	50
D	$\frac{3}{4}$	$\frac{1}{2}$	50
	$1\frac{1}{2}$	$\frac{1}{4}$	50
	2	$\frac{1}{2}$	50

4. The sample for the test shall be placed in the cast-iron cylinder of the Deval abrasion-testing machine as specified for the standard abrasion test for 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.⁷

5. 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 No. 12 sieve, conforming to the requirements of the standard specifications for sieves for testing purposes of the American Society for Testing Materials (serial designation E 11-26 (3)). The material retained upon the sieve shall be washed, dried, and weighed. The difference between this weight and the weight of the original sample, expressed as a percentage of the original weight, shall be considered as the loss by abrasion.

6. When the gravel has a specific gravity below 2.20, a sample of 4,000 grams, instead of 5,000 grams, shall be used for the test. The testing procedure shall be the same in all other respects.

7. When the gravel, as used in the work, contains as much as 25 per cent of material finer than one-half inch, but is of such size that either grading A, B, or C would be used for the abrasion test, a second abrasion test shall be made, using grading D, if, in the opinion of the engineer, the particles finer than one-half inch are not at least equal in hardness to those coarser than one-half inch.

⁷ These spheres are the same as those used in the standard rattler test for paving brick.

B. TEST FOR GRAVEL CONTAINING CRUSHED PIECES³

8. Gravel containing more than about 10 per cent of crushed pieces shall, for the purpose of this test, be considered as crushed gravel. In such cases the abrasion test shall be made on a representative sample of the whole, including the crushed pieces, following the procedure described in paragraphs 1 to 7. The percentage, by weight, of crushed pieces shall be determined and the permissible percentage of wear which shall govern for any given sample shall be calculated from the following formula:

$$W = \frac{AL + (100 - A)L'}{100}$$

in which

- A = percentage of uncrushed pieces,
 $100 - A$ = percentage of crushed pieces,
 L = maximum percentage of wear permitted by the specifications for gravel containing no crushed pieces,
 L' = maximum percentage of wear permitted by the specifications for gravel consisting entirely of crushed pieces,
 W = permissible percentage of wear.

5. METHOD OF TEST FOR TOUGHNESS OF ROCK

(A. S. T. M. standard method (4), serial designation D 3-18)

1. Toughness, as applied to rock, is the resistance offered to fracture under impact, expressed as the final height of blow required of a standard hammer to cause fracture of a cylindrical test specimen of given dimensions.

2. 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, throughout the test, be tangent to the spherical end of the plunger at its lowest point.

3. Quarry samples of rock from which test specimens are to be prepared shall measure at least six 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 should 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 should 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.

4. Specimens for test shall be cylinders prepared as described in section 5, 25 millimeters in height and from 24 to 25 millimeters in diameter. Three test specimens shall constitute a test set. The ends of the specimens shall be plane surfaces at right angles to the axis of the cylinder.

5. 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

³ For the purpose of this test, a crushed gravel fragment may be considered as a fragment of gravel having at least one fractured face.

subject the material to undue stresses and which will insure the specified dimensions. The ends of the cylinders may be sawed by means of a band or diamond saw,¹⁰ 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.

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 an increase of 1-centimeter fall for each succeeding blow until failure of the test specimen occurs.

7. The height of the blow in centimeters at failure shall be 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. In cases where 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.

6. METHOD OF TEST FOR TOUGHNESS OF GRAVEL

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 (11).

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
$\frac{3}{4}$	2	3
1	3	4
$1\frac{1}{4}$	4	5
$1\frac{1}{2}$	5	6

Failure in this test shall be construed as the actual crushing or splitting of a fragment under a single blow of the hammer.

7. METHOD OF DETERMINING QUALITY OF GLACIAL GRAVELS BY LITHOLOGICAL COUNT OF PARTICLES

From 100 to 200 pieces of gravel stones, selected at random, ranging in size from one-half inch to two 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.

⁹ The form of diamond drill described in Department Bulletin No. 347, U. S. Department of Agriculture (10, p. 6-7), is recommended, and should prove satisfactory if the instructions are strictly followed.

¹⁰ A satisfactory form of diamond saw is described in Department Bulletin No. 347, U. S. Department of Agriculture (10, p. 7-9).

8. METHOD OF TEST FOR QUANTITY OF SOFT PEBBLES IN GRAVEL¹¹

1. The method consists in determining the resistance of the pebbles to uniform static pressure.

2. A sample of at least 1,000 grams shall be separated into three sizes and subjected to uniform pressure without impact, as follows:

	Pounds
0.185 inch to 0.375 inch.....	35
0.375 inch to 0.750 inch.....	60
0.750 inch to maximum size.....	75

3. Pebbles crushing under these loads shall be classed as soft pebbles, and shall be reported as a percentage of the original sample by weight.

4. Disintegrated rocks or other rocks which will obviously break up into fine material during the screening or mixing operation, shall not be considered in this test.

9. METHOD OF TEST FOR SOUNDNESS OF COARSE AGGREGATE

(Sodium-sulphate soundness test)

1. 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 — times. The operation of immersing, heating, reimmersing, etc., shall be continuous. Note the condition of the rock as to soundness at the end of the test.

2. Samples which exhibit marked disintegration shall be considered to have failed in this test.¹²

10. METHOD OF TEST FOR PERCENTAGE OF SHALE IN AGGREGATE

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.

NOTE.—Attention is called to the fact that this method of test is not applicable when the specific gravity of the shale exceeds the specific gravity of the solution. In cases where a solution of very high density is required, a mixture of bromoform and benzol may be employed.

¹¹A satisfactory instrument for classifying soft stone by this method was devised by the late W. H. Douglass of the Iowa Highway Commission. A description of this apparatus follows:

The Douglass stone meter consists of a jack acting through the specimen to compress a calibrated spring. The jack, which is of the rack and pinion type, is located in the base of the machine and is actuated by means of a hand lever attached to the shaft of the pinion. To the base are attached two vertical standards which are attached to each other by two cross members, one at the top and one near the center. The coil compression spring is located between these two cross members. The top of the spring bears against the top cross bar and the bottom against a collar on the movable plunger which passes through both cross members and the spring. To the lower end of the plunger is screwed a mechanic's jack which allows adjustment for the various sizes of stones and also carries a ball and socket head. The top end of the plunger is graduated to read the desired pressures as it passes through the top cross member.

¹²A correct interpretation of the results of the sodium-sulphate soundness test will be greatly assisted by a visual examination of the ledge from which the sample was obtained. Failure in the test, when accompanied by evidence of weathering of the exposed surface, may be considered sufficient ground for rejection.

11. METHOD OF TEST FOR QUANTITY OF CLAY AND SILT IN GRAVEL [FOR HIGHWAY CONSTRUCTION]

(A. S. T. M. standard method (4), serial designation D 72-21, except as indicated. See p. 1)

1. This test covers 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.)

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 gravel. This is repeated until the wash water is clear. The washed material 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 gravel determined.

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:

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

12. METHOD OF DECANTATION TEST FOR SAND AND OTHER FINE AGGREGATES

(A. S. T. M. tentative standard method (5), serial designation D 136-22 T)

1. This method of test covers the determination of the total quantity of silt, loam, clay, etc., in sand and other fine aggregates.¹³

2. The pan or vessel to be used in the determination shall be approximately 9 inches (230 millimeters) in diameter and not less than 4 inches (102 millimeters) in depth.

3. The sample must contain sufficient moisture to prevent segregation and shall be thoroughly mixed. A representative portion of the sample sufficient to yield approximately 500 grams of dried material shall then be dried to a constant weight at a temperature not exceeding 110° C. (230° F.).

4. The dried material shall be placed in the pan and sufficient water added to cover the sample (about 225 cubic centimeters). The contents of the pan shall be agitated vigorously for 15 seconds, and then be allowed to settle for 15 seconds, after which the water shall be poured off, care being taken not to pour off any sand. This operation shall be repeated until the wash water is clear. As a precaution, the wash water shall be poured through a No. 200 sieve and any material retained thereon returned to the washed sample. The washed sand shall be dried to a constant weight at a temperature not exceeding 110° C. (230° F.) and weighed.

5. The results shall be calculated from the formula:

$$\text{Percentage of silt, clay, loam, etc.} = \frac{\text{Original dry weight} - \text{weight after washing}}{\text{Original dry weight}} \times 100$$

¹³ This determination of the percentage of silt, clay, loam, etc., will include all water-soluble material present, the percentage of which may be determined separately if desired.

6. When check determinations are desired, the wash water shall be evaporated to dryness, the residue weighed, and the percentage calculated from the formula:

$$\text{Percentage of silt, loam, clay, etc.} = \frac{\text{Weight of residue}}{\text{Original dry weight}} \times 100$$

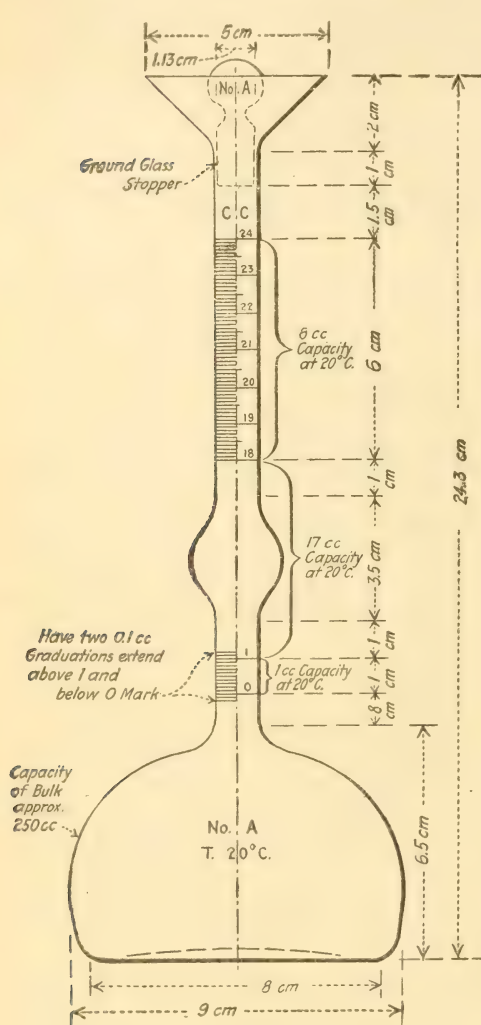


Fig. 7.—Le Chatelier apparatus

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

(A. S. T. M. standard method (4), serial designation D 55-25, except as indicated. (See p. 1)

LE CHATELIER TEST

1. The determination of specific gravity shall be made with a standardized Le Chatelier apparatus which conforms to the requirements illustrated in Figure 7. This apparatus is standardized by the United States Bureau of Standards. Kerosene or water may be used in making this determination.

2. (a) The flask shall be filled with either of these liquids to a point on the stem between zero and 1 cubic centimeter and a reading taken. From 50 to 64 grams (depending upon its approximate specific gravity and to bring the reading on the scale) 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 in the graduated stem shall be read, and the difference between readings is the volume displaced by the known weight of the material.

(b) The specific gravity shall then be calculated from the formula:

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

(c) 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, which shall not exceed 0.5° C. The results of repeated tests should agree within 0.01.

NOTE.—This method is not accurate when sand containing a relatively large proportion of absorptive grains is tested.

14. METHOD OF TEST FOR APPARENT SPECIFIC GRAVITY AND ABSORPTION OF STONE AND 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 $1\frac{1}{4}$ 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 $2\frac{1}{2}$ 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.

15. METHOD OF TEST FOR APPARENT SPECIFIC GRAVITY AND ABSORPTION OR FREE MOISTURE CONTENT OF SAND, GRAVEL, STONE, OR OTHER NONBITUMINOUS HIGHWAY MATERIALS

PYCNOMETER METHOD

1. This method is designed primarily for the purpose of determining the apparent specific gravity of saturated surface-dry materials and the absorption or the free moisture content of materials as used in the field. While this method has been adapted for field use, it is suggested as a convenient method for making these determinations in the laboratory.

2. The special apparatus used consists of a pycnometer which shall conform to the following conditions.

(a) The opening at the top shall be approximately three-eighths inch in diameter.

(b) The shape of the apparatus shall be such as will facilitate the escape of air bubbles.

(c) It shall have an opening for the introduction of samples which will admit particles as large as 2 inches.

(d) It shall be capable of being cleaned quickly and easily.

APPARENT SPECIFIC GRAVITY

3. (a) Coarse material.—The apparent specific gravity of coarse material shall be determined upon material that has been immersed in water at 70° F. for 24 hours. The pieces shall be individually surface dried with a towel. Two thousand grams of this surface-dried material shall be weighed out to the nearest 0.5 gram. This sample shall be placed in the pycnometer and the pycnometer partly filled with water at 70° F. The pycnometer shall be gently shaken and rolled to remove entrained air. The pycnometer shall then be exactly filled with water and its outside surface dried. The pycnometer and its contents shall then be weighed to the nearest 0.5 gram. The apparent specific gravity shall be computed from the formula

$$\text{Specific gravity} = \frac{S}{P + S - W}$$

where

S = the weight of the sample in grams,

P = the weight in grams of the pycnometer full of water,

W = the weight in grams of the pycnometer plus the sample plus sufficient water to fill the pycnometer.

(b) Fine material.—The apparent specific gravity of fine material shall be determined in the same way as for coarse material except that the sample shall consist of air-dried material and shall consist of 1,000 grams.

ABSORPTION OR FREE WATER CONTENT

4. The absorption or the free water content of material shall be determined upon a sample consisting of material in the condition in which it is to be used.

5. Two thousand grams of the sample shall be weighed out to the nearest 0.5 gram. This sample shall be placed in the pycnometer and the pycnometer partly filled with water at 70° F. The pycnometer shall be gently shaken and rolled to remove entrained air. The pycnometer shall then be exactly filled with water and its outside surface dried. The pycnometer and its contents shall then be weighed to the nearest 0.5 gram.

6. The absorption or the free moisture content of the sample shall be computed from the following formula:

$$\text{Percentage of moisture or absorption}^{14} = \frac{100}{S} (W - W_1) \frac{G_s}{G_s - 1}$$

Where S = the weight of the sample in grams,

$$W = P + S - \frac{S}{G_s}$$

P = the weight in grams of the pycnometer filled with water,

W_1 = the weight of the pycnometer containing the sample and sufficient water to fill the remaining space,

G_s = the specific gravity of the saturated surface dry material.

7. A pycnometer that has been used successfully by the Iowa Highway Commission consists of an ordinary glass Mason fruit jar with a special cap. This cap is the frustum of a cone superimposed upon a ring which will screw upon the top of the jar. This cap may be made from the screw ring from a Kerr fruit-jar cap to which is soldered a tin cone with a slope of 60° and a three-eighths-inch hole at the top. A more satisfactory cap could be cast from brass or some other metal that does not corrode. In using a pycnometer of this kind the cap and the jar should be marked so that the cap will be screwed on to the same position each time so that the volume of the apparatus will not be varied.

16. METHOD OF TEST FOR FIELD DETERMINATION OF APPROXIMATE APPARENT SPECIFIC GRAVITY OF FINE AGGREGATE

(A. S. T. M. tentative standard method, (5) serial designation C 68-27 T)

1. This method of test is intended for use in the field for making approximate determinations of the apparent specific gravity of fine aggregate.

2. The apparatus shall consist of the following:

(a) Balance.—A balance, preferably of the torsion type, having a capacity of 2 kilograms or more and sensitive to 0.5 gram or less.

(b) Flask.—A special graduated flask of the type, and conforming to the dimensions, shown in Figure 8.

3. A 1-kilogram sample shall be selected which shall be as truly representative of the fine aggregate as possible. It shall be spread out on a flat surface and air dried until the surface moisture has evaporated and the sample is free flowing. The sample shall then be thoroughly mixed and 500 grams accurately weighed out.

4. The graduated flask shall be filled to the 200-cubic centimeter mark on the lower neck with water at room temperature. The 500-gram sample of fine aggregate shall then be slowly poured into the flask, and the flask and contents agitated while introducing the aggregate to free any entrained air bubbles. The combined volume in cubic centimeters of the water and fine aggregate shall be read on the scale on the upper neck of the flask.

¹⁴ If the algebraic sign of the result is positive, the material contains free water. If the sign is negative, the material absorbs water.

5. The approximate apparent specific gravity of the fine aggregate may be calculated from the formula:

Approximate apparent specific gravity = $\frac{500}{V-200}$ where V = the combined volume in cubic centimeters of the water and fine aggregate in the flask.

6. Duplicate determinations should check to within 0.05. Variations in the amount of water evaporated from the moist sample as described in section 3 beyond the point at which the surface moisture has disappeared and the sample is free flowing and apparently dry will cause variations in results of from 0.5 to 1 per cent depending upon the absorption of the aggregate.

17. METHOD OF TEST FOR FIELD DETERMINATION OF APPROXIMATE PERCENTAGE OF VOIDS IN FINE AGGREGATE

(A. S. T. M. tentative standard method (5), serial designation C 69-27 T)

1. This method of test is intended for use in the field for making approximate determinations of the percentage of voids in fine aggregate when inundated with water.

2. The apparatus shall consist of the following:

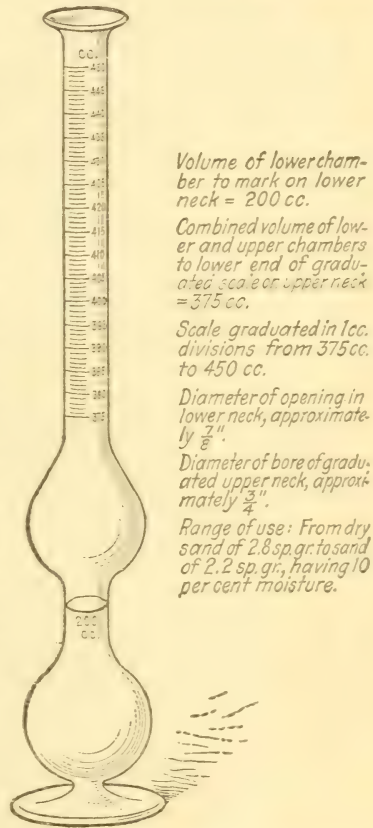
(a) Balance.—A balance, preferably of the torsion type, having a capacity of 2 kilograms or more and sensitive to 0.5 gram or less.

(b) Flask.—A special graduated flask of the type and conforming to the dimensions shown in Figure 8.

3. A sample weighing about 2 kilograms shall be selected which shall be as truly representative of the fine aggregate as possible. It shall be well mixed and spread out on a flat surface and air dried until the surface moisture has evaporated and the sample is free flowing.

4. About 100 cubic centimeters of water shall be placed in the flask. Aggregate from the room-dry sample shall then be introduced slowly into the flask until the flask is filled to the 400-cubic centimeter mark on the upper neck. Water shall be added as required to keep the aggregate inundated as the filling proceeds. When the flask is filled to the 400-cubic centimeter mark, both water and aggregate shall be at the same level. The aggregate in the flask shall not be agitated during or after the filling operation. The flask and contents shall be weighed to the nearest gram, and the weight of the flask shall be deducted, unless its weight has been counterbalanced on the scale at the time of weighing.

5. (a) This method requires that the apparent specific gravity be known of the fine aggregate in a room-dry, free-flowing condition, but with the voids within the particles of the aggregate containing moisture. If the apparent specific gravity of the aggregate in this condition is not known, it shall be determined in accordance with the tentative method of test for field determination of approximate apparent specific gravity of fine aggregate (serial designation C 68-27 T) of the American Society for Testing Materials (5).



Volume of lower chamber to mark on lower neck = 200 cc.

Combined volume of lower and upper chambers to lower end of graduated scale or upper neck = 375 cc.

Scale graduated in 1cc. divisions from 375 cc. to 450 cc.

Diameter of opening in lower neck, approximately $\frac{7}{8}$ ".

Diameter of bore of graduated upper neck, approximately $\frac{3}{4}$ ".

Range of use: From dry sand of 2.8 sp. gr. to sand of 2.2 sp. gr., having 10 per cent moisture.

FIG. 8.—Graduated flask for field testing of fine aggregate

(b) The approximate percentage of voids in the inundated fine aggregate may be calculated from the formula:

$$V = \frac{400 \times \text{specific gravity} - W}{400 (\text{specific gravity} - 1)} \times 100$$

where

V = the approximate percentage of voids,
 Specific gravity = apparent specific gravity of the fine aggregate,
 W = combined weight in grams of 400 cubic centimeters of water and fine aggregate in the flask.

6. Duplicate determinations should check to within 1 per cent. Variations in the composition of the rock grains of the fine aggregate which result in variations of 0.05 in the approximate apparent specific gravity of the aggregate will cause an inaccuracy in results equal to from 2 to 3 per cent of voids.

18. METHOD OF TEST FOR FIELD DETERMINATION OF SURFACE MOISTURE IN FINE AGGREGATE

(A. S. T. M. tentative standard method (5), serial designation C 70-27 T)

1. This method of test is intended for use in the field for making approximate determinations of the percentage of surface moisture in fine aggregate for use in computing the allowance to be made for water in the fine aggregate when proportioning concrete by the maximum water content method.

This method determines only surface moisture; that is, moisture on the outside of the particles. The moisture absorbed within the particles does not add to the volume of the particles and, therefore, does not make itself evident in this test.

2. The apparatus shall consist of the following:

(a) Balance.—A balance, preferably of the torsion type, having a capacity of 2 kilograms or more and sensitive to 0.5 gram or less.

(b) Flask.—A special graduated flask of the type, and conforming to the dimensions, shown in Figure 8.

3. A sample weighing about 1 kilogram shall be selected which shall be as truly representative of the fine aggregate as possible. It shall be well mixed and spread out on a flat nonabsorbent surface. Five hundred grams shall be immediately weighed out, permitting moisture to evaporate as little as possible from the sample.

4. The graduated flask shall be filled to the 200-cubic centimeter mark on the lower neck with water at room temperature. The 500-gram sample of damp aggregate shall then be slowly poured into the flask, and the contents of the flask agitated or stirred to free any entrained air bubbles. The combined volume, in cubic centimeters, of the water and fine aggregate shall be read on the scale on the upper neck of the flask.

5. (a) This method requires that the apparent specific gravity be known of the fine aggregate in a room-dry, free-flowing condition, but with the voids within the particles of the aggregate containing moisture. If the apparent specific gravity of the aggregate in this condition is not known it shall be determined in accordance with the tentative method of test for field determination of approximate apparent specific gravity of fine aggregate (serial designation C 68-27 T) of the American Society for Testing Materials (5).

(b) The percentage of surface moisture in the fine aggregate (air-dry basis) may be calculated from the formula:

$$\text{Percentage of surface moisture} = \frac{V - \frac{500}{\text{specific gravity}} - 200}{200 + 500 - V} \times 100$$

where

V = the combined volume in cubic centimeters of the water and fine aggregate in the flask,

Specific gravity = approximate apparent specific gravity of the fine aggregate.

6. Duplicate determinations should check to within 0.5 per cent. Variations in the composition of the rock grains of the fine aggregate which result in variations of 0.05 in the approximate apparent specific gravity of the aggregate will cause inaccuracy in results equal to from 1 to 1.5 per cent of moisture.

19. METHOD OF TEST FOR UNIT WEIGHT OF AGGREGATE FOR CONCRETE

(A. S. T. M. method (4), serial designation C 29-27)

1. This method is intended to cover the determination of unit weight of fine, coarse, or mixed aggregate for concrete.

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.

(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 of 0.1, 0.5, 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 (cubic foot)	Inside diameter	Inside height	Minimum thickness of metal, U. S. gauge	Diameter of largest particles of aggregate
	Inches	Inches	No.	Inches
0.1	6.00	6.10	11	Under 1/2.
0.5	10.00	11.00	8	Under 1 1/2.
1	14.00	11.23	5	Over 1 1/2.

(c) Tamping rod.—The tamping rod shall be a straight metal rod 5/8 inch in diameter and 24 inches in length, 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.) (62.355 pounds per cubic foot) by the weight of water at 16.7° C. (62° F.) required to fill the measure.

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 puddled with the tamping rod with 25 strokes, evenly distributed over the surface. The measure shall be filled two-thirds full and again puddled with 25 strokes as before. The measure shall then be filled to overflowing, puddled 25 times, and the surplus aggregate struck off, using the tamping rod as a straightedge.

In puddling the first layer the rod should not be permitted to forcibly strike the bottom of the measure. In puddling 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.

(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.

20. METHOD OF TEST FOR DETERMINATION OF VOIDS IN FINE AGGREGATE FOR CONCRETE

(A. S. T. M. standard method (4), serial designation C 30-22)

1. The voids in fine aggregate for cement concrete shall be determined by the formula:

$$\text{Percentage of voids} = \frac{(\text{Sp. gr.} \times 62.355) - \text{wt.}}{\text{Sp. gr.} \times 62.355} \times 100$$

2. The symbol "sp. gr." represents the apparent specific gravity of the fine aggregate as determined by the standard method of test for apparent specific gravity of sand, stone, and slag screenings, and other fine nonbituminous high-

way materials (serial designation D 55) of the American Society for Testing Materials (see p. 22) (4).

3. The quantity 62.355 is the weight in pounds of 1 cubic foot of water at the standard temperature of 16.7° C. (62° F.).

4. The symbol "wt." represents the weight in pounds per cubic foot of the fine aggregate as determined by the standard method of test for unit weight of aggregate for concrete (serial designation C 29, see p. 27) of the American Society for Testing Materials (4).

21. METHOD OF TEST FOR ORGANIC IMPURITIES IN SANDS FOR CONCRETE

(A. S. T. M. standard method (4), serial designation C 40-27, except as indicated. See p. 1)

1. This method of test is an approximate method of determining the presence of injurious organic compounds in natural sands which are to be used in cement mortar or concrete. The principal value of the test is to furnish a warning that further tests of the sands are necessary before they are approved for use.

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

3. (a) A 12-ounce graduated clear glass bottle shall be filled to the 4½-ounce mark with the sand to be tested.

(b) A 3 per cent solution of sodium hydroxide in water shall be added until the volume of the sand and liquid indicated after shaking is 7 liquid ounces.

(c) The bottle shall be stoppered, shaken vigorously, and then allowed to stand for 24 hours.

4. 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 97.5 cubic centimeters of a 3 per cent sodium hydroxide solution. This shall be placed in a 12-ounce bottle, stoppered, shaken vigorously, and allowed to stand for 24 hours.

5. (a) After standing 24 hours, the color of the clear liquid above the sand shall be compared with the color of the standard color solution prepared at the same time and in accordance with the method described in section 4, or with a glass of color similar to the color of the standard solution.

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

6. *In lieu of the method given in paragraph 5 (a) above, the color of the clear liquid above the sand may be compared with the colors given in Plate 2.*

NOTE.—Where chemically pure sodium hydroxide is not available, ordinarily available commercial soda lye may be used.

22. METHODS OF MAKING COMPRESSION TESTS OF CONCRETE

(A. S. T. M. standard methods (4), serial designation C 39-27)

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 compression specimens, but may be used with slight modification in making tests of concrete for wearing resistance, bond between concrete and steel, impermeability, etc. The investigation of machine-mixed concrete will require certain obvious changes in the methods. For methods of conducting compression tests of concrete specimens made during the progress of construction work, see the standard method of making and storing specimens of concrete in the field (serial designation C 31, see p. 31) of the American Society for Testing Materials (4).

2. Materials shall be brought to room temperature (18° to 24° C., 65° to 75° F.) before beginning the 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



FIG. 1

Sands suitable for use in high-grade concrete.



FIG. 2

Sands which may be used in unimportant concrete work.

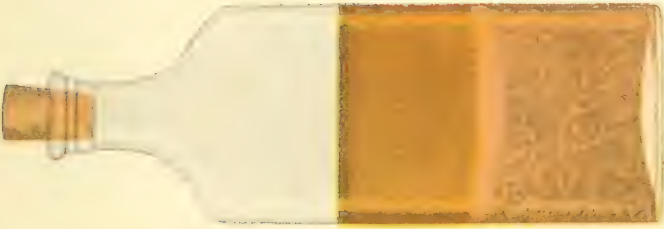


FIG. 3

Sands which should never be used in concrete.



FIG. 4

An unusually bad sand, soil or loam.

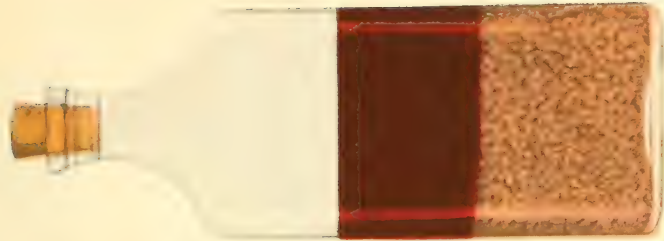


FIG. 5

CATEGORIES OF TREATED SANDS WITH SUGGESTED RANGES OF APPLICATION.



shall be sieved through a No. 16 (1,190-micron) sieve and all lumps rejected. Aggregates shall be in a room-dry condition when used in concrete tests. In general, aggregates should be separated on the No. 4 (4,760-micron), $\frac{3}{8}$ -inch and $1\frac{1}{2}$ -inch sieves and recombined to the average original sieve analysis for each batch. Fine aggregate should be separated into different sizes also in cases where unusual gradings are being studied.

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 tested in accordance with the methods described in the standard specifications and tests for Portland cement (serial designation C 9, see p. 3) of the American Society for Testing Materials (4).

5. Fine aggregates (passing through a No. 4 (4,760-micron) sieve) shall be subjected, when required, to the following tests of the American Society for Testing Materials:

(a) Sieve-analysis test made in accordance with the standard method of test for sieve analysis of aggregates for concrete (serial designation C 41, see p. 36 (4)).

(b) Test for organic impurities made in accordance with the standard method of test for organic impurities in sands for concrete (serial designation C 40, see p. 28 (4)).

(c) Test for quantity of silt, clay, and dust made in accordance with the tentative method of decantation test for sand and other fine aggregates (serial designation D 136-22 T, see p. 21 (4)).

(d) Test for unit weight made in accordance with the standard method of test for unit weight of aggregate for concrete (serial designation C 29, see p. 27 (4)).

(e) Strength test of 1:3 mortar by weight at 7 and 28 days in comparison with standard sand mortar in accordance with standard specifications and tests for Portland cement (serial designation C 9, see p. 3 (4)).

6. Coarse aggregates (retained on a No. 4 (4,760-micron) sieve) shall be subjected when required to the following tests:

(a) Sieve analysis test as specified in section 5 (a).

(b) Test for quantity of silt, clay, and dust made in accordance with standard method of test for quantity of clay and silt in gravel for highway construction (serial designation D 72, see p. 21) of the American Society for Testing Materials (4).

(c) Test for unit weight as specified in 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 of fine aggregate, and — volumes of coarse aggregate, or (c) 1 part by weight of cement, — parts by weight of fine aggregate, and — parts by weight of coarse aggregate.¹⁵

9. Compression tests of concrete shall be made on cylinders of 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 a single test specimen. The batch

¹⁵ It is impracticable to give a general method for proportioning concrete for experimental purposes; the details will necessarily vary widely with the purpose for which the tests are made. The following procedure is suggested for specific cases: (a) Vary the cement content by 10 per cent intervals above and below assumed quantity. (b) Vary the proportions of fine to coarse aggregate, measured separately, at intervals of 10 per cent. (c) Vary the quantity of mixing water by intervals of 10 per cent.

shall preferably be mixed in a shallow metal pan with a 10-inch bricklayer's trowel which has been blunted by cutting off about $2\frac{1}{2}$ inches of the point, 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 mass shall be mixed thoroughly until the resulting concrete 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:

(a) Slump test made in accordance with the tentative method of test for consistency of Portland cement concrete (serial designation D 138-26 T) of the American Society for Testing Materials (5).

(b) Flow test made by placing a metal mold in the shape of a frustrum of a cone $6\frac{3}{4}$ inches in top diameter, 10 inches in bottom diameter, 5 inches in depth, on the table of the flow apparatus.¹⁶ The fresh concrete shall be placed in the mold in two layers. Each layer shall be puddled as described in section 13. Immediately after molding the surplus concrete shall be struck off with a trowel and the mold shall be removed by a steady upward pull; the table raised one-half inch and dropped 15 times in about 10 seconds by means of a suitable cam and crank. The spread of the fresh concrete due to this treatment expressed as a percentage of the original bottom diameter of the cone is the "flow."

12. The molds shall preferably be of metal. Each mold 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 molds so that the mixing water will not escape during molding.¹⁷

13. Concrete test specimens shall be molded by placing the fresh concrete in the mold in layers 3 to 4 inches in thickness. Each layer shall be puddled with 25 strokes with a $\frac{5}{8}$ -inch rod 24 inches in length, bullet-pointed at the lower end. After the top layer has been puddled the surplus concrete shall be struck off with a trowel and the mold covered with a piece of plate glass at least one-fourth inch in thickness or a machined metal plate, which will be used later in capping the test specimen.

14. 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 shall be formed by means of a piece of plate glass one-fourth inch in thickness or a machined metal plate one-half inch in thickness and of a diameter 2 or 3 inches larger than that of the mold. The plate shall be worked on the fresh cement paste until it rests on top of the mold. The cement for capping should be mixed to a stiff paste two to four hours 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 may be avoided by oiling the plates or by inserting a sheet of paraffined paper.

15. Concrete test specimens shall be removed from the molds 20 to 48 hours after forming, 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 18° to 24° C. (65° 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 specimens should be made on different days in investigations in which accurate comparisons are desired.

18. Compression tests shall be made immediately upon removal of the concrete test specimens from the curing room; that is, the test specimens shall be loaded in a damp condition. The length and average diameter of the test

¹⁶ For a description and illustration of one design for a flow table, see: (17; 18, p. 242).

¹⁷ The best type of mold consists of lengths of cold-drawn steel tubing, split along one element and closed by means of a circumferential band and bolt. Satisfactory molds can be made from 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.

specimen shall be measured in inches and hundredths; two diameters shall be measured at right angles near the midlength. The test specimen shall be weighed immediately before testing.

19. In general, only the ultimate compressive 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 specimen; cushioning materials shall not be used. An adjustable bearing block shall be used to transmit the load to the test specimen. The bearing block shall be used on top of the specimen in vertical testing machines. The diameter of the bearing block shall be the same or slightly larger than that of the test specimen. The upper or lower 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 specimen.

20. The load shall be applied uniformly and without shock. The moving head of the testing machine should travel at the rate of about 0.05 inch per minute when the machine is running idle.

21. The total load indicated by the testing machine at failure of the test specimen 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.

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

23. Density and yield of concrete when required shall be calculated from the unit volumes of the constituent materials and the volume of the fresh or set concrete as desired. 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 the volume of aggregate mixed as used.

24. The report 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, where methods deviate from the proposed standards.
- (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 specimens.
- (i) The date of molding and testing each cylinder.
- (j) The compressive strength in pounds per square inch of each test specimen and the average of tests in a set.
- (k) A description of failure and appearance of concrete on each test specimen.
- (l) The unit weight, density, and yield of the concrete.

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

(A. S. T. M. standard method (4), serial designation C 31-27, except as indicated. See p. 1)

1. This method is intended to apply to the molding and storing of test specimens of concrete sampled from concrete being used in construction.

2. The test specimens shall be cylindrical in form with the length twice the diameter. 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.

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.¹⁸

¹⁸ See footnote 17.

(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 properly support the mold 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 when molded.

(c) Suggestions for suitable molds are shown in Figures 9, 10, and 11.

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

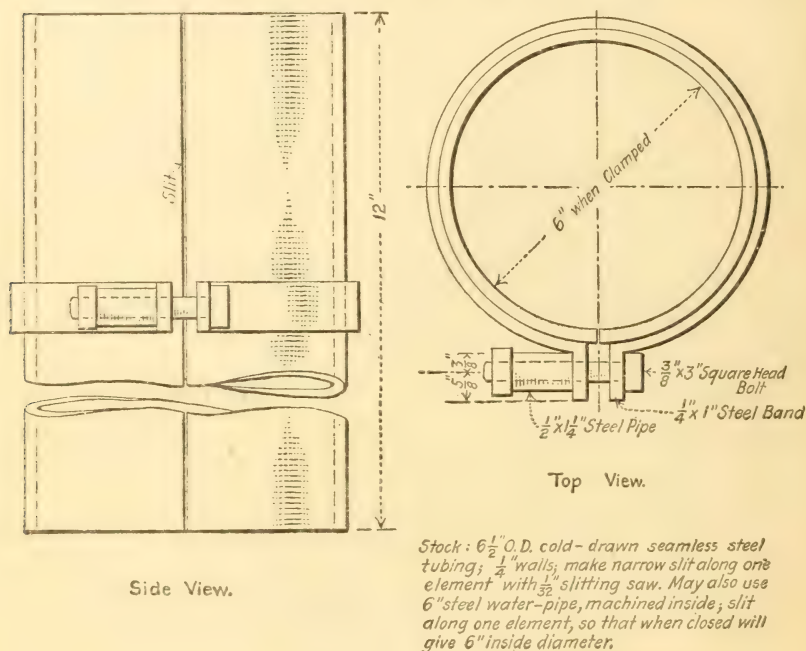


FIG. 9.—Type of mold

transporting to the place of molding. Care shall be taken to see that each test specimen represents the total mixture of the concrete from a given 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 occurring during transportation, each sample shall be dumped into a nonabsorbent water-tight receptacle and, after slight mixing, immediately placed in the mold.

(b) The test specimens shall be molded by placing the concrete in the mold in layers 3 to 4 inches in thickness. Each layer shall be puddled with 25 strokes with a 3/8-inch rod 24 inches in length bullet-pointed 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 may later be used in capping the test specimen.

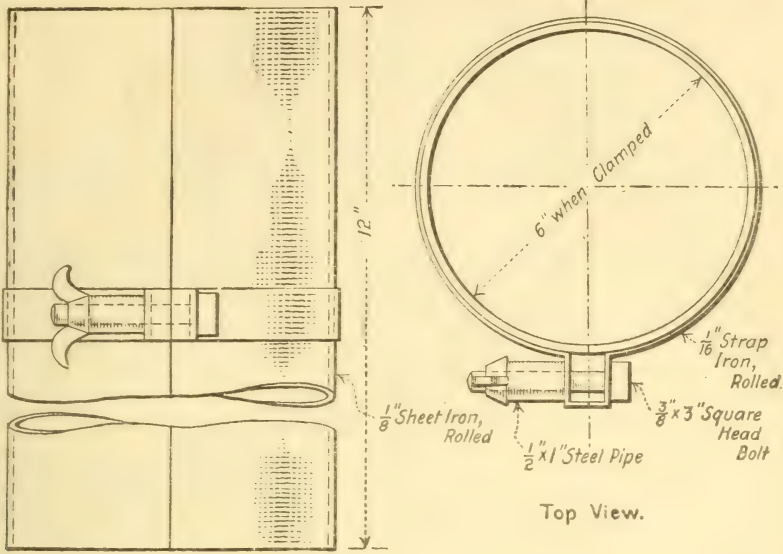


FIG. 10.—Type of mold

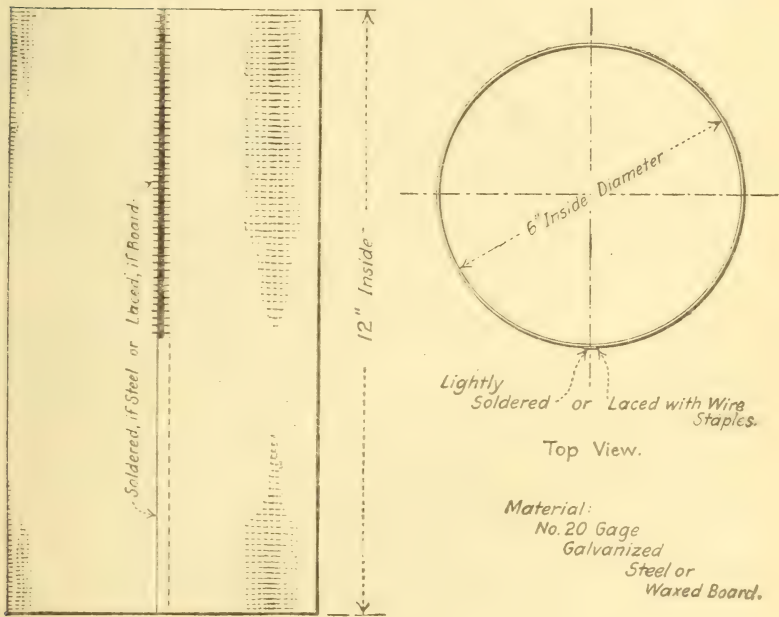


FIG. 11.—Type of mold

6. (a) 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 shall be formed by means of a piece of plate glass one-fourth inch in thickness or a machined metal plate one-half inch in thickness and of a diameter 2 or 3 inches larger than that of the mold. The plate shall be worked on the fresh cement paste until it rests on top of the mold. The cement for capping should be mixed to a stiff paste two to four hours 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 may be avoided by oiling the plates or by inserting a sheet of paraffined paper.

(b) When directed, specimens may be capped after having been received in the laboratory.

7. (a) At the end of 48 hours the test specimens shall be removed from the molds and buried in damp sand except in case the molds shown in Figure 11 are used, when they may be buried in damp sand without removal of the mold, thus permitting shipping in the molds.

(b) When directed, specimens may be cured in a manner similar to the structure.

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 seven-day test be required, the test specimens shall remain at the work as long as possible to harden and then shall be shipped so as to arrive at the laboratory in time for test on the required date.

(c) Test specimens shall be protected from drying after removal from damp storage and before testing.

24. METHODS OF SECURING SPECIMENS OF HARDENED CONCRETE FROM THE STRUCTURE

(A. S. T. M. standard method (4), serial designation C 42-27)

1. These methods and precautions are intended to 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 and the coarse aggregate of the specimen.

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 place 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 or 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 to parallel plane surfaces by capping with a thin layer of mortar richer than the mortar of the specimen or with a mixture of cement and calcined gypsum (plaster of Paris).

7. In case the dimensions of a specimen are necessarily appreciably different from the recommended shape which requires a height twice the diameter, allowance for the ratio of height to diameter must be made in determining the

strength. When the height is more than twice the diameter; that is, when the ratio is greater than two, no correction need be made. For other ratios corrections shall be made as indicated in the following table. Values not given in the table shall be determined by interpolation:

Ratio of height of cylinder to diameter $\left(\frac{h}{d}\right)$

1.75, multiply	crushing strength by-----	0.98
1.50, multiply	crushing strength by-----	.95
1.25, multiply	crushing strength by-----	.94
1.10, multiply	crushing strength by-----	.90
1.00, multiply	crushing strength by-----	.85
0.75, multiply	crushing strength by-----	.70
0.50, multiply	crushing strength by-----	.50

8. The direction of the application of the load in the compression test shall be stated with reference to the direction of the compaction of the mass of concrete in the structure, and where it is practicable the compression load shall be applied in the same direction. In order that the tests be made under uniform conditions as to moisture content, the test specimens shall be completely submerged in water for 48 hours and the compression test shall be made immediately thereafter.

25. METHOD OF TEST FOR ABSORPTION 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.

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 two-hour periods until checks 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.

26. METHOD OF TEST FOR QUALITY OF WATER TO BE USED IN CONCRETE

1. Acidity and alkalinity.—The acidity or alkalinity shall be determined with standard solutions of one-tenth normal alkali or acid, respectively, 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.

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.

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 percentage 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 distilled water 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 tests 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.)

27. METHOD OF TEST FOR SIEVE ANALYSIS OF AGGREGATES FOR CONCRETE¹⁹

(A. S. T. M. standard method (4), serial designation C 41-24, except as indicated. See p. 1)

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]. *From 100 to 500 gram samples, depending upon the coarseness of the sand.*

NOTE.—When samples contain more than 2 per cent silt, it is recommended that an elutriation test be made prior to and on the same sample as the sieve analysis.

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

TABLE 3.—Requirements for sieve openings and wire diameters with permissible variations¹

Sieve number or size in inches	Sieve opening		Wire diameter		Tolerance (per cent)			Maximum opening
	Milli-meters	Inches	Milli-meters	Inches	Average opening	Wire diameter		
						Under	Over	
No. 100 (149 microns)-----	0.149	0.0059	0.102	0.0040	±6	15	35	40
No. 50 (297 microns)-----	.297	.0117	.188	.0074	±6	15	35	40
No. 30 (590 microns)-----	.59	.0232	.33	.0130	±5	15	30	25
No. 16 (1,190 microns)-----	1.19	.0469	.54	.0213	±3	15	30	10
No. 8 (2,380 microns)-----	2.38	.0937	.84	.0331	±3	15	30	10
No. 4 (4,760 microns)-----	4.76	.187	1.27	.050	±3	15	30	10
3/8-inch-----	9.5	.375	2.33	.092	±3	10	10	10
3/4-inch-----	19.0	.75	3.42	.135	±3	10	10	10
1-inch-----	25.4	1.00	4.12	.162	±3	10	10	10
1 1/2-inch-----	38.0	1.50	4.50	.177	±3	10	10	10
2-inch-----	50.8	2.00	4.88	.192	±3	10	10	10
3-inch-----	76.0	3.00	6.3	.25	±3	10	10	10

¹ The dimensions and tolerances for these sieves conform to the requirements of the standard specifications for sieves for testing purposes (serial designation E 11) of the American Society for Testing Materials (3, p. 789).

¹⁹ This method is intended primarily for use in connection with the sieve analysis of either coarse aggregates or mixtures of fine and coarse aggregates for various types of road construction, including concrete, when the sizes larger than No. 4 are specified on the basis of sieves with square openings.

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

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 sieving.

(b) The size of wire and sieve openings shall conform to the requirements given in Table 3.

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

(b) Each size shall be weighed on a balance or scale which is sensitive to 0.001 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 (4,760 microns) sieve, or more than 15 per cent of a coarse aggregate is finer than the No. 4 (4,760 microns) sieve, the sieve analysis of the portions finer and coarser than this sieve shall be reported separately.

28. METHOD OF MECHANICAL ANALYSIS OF SAND OR OTHER FINE HIGHWAY MATERIAL

(A. S. T. M. standard method (1), serial designation D 7-27, except as indicated. See p. 1)

1. A representative test sample of the aggregate weighing [50 grams] *not less than 100 grams* shall be taken.

NOTE.—When samples contain more than 2 per cent silt it is recommended that an elutriation test be made prior to and on the same sample as the sieve analysis.

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

3. (a) The sample shall be passed through each of the standard sieves specified in Table 4.

TABLE 4.—Requirements for sieve openings and wire diameters with permissible variations¹

Mesh designation, United States standard sieve series No. 2	Sieve opening		Wire diameter		Tolerance in average opening	Tolerance on wire diameter		Tolerance in maximum opening
	Milli-meters	Inches	Milli-meters	Inches		Under	Over	
10.....	2.00	0.0787	0.76	0.0299	Per cent ±3	Per cent 15	Per cent 30	Per cent 10
20.....	.84	.0331	.42	.0165	±5	15	30	25
30.....	.59	.0232	.33	.0130	±5	15	30	25
40.....	.42	.0165	.25	.0098	±5	15	30	25
50.....	.297	.0117	.188	.0074	±6	15	35	40
60.....	.177	.0070	.119	.0047	±6	15	35	40
100.....	.149	.0059	.102	.0040	±6	15	35	40
200.....	.074	.0029	.053	.0021	±8	15	35	60

¹ The dimensions and tolerances for these sieves conform to the requirements of the standard specifications for sieves for testing purposes (serial designation E 11) of the American Society for Testing Materials (3, p. 789).

² The order in which the sieves are to be used in the process of sieving is immaterial and shall be left optional, but in reporting results the order in which the sieves have been used shall be stated.

(b) The percentage by weight retained on each sieve shall be determined and the sieving on each sieve shall be continued until less than 1 per cent of the weight retained on each sieve shall pass through the sieve during the last minute of sieving.

4. The percentages in mechanical analysis shall be reported in the following manner:

	Per cent
Passing the No. 200 sieve-----	-----
Passing the No. 100 sieve and retained on the No. 200 sieve-----	-----
Passing the No. 80 sieve and retained on the No. 100 sieve-----	-----
Passing the No. 50 sieve and retained on the No. 80 sieve-----	-----
-----	-----
-----	-----
-----	-----
-----	-----
	100.00

29. METHOD OF MECHANICAL ANALYSIS OF COARSE AGGREGATES²⁰

(A. S. T. M. standard method (4), serial designation D 18-16 except as indicated. See p. 1)

The method shall consist of (1) drying at not over 110° C. (230° F.) to a 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 size screens having circular openings as are required or called for by the specifications, screens to be used in the order named: 8.89 centimeters (3½ inches), 7.62 centimeters (3 inches), 6.35 centimeters, (2½ inches), 5.08 centimeters (2 inches), 3.81 centimeters (1½ inches), 3.18 centimeters (1¼ inches), 2.54 centimeters (1 inch), 1.90 centimeters (¾ inch), 1.27 centimeter (½ inch), and 0.64 centimeter (¼ inch); (3) determining the percentage by weight retained on each screen; and (4) recording the mechanical analysis in the following manner.²¹

	Per cent
Passing 0.64-centimeter (¼-inch) screen-----	-----
Passing 1.27-centimeter (½-inch) screen and retained on a 0.64-centimeter (¼-inch) screen-----	-----
Passing 1.90-centimeter (¾-inch) screen and retained on a 1.27-centimeter (½-inch) screen-----	-----
Passing 2.54-centimeter (1-inch) screen and retained on a 1.90-centimeter (¾-inch) screen-----	-----
-----	-----
-----	-----
-----	-----
	100.00

30. METHOD OF MECHANICAL ANALYSIS OF EXTRACTED AGGREGATES

1. 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.

²⁰ This method is intended primarily for use in connection with the mechanical analysis of coarse aggregates for various types of road construction, including concrete, when the sizes are specified on the basis of round hole screens.

²¹ Attention is called to the fact that the 1-inch screen is not necessarily the largest screen which may be used.

2. The mechanical analysis shall be recorded 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-----	-----
Passing 1/2-inch screen and retained on 10-mesh sieve-----	-----
Passing 3/8-inch screen and retained on 1/4-inch screen-----	-----
Passing 3/4-inch screen and retained on 1/2-inch screen-----	-----
	100.00

3. 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.

31. METHOD OF SAMPLING PAVING BRICK

1. 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.

2. 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.

3. (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.

(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.

(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.

4. 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.

5. 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.

32. METHODS OF TESTING BRICK

(A. S. T. M. tentative standard method (5), serial designation C 67-27 T, except as indicated. See p. 1)

1. These methods cover the following tests for brick :

- The compression test ;
- The flexure or cross-bending test ;
- The rattler test.

COMPRESSION TEST

APPARATUS

2. Any form of standard testing machine may be used for the compression test, together with a spherical bearing block large enough entirely to cover the specimen.

TEST SPECIMEN

3. The test specimen shall consist of a half brick.²² Any method which will produce a specimen with approximately plane and parallel ends, without shattering the specimen, may be used.

4. A half brick to be used for the compression test shall be prepared as follows: The two surfaces intended to receive the load from the testing machine shall receive a thin coating of shellac and be allowed to dry thoroughly. One of the shellacked bearing surfaces shall then receive a thin coat of a neat mortar of calcined gypsum (plaster of Paris). This mortared face of the

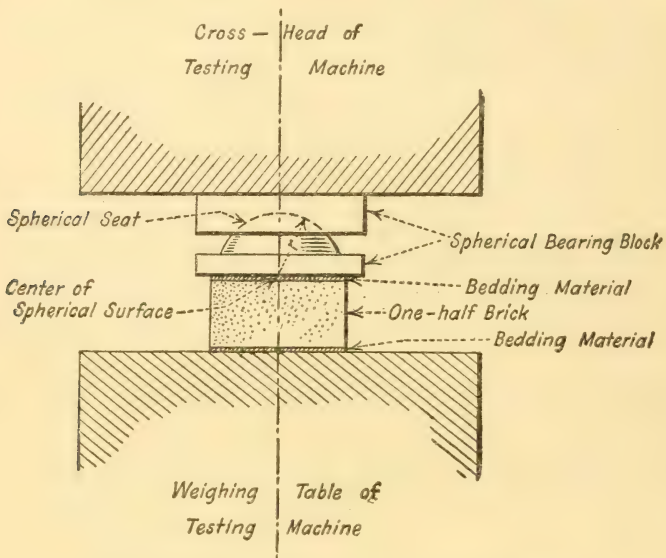


FIG. 12.—Special bearing block for use in testing brick

specimen shall then be pressed on plate glass or a machined metal plate, previously oiled or covered with waxed paper, and the bedding material allowed to harden. This procedure shall be repeated with the other shellacked surface. Care shall be taken that the opposite faces so coated with bedding material shall be parallel. Depressions or recessed or paneled bricks shall be filled with neat Portland-cement mortar, which shall stand at least 24 hours before testing.

5. Compression tests shall be made on at least five half-brick test specimens from different bricks.

PROCEDURE

6. (a) The half-brick test specimen shall be tested, placed flat.

(b) The bearing block shall be used on top of the test specimen in vertical testing machines. The center of the sphere of the bearing block shall be used in contact with the top bearing surface of the specimen, and in the vertical axis of the specimen. (Fig. 12.) The spherical bearing block shall be kept thoroughly lubricated to insure accurate adjustment, which should be made by hand under a small initial load.

²² This permits the use of a suitable half brick from the flexure test.

No.	Name	Age
1	[Faint Name]	[Faint Age]
2	[Faint Name]	[Faint Age]
3	[Faint Name]	[Faint Age]
4	[Faint Name]	[Faint Age]
5	[Faint Name]	[Faint Age]
6	[Faint Name]	[Faint Age]
7	[Faint Name]	[Faint Age]
8	[Faint Name]	[Faint Age]
9	[Faint Name]	[Faint Age]
10	[Faint Name]	[Faint Age]

(c) The speed of the moving head of the testing machine, during the application of the load, shall not be more than 0.05 inch per minute.
 (d) During the test the beam of the testing machine shall be kept constantly in a floating position.

7. The compressive strength in pounds per square inch shall be reported as the total maximum load, W , in pounds, indicated by the testing machine, divided by the area, A , in square inches, of the minimum section of the specimen perpendicular to the line of application of the load.

The compressive strength in pounds per square inch shall be calculated from the formula:

$$\text{Compressive strength} = \frac{W}{A}$$

FLEXURE TEST

APPARATUS

8. (a) Any form of standard testing machine with knife edges as described in paragraph (b) may be used for the flexure test.²³

(b) Knife edges shall be straight and have full contact with the specimen or bearing plate, and they shall have a length at least equal to the width of the brick to be tested. Their bases shall be so curved or rounded as to permit

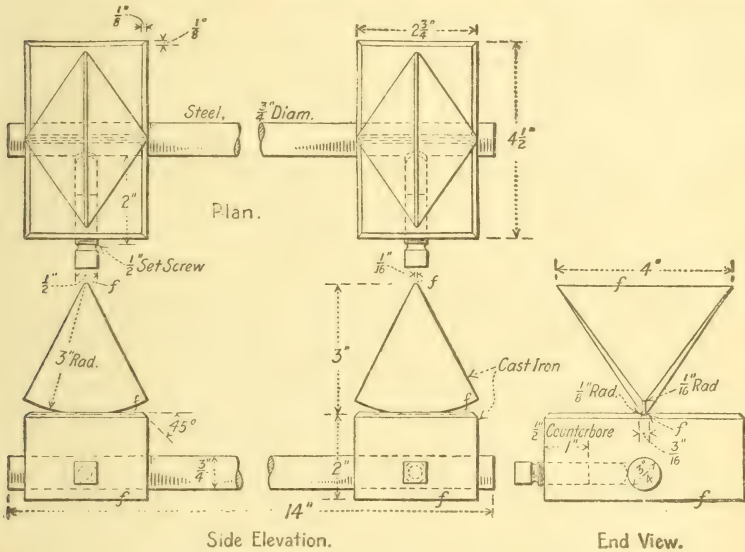


FIG. 13.—Knife edges use in testing brick

free movement of the working edges, both longitudinally and transversely. The use of knife edges of the type shown in Figure 13 is recommended, although other types may be used which will provide free movement and at the same time insure full and even bearing at all times during the test.

TEST SPECIMEN

9. The test specimen shall be a whole brick.
10. Flexure tests shall be made on at least five whole bricks.

PROCEDURE

11. (a) A test specimen brick shall be placed flatwise on the knife edges with a span of 7 inches and the load applied at midspan.

²³ Portable apparatus properly calibrated may be used.

(b) A steel bearing piece one-half inch square and of a length equal to the full width of the specimen shall be placed between the upper knife edge and the specimen.

(c) The speed of travel of the head of the testing machine, during the application of the load, shall not be more than 0.05 inch per minute.

12. The modulus of rupture, R , in pounds per square inch shall be reported as calculated from the following formula:

$$R = \frac{3Wl}{2bd^2}$$

where

W = the total load in pounds at which the brick failed,

l = the distance between the supports in inches,

b = the width of the brick in inches, and

d = the depth of the brick in inches.

RATTLER TEST

APPARATUS

13. The machine used in the rattler test 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 described in sections 14 to 18. In Figure 14 is illustrated a rattler which will meet the requirements specified, and to which reference should be made.

14. The barrel of the machine shall consist of the heads, headliners, staves, and stave liners conforming to the following requirements:

(a) *Heads.*—The heads may be cast in one piece with the trunnions which shall be $2\frac{1}{8}$ 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 key seated 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 $15\frac{3}{8}$ inches in the head for the driving end of the rattler and $11\frac{3}{8}$ inches for the other head, and the distance from the face of the hubs to the inside face of the heads shall be $5\frac{1}{2}$ inches.

The heads shall not be less than three-fourths nor more than seven-eighths inch in thickness. In outline, each head shall be a regular 14-sided polygon inscribed in a circle $28\frac{3}{8}$ inches in diameter. Each head shall be provided with flanges not less than three-fourths inch in thickness and extending outward $2\frac{1}{2}$ 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-fourths-inch bolts at each end of each stave, the slots to be thirteen-sixteenths inch in width and $2\frac{3}{4}$ inches center to center. Each slot shall be provided with a recess for the bolt head, which shall act to prevent the turning of the same. Between each two slots there shall be a brace three-eighths inch in thickness extending down the outward side of the head not less than 2 inches.

(b) *Headliners.*—There shall be for each head a cast-iron headliner, 1 inch in thickness and conforming to the outline of the head, but inscribed in a circle $28\frac{3}{8}$ inches in diameter. This headliner shall be fastened to the head by seven five-eighths-inch cap screws, through the head from the outside. Whenever those headliners 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 headliners shall be hard machinery iron and should contain not less than 1 per cent of combined carbon.

(c) *Staves.*—The staves shall be made of 6-inch medium-carbon steel structural channels $27\frac{1}{4}$ inches in length 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 $1\frac{3}{8}$ inches either way from the longitudinal center line. The spaces between the staves shall be as uniform as practicable, but shall not exceed five-sixteenths inch.

(d) *Staveliners.*—The interior or flat side of each stave shall be protected by a liner three-eighths inch thick by $5\frac{1}{2}$ inches wide by $19\frac{3}{4}$ inches long. The

liner shall consist of medium-carbon 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 counter-sunk on the face of the liner and the rivets shall be driven hot and chipped off flush with the surface of the liners. These lines shall be inspected from time to time, and if found loose shall be at once riveted.

15. 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 proportion as provided in section 19, and shall then be run for 18,000 revolutions at the rate of speed specified in section 22 (a). The shot shall then be removed and a standard shot charge inserted, after which the rattler may be charged with brick for a test.

16. 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 conditions 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 in length, inside measurement, between headliners. The liners of the staves shall be so placed as to drop between the headliners. The staves shall be bolted tightly to the heads by four $\frac{3}{4}$ -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.

17. 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 bots at not less than four points.

18. The barrel shall be driven by gearing whose ratio of driver to driven is not less than one to four. The countershaft upon which the driving pinion is mounted shall not be less than $1\frac{1}{2}$ inches in diameter, with bearing not less than 6 inches in length. If a belt drive is used, the pulley shall be not 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.

19. The abrasive charge shall consist of cast-iron spheres of two sizes.

(a) **Large spheres.**—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. 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 substituted by new ones at any single time, and that in so far as possible the large spheres shall compose a graduated series in various stage of wear.

(b) **Small spheres.**—When new, the smaller spheres shall be 1.875 inches in diameter and shall weigh approximately 0.95 pound (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 nearly 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-fourth 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 plate mentioned 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.

20. The cast-iron abrasive spheres shall conform to the following requirements as to chemical composition:

	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 be obtained by the user before introducing the spheres into the charge, and unless the analysis conforms to the requirements specified the batch of spheres shall be rejected.

21. The scales used shall be placed on a solid level foundation and shall be sufficiently sensitive to determine the initial weight of 10 bricks to the nearest 0.1 pound. The scale shall be tested by a standard-test weight at intervals of not more than every 10 tests.

TEST SPECIMEN

22. The number of brick per test shall be 10 whole bricks for all weights of brick, and the brick shall be weighed to the nearest 0.1 pound. 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.

PROCEDURE

23. (a) The sample, together with the abrasive charge, shall be placed in the rattler and the rattler closed. The rattler shall be rotated at a uniform rate of not less than 23.5 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 starting and stopping. Only one start and stop per test is in general acceptable. If, from accidental causes, the rattler is stopped and started more than once during a test, and the percentage loss exceeds the maximum permissible under the specifications, the test shall be disqualified and another made.

(b) Any test, at the expiration of which a stave liner is found detached from the stave or seriously out of position, shall be rejected.

24. The loss shall be calculated as a percentage of the initial weight of the brick composing the charge, by weighing the brick remaining in the rattler at the end of the test, deducting this weight from the initial weight of the brick, dividing this difference by the original weight of the brick and multiplying by 100.²⁴

$$\text{Percentage of loss} = \frac{\text{Original weight} - \text{weight after testing}}{\text{Original weight}} \times 100$$

In weighing the rattled brick, any piece weighing less than 1 pound shall be rejected.

25. The percentages of loss, as calculated in accordance with section 24, shall be corrected by subtracting from it the differential obtained from the following tabulation, which corresponds to the initial weight of the 10 bricks composing the test charge.²⁵ The corrected result shall be reported as the corrected percentage of loss.

Weight of 10 brick before test, pounds	Correction differential, per cent
95 or over-----	0
90 to 94, inclusive-----	1.0
85 to 89, inclusive-----	1.5
80 to 84, inclusive-----	2.5
75 to 79, inclusive-----	3.0
70 to 74, inclusive-----	3.5
65 to 69, inclusive-----	4.0
60 to 64, inclusive-----	5.0
59 or under-----	6.0

In all cases where the initial weight of 10 brick in pounds and tenths of a pound lies between the upper limit of one grade and the lower limit of the

²⁴ The percentage of loss to be taken as the standard remains within the province of the contracting parties. For their information, the average percentage of loss after correction as provided in section 25 should not exceed 22 per cent for heavy traffic and 24 per cent for light traffic.

²⁵ At the option of the purchaser, the results of the rattler test may be reported direct without correction for size of brick.

next heavier grade, the brick shall be classified in the heavier grade when the fractional part of the weight is 0.5 pound or more.

RECORD

26. A complete and continuous record shall be kept of the operation of all rattlers working under these tentative methods of testing. 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 bricks by which they were identified.
 5. The number of bricks 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, width, and thickness of the bricks.
 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 to conform to the requirements 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 stove 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 counting machine.
 16. Certificate of the operator as to number of stops and starts made in each test.
 17. The initial collective weight of the 10 bricks composing the charge and their collective weight after rattling.
 18. The loss calculated as a percentage of the initial weight and the calculation itself, the correction factor, and the corrected percentage of loss.
 19. The number of broken bricks 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 certificate of the operator that the test was made in accordance with the requirements of the American Society for Testing Materials and that the record is a true record.
 22. The signature of the operator or person responsible for the test.
 23. The location of the laboratory where the test was made and name of the owner.
 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 duplicate, but the original record shall always be preserved intact and complete.²⁶

REPORT OF STANDARD RATTLER TEST OF PAVING BRICK

IDENTIFICATION DATA

Name of firm furnishing sample.....
 Name of firm manufacturing sample.....
 Street or job which sample represents.....
 Brands or marks on the brick.....
 Quantity furnished..... drying treatment.....
 Date received..... date tested.....
 Length....., width....., thickness.....

²⁶ For the convenience of those making use of the test, the accompanying blank form, which provides space for the necessary data, is furnished and its use is recommended.

STANDARDIZATION DATA

Weight of charge (after standardization)	Condition of locknuts 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.....				

Number of charges tested since last inspection.....

RUNNING DATA

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

WEIGHTS AND CALCULATIONS

	Percentage loss ¹
Initial weight of 10 brick.....	
Final weight of 10 brick.....	
Loss in weight.....	
Percentage loss.....	
Correction differential.....	
Corrected percentage of loss.....	

¹ The calculation must appear.

Number of broken brick and remarks on same.....

I certify that the foregoing test was made in accordance with the methods of testing of the American Society for Testing Materials and is a true record.

Signature of tester:

Date..... Location of laboratory.....

Owner.....

Serial No.

33. METHOD OF TESTING CAST IRON, CONCRETE, AND VITRIFIED CLAY CULVERT PIPE

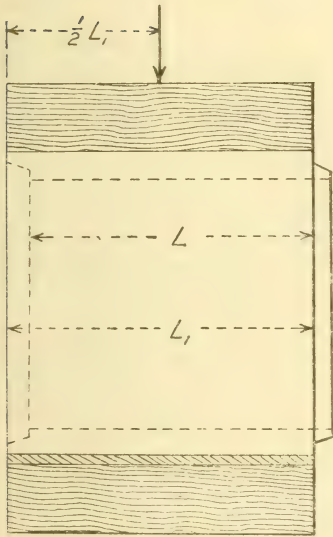
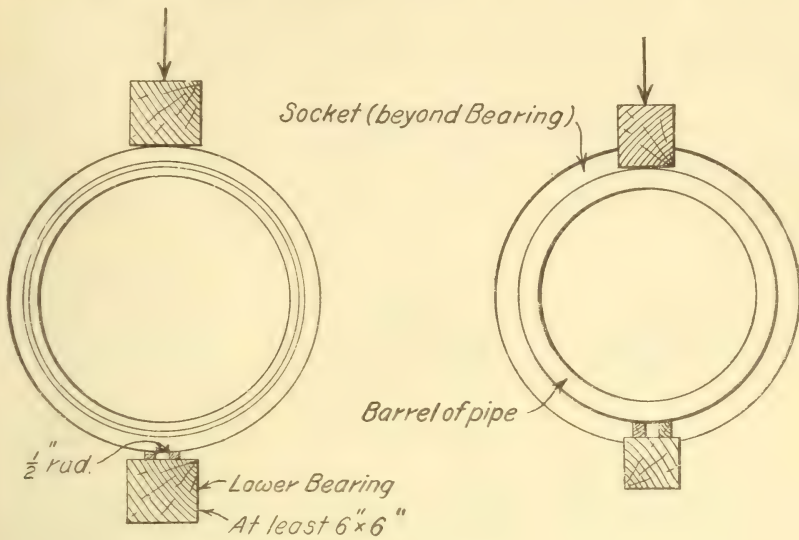
STRENGTH TESTS ²⁷

1. In making strength tests either the three-edge bearing method or the sand-bearing method may be used.

2. When tested by the three-edge bearing method (figs. 15 and 16), a machine shall be used which gives a uniform deflection throughout the full length of the pipe. The lower bearing for the pipe shall consist of two wooden strips with vertical sides having their interior top corners rounded to a radius of approximately one-half inch. The strips shall be straight and shall be securely fastened to a rigid block with the interior vertical sides spaced a distance apart not less than one-half inch nor more than 1 inch for each foot of diameter of pipe. The upper bearing shall be a rigid wooden block, straight and true from end to end. The upper and lower bearings shall extend the full length of pipe exclusive of bell. The pipe shall be placed symmetrically between the two bearings as illustrated in Figures 15 and 16.

²⁷ Methods recommended by the joint concrete culvert pipe committee in its report of February, 1926 (12).

3. When sand bearings are used (figs. 17 and 18), 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



Side View

FIG. 15.—Arrangement for three-edge bearing test of pipe, without bell and spigot

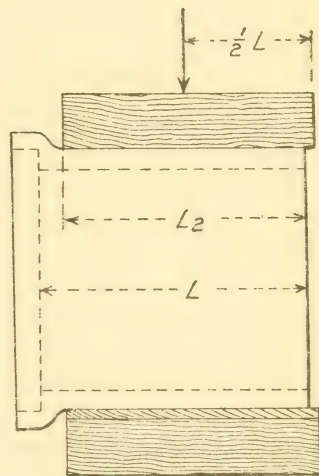
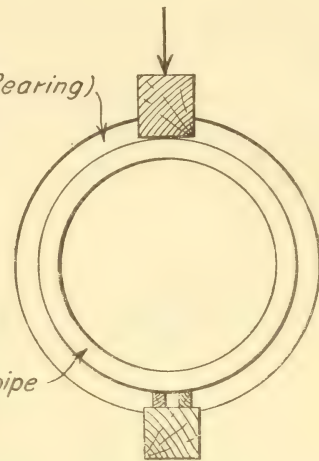


FIG. 16.—Arrangement for three-edge test of bell-and-spigot pipe

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.

4. The sand used shall be clean and dry and shall be such as will pass a No. 4 screen.

5. The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck level with a straight edge, 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, or in such manner as to produce uniform deflection throughout the full length of the pipe. 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

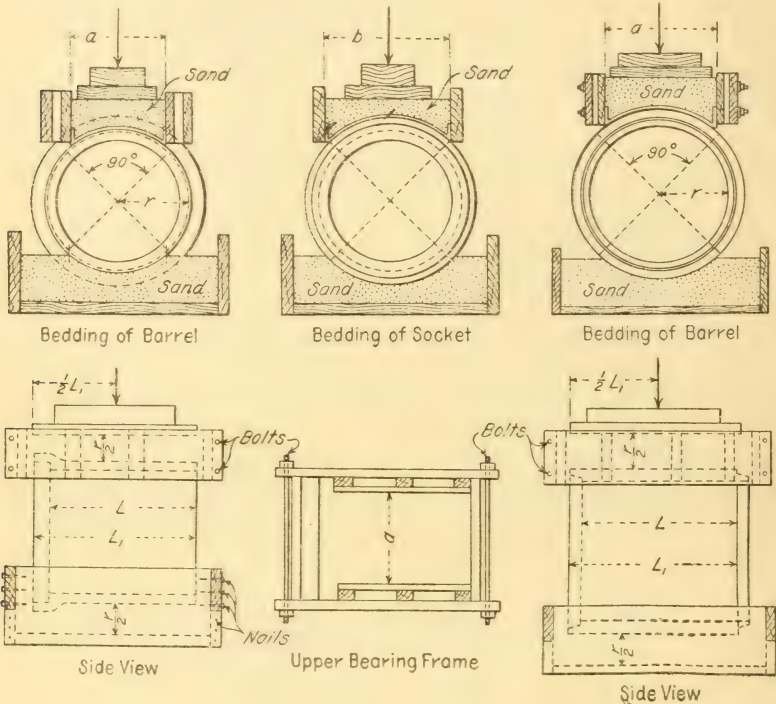


FIG. 17.—Arrangement for sand-bearing test of bell-and-spigot pipe

FIG. 18.—Arrangement for sand-bearing test of pipe without bell and spigot

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.

6. 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 inside of 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.

7. 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. 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. The

load shall be applied continuously until the ultimate strength of the pipe is reached. The load at ultimate strength and also when the first crack appears shall be observed and recorded.

8. In testing pipe which is "out of line" the lines of the bearings chosen shall be from those which appear to give the most favorable conditions for fair test.

ABSORPTION TESTS²⁸ (CONCRETE AND VITRIFIED CLAY PIPE)

9. Not less than three separate test specimens from each of five separate pipe shall be taken as a "standard sample" for the absorption test. Of the three specimens from each pipe, one shall be taken from one end, another from the opposite end, and the third shall be taken from the middle portion of the pipe. Each specimen shall be of from 12 to 20 square inches in area, measured upon the exterior or convex side, and shall be as nearly square as the nature of the material will readily permit. The specimens shall be obtained by breaking the pipe, and shall be apparently sound, solid pieces of the wall of the pipe, and shall not show cracks or fissures or shattered edges due to the shock of breaking or cutting. The specimens may be obtained from the broken pieces of the pipe used in the strength test, if the restrictions as to the size and location of the specimens can be duly observed. The specimens shall be so marked as to permit the identity of each one to be ascertained at any stage of the test.

10. Preparatory to the absorption test, all specimens shall 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 two-hour periods until check weights are obtained.

11. The balance used shall be sensitive to 0.5 grams when loaded with 1 kilogram, and weighings shall be read at least to the nearest gram. Where other than metric weights are used the same order of accuracy must be obtained.

12. In reweighing after immersion, the specimens shall be removed from the water, not allowed to drain for more than one minute, the superficial water removed by towel or blotting paper, and the specimens at once put upon the balance.

13. Specimens after weighing shall be placed in a suitable woven-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.).

14. 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 15 or more specimens comprising the standard sample, the maximum and the minimum single observations entering into the mean, and the variation between the maximum and the minimum of the three specimens of each pipe represented in the standard sample.

FREEZING AND THAWING TESTS²⁹ (CONCRETE AND VITRIFIED CLAY PIPE)

15. The test specimens employed in making the freezing and thawing test shall be another set selected as specified in section 9.

16. The specimens shall be dried as specified in section 10.

17. The same scales and weights as are specified in section 11 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 11 shall be used.

18. 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 11.

²⁸ From A. S. T. M. standard specification, serial designation C 4-24 (4).

²⁹ From A. S. T. M. specifications, serial designation C 4-24 (4).

19. 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 test 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,³⁰ 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 the artificial freezing apparatus is employed³¹ the apparatus shall have sufficient heat-absorbent capacity to enable the temperature of the freezing chamber to be brought to -10° C. ($+14^{\circ}$ 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 a 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.

34. METHOD OF SAMPLING AND TESTING DRAIN TILE

(A. S. T. M. standard method (j), serial designation C 4-24, sections 8 to 30, inclusive, except as indicated. See p. 1)

8. The specimens of tile shall all be selected at the factory or at the shipping destination, or at the trench, at the option of the purchaser. The selection shall be made by a competent inspector employed by the purchaser. The inspector shall divide the tile into subclasses if lack of uniformity in any important particular warrants such division, and shall select enough representative specimens of tile from each subclass for a complete set of standard physical tests.

9. A standard physical test shall comprise tests of five individual tiles. Specimens of tile may be selected by the inspector in such number as he judges necessary to determine fairly the quality of all the tile. The manufacturer or other seller shall furnish specimens of tile without separate charge up to 0.5 per cent of the whole number of tile, and the purchaser shall pay for all in excess of that percentage at the same rate as for other tile.

(A) STRENGTH TESTS OF DRAIN TILE

10. The specimens of tile shall be unbroken, full-size tile.

11. The walls of the tile shall, at the time of testing, be as thoroughly wet as will result from completely covering with hay, cloth, or similar absorbent material, and keeping the covering wet for not less than 12 hours.³²

12. No specimen of tile shall be exposed to water or air temperatures lower than 40° F. from the beginning of wetting until tested. Frozen tile shall be completely thawed before the wetting begins.

13. Each specimen of tile shall be weighed on reliable scales just prior to testing, and the weights shall be reported.

³⁰ A satisfactory type of freezing apparatus is illustrated in connection with A. S. T. M. method, serial designation C 4-24 (j). (See figs. 22 and 23.)

³¹ Artificial freezing will generally be necessary. It may be conducted in a commercial refrigerating room at 0° F. temperature or in an artificial freezing box similar to the one shown in Figure 23, in which 0° F. temperatures can readily be produced by the use of salt and ice.

³² In the case of routine testing, the preliminary wetting of the walls of the tile may be omitted.

14. Any machine or hand method which will apply the load continuously, or in increments not exceeding 5 per cent of the estimated total breaking load, may be used in making the test. The tile shall not be allowed to stand under load longer than is required for observing and recording the loads. All solid parts of the bearing frames and bearing blocks shall be so rigid that the distribution of the load will not be affected appreciably by the deformation of any part. All bearings and the specimens of tile shall be so accurately centered as to secure a symmetrical distribution of the loading on each side of the center of the tile in every direction.

15. The purchaser shall choose (1) sand bearings, (2) two-edge bearings, or (3) three edge bearings for use in making strength tests of draintile. (See sections 18, 19, and 20.)

16. The tests results shall be calculated and reported, in pounds per linear foot of tile, in terms of the "ordinary supporting strength."³³

The ordinary supporting strength shall be calculated by multiplying the test breaking loads by the following factors: For sand bearings, 1.00; for two-edge bearings, 1.50; for three-edge bearings, 1.50.

The results of the strength tests shall be reported separately for each of the five individual specimens of tile constituting a standard test, together with the average.

17. The modulus of rupture may or may not be calculated and reported, at the option of the purchaser. When reported it shall be calculated by the equations:³⁴

$$M = 0.20 \tau \frac{W}{t^2} \text{-----} (1)$$

$$f = \frac{6 M}{t^2} \text{-----} (2)$$

where M =maximum bending moment in wall in pound-inches per inch of length, τ =radius of middle line of tile wall in inches, W =ordinary supporting strength, calculated as prescribed in section 16, in pounds per linear foot of tile, f =modulus of rupture in pounds per square inch, and t =thickness of tile wall in inches.

Five-eighths of the weight of the tile per linear foot for sand bearings, or three-fourths for two-edge or three-edge bearings, shall be added to W in computing the maximum bending moment M , when such addition exceeds 5 per cent of W . The value of t used shall be the average thickness of the wall at the top of the tile or that at the bottom, selecting the lesser of the two.

18. When sand bearings (fig. 19) are used, the ends of each specimen of tile shall be accurately marked in quarters of the circumference prior to the test. Specimens shall be carefully bedded, above and below, in sand, for one-fourth the circumference of the tile measured on the middle line of the wall. The depth of bedding above and below the tile at the thinnest points shall be one-half the radius of the middle line of the wall.

The sand used shall be clean, and shall be such as will pass a No. 4 sieve.

The top bearing frame shall not be allowed to come in contact with the tile nor 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 weight shall be piled symmetrically about a vertical line through the center of the tile, 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 the sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with

³³The "ordinary supporting strength," when calculated as specified in section 16, is approximately equal to the actual supporting strength of a tile when laid in a ditch by the "ordinary" method.

³⁴The coefficient of 0.20 in equation (1) approximates the value found by theoretical analysis and also that determined by extended tests.

the tile 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 tile.

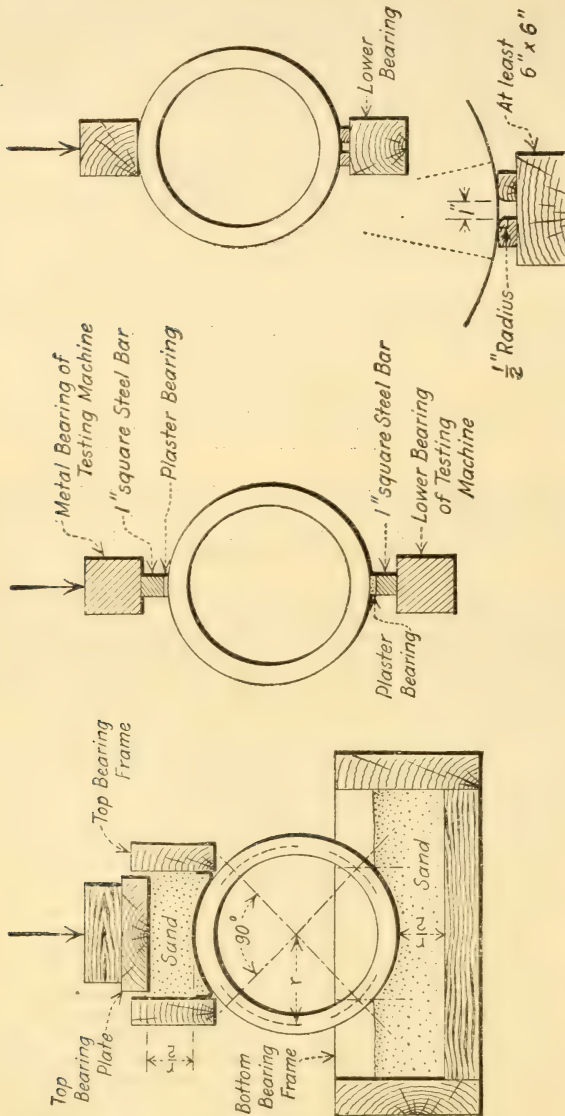


FIG. 20.—Two-edge bearings

FIG. 19.—Sand bearings

Detail of Lower Bearing.

FIG. 21.—Three-edge bearings

19. When two-edge bearings (fig. 20) are used, the ends of each specimen of tile shall be accurately marked in halves of the circumference prior to the test.

The tile to be tested shall be supported by a metallic knife bearing 1 inch wide and extending the length of the tile. Before the tile is placed, a fillet of plaster of Paris and sand 1 inch wide, and thick enough to compensate for all the inequalities of the tile barrel, shall be cast on the surface of the knife-edge

bearing. The tile 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 tile 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 test without lost motion, vibration, or sudden shock.

20. When three-edge bearings (fig. 21) are used, the ends of each specimen of tile shall be accurately marked in halves of the circumference prior to the test.

The 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 tile which is "out of straight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair bearings.

(B) ABSORPTION TESTS OF DRAIN TILE

21. Not less than three separate test specimens from each of five separate tiles shall be taken as a "standard sample" for the absorption test. Of the three specimens from each tile, one shall be taken from one end, another from the opposite end, and the third shall be taken from the middle portion of the tile. Each specimen shall be of from 12 to 20 square inches in area, measured upon the exterior or convex side, and shall be as nearly square as the nature of the material will readily permit. The specimens shall be obtained by breaking the tile, and shall be apparently sound, solid pieces of the wall of the tile, and shall not show cracks or fissures or shattered edges due to the shock of breaking or cutting. The specimens may be obtained from the broken pieces of the tiles used in the strength test, if the restrictions as to the size and location of the specimens can be duly observed. The specimens shall be so marked as to permit the identity of each one to be ascertained at any stage of the test.

22. Preparatory to the absorption test, all specimens shall be first 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 two-hour periods until check weights are obtained.

23. The balance used shall be sensitive to 0.5 gram when loaded with 1 kilogram, and weighings shall be read at least to the nearest gram. Where other than metric weights are used, the same order of accuracy must be obtained.

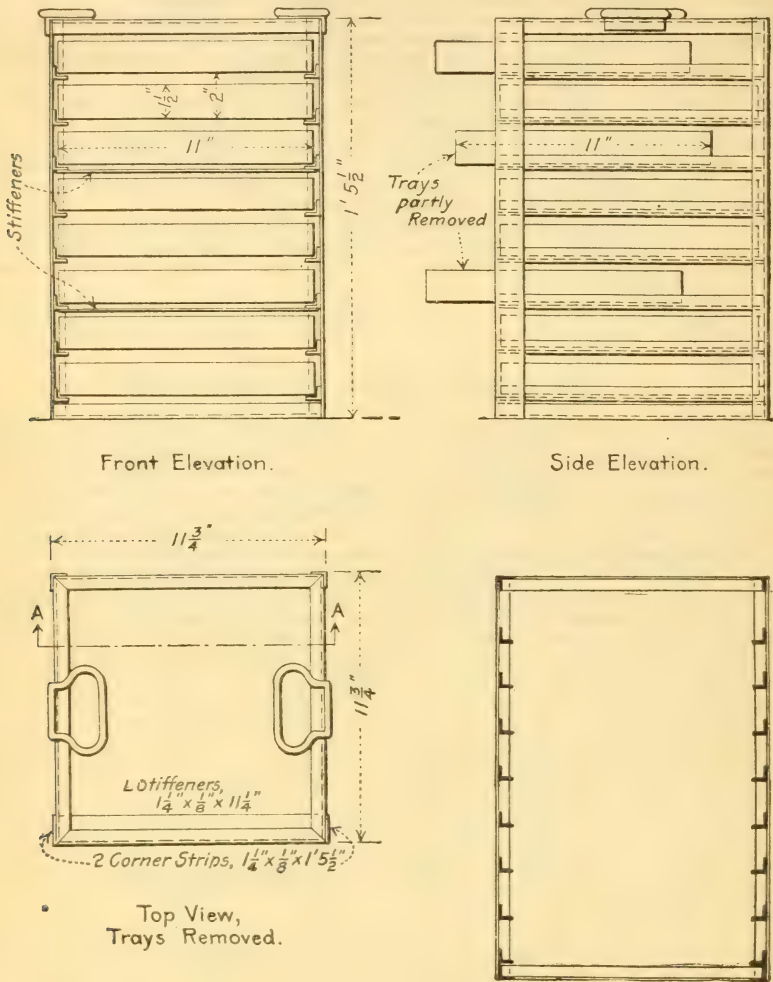
In reweighing after immersion, the specimens shall be removed from the water, not allowed to drain for more than one minute, the superficial water removed by towel or blotting paper, and the specimens at once put upon the balance.

24. Specimens after weighing shall be placed in a suitable woven-wire receptacle, packed tightly enough to prevent jostling, covered with distilled water or rainwater, 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.).

25. 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 15 or more specimens comprising the standard sample, the maximum and the minimum single observations entering into the mean, and the variation between the maximum and the minimum of the three specimens of each tile represented in the standard sample.

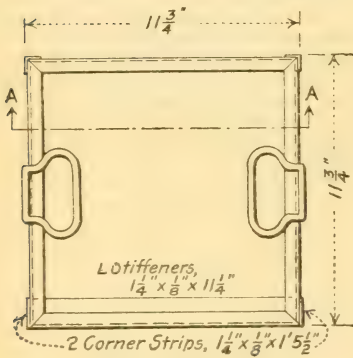
(C) FREEZING AND THAWING TESTS OF DRAIN TILE

26. The test specimens employed in making the freezing and thawing test shall be another set selected as specified in section 21.



Front Elevation.

Side Elevation.



Top View,
Trays Removed.



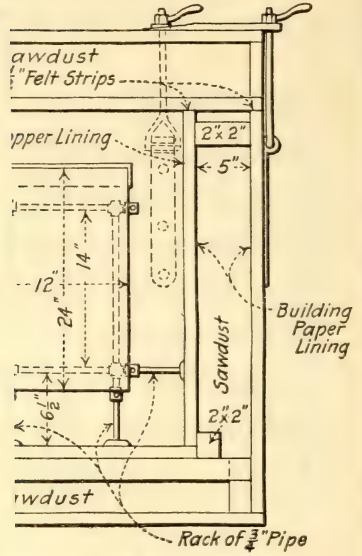
Section A-A,
Trays Removed.

- Notes:
- Rack Constructed of $\frac{5}{8}$ " x $\frac{5}{8}$ " x $\frac{1}{8}$ " Galvanized Angles, except as Noted. All Connections Riveted or Soldered.
 - Trays, $11 \times 11 \times 1\frac{1}{2}$ " outside, Made of No. 17 Galvanized Steel.

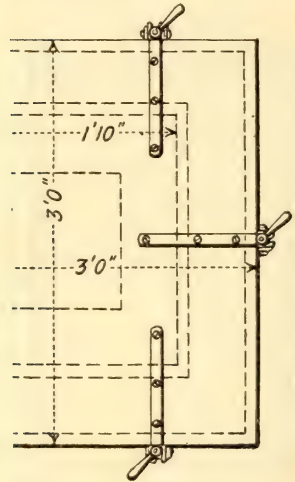
FIG. 22.—Suggested plans for freezing crate and trays

27. The specimens shall be dried as specified in section 22.

28. The same scales and weights as are specified in section 23 for the absorption test or others of equivalent sensitiveness and accuracy shall be employed



Section B-B



View, Top in Place

SUGGESTED PLANS
 FOR
FREEZING BOX
 TO HOLD SALT AND ICE TO FREEZE

for the weighings required in the freezing and thawing test. The same procedure in weighings and reweighing as specified in section 23 shall be used.

29. 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 23.

30. 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 test 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,³⁵ 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 employed³⁶ 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 a 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.

35. METHODS OF MAKING 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.

(A) TENSION TEST

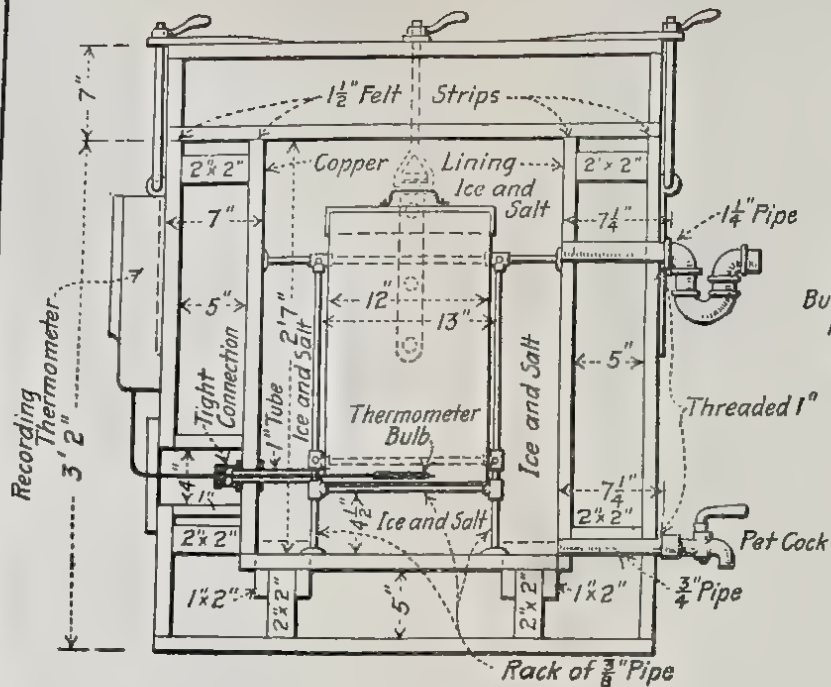
3. The briquets for the tension test shall be molded in the standard mold as used for testing Portland cement. Single or gang molds may be used. They shall be wiped with an oily cloth before using.

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.

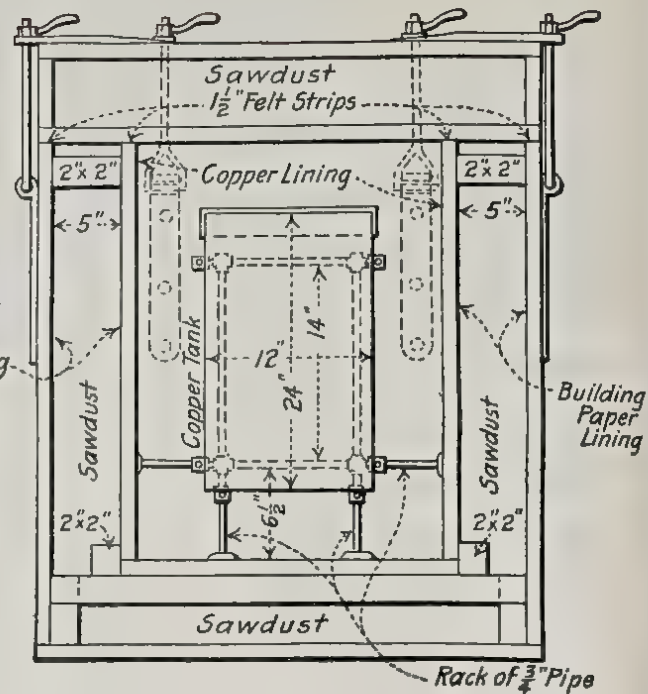
5. Tests shall be made with any standard machine. The briquets shall be tested as soon as they are removed from the water. The bearing surfaces of the clips and briquets shall be free from grains of sand or dirt. The briquets shall be carefully centered and the load applied continuously at the rate of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

³⁵ Figure 22 shows a crate and trays suitable for use in the box for artificial freezing illustrated in Figure 25.

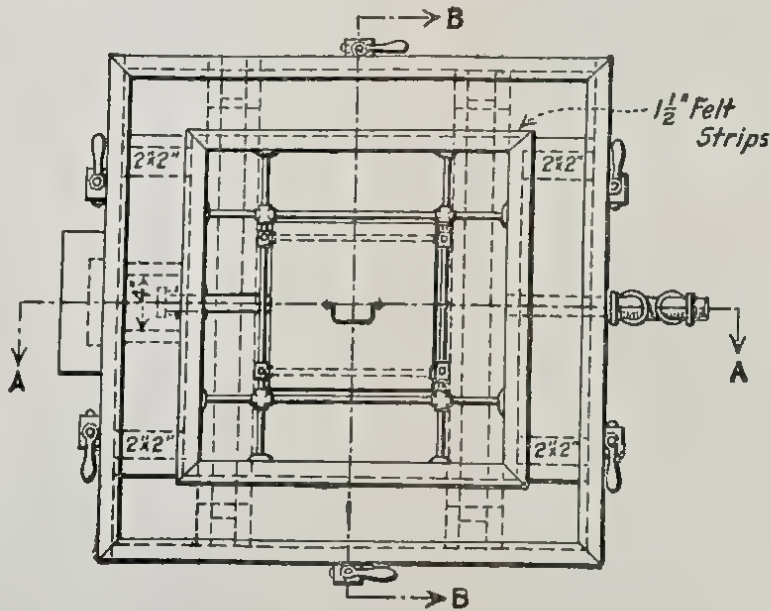
³⁶ Artificial freezing will generally be necessary. It may be conducted in a commercial 0° F. refrigerating room or in an artificial freezing box similar to the one shown in Figure 23, in which 0° F. temperature can readily be produced by the use of salt and ice.



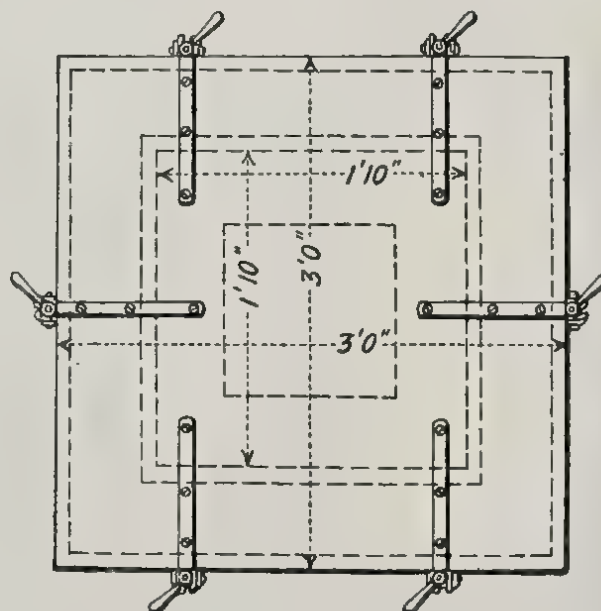
Section A-A.



Section B-B



Plan View,
Top Removed.



Plan View, Top in Place

Note: Box to be Constructed of Seasoned White Pine, free from Defects, or other Suitable Timber, 1"x6", unless otherwise Specified.

SUGGESTED PLANS
FOR
FREEZING BOX
USING SALT AND ICE TO FREEZE

FIG. 23.—Suggested plans for freezing box using salt and ice to freeze

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.

(B) COMPRESSION TEST

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.

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.

(C) STORAGE OF TEST PIECES

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 pounds per cubic foot.

Required, 75 cubic inches of mortar.

Quantity of sand, $\frac{75}{1728} \times 100 \times 453.6 = 1,969$ grams.Quantity of cement, $\frac{25}{1728} \times 94 \times 453.6 = 617$ grams.**36. METHOD OF SAMPLING MINERAL FILLER**

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

37. 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.

38. METHOD OF PROPORTIONING NATURAL SAND AND GRAVEL 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.

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) $1:2:3\frac{1}{2}=94:2 \text{ times } 95:3\frac{1}{2} \text{ times } 110.$
 $=94:190:385$ proportion by weight.
 $=1:2.02:4.10=1:6.12.$
- (2) Percentage of sand in base mixture aggregate $=\frac{190}{190+385} \times 100=33$
 Percentage of sand in new mixture $=60$
- (3) $60-33=27.$
 $27 \text{ times } 0.05=1.35.$
 $6.12-1.35=4.77$, 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) $1:2.86:1.91=94 \text{ pounds}:269 \text{ pounds}:180 \text{ pounds.}$
 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 $1:4.77=94 \text{ pounds to } 449 \text{ pounds.}$

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 33³⁷

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.³⁸

39. METHODS OF TESTS FOR 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.

³⁷ 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.

³⁸ For a theoretical discussion of the method of proportioning see Bulletin No. 60, Engineering Experiment Station, Iowa State College (6) and also a paper by R. W. Crum, Proceedings American Society for Testing Materials, 1922 (7).

PREPARATION OF SPECIMEN FOR TEST

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.

40. METHODS OF SAMPLING BITUMINOUS MATERIALS

(A. S. T. M. standard method (4), serial designation D 140-25)

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 practical, bituminous materials 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 of 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.

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.

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 discarded 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

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 opening should 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-gallon 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 MATERIALS FLOW BY GRAVITY

9. Materials 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.

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.

12. (a) Semisolid or solid materials.—Where the lot of material to be sampled is obviously from a single run or batch of the producer, one package or cake shall be selected at random and sampled as described in the following paragraph. Where the lot of material to be sampled is not obviously from a single run or batch of the producer, or where the single sample selected as described above fails on test to conform to the requirements of the specifications, a number of packages or cakes shall be selected at random equivalent to the cube root of the total number of packages or cakes in the lot. For convenience, Table 6 is given.

TABLE 6.—Number of samples of bituminous material to be selected for shipments of various sizes

Packages or cakes in shipment	Packages or cakes selected	Packages or cakes in shipment	Packages or cakes selected
2 to 8.....	2	217 to 343.....	7
9 to 27.....	3	344 to 512.....	8
28 to 64.....	4	513 to 729.....	9
65 to 125.....	5	730 to 1,000.....	10
126 to 216.....	6	1,001 to 1,331.....	11

Samples shall be taken from at least 3 inches below the surface and at least 3 inches from the side of the container or cake or from the center of a cake. 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. An auger, or brace and $\frac{3}{4}$ -inch bit, or other suitable means may also be used. When more than one package or cake in a lot is sampled, each individual sample shall be not less than one-fourth pound in weight. When the lot of material is obviously from a single run or batch of the producer all samples from the lot shall be melted and thoroughly mixed, and an average sample taken from the combined material for examination. In case more than a single run or batch of the producer is present and the batches can be clearly differentiated, a composite sample shall be prepared for examination from each batch. Where it is not possible to differentiate between the various batches each sample shall be examined separately.

(b) Liquid materials.—Samples of liquid bituminous materials from barrels or drums shall be secured by taking 1 quart of material from packages selected at random according to the cube root method described in paragraph (a). When the lot of material is obviously from a single run or batch of the producer the samples shall be combined and thoroughly mixed and an average sample taken from the combined material. In case more than one run or batch is present and can be clearly differentiated, a composite sample shall be prepared from each batch. Where it is impossible to differentiate between the various batches or runs, each sample shall be examined separately.

13. Solid bituminous materials in crushed fragments or powder, such as gilsonite, grahamite, etc., shall be sampled in accordance with the standard method of sampling coal (serial designation D 21) of the American Society for Testing Materials (4).

41. 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 of sheet asphalt mixtures 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 in the following manner: These composite samples shall be taken by collecting small quantities at different times from not less than ten and preferably more separate batches, molding the portions into the container provided for the purpose.

In the sampling of bituminous concrete either of the two methods given below shall be used in order to obtain a composite sample of not less than 5 pounds, the amount being dependent on the maximum size of the coarse aggregate. Small portions from a number of batches are taken and stored on a metal plate until near the end of the day, when the entire mass is reheated, care being exercised not to apply too great heat, mixed thoroughly and the day's sample selected in small portions from this mass. In case of dense bituminous-concrete mixtures the samples may be taken by collecting portions from one or more batches on a large shovel, thoroughly mixing while still warm and taking a small scoopful which is placed directly in the carton. This operation is to be repeated as often as possible, thus representing a maximum number of batches. In either of these methods care must be taken to avoid segregation of either stone or bituminous mortar. In selecting the portions of the sample, care should be taken to scrape away the top material in the load, so as to avoid dust or any segregated unrepresentative mixture.

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

42. METHODS OF SAMPLING AND TESTING PREMOLDED JOINT FILLERS

One section at least 1 foot long and the full width of the joint shall be submitted from each consignment and shall be tested in accordance with the following method:

(a) Absorption.—A sample 2 by 6 inches is cut from the joint material in such a manner that all edges are freshly cut and weighed. The specimen is then

immersed in water for 24 hours, removed, and the surface water wiped off with a slightly dampened cloth. The specimen is then quickly weighed and the percentage of absorption computed.

(b) Brittleness.—A sample 2 by 6 inches is cut from the joint material parallel to the lay of the fiber and maintained at a temperature of from 4° to 6° C. in water for at least two hours prior to testing. It is then clamped between two boards so that expansion joint cantilevers 3½ inches and is held in any suitable support. A cast-iron ball, weighing 0.957 pound and having a diameter of 1.875 inches, is suspended by a cord which is tied to an eyelet soldered to the ball. For samples having a thickness of nine-sixteenths inch or less, the ball is suspended 1 foot above the center of the projected portion of the specimen. For samples over nine-sixteenths inch in thickness the ball is suspended 2 feet above the specimen. The ball is released by burning the string above the eyelet.

(c) Distortion.—A sample 2 by 6 inches, absolutely flat and straight, which has been cut parallel to the lay of the fiber, is clamped between two blocks so that the expansion joint cantilevers 3½ inches. The clamp with the expansion joint is then placed in an oven maintained at 125° F. for two hours. The deflection from the horizontal is then measured.

43. METHODS OF TEST FOR SPECIFIC GRAVITY OF BITUMINOUS MATERIALS

1. The specific gravity of bituminous materials shall be expressed preferably 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:

$$\text{Specific gravity } 25^{\circ}/25^{\circ} \text{ C. (77}^{\circ}/77^{\circ} \text{ F.)}$$

HYDROMETER METHOD

(Used for thin fluid bitumens)

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:

$$\text{Specific gravity } 25^{\circ}/25^{\circ} \text{ C.} = \text{specific gravity } 25^{\circ}/15.5^{\circ} \text{ C.} \times 1.002$$

PYCNOMETER METHOD

(Used for viscous fluid and semisolid bitumens and emulsions)

4. The determination of specific gravity shall be made with a pycnometer (Hubbard-Carmick type recommended) 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.

DISPLACEMENT METHOD

(Used for hard, solid bitumens)

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:

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

DISPLACEMENT METHOD

(Used 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:

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

44. METHOD OF TEST FOR PERCENTAGE 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.

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 allowed to dry over the top of the oven until no odor of carbon disulphide is detected and then 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 subsidence 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.³⁹

45. METHOD OF TEST FOR PERCENTAGE OF BITUMEN INSOLUBLE IN CARBON TETRACHLORIDE

1. This determination is conducted in exactly the same manner as described under "Method of test for percentage of bitumen" (see p. 64), 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 "Method of test for percentage of bitumen insoluble in paraffin naphtha." (See p. 66.)

³⁹ This method for recovery of mineral material should also be used in case of asphalt topping and rock asphalt if analyzed as previously described.

46. METHOD OF TEST FOR PERCENTAGE 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:

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

47. METHOD OF TEST FOR LOSS ON HEATING OF OIL AND ASPHALTIC COMPOUNDS

(A. S. T. M. standard method (4), serial designation D 6-27, except as indicated. See p. 1)

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

APPARATUS

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 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 to 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. (A recommended form of aluminum shelf is shown in fig. 24.)

3. The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in centigrade degrees, the range being 155 to 170° C.

Type: Etched stem, glass.

Liquid: Mercury.

Range and subdivision: 155 to 170° C. in 0.5° C.

Total length: 150 to 154 millimeters (5.90 to 6.06 inches).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 millimeters (0.24 to 0.28 inch).

Bulb: Corning normal or equally suitable thermometric glass. Length, 10 to 15 millimeters (0.39 to 0.59 inch). Diameter, 5 to 6 millimeters (0.196 to 0.236 inch).

Distance to 0° C. line from bottom of bulb: 33 to 38 millimeters (1.30 to 1.49 inches).

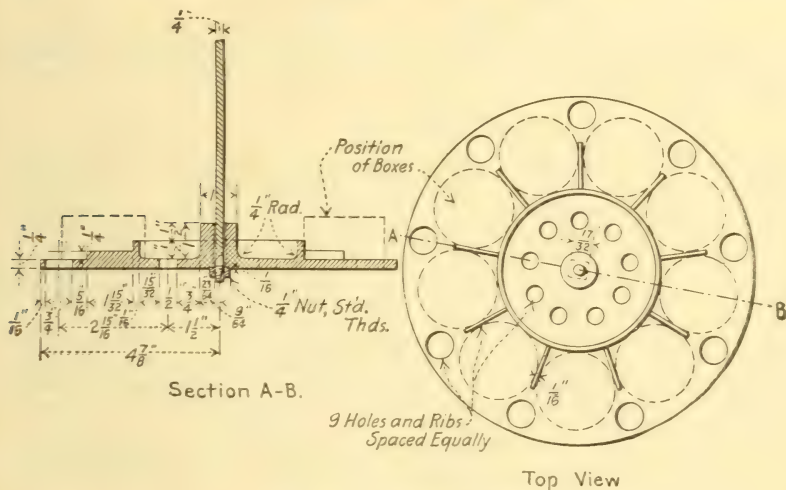


FIG. 24.—Aluminum shelf

Distance to 155° C. line from bottom of bulb: 70 to 80 millimeters (2.75 to 3.15 inches).

Distance to 170° C. line from top of thermometer: 20 to 30 millimeters (0.79 to 1.18 inches).

Contraction chamber: To be long, narrow type, top to be not more than 65 millimeters (2.56 inches) above bottom of the bulb.

Expansion chamber: To permit heating the thermometer at least 25° C. above the highest temperature on the scale.

Filling above mercury: Nitrogen gas.

Top finish: Glass ring.

Graduation: All lines and figures clear cut and distinct. Each whole degree centigrade line to be longer than the remaining lines. Graduations are to be numbered at 0°, 155°, 163°, and 170° C.

Immersion: Total.

Special marking: "A. S. T. M. loss on heat," a serial number and the manufacturer's name or trade-mark shall be etched on the stem.

Scale error: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 0.5° C.

Standardization: The thermometer shall be standardized at three points, including 163° C., for total immersion.

Case: The thermometer shall be supplied in a suitable case, on which shall appear the marking: "A. S. T. M. loss on heat, 155° to 170° C."

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gauge.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

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 road oils the container may be a 2-ounce Gill-style flat-bottomed can approximately 60 millimeters in diameter and 20 millimeters deep.*

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.

PROCEDURE

6. Fifty grams of the water-free material to be tested shall be weighed into a tared container conforming to the requirements of section 4. *For road oils 20 grams of material may be used.* The oven shall be brought to a temperature of 163° C. (325° F.), and the tin box containing the sample placed 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. The oven shall then be closed and the shelf rotated five to six revolutions per minute during the entire test. The temperature shall be maintained at 163° C. (325° F.) for five hours, then the sample shall be removed from the oven, cooled and weighed, and the loss due to volatilization calculated.

7. *For a determination of the loss on heating at 100° C. (212° F.) use procedure outlined in paragraph 6, except that the oven is to be maintained at a temperature of 100° C. (212° F.).*

8. 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.

9. 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 in section 10. Results obtained on samples showing evidences of foaming during the test shall be rejected.

ACCURACY

10. 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 shown in Table 7.

TABLE 7.—Volatilization loss on heating of oil and asphaltic compounds

Volatilization loss	Numerical correction	True volatilization loss
<i>Per cent</i>		<i>Per cent</i>
5.0	±0.50	4.50 to 5.50
5.5	±0.51	4.91 to 6.01
6.0	±0.52	5.48 to 6.52
10.0	±0.60	9.40 to 10.60
15.0	±0.70	14.30 to 15.70
25.0	±0.90	24.10 to 25.90
40.0	±1.20	38.80 to 41.20

NOTE.—If additional periods of heating are desired, it is recommended that they be made in successive increments of five hours each.

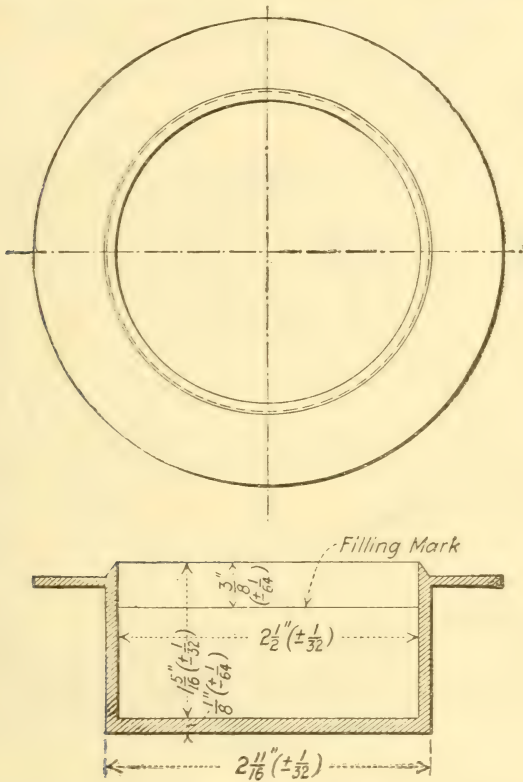


FIG. 25.—Cleveland open cup

When the penetration of the sample after heating is 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. Then bring it to the standard temperature and test as prescribed under the directions for the standard method of test for penetration of bituminous materials (serial designation, D 5) of the American Society for Testing Materials (1). (See p. 72.)

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.

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

(A. S. T. M. standard method (4), serial designation D 92-24, except as indicated.
See p. 1)

1. The open cup flash and fire test on all products except fuel oils and those having an open cup flash below $[175^{\circ} \text{ F.}] 158^{\circ} \text{ F.}$ 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

2. The cup shall be supported by a metal plate one-fourth inch (0.635 centimeter) in thickness and 6 inches (15.24 centimeters) in width. The plate shall

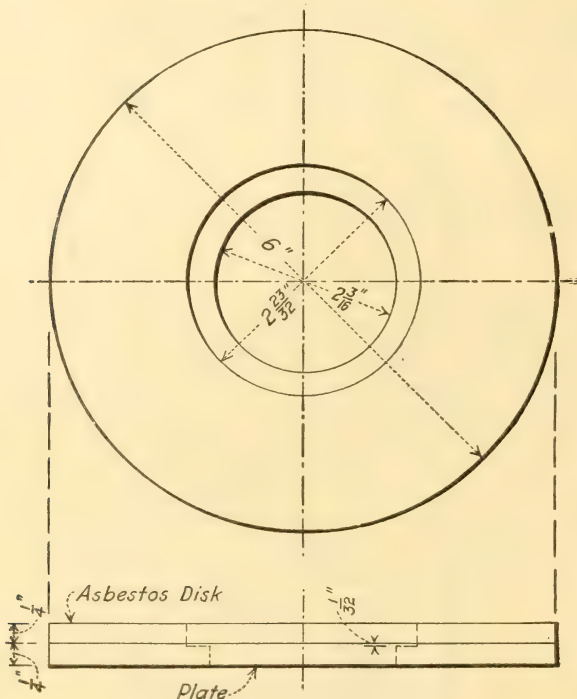


FIG. 26.—Heating plate

be of brass, cast iron, wrought iron, or steel. In the center of the plate there shall be a plane depression one-thirty-second inch (0.079 centimeter) in depth, and of just sufficient diameter to fit the cup. There shall be a circular opening $2\frac{1}{8}$ inches (5.50 centimeters) in diameter, cut through the plate, centering with the center of the above-mentioned depression. The plate shall be covered with a sheet of hard asbestos board one-fourth inch in thickness, 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. Heat may be supplied from any convenient source. The use of a gas burner, electric heater, or alcohol lamp is permitted but under no circumstances are products of combustion or free flame allowed to come up around the cup. The source of heat shall be centered under the opening in the plate and shall be of a type that will not produce local superheating. 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 style 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.

3. The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either centigrade or Fahrenheit degrees as specified, the ranges being -6 to $+400^{\circ}$ C., or $+20$ to $+760^{\circ}$ F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and subdivision: -6 to $+400^{\circ}$ C. in 2° C. or $+20$ to $+760^{\circ}$ F. in 5° F.

Total length: 303 to 307 millimeters (11.92 to 12.08 inches).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 millimeters (0.24 to 0.28 inch).

Bulb: Corning normal or equally suitable thermometric glass. Length, not over 13 millimeters (0.51 inch). Diameter, not greater than stem.

Distance to -6° C. or 20° F. line from bottom of bulb. 40 to 50 millimeters (1.57 to 1.97 inches).

Distance to 400° C. or 760° F. line from top of thermometer: 30 to 45 millimeters (1.18 to 1.77 inches).

Filling above mercury: Nitrogen gas.

Top finish: Red glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. The first and each succeeding 10° F. line to be longer than the intermediate lines. Graduations to be numbered at each multiple of 20° .

Immersion: 25 millimeters or 1 inch. The words "1-inch immersion" on Fahrenheit thermometers or "25 millimeters immersion" on centigrade thermometers and a line around the stem 25.4 millimeters or 1.00 inch above the bottom of the bulb shall be etched on the thermometer.

Special marking: "A. S. T. M. open flash," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

Scale error: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 1° C. up to 372° C. or $2\frac{1}{2}^{\circ}$ F. up to 700° F.

Standardization: The thermometer shall be standardized at the ice point and at intervals of approximately 100° F. for 25 millimeters or 1-inch immersion and for the following temperatures of the emergent mercury column:

Thermometer reading:	Average temperature of emergent mercury column
100° C., 200° F.	44° C., 110° F.
150° C., 300° F.	54° C., 129° F.
200° C., 400° F.	64° C., 150° F.
250° C., 500° F.	77° C., 175° F.
300° C., 600° F.	91° C., 205° F.
350° C., 700° F.	108° C., 240° F.

Test for permanency of range: After being subjected to a temperature between 360° to 370° C. or 680° to 700° F. for 24 hours, the accuracy shall be within the limit specified.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A. S. T. M. open flash, -6 to $+400^{\circ}$ C." or "A. S. T. M. open flash, $+20$ to $+760^{\circ}$ F.," according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply: The total length is the over-all length of the finished instrument. The diameter is that measured with a ring gauge. The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing. The top of the thermometer is the top of the finished instrument.

PROCEDURE

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-fourth inch (0.635 centimeter)⁴⁰ 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

⁴⁰ 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.

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 (0.79 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 one second.

(e) The oil shall be heated at a rate not exceeding 30° F. per minute temperature rise, till a point is reached approximately 100° F. below the probable flash point of the oil. Thereafter the rate of heating shall be decreased and for at least the last 50° F. before the flash point is reached, the rate shall be 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 of 9° to 11° F. per minute, 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 five seconds. The method of application of the flame shall be the same as for flash point. The temperature read at the time of the flame application which causes burning for a period of five seconds or more shall be recorded 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.

49. METHOD OF TEST FOR PENETRATION OF BITUMINOUS MATERIALS

(A. S. T. M. standard method (4), serial designation D 5-25, except as indicated. See p. 1)

1. Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically 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, 25° C. (77° F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

APPARATUS

2. The container for holding the material to be tested shall be a flat-bottom, cylindrical dish, 55 millimeters (2 $\frac{3}{8}$ inches) in diameter and 35 millimeters (1 $\frac{3}{8}$ inches) deep.⁴¹

3. The needle (fig. 27) for this test shall be made from a cylindrical steel rod approximately 50.8 millimeters (2 inches) long, and having a diameter of 1 to 1.02 millimeters. This shall be symmetrically tapered at one end to a cone approximately 6.35 millimeters (one-fourth inch) in height and whose angle shall be within the range of 8° 40' and 9° 40'. After tapering, the point shall be "blunted" by grinding off to a truncated cone, the smaller base

⁴¹ This requirement is fulfilled by the American Can Co.'s Gill-syle ointment box, deep pattern, 3-ounce capacity.

of which shall be from 0.14 to 0.16 millimeters in diameter. The finished needle shall be hardened and highly polished. A *Roberts No. 2 parabola needle checked against the standard needle may be used.*⁴²

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.

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.

6. The transfer dish for 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.

PREPARATION OF SAMPLE

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

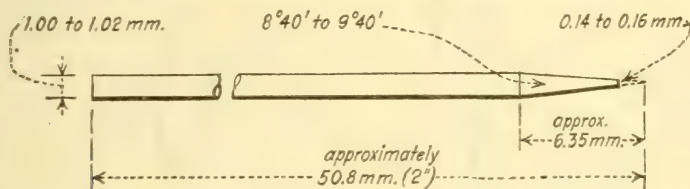


Fig. 27.—Needle for penetration test

It shall then be placed in the water bath along with the transfer dish and allowed to remain one hour.

PROCEDURE

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 to completely cover the container. The transfer dish containing the sample shall then 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-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-gram weight, 60 seconds.

At 46.1° C. (115° F.) 50-gram weight, 5 seconds.

⁴²The U. S. Bureau of Standards will measure and certify the accuracy of penetration needles in accordance with the tolerances given.

50. METHOD OF FLOAT TEST FOR BITUMINOUS MATERIALS

(A. S. T. M. standard method (4), serial Designation D 139-27)

APPARATUS

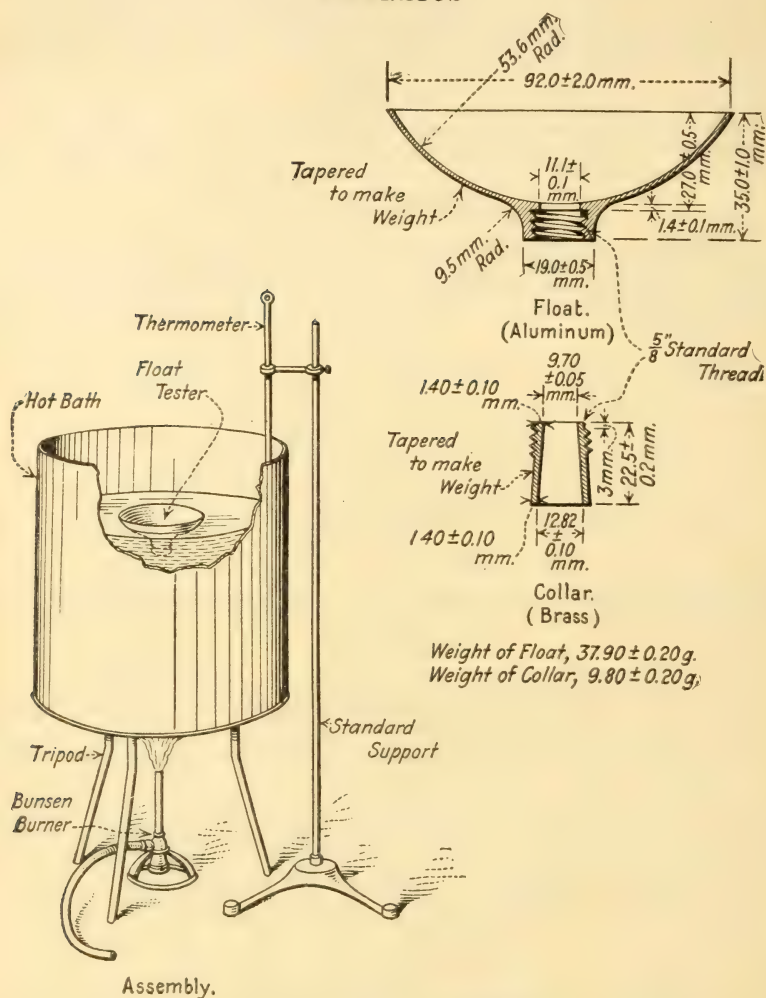


FIG. 28.—Float-test apparatus

1. The float (fig. 28) shall be made of aluminum or aluminum alloy and shall be in accordance with the following requirements:

	Minimum	Normal	Maximum
Weight of float, grams.....	37.7	37.9	38.1
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, millimeters.....	1.3	1.4	1.5
Diameter of opening, millimeters.....	11.0	11.1	11.2

2. The collar (fig. 28) shall be made of brass and shall be in accordance with the following requirements:

	Minimum	Normal	Maximum
Weight of collar, grams.....	9.60	9.80	10.00
Over-all height of collar, millimeters.....	22.3	22.5	22.7
Inside diameter at bottom, millimeters.....	12.72	12.82	12.92
Inside diameter at top, millimeters.....	9.65	9.70	9.75

The top of the collar shall screw up tightly against the lower side of the shoulder.

3. The assembled float and collar, with the collar filled flush with the bottom and weighted to a total weight of 53.2 grams, shall float upon water with the rim 8.5 ± 1.5 millimeters above the surface of the water. Dimensions of the apparatus additional to those required above are given in Figure 28.

4. The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either centigrade or Fahrenheit degrees as specified, the ranges being -2° to $+80^{\circ}$ C. or $+30^{\circ}$ to $+180^{\circ}$ F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and subdivision: -2° to $+80^{\circ}$ C. in 0.2° C. or $+30^{\circ}$ to $+180^{\circ}$ F. in 0.5° F.

Total length: 378 to 384 millimeters (14.88 to 15.12 inches).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 millimeters (0.24 to 0.28 inches).

Bulb: Corning normal or equally suitable thermometric glass. Length, 9 to 14 millimeters (0.35 to 0.55 inch). Diameter, 4.5 to 5.5 millimeters (0.18 to 0.22 inch).

Distance to 0° C. or 32° F. line from bottom of bulb: 75 to 90 millimeters (2.95 to 3.54 inches).

Distance to 80° C. or 176° F. line from top of thermometer: 30 to 45 millimeters (1.18 to 1.77 inches).

Expansion chamber: To permit heating the thermometer at least 50° C. (90° F.) above highest temperature on scale.

Filling above mercury: Nitrogen gas.

Top finish: Glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. Each whole degree centigrade or Fahrenheit line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° C. or 5° F.

Immersion: Total.

Special marking: "A. S. T. M. low S. P.," a serial number and the manufacturer's name or trade-mark shall be etched on the thermometer.

Scale error: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.2° or 0.4° F.

Standardization: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 20° C. or 40° F.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A. S. T. M. low S. P., -2 to $+80^{\circ}$ C." or "A. S. T. M. low S. P., $+30$ to $+180^{\circ}$ F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply: The total length is the over-all length of the finished instrument. The diameter is that measured with a ring gauge. The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing. The top of the thermometer is the top of the finished instrument.

5. The diameter of the bath and the depth of water shall be at least 185 millimeters.

PREPARATION OF SAMPLE

6. 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 completely melted at the lowest possible temperature that will bring it to a sufficiently fluid condition for pouring, excepting creosote-oil

residues, which shall be mixed and poured at a temperature of 100° to 125° C. It shall be stirred thoroughly until it is homogeneous and free from air bubbles. The sample shall then be poured into the collar in any convenient manner until slightly more than level with the top.

Asphalt and asphalt products.—Asphalt and asphalt products shall be cooled to room temperature, placed in water maintained at 5° C. for five 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., $\pm 1^\circ$ C., and left in this bath for at least 15 minutes.

Tar products.—Tar products shall be immediately immersed in ice water maintained at 5° C. for five 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., $\pm 1^\circ$ C., and left in this bath for at least 15 minutes.

PROCEDURE

7. (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 30 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 one 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 placing the apparatus on the water and 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.

51. METHOD OF TEST FOR DUCTILITY OF BITUMINOUS MATERIALS

(A. S. T. M. tentative standard method (5), serial designation D 113-26 T)

1. The ductility of an asphalt cement or semisolid bitumen is measured by the distance to which it will elongate before breaking when two ends of a briquet of the material are pulled apart at a specified rate of speed and at a specified temperature, which for a normal test shall be 5 centimeters per minute at 25° C. (77° F.).

APPARATUS

2. The mold shall be as shown in Figure 29. 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:

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

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.

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 rate of speed of 5 centimeters per minute.

PROCEDURE

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 *aa'* 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 be left to cool to room temperature and then placed in water maintained at 25° C. (77° 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 one and one-half hours, when the briquet shall be removed from the plate and the side pieces detached.

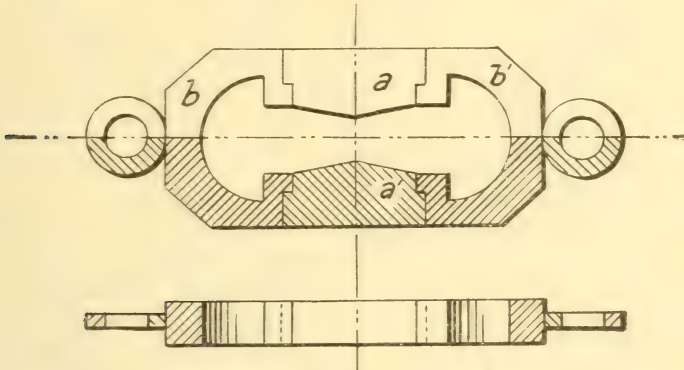


FIG. 29.—Ductility mold

7. The rings at each end of the clip shall then be attached to the pins or hooks in the ductility machine and the two clips pulled apart at a uniform rate of 5 centimeters per minute (± 5 per cent) until the briquet ruptures. The distance through which the clips have been pulled to produce rupture shall then be measured in centimeters. While the test is being made, the water in the tank of the ductility machine shall cover the sample by at least 2.5 centimeters and shall be kept continuously at a temperature within 0.5° C. of 25° C. (77° F.).

A normal test is one in which the material between the two clips pulls out to a point or thread until rupture occurs at the point where the thread has practically no cross-sectional area. The average of three normal tests shall be taken and reported as the ductility of the sample.

If a normal test is not obtainable on three successive tests, the ductility should be reported as being unobtainable under the conditions of the test.

52. METHOD OF TEST FOR DISTILLATION OF TARS AND TAR PRODUCTS

(A. S. T. M. tentative standard method (5), serial designation D 20-27 T)

APPARATUS

1. The apparatus consists of a flask, condenser tube, shield, receivers, and thermometers, as specified in sections 2 to 6.

2. Flask.—The distillation flask, Figure 30, shall be a side-neck distilling flask, having the following dimensions:

	Millimeters
Diameter of bulb, outside.....	86 ± 1.5
Diameter of neck, inside.....	22 ± 1.0
Diameter of tubulature, inside.....	10 ± 0.5
Length of neck.....	43 ± 1.0
Distance, top of neck to tubulature.....	25 ± 1.0
Length of tubulature.....	220 ± 5.0
Angle of tubulature.....	degrees 75 ± 1.0

3. Condenser tube.—The condenser tube shall be a suitable form of tapered glass tubing of the following dimensions:

Outside diameter of small end.....	12.5 millimeters; permissible variation ± 1.5
Outside diameter of large end.....	28.5 millimeters; permissible variation ± 3.0
Length.....	360.0 millimeters; permissible variation ± 4.0
Length of tapered part.....	100.0 millimeters; permissible variation ± 5.0

4. Shield.—A galvanized-iron shield, lined with $\frac{1}{8}$ -inch asbestos, of the form and dimensions shown in Figure 31, shall be used to protect the flask from air currents and to prevent radiation. The cover (top) may be of transit board, galvanized iron lined with $\frac{1}{8}$ -inch asbestos, or of other suitable insulating material.

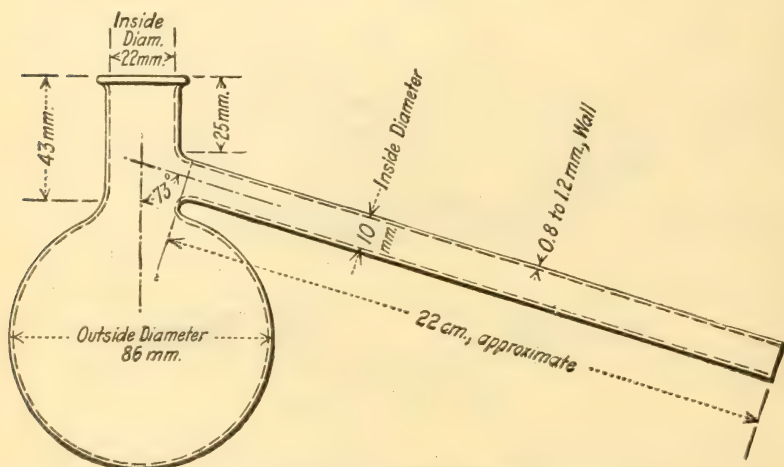


FIG. 30.—Distillation flask

5. Receiver.—The distillates will be collected in tared Erlenmeyer flasks having a capacity of 50 to 100 milliliters.

6. Thermometer.—The thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated in either centigrade or Fahrenheit degrees as specified, the range being from 0° to 400° C. or 30° to 760° F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and subdivision: 0° to 400° C. in 1° C. or 30° to 760° F. in 2° F.

Total length: 378 to 384 millimeters (14.88 to 15.12 inches).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 millimeters (0.24 to 0.28 inch).

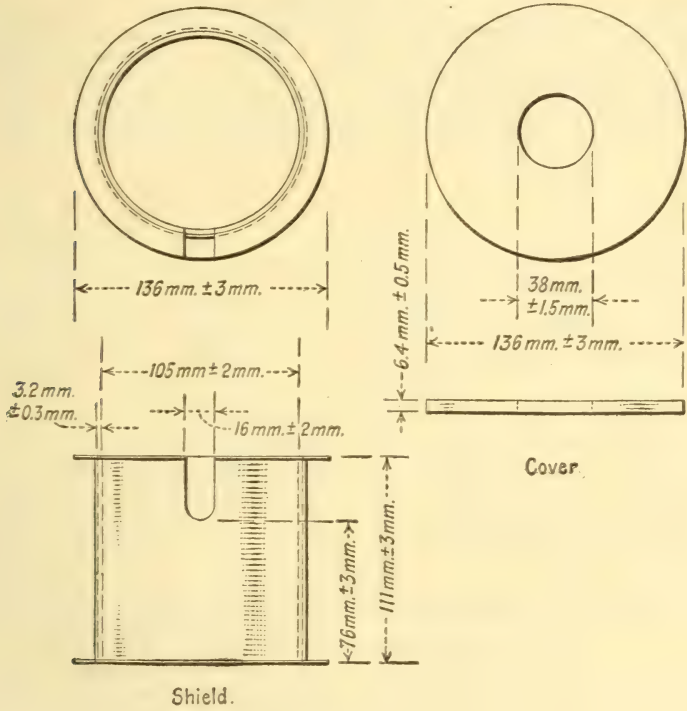
Bulb: Corning normal or equally suitable thermometric glass. Length, 10 to 15 millimeters (0.39 to 0.59 inch). Diameter, 5 to 6 millimeters (0.20 to 0.24 inch).

Distance to 0° C. or 32° F. line from bottom of bulb: 25 to 35 millimeters (0.98 to 1.38 inches).

Distance to 400° C. or 752° F. line from top of thermometer: 30 to 45 millimeters (1.18 to 1.77 inches).

Filling above mercury; Nitrogen gas.

Top finish: Glass ring.
 Graduation: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.
 Immersion: Total.
 Special marking: "A. S. T. M. high distillation," a serial number, and the manufacturer's name or trade-mark shall be etched on the stem.
 Scale error: The error at any point of the scale up to 370° C. or 700° F. when the thermometer is standardized as provided below shall not exceed 1° C. or 2° F.
 Standardization: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point, and at temperature intervals of approximately 50° C. or 100° F. up to 370° C. or 700° F.



Shield.
*Flanged Open-End Cylinder
 Made of 22 gage Galvanized
 Iron with 1/8-in. Asbestos Lining
 Riveted to Metal.*

FIG. 31.—Shield

Test for permanency of range: After being subjected to a temperature between 360° and 370° C. or 680° and 700° F. for 24 hours, the accuracy shall be within the limit specified.

Case: The thermometer shall be supplied in a suitable case, on which shall appear the marking: "A. S. T. M. high distillation, 0° to 400° C." or "A. S. T. M. high distillation, 30° to 760° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications, the following definitions apply: The total length is the over-all length of the finished instrument. The diameter is that measured with a ring gage. The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing. The top of the thermometer is the top of the finished instrument.

PREPARATION OF SAMPLE

7. 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.

8. The material may be tested for distillation without dehydration, if water is present not to exceed 2 per cent. If water is present in excess of 2 per cent, the bituminous material shall be dehydrated before distillation in accordance with the method for dehydration as given in sections 2 to 4 of the standard methods of sampling and analysis of creosote oil (serial designation D 38-27) of the American Society for Testing Materials (see p. 93).

PROCEDURE

9. (a) The flask shall be supported on a tripod or rings over two sheets of 20-mesh gauze, 150 millimeters square, as shown in Figure 32. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the neck with the top of the bulb level with the lowest point of juncture of the tubulature and neck of the flask.

(b) The axis of the flask through the neck shall be vertical.

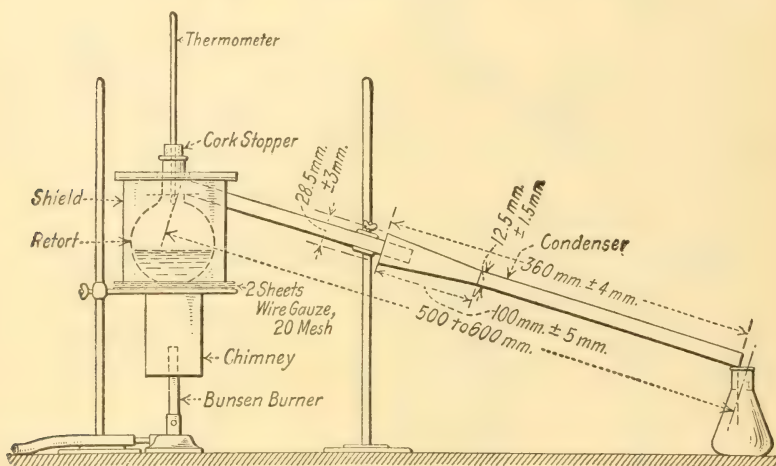


FIG. 32.—Distillation apparatus assembly

(c) The distance from the bulb of the thermometer to the outlet end of the condenser tube shall be not more than 600 nor less than 500 millimeters. The burner should be protected from draughts by a suitable shield or chimney. (Fig. 32.)

10. (a) One hundred grams (100 grams \pm 0.1 gram) of the sample shall be weighed into the flask, the apparatus assembled and heat applied so that the first drop comes over in from 5 to 15 minutes. The distillation shall be conducted at the rate of between 50 and 70 drops per minute and the distillate collected in weighed receivers. The condenser tube shall be warmed whenever necessary to prevent accumulation of solid distillates. The fraction shall be collected at the points designated by the specifications. The receivers shall be changed when the thermometer indicates the maximum temperature for each fraction. When the maximum specified temperature of the test is indicated by the thermometer, the flame shall be removed and any oil which has condensed in the condenser tube shall be drained into the last fraction.

(b) The residue shall remain in the flask with the cork and thermometer in position until no vapors are visible and it shall then be weighed. If tests of the residue are required, the flask shall then be inclined so that the residue will flow around the sides, thus collecting any condensed vapors that may be on the

sides of the flask, after which the residue shall be poured into a suitable receptacle and covered. If the residue becomes so cool that it can not be poured readily from the flask, it shall be reheated to a temperature not exceeding 125° C. by holding the bulb of the flask in a suitable bath 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.

(c) 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.

(d) The results of the distillation test shall be reported in percentages by weight of water-free material. The following fractions are usually reported:

Up to 170° C.
170° to 235° C.
235° to 270° C.
270° to 300° C.
Residue

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

(A. S. T. M. standard method (4), serial designation D 36-26, except as indicated. See p. 1)

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.

APPARATUS

2. The apparatus shall consist of the following:

(a) A brass ring 15.875 millimeters (five-eighths inch) in inside diameter and 6.35 millimeters (one-fourth inch) deep; thickness of wall, 2.38 millimeters (three thirty-seconds inch); permissible variation on inside diameter and thickness of ring 0.25 millimeter (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=0.072 inch). (Fig. 33.)

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

(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.)

(d) A thermometer which shall conform to the following requirements. These specifications cover a special thermometer graduated in either centigrade or Fahrenheit degrees as specified, the ranges being -2 to +80° C. or +30 to +180 F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and subdivision: -2° to +80° C. in 0.2° C. or +30° to +180° F. in 0.5° F.

Total length: 378 to 384 millimeters (14.88 to 15.12 inches).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 millimeters (0.24 to 0.28 inch).

Bulb: Corning normal or equally suitable thermometric glass. Length, 9 to 14 millimeters (0.35 to 0.55 inch). Diameter, 4.5 to 5.5 millimeters (0.18 to 0.22 inch).

Distance to 0° C. or 32° F. line from bottom of bulb: 75 to 90 millimeters (2.95 to 3.54 inches).

Distance to 80° C. or 176° F. line from top of thermometer: 30 to 45 millimeters (1.18 to 1.77 inches).

Expansion chamber: To permit heating the thermometer at least 50° C. (90° F.) above highest temperature on scale.

Filling above mercury: Nitrogen gas.

Top finish: Glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. Each whole degree centigrade or Fahrenheit line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° C. or 5° F.

Immersion: Total.

Special marking: "A. S. T. M. low S. P.," a serial number and the manufacturer's name or trade-mark shall be etched on the thermometer.

Scale error: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.2° C. or 0.4° F.

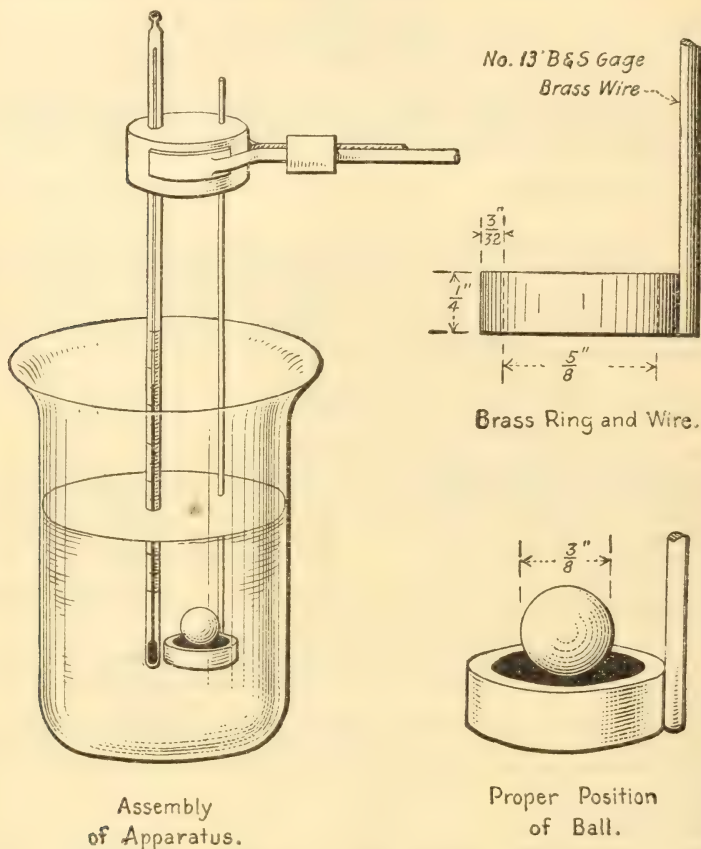


FIG. 33.—Apparatus for ring-and-ball method

Standardization: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 20° C. or 40° F.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A. S. T. M. low S. P., -2° to +80° C.," or "A. S. T. M. low S. P., +30° to +180° F.," according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply: The total length is the over-all length of the finished instrument. The diameter is that measured with a ring gauge. The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing. The top of the thermometer is the top of the finished instrument.

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.

PROCEDURE

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

4. Fill the glass vessel to a depth of substantially 8.25 centimeters (3.25 inches) with freshly boiled, distilled water at 5° C. (41° F.). Suspend the ring containing the sample 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 centimeters (2 inches) below the surface of the water. Place the ball in the water but not on the specimen. Suspend the thermometer so that the bottom of the bulb is level with the bottom of the ring and within 0.635 centimeter (one-fourth inch) but not touching the ring. Maintain the temperature of the water at 5° C. (41° F.) for 15 minutes. With suitable forceps place the ball in the center of the upper surface of the bitumen in the ring, thus completing the assembly as in Figure 33.

NOTE.—The illustration shows only one ring, but more than one 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. No correction shall be made for emergent stem.

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

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

8. The same method as given under (A) shall be employed except that U. S. P. glycerin shall be used instead of water, and the starting point of the glycerin bath shall be 32° C. (89.6° F.). The bath shall be brought to this temperature and thoroughly agitated, then the apparatus and specimens shall be placed in the bath, which shall be maintained under agitation at the starting temperature for 15 minutes, after which the assembly shall be completed by placing the ball on the center of the specimen and the test carried on as in (A). In applying the heat, the ring apparatus shall be placed off the center of the container and the burner placed midway between the center and edge of the beaker away from the specimen.

The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either centigrade or Fahrenheit degrees as specified, the ranges being 30° to 160° C. or 85° to 320° F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and subdivision: 30° to 160° C. in 0.5° C. or 85° to 320° F. in 1° F.

Total length: 378 to 384 millimeters (14.88 to 15.12 inches).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 millimeters (0.24 to 0.28 inch).

Bulb: Corning normal or equally suitable thermometric glass. Length, 9 to 14 millimeters (0.35 to 0.55 inch). Diameter, 4.5 to 5.5 millimeters (0.18 to 0.22 inch).

Distance to 30° C. or 86° F. line from bottom of bulb: 75 to 90 millimeters (2.95 to 3.54 inches).

Distance to 160° C. or 320° F. line from top of thermometer: 30 to 45 millimeters (1.18 to 1.77 inches).

Expansion chamber: To permit heating the thermometer at least 50° C. (90° F.) above the highest temperature on the scale.

Filling above mercury: Nitrogen gas.

Top finish: Glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. Each whole degree centigrade line or the first and each succeeding 5° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 5° C. or 10° F.

Immersion: Total.

Special marking: "A. S. T. M. high S. P.," a serial number and the manufacturer's name or trade-mark shall be etched on the thermometer.

Scale error: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.3° C. or 0.5° F.

Standardization: The thermometer shall be standardized, immersed in the testing bath to the top of the mercury column, at intervals of approximately 40° C. or 70° F.

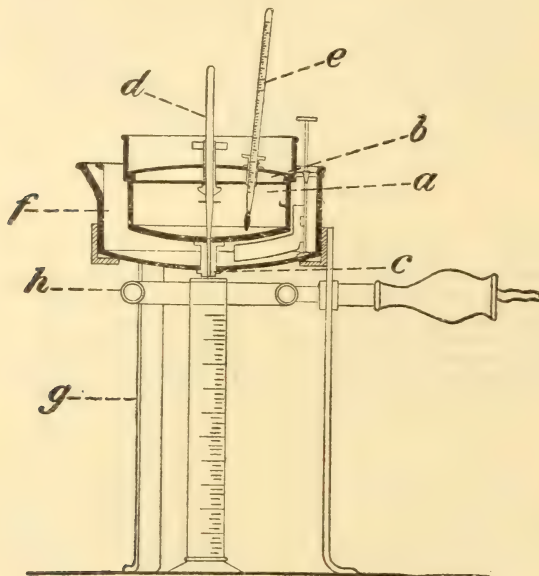


FIG. 34.—Engler viscosimeter

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A. S. T. M. high S. P., 30 to 160° C.," or "A. S. T. M. high S. P., 85 to 320° F.," according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply: The total length is the over-all length of the finished instrument. The diameter is that measured with a ring gauge. The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing. The top of the thermometer is the top of the finished instrument.

PRECAUTIONS

9. The use of freshly boiled distilled water is essential, as otherwise air bubbles may form on the specimen and effect 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.

ACCURACY

10. The limit of accuracy of the test is $\pm 0.5^{\circ}$ C. (0.9° F.).

54. METHOD OF TEST FOR SPECIFIC VISCOSITY

1. 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 34, 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.

2. 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 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.

3. 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:

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

55. METHOD OF TEST FOR WATER IN PETROLEUM PRODUCTS AND OTHER BITUMINOUS MATERIALS

(A. S. T. M. standard method (4), serial designation D 95-27)

1. This method of test determines water existing in a sample of bituminous material by distilling the sample with a volatile solvent. This method is suitable for a variety of materials but is especially applicable to petroleum, fuel oil, road oil, coal tar, water-gas tar, coke-oven tar, and other petroleum products or bituminous materials.

APPARATUS

2. The apparatus shall consist of a metal still or glass flask, heated by suitable means and provided with a reflux condenser discharging into a trap connected to the still or flask. The trap serves to collect and measure the condensed water and to return the solvent to the still.

The type of distilling apparatus used is not an essential feature of this method, but glass has been generally used for petroleum products and the metal still for road materials and tars.

3. (a) The metal still (fig. 35, a) shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the top to which the head is tightly attached by means of a clamp. The head shall be of metal, preferably of brass or copper, and be provided with a tubulation 1 inch in inside diameter.

(b) The glass flask (fig. 35, b) shall be of the short-neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 cubic centimeters.

4. The burner used with the metal still shall be a ring gas burner, 4 inches (100 millimeters) in inside diameter. With the glass flask, an ordinary gas burner or electric heater may be used as the source of heat.

5. The condenser shall be of the water-cooled, reflux, glass-tube type, having a condenser jacket not less than 400 millimeters (15¾ inches) in length with an inner tube 12 to 16 millimeters (one-half to five-eighths inch) in diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 60°.

6. The trap shall be made of well-annealed glass constructed in accordance with Figure 38 c, and shall be graduated as shown from 0 to 10 cubic centimeters in 0.1 cubic centimeter divisions. The error of any indicated capacity shall not be greater than 0.05 cubic centimeter.

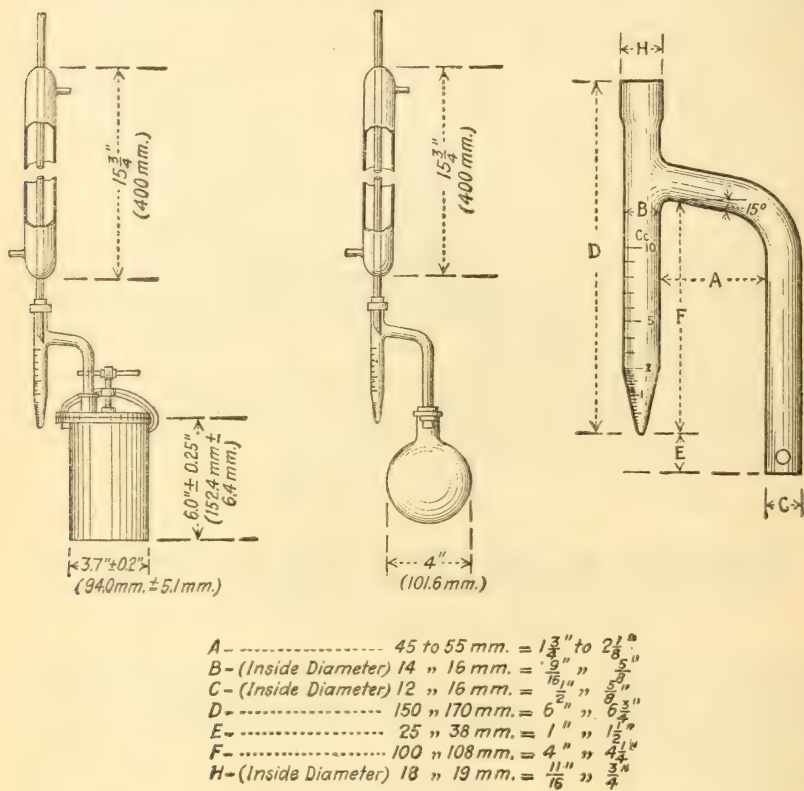


FIG. 35.—Apparatus for use in tests for water in petroleum products and other bituminous materials

The outside diameters should be preferably 2.5 to 3.5 millimeters (three thirty-seconds to one-eighth inch) greater than the inside diameters specified.

7. (a) The solvent used when testing petroleum products or bituminous materials derived from petroleum shall be gasoline free from water and shall conform to the following distillation requirements, determined in accordance with the standard method of test for distillation of gasoline, naphtha, kerosene, and similar petroleum products (serial designation D 86) of the American Society for Testing Materials (4) :

Five per cent shall distill at a temperature not below 194° F. (90° C.) nor above 212° F. (100° C.).

Ninety per cent shall distill below 410° F. (210° C.).

(b) The solvent used when testing bituminous materials derived from coal tar, water-gas tar, etc., shall be a coal-tar naphtha or a light oil and shall conform to the following distillation requirements, determined in accordance with the standard method of test for distillation of gasoline, naphtha, kerosene, and similar petroleum products (serial designation D 86) of the American Society for Testing Materials (4):

Ninety-eight per cent shall distill between 248° F. (120° C.) and 482° F. (250° C.).

SAMPLE

8. The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this requirement shall not be permitted.

NOTE.—The difficulties in obtaining proper representative samples for this determination are unusually great, so that the importance of sampling can not be too strongly emphasized.

PROCEDURE

9. When the sample to be tested contains less than 10 per cent of water, exactly 100 cubic centimeters of the material to be tested shall be placed into the still or flask and thoroughly mixed with an equal volume of solvent by swirling, proper care being taken to avoid any loss of material. If the material is measured by volume, an accurate 100-cubic centimeter graduated cylinder shall be used and the contents transferred to the still by rinsing with one 50-cubic centimeter portion of solvent followed by two successive 25-cubic centimeter portions of solvent, the cylinder being allowed to drain each time. When the sample to be tested contains more than 10 per cent of water, the volume of material used shall be decreased to that which will yield somewhat less than 10 cubic centimeters of water.

NOTE.—In special cases where the water content exceeds 10 per cent and it is not desirable to reduce the size of the sample to that which will yield somewhat less than 10 cubic centimeters of water, a distilling-tube receiver graduated from 0 to 25 cubic centimeters may be used. This tube shall be graduated from 0 to 2 cubic centimeters in 0.1 cubic centimeter, from 2 to 5 cubic centimeters in 0.2 cubic centimeter, and from 5 to 25 cubic centimeters in 0.5 cubic centimeter.

The connections between the still or flask, trap, and condenser shall be made by means of tight-fitting corks as shown in Figure 35. When the metal still is used, a heavy paper gasket moistened with the solvent shall be inserted between the lid and flange before attaching the clamp. A loose cotton plug shall be inserted in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

Heat shall then be applied and so regulated that the condensed distillate falls from the end of the condenser at the rate of from 2 to 5 drops per second. The ring burner used with the metal still should be placed about 3 inches above the bottom of the still at the beginning of the distillation and gradually lowered as the distillation proceeds.

The distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the tray. This operation usually requires less than an hour. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

10. The volume of condensed water measured in the trap at room temperature multiplied by 100 and divided by the volume of the sample used shall be the percentage of water and shall be reported as "——— per cent water by volume, A. S. T. M. method."

ACCURACY

11. The accuracy to be expected with this method is that duplicate determinations of water should not differ from each other by more than one division on the trap.

56. METHOD OF TEST FOR PERCENTAGE OF RESIDUE OF DESIRED PENETRATION

1. 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.

2. Ordinarily a residue shall be considered as satisfactorily obtained on the basis of 100 penetration being specified when its penetration is within 25 points of that desired. When it is necessary to determine more precisely the per cent of reesidue for a specified penetration, such percentage shall be computed by interpolation between percentages of two residues, one having a penetration greater, and one having a penetration lower than that specified.

57. CALCIUM-CHLORIDE METHOD FOR DETERMINATION OF PERCENTAGE OF WATER IN BITUMINOUS EMULSIONS

(Not applicable to clay 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 calcium 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.

$$\text{Calculations: Per cent water} = \frac{A-B}{C} \times 100$$

A = volume of supernatant water,

B = volume of 10 per cent calcium chloride, solution originally used,

C = weight of emulsion taken for water determination.

58. METHOD FOR EXAMINATION OF BITUMINOUS MIXTURES

CENTRIFUGAL METHOD

1. 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-quarter inch in diameter or 200 grams of aggregate with all particles smaller than three-quarter 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 extracted, 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 manilla 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.

2. 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.

3. 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 bitumen that are necessary are then made upon it.

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

5. 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.

HOT EXTRACTION METHOD

6. 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.

7. 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

8. 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 tester. This method is based on decantation of the supernatant solvent. (The sample shall consist of not less than 25 grams.) 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 shall be made as given under the centrifugal method.

9. Mechanical analysis of the extracted aggregate shall be conducted in accordance with method No. 30.

59. METHODS OF TESTING BITUMINOUS EMULSIONS

(A. S. T. M. tentative standard method (5), serial designation D 244-26 T, except as indicated. See p. 1)

1. For testing purposes emulsions shall be classified as follows:
 - I. Emulsified light oils or liquid petroleum products intended for dust-laying purposes.
 - II. Emulsified asphaltic materials the asphalt content of which is of a consistency suitable for construction or repair.
 - (a) Containing little or no mineral matter.
 - (b) Containing appreciable quantities of mineral matter.

METHOD FOR DETERMINING MISCIBILITY OF BITUMINOUS EMULSIONS WITH WATER

(Applicable to both Classes I and II)

2. To about 50 cubic centimeters of the emulsion shall be gradually added about 150 cubic centimeters of distilled water, stirring the mixture while adding the water. The temperature is not important, but should be between 21 and 25° C. (70 and 79° F.). The mixture shall be allowed to stand for two hours and then examined for any appreciable separation.

STONE COATING TEST

(Applicable only to emulsions of Class II (a))

3. Four hundred and sixty-five grams of clean stone (one-fourth to three-fourths inch in size, not more than 5 per cent passing a one-fourth-inch screen and 100 per cent passing a three-fourth inch screen), see tentative specifications for commercial sizes of broken stone and broken slag for highway con-

struction (serial designation D 63-23 T) of the American Society for Testing Materials (5), of either trap or hard limestone shall be placed in a No. 10 sieve, drenched and washed with distilled water, allowed to drain for three minutes, and transferred to a suitable pan in which the mixture is to be made. (A 6-inch hemispherical iron dish has been found suitable and satisfactory for this purpose.) Thirty-five grams of the emulsion shall then be added to and mixed vigorously with the stone for three minutes. (A steel spatula is satisfactory for mixing.) There shall be no signs of appreciable separation of the asphalt contained in the emulsion during the three-minute mixing period.

METHOD FOR DETERMINATION OF WATER, OIL DISTILLED WITH WATER, AND PREPARATION OF ASPHALTIC BASE FOR FURTHER TESTS

(A) METHOD APPLICABLE TO EMULSIONS OF CLASS II (a) ONLY⁴³

4. The apparatus shall consist of a copper still 6 inches by 3½ inches in inside diameter with an air-adjustable ring burner to fit the still. The still shall be provided with a connecting tube of approximately 12-millimeter diameter glass tubing, a tin shield, a condenser trough, a condenser tube, and a suitable graduated cylinder. A thermometer 0° to 400° C. shall be provided conforming to the requirements for the thermometer specified in section 6 of the tentative method of test for distillation of creosote oil (serial designation D 246-27 T) of the American Society for Testing Materials (5). The apparatus shall be set up as shown in Figure 36. The still shall be charged with 200 grams of the well-mixed sample. An oiled-paper gasket shall be applied to the flange at the top of the still and the cover clamped on securely. The thermometer shall be placed in position so that the tip of the bulb is one-half inch from the inside bottom of the still. A gentle heat shall be applied to the top of the still by the ring burner. Just sufficient heat shall be applied to the connecting tube to prevent condensation of water therein.

The heat shall be applied to the extreme upper part of the still until practically all of the water has distilled over, or until a temperature of 121° C. (250° F.) has been reached. The ring burner shall then be lowered very gradually to a position midway between the top and bottom of the still and held there until the temperature is about 176° C. (349° F.). The ring burner shall then be lowered more rapidly to within one-fourth inch of the bottom of the still and the temperature then raised to 260° C. (500° F.) and maintained for 15 minutes. This period of heating is important to insure a smooth homogeneous residue in the still.

The cover shall then be removed from the still and the hot residue poured into a ductility mold and into a standard 3-ounce tin box for the penetration test, the receptacles being left uncovered. While cooling any excess shall be poured into suitable containers for any additional tests desired.

Any water or oil remaining in the condenser tube shall be forced out carefully into the graduated cylinder by means of a rubber stopper fixed on the end of a long rod.

The distillation should be completed in not less than one hour and in not more than one and one-fourth hours.

If the residue is not homogeneous and appears granular, it shall be rejected and the distillation repeated upon a fresh sample, maintaining the temperature at 260° C. (500° F.) for slightly more than 15 minutes.

The number of cubic centimeters of water divided by 2 is the percentage of weight of water.

The weight of residue remaining in the still divided by 2 is the percentage of nonvolatile matter under the conditions of this test.

The sum of the percentage of water and nonvolatile matter deducted from 100 represents distilled oils.

(B) METHOD APPLICABLE TO EMULSIONS OF CLASS II (b)

Water.—The water shall be determined by the distillation method above described.

⁴³ Method No. 57 (page 88) may be used as an alternate for the method of determining water herein specified.

Oils.—Two 50-gram samples of well-mixed emulsion shall be placed in a standard 3-ounce tin box, in an oven at 163° C. (325° F.) for five hours, and the loss determined. If the loss is greater than the percentage of water (see preceding paragraph) it would indicate the presence of volatile oils and the difference in the two determinations would closely approximate the percentage of volatile oil.

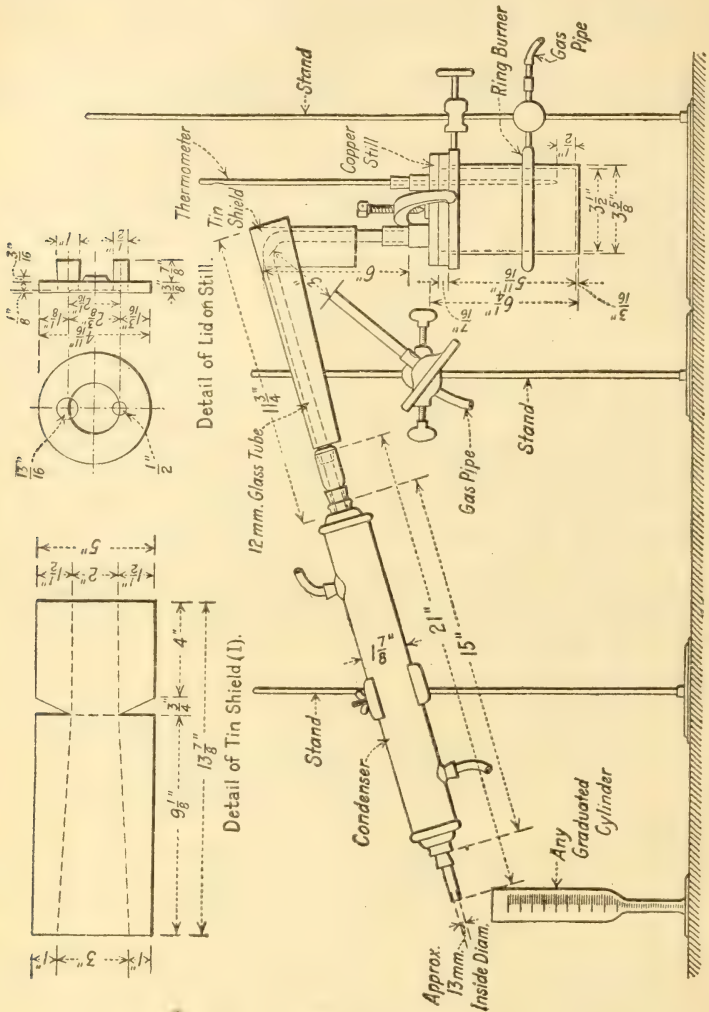


FIG. 36.—Assembly for distillation test for bituminous emulsions

The residue of the 50-gram sample shall be dissolved in benzol, filtered, and the filtrate evaporated on a water bath until practically all the benzol is evaporated, then placed in an oven at 105° C. (221° F.) for 30 minutes and finally heated on a hot plate to 121° C. (250° F.) with constant stirring. The consistency, ductility, or other tests desired are made upon this residue.

Mineral matter.—The mineral matter on the filter paper from the extraction shall be dried and weighed or a separate determination on a smaller sample of the original emulsion may be made.

60. METHODS OF SAMPLING AND ANALYSIS OF CREOSOTE OIL

(A. S. T. M. standard method (4), serial designation D38-27)

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 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

2. (a) Still.—A vertical, cylindrical copper still, with removable flanged top and yoke, of the form and approximate dimensions shown in Figure 37 shall be used.

(b) Thermometer.—The thermometer shall conform to the following requirements. These specifications cover a total immersion thermometer graduated in either centigrade or Fahrenheit degrees as specified, the ranges being 0° to 400° C. or 30° to 760° F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and subdivision: 0° to 400° C. in 1° C. or 30° to 760° F. in 2° F.

Total length: 378 to 384 millimeters (14.88 to 15.12 inches).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 millimeters (0.24 to 0.28 inch).

Bulb: Corning normal or equally suitable thermometric glass. Length, 10 to 15 millimeters (0.39 to 0.59 inch). Diameter, 5 to 6 millimeters (0.20 to 0.24 inch).

Distance to 0° C. or 32° F. line from bottom of bulb: 25 to 35 millimeters (0.98 to 1.38 inches).

Distance to 400° C. or 752° F. line from top of thermometer: 30 to 45 millimeters (1.18 to 1.77 inches).

Filling above mercury: Nitrogen gas.

Top finish: Glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

Immersion: Total.

Special marking: "A. S. T. M. High Distillation," a serial number, and the manufacturer's name or trade-mark shall be etched on the stem.

Scale error: The error at any point of the scale up to 370° C. or 700° F., when the thermometer is standardized as provided below, shall not exceed 1° C. or 2° F.

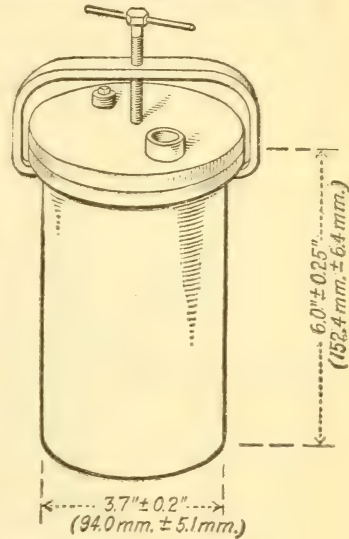


FIG. 37.—Copper still

Standardization: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 370° C. or 700° F.

Test for permanency of range: After being subjected to a temperature between 360° and 370° C. or 680° and 700° F. for 24 hours, the accuracy shall be within the limit specified.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A. S. T. M. high distillation, 0° to 400° C." or "A. S. T. M. high distillation, 30° to 760° F.," according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply: The total length is the over-all length of the finished instrument. The diameter is that measured with a ring gauge. The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing. The top of the thermometer is the top of the finished instrument.

NOTE.—Inasmuch as those dimensions given above which differ from the corresponding dimensions that appeared in methods D 38 until their revision in 1924 are of such a nature as to affect the facility of making but not the accuracy of the thermometer, any thermometer now in use complying with the former requirements of D 38 shall be accepted as satisfactory by producers and users of creosote oil; provided that all thermometers hereafter purchased shall be in accordance with the new requirements.

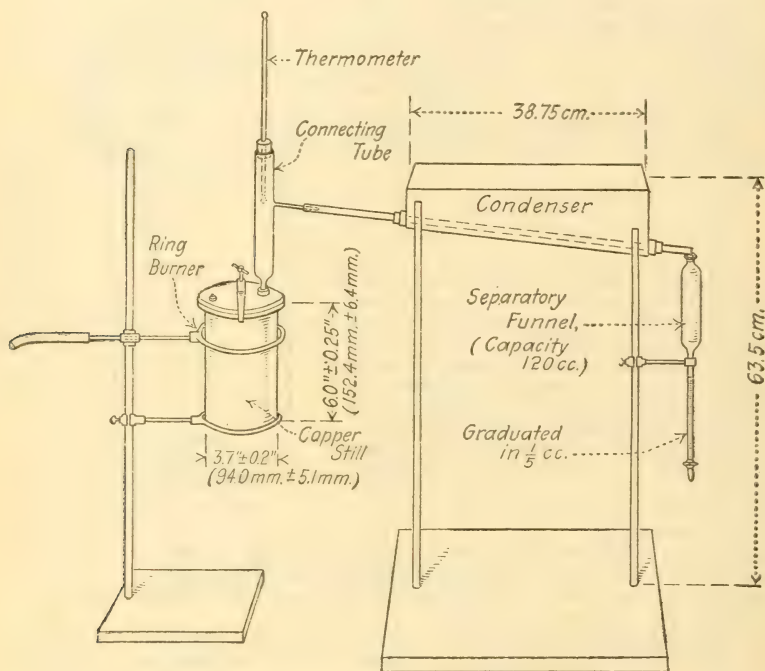


FIG. 38.—Assembled apparatus for water test

(c) Condenser.—A copper trough condenser shall be used, with straight-walled glass tube, having approximately the form and dimensions shown in Figure 38.

(d) Separatory funnel.—A separatory funnel of the form shown in Figure 38 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 38.

4. When any measurable amount of water is present in the distillate below 210° C. as determined in accordance with the tentative method of test for distillation of creosote oil (serial designation D 246-27 T) of the American Society for Testing Materials (5) (see page 98) 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:

Measure 200 cubic centimeter of oil in 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 off-take 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.

5. The amount of water may be determined in accordance with the standard method of test for water in petroleum products and other bituminous materials (serial designation D 95) of the American Society for Testing Materials ($\frac{1}{2}$). (See p. 85.)⁴⁴

MATTER INSOLUBLE IN BENZOL

6. (a) **Extractor.**—The extractor may be of the form shown in Figure 39 or any similar form in which the oil is subjected to direct washing by the boiling vapors of the solvent.

(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 by 80 R. A. 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 under surface 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.

7. 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 descending 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.

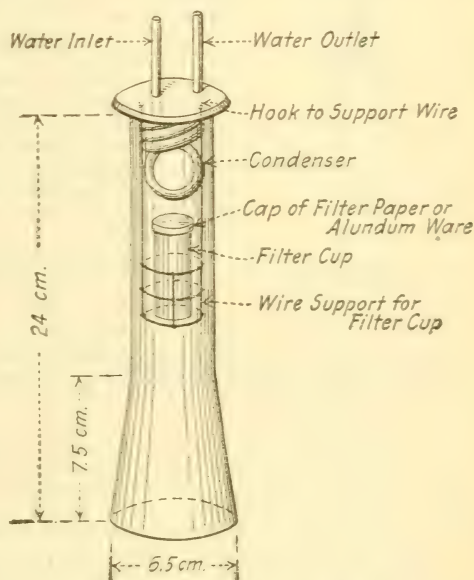


FIG. 39.—Extraction flask

⁴⁴This method is of particular service where the determination of water only is involved and the dehydrated material is not to be used subsequently for analysis.

SPECIFIC GRAVITY

8. (a) Hydrometer.—The hydrometer shall be of the form shown in Figure 40. It 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 to 1.08 and 1.07 to 1.15 will suffice.

(b) Cylinder.—The cylinder shall be of the form shown in Figure 41. It shall have the following dimensions:

Length-----	300 millimeters; permissible variation, 30 millimeters.
Diameter-----	32 millimeters; permissible variation, 3 millimeters.

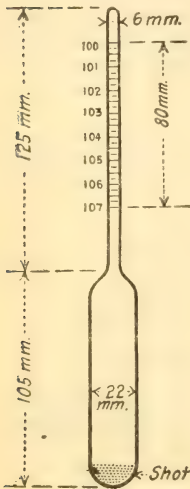


Fig. 40.—Hydrometer

9. 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

10. The distillation of creosote oil shall be determined in accordance with the tentative method of test for distillation of creosote oil (serial designation D 246-27 T) of the American Society for Testing Materials (5). (See p. 98.)

SPECIFIC GRAVITY OF FRACTIONS

11. 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.

(a) LIQUID FRACTIONS

12. The Westphal balance may be used.

13. 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

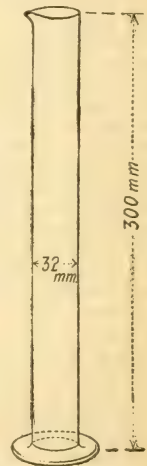


Fig. 41.—Cylinder

the balance be accurately made. The plummet can readily be made in the laboratory from a piece of ordinary glass tubing 7 millimeters in 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 blowpipe flame. The plummet shall have a length of about 55 to 60 millimeters over all and shall weigh between 10 and 12 grams.

(b) SOLID AND SEMISOLID FRACTIONS

14. A pan of the form shown in Figure 42, having the following approximate dimensions, may be used:

	Millimeters
Diameter of base.....	20
Diameter of top.....	25
Depth.....	12
Diameter of wire.....	1
Total weight.....	grams... 7

The pan and wires are made of platinum or nickel.

15. 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 fractions 315° to 355° C.

FLOAT TEST OF RESIDUE

16. The residue remaining in the retort after the distillation test shall remain until it reaches a temperature between 100° and 125° C. The consistency of the residue shall then be determined in accordance with the standard method of float test for bituminous materials (serial designation D-139) of the American Society for Testing Materials (4). (See p. 74.)

NOTE.—Care must be taken at the end of the distillation test to see that the vapor temperature as indicated on the thermometer does not rise above 355° C.; an excess temperature of only 1° or 2° C. at this point makes the float test invalid.

COKE RESIDUE

17. The coke residue of creosote oil shall be determined in accordance with the standard method of test for coke residue of creosote oil (serial designation D 168) of the American Society for Testing Materials (4). (See below.)



FIG. 42.—Platinum or nickel pan

61. METHOD OF TEST FOR COKE RESIDUE OF CREOSOTE OIL

(A. S. T. M. standard method (4), serial designation D 168-27)

1. Crucible.—The crucible shall be of platinum and shall have a capacity of 20 to 30 cubic centimeters. The cover of the crucible shall be of the inverted type, having a depth of about 1 centimeter, the wall tightly fitting the crucible except for a slight crease.

2. The residue resulting from the distillation test, carried out in accordance with the tentative method of test for distillation of creosote oil (serial designation D 246-27 T) of the American Society for Testing Materials (5) (see p. 98), shall be poured directly into the tared crucible or into a tin box wherein it may be heated on a water or steam bath, but not over a flame. About 1 gram of the residue shall be weighed into the covered crucible and then placed, on a platinum, nichrome, or fire-clay triangle over a Bunsen burner, with the bottom of the crucible 6 to 8 centimeters from the top of the burner. The burner flame shall be regulated to a height of 20 centimeters while burning free and the crucible shall be exposed to the full flame for seven minutes. A Meker burner may be used, or the crucible may be heated for several minutes in an electric

furnace. Whatever the method of heating employed, the temperature during the entire seven-minute period shall be not less than 950° C. and should be as near that temperature as possible. At the end of this period the flame shall be removed, the crucible transferred to a desiccator and permitted to cool, after which, it shall be weighed. The residue in the crucible after ignition shall be reported as "fixed carbon" (that is, coke).

3. The test shall be conducted in a part of the laboratory free from drafts.

4. The percentage of coke obtained in accordance with section 2 shall be calculated on the basis of the original sample of oil.

EXAMPLE.—With a retort distillation of 29 per cent of residue at 355° C., the residue containing 28 per cent of fixed carbon:

$$\text{Coke in oil} = \frac{29 \times 28}{100} = 8.1 \text{ per cent.}$$

62. METHOD OF TEST FOR DISTILLATION OF CREOSOTE OIL

(A. S. T. M. standard method (4), serial designation D 246-27 T)

This is the same method as given under method No. 52 (see p. 77), method of test for distillation of tars and tar products.

63. METHOD OF SAMPLING AND TESTING 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.

ANALYSES

3. The sample for analysis shall be obtained by boring into the end grain of all the blocks, according to Figure 43.

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.

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.

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.

64. METHOD OF 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.

2. The inspector shall determine the gauge of the metal, inside diameter, depth and spacing of the corrugations, width of lap, rivet spacing, weight per

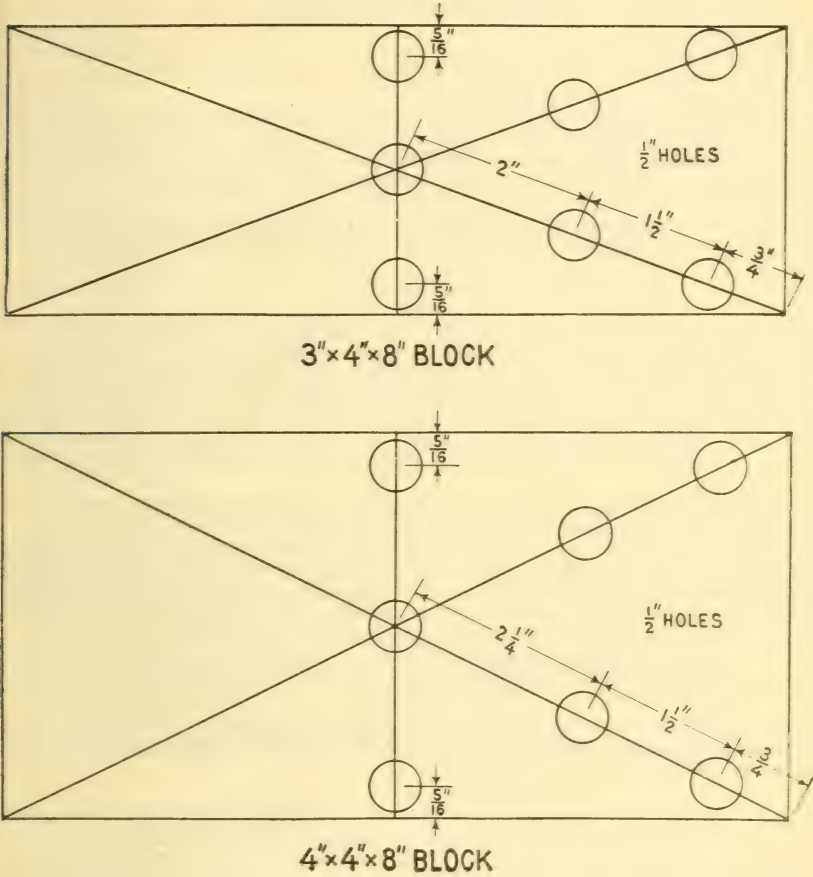


FIG. 43.—Method of boring wood blocks for sample. Borings made in end grain with 1/2-inch bit or drill. Three borings on center line made halfway through block; other borings through block.

linear foot of the finished pipe, condition of spelter coating and other requirements as called for under the specifications. 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, including rivets 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.

5. Chemical analysis to be conducted in accordance with the standard method of chemical analysis of plain carbon steel, A. S. T. M. A 33-24 (3).

65. METHODS FOR DETERMINING WEIGHT OF COATING ON ZINC-COATED ARTICLES

(A. S. T. M. standard method (3), serial designation A 90-24, except as indicated. See p. 1)

SHOP WEIGHING TESTS

Select a pickled and washed sheet. Stand the sheet on edge and allow to drain for two minutes. The water remaining on the sheet will then amount to approximately 0.01 pound per square foot.

Weigh this sheet with an accuracy of 0.05 pound, and subtract the weight of the water to obtain the real weight of the uncoated sheet. After galvanizing, reweigh the sheet. The increase in weight represents the coating.

Where greater accuracy is desired, wash, dry, and weigh 10 sheets: then coat and reweigh.

To determine the weight of coating in ounces per square foot, divide the weight of coating in ounces by the area in square feet in the sheet or sheets.

SAMPLING

(a) Sheets.—The laboratory samples for determining the average weight of coating upon a galvanized sheet shall be taken as follows:

A 2½-inch strip shall be cut transversely or diagonally across the middle of the sheet with its ends approximately 1 inch from each edge. Three samples, 2¼ by 2¼ inches, or of equivalent area, shall be accurately cut from the middle and the two ends of this strip. These samples shall be cleaned with gasoline or benzol, then with alcohol and dried thoroughly before proceeding with the test.

The weight of coating in grams on a 2¼ by 2¼ inch piece (5.08 square inches) is numerically equal to the coating in ounces per square foot.

(b) Wire.—Select either exactly 1 foot of wire or a definite length of wire as given in Table 8, depending upon the diameter of the wire after stripping. Clean the wire with gasoline or benzol, then with alcohol, and dry thoroughly.

When using exactly 12 inches of wire for the test and obtaining the loss of weight (weight of coating) in grams, the formula is as follows:

$$\frac{\text{Loss of weight in grams}}{7.422 \times \text{diameter in inches of stripped wire}} = \text{Ounces per square foot of actual surface.}$$

It is sometimes more convenient to use a length of wire, as given in Table 8, such that the loss in weight (weight of coating) in grams will numerically equal the ounces per square foot of actual surface.

TABLE 8.—Lengths of wire to give grams of coating which are numerically equal to ounces per square foot of actual surface

Size of wire B. W. G.	Diameter		Length for test		Size of wire B. W. G.	Diameter		Length for test	
	Inches	Inches	Inches	Centi-meters		Inches	Inches	Centi-meters	
0.....	0.340	4¾	12.1		10.....	0.134	12½	30.6	
1.....	.300	5½	13.7		11.....	.120	13½	34.2	
2.....	.284	5¼	14.5		12.....	.109	14½	37.7	
3.....	.259	6¼	15.9		13.....	.095	17	43.2	
4.....	.238	6¼	17.3		14.....	.083	19½	49.5	
5.....	.220	7½	18.7		15.....	.072	22½	57.0	
6.....	.203	7¼	20.2		16.....	.065	24½	63.2	
7.....	.180	9	22.8		17.....	.058	27½	70.8	
8.....	.165	9¼	24.9		18.....	.049	33	83.8	
9.....	.148	10½	27.7						

By multiplying by 2, the figures for coating in ounces per square foot of actual surface may be compared with the figures for coating in ounces per square foot on zinc-coated sheets.

HYDROCHLORIC ACID-ANTIMONY CHLORIDE METHOD (STANDARD METHOD)

SOLUTIONS REQUIRED

Antimony chloride solution.—Dissolve 20 grams of antimony trioxide or 32 grams of $SbCl_3$ in 1,000 cubic centimeters of HCl (specific gravity *approximately* 1.19).

Hydrochloric acid.—Concentrated HCl (specific gravity *approximately* 1.19).

METHOD

(a) Sheets.—Cut three $2\frac{1}{4}$ by $2\frac{1}{4}$ inches (*or equivalent area*), laboratory samples, as described under "Sampling," and weigh together⁴⁵ after cleaning and drying. They should be immersed singly in 100 cubic centimeters of hydrochloric acid (specific gravity *approximately* 1.19), to which has been added 5 cubic centimeters of antimony chloride solution, and allowed to remain therein until the evolution of hydrogen has ceased or until only a few bubbles are being evolved. This requires only about 15 to 30 seconds, except in the case of sherardized coatings, which require a somewhat longer time. The same 100 cubic centimeters of hydrochloric acid can be used for at least five samples, but 5 cubic centimeters of the antimony chloride solution should be added before the immersion of each sample. After stripping the samples should be washed and scrubbed under running water, dried with a towel, and laid in a warm place for a short time. The samples should again be weighed together⁴⁵ and the number of grams lost divided by the number of samples taken. Each gram then corresponds numerically to 1 ounce of coating per square foot.

(b) Wire.—A small section of the zinc-coated wire should be stripped in hydrochloric acid containing antimony chloride and the diameter of the stripped wire determined.

To determine the weight of coating the sample is carefully weighed to 0.01 gram. The wire is then placed in a tall cylinder containing hydrochloric acid (specific gravity *approximately* 1.19) to which has been added 2 to 3 cubic centimeters of antimony chloride solution of the same strength as is used in the testing of sheets. After stripping the wire is washed under running water, dried, and laid in a warm place for a short time. The loss in weight represents the zinc coating, and if the length of wire designated in Table 8 is used the coating in grams will be numerically equal to the coating in ounces per square foot of actual surface. In order to compare this figure with that given for sheets it must be multiplied by 2.

BASIC LEAD ACETATE METHOD (ALTERNATE METHOD)

SOLUTIONS REQUIRED

Lead acetate solution.—Dissolve 400 grams of crystallized lead acetate in 1 liter of water. When dissolved add 4 grams of finely powdered litharge, and after thorough agitation allow the solution to settle and decant the clear portion for use.

METHOD

Cut three $2\frac{1}{4}$ by $2\frac{1}{4}$ inch (*or of equivalent area*) laboratory samples, clean and weigh as described in the hydrochloric acid-antimony chloride method.⁴⁶ The samples should then be submerged separately in a tumbler containing sufficient lead acetate solution to cover the samples. The samples should be

⁴⁵ If it is of interest to know the individual weight of coating on each piece, each must be analyzed individually instead of collectively, as described.

⁴⁶ See footnote 45.

maintained in an upright position in order to enable the lead acetate to attack all portions.

After being immersed about three minutes the sample should be taken out and the adherent lead carefully removed under running water in order that no burnishing action will cause the lead to plate onto the sample. Four immersions of three minutes each are usually sufficient to remove all zinc from the sample, when the bright surface of the iron will be exposed. The test pieces should then be washed in water, dried carefully, and weighed. The loss in grams divided by the number of samples represents the weight of coating in grams, which is numerically equal to the weight of coating in ounces per square foot.

SULPHURIC ACID PERMANGANATE METHOD (ALTERNATE METHOD)

SOLUTIONS REQUIRED

Distilled water should be used in all cases where water is mentioned as a reagent.

Standard potassium permanganate, one-tenth normal.—Dissolve pure, crystallized potassium permanganate in water in the proportion of 3.2 grams per liter. Allow to stand at least two weeks and filter through asbestos. Standardize the solution in the following manner:

Weigh accurately 0.45 to 0.5 grams of United States Bureau of Standards' sodium oxalate. Place in a 400-cubic centimeter beaker and dissolve in 250 cubic centimeters of water at 80° to 90° C. Add 10 cubic centimeters of sulphuric acid composed of 5 cubic centimeters of water and 5 cubic centimeters of chemically pure H_2SO_4 (specific gravity 1.84) and titrate the solution at once with the potassium permanganate solution, stirring the liquid vigorously and continuously. The permanganate must not be added more rapidly than 10 to 15 cubic centimeters per minute, and the last 0.5 to 1 cubic centimeter must be added dropwise, with particular care to allow each drop to be completely decolorized before the next is introduced. The solution should not be below 60° C. by the time the end point is reached. More rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered electric hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient. Calculate the iron equivalent of the permanganate solution, using the iron value furnished by the Bureau of Standards for the sodium oxalate used.

Sulphuric acid.—Mix 100 cubic centimeters chemically pure H_2SO_4 (specific gravity 1.84) with 900 cubic centimeters of water.

METHOD

Carefully weigh the $2\frac{1}{4}$ by $2\frac{1}{4}$ inches (*or equivalent area*) piece, containing exactly 5.08 square inches.

After determining the weight of the plate, place the piece in a 250-cubic centimeter breaker (Griffin form). Allow the piece to rest on or come in contact with a small piece of platinum foil. Add 125 cubic centimeters or a sufficient amount of the sulphuric-acid solution to cover the inclined piece. Cover the beaker with a watch glass and allow the zinc coating to dissolve. In from 10 to 20 minutes the zinc will be dissolved, as will be indicated by the cessation of the rapid evolution of gas, disregarding the small amount of gas coming mainly from the edges of the plate, due to the action of the acid on the base plate. Wash the plate with a jet of water from a wash bottle, allowing the washings to run into the beaker. Wipe the plate dry, and immediately titrate the solution in the beaker with the standard potassium permanganate solution and calculate the weight of iron dissolved with the zinc coating. Weigh the plate and subtract this weight from the weight of the plate before removal of the coating. From the difference deduct the weight of iron found to have been dissolved with the coating, which will give the weight in grams of zinc coating on 5.08 square inches of the sample. The weight of the zinc coating in grams on the $2\frac{1}{4}$ by $2\frac{1}{4}$ inch (*or equivalent area*) piece will be numerically equal to the weight of zinc coating in ounces per square foot.

APPENDIX

FIELD TESTS FOR DETERMINATION OF ZINC-COATED ARTICLES

PREECE OR COPPER-SULPHATE DIP TEST

The samples for the Preece test may be of any size, but should be free from as much cut surface as possible and should be carefully cleaned before starting the test. The samples are then dipped into not less than 100 cubic centimeters of copper-sulphate solution maintained at a temperature of 18° C. The samples are allowed to remain exactly one minute in the solution. They are then washed in running water and the copper deposit lightly rubbed off. Successive immersions of one minute each are continued, with the washing and cleaning of the sample between each one until the appearance of bright adherent copper indicates that the iron beneath the coating has been exposed. Should a small amount of copper be coated adherently to the zinc coating it must not be mistaken for the end point in which the copper plates out onto the iron. The appearance of copper within 1 inch of a cut surface is likewise not considered to be the end point of the test.

The copper-sulphate solution is prepared by dissolving 36 parts of commercial copper sulphate crystals in 100 parts of water, then adding enough cupric oxide to neutralize any free acid. The solution is filtered or allowed to settle and decanted, then diluted with water until its specific gravity is 1.186 at 18° C. It should always be used at this temperature.

When material is tested by the Preece or copper-sulphate method the number of one-minute immersions which the sample shall withstand is specified.

The amount of coating removed by a one-minute immersion in the copper-sulphate solution is, roughly, between 0.20 and 0.25 ounce per square foot of actual surface, or double this per square foot of sheets.

This test is not as accurate in determining the average weight of coating upon a sample as is the hydrochloric acid-antimony chloride method, but is useful, however, in determining which are the thinnest portions of the coating.

THERMAL METHOD ⁴⁷

This method makes possible the determination of the zinc coating on iron and steel sheets and other articles without necessitating the use of an analytical balance. An accurately measured galvanized sample is immersed in a measured amount of acid, the maximum temperature rise being recorded. Each 0.1° rise in temperature is equivalent to a definite weight of zinc.

The apparatus necessary for conducting this test is as follows:

1. Testing jar, about 2½ inches in inside diameter, with a capacity of about 400 cubic centimeters flaring at the top if possible.
2. Thermometer graduated in 0.1° C., with a range of about 50° C.
3. Three hundred cubic centimeter graduated cylinder.
4. Glass-stoppered bottle containing concentrated hydrochloric acid (specific gravity *approximately* 1.19).
5. Rule for measuring: The sample is cleaned with gasoline, dried, and accurately measured. For testing zinc-coated sheet the usual 2¼ by 2¼ inch (*or of equivalent area*) specimen may be used, and for wire suitable lengths may be used as given in Table 8.

A measured amount of hydrochloric acid (usually 200 cubic centimeters) is poured into the testing jar. The temperature of the acid is recorded after stirring and the sample is immersed in the acid, care being taken that the acid does not overflow the jar. During this time, and until the coating has been stripped from the piece, the temperature is watched until the highest point is reached and the temperature starts to decline. The rise in temperature is then recorded, and from a table of factors prepared for various quantities of acid the amount of zinc may be calculated. From the number of grams of zinc and the area of the piece in square inches the weight of coating may be calculated, as follows:

$$\text{Coating, ounces per square foot.} = \frac{\text{Weight of zinc in grams}}{\text{actual surface}} \times \frac{5.08 \text{ square inches.}}{\text{Area, square inches.}}$$

⁴⁷ Described in paper by D. M. STRICKLAND (15).

In this formula the total area—that is, both sides of the sheet—is measured. To place this on the ordinary sheet basis, as expressed on zinc-coated sheet, the above figure should be doubled. For very light coatings (about 1 ounce per square foot) 100 cubic centimeters of acid are sufficient. With coatings of $2\frac{1}{2}$ ounces per square foot or heavier 300 cubic centimeters of acid should be used. To standardize the equipment five or six zinc-coated specimens of any convenient gauge or size are weighed, the rise in temperature determined for the apparatus, thermometer, and volume of acid. The samples are washed, dried, and reweighed and the difference in weight noted. The weight loss in grams is divided by the corresponding rise in temperature in degrees. When carefully determined the average of the above five or six determinations will give a conversion factor which is reliable for the equipment used and for the volume of acid employed. Factors may be determined for any volume of acid in like manner.

EVOLUTION OF HYDROGEN METHOD

This method consists in measuring the hydrogen evolved from the action of hydrochloric acid containing antimony chloride upon a definite area of galvanized surface. By this method it is not required to cut any portion of the article to be tested, as with the Cushman coating tester, a $2\frac{1}{2}$ -inch ring is sealed to a portion of the surface. Acid is then added, which attacks the coating within the area of the ring, and the evolved hydrogen is measured by a suitable gas burette. After removal of the spent acid the stripped spot is washed, dried, and may either be painted or coated with a suitable metallic coating. The article is, therefore, not mutilated. A full description of the test is found in the 1920 Proceedings of the American Society for Testing Materials (8).

The results obtained by this method check very closely with those obtained by the regular hydrochloric acid—antimony chloride method. The main objection to this method is the fact that the coating upon only one side of a sheet is obtained, and as the coating upon galvanized sheets is not always exactly the same on each side doubling the figures obtained on one side of the sheet will not always give the real amount of coating on both sides of the sheet.

This tester finds use, however, when it is necessary to test sheets or culverts already installed and where it is desired that the articles shall not be mutilated, as would be necessary in taking a regular sample.

66. METHOD OF TEST FOR UNIFORMITY OF GALVANIZING OR SPELTER COATING ON WIRE

1. This method gives in detail the test to be applied to galvanized wire.
2. 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.
3. Cleaning.—The sample shall be cleaned before testing, first with carbon tetrachloride, 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.
5. 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. 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. Samples.—Not more than seven wires shall be simultaneously immersed in the specified quantity of solution.

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. 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.

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. Rejection.—If after the test described in paragraphs 12, 13, and 14 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.

67. STANDARD METHODS OF VERIFICATION OF TESTING MACHINES

(A. S. T. M. standard method (3), serial designation E 4-27)

DEFINITIONS

1. (a) Testing machine.—A mechanical device for applying a load (force) to a specimen.⁴⁸

(b) Error.—In the case of a testing machine, the value obtained by subtracting the correct value of the quantity measured (usually load) from the indicated value as given by the testing machine.⁴⁹

(c) Percentage of error.—In the case of a testing machine, the ratio, expressed in per cent, of the error to the correct value of the quantity measured.

(d) Correction.—In the case of a testing machine, the value obtained by subtracting the indicated value from the correct value of the quantity measured.

NOTE.—The correction has the same magnitude as the error but the opposite sign. It is recommended that, except for special cases, no corrections be used on machines tested and found to have errors within the tolerances given in these methods.

(e) Tolerance.—In the case of a testing machine, the maximum allowable error in the value of the quantity indicated.

NOTE.—It is convenient to express tolerance in terms of percentage of error. The numerical value of the tolerance for a testing machine is so stated hereafter in these methods.

⁴⁸ Usually the magnitude of the load can be changed at the will of the operator. Many testing machines are arranged to measure the load, but this is not always the case, especially with impact machines and machines for testing ductility.

⁴⁹ The error may be positive or negative.

The word "accurate" applied to a testing machine shall be used without numerical values. The word "error" shall be used with numerical values. For example: "An accurate testing machine was used for the investigation"; but "The error of the testing machine at a load of 30,000 pounds was 15 pounds." A testing machine is said to be accurate if the indicated load is within the specified tolerance of the actual load.

NOTE.—The accuracy of a testing machine should not be confused with sensitiveness. For example, a testing machine might be very sensitive—that is, it might indicate quickly and definitely small changes in the load—but, nevertheless, be very inaccurate. On the other hand, the accuracy of the results is, in general, limited by the sensibility.

(f) **Loading range.**—In the case of a testing machine, the range of indicated loads for which the testing machine gives results within the specified tolerances. The loading range shall not include loads either greater than the largest load or less than the smallest load applied in verifying the testing machine.

METHODS FOR VERIFYING TESTING MACHINES THAT MEASURE LOAD

2. Wherever practicable, testing machines shall be verified by applying standard weights directly to the weighing mechanism of the testing machine, or by the use of standardized proving levers in connection with standard weights. The test loads selected shall be applied in both ascending and descending order, and the indications for both the ascending series and the descending series of loads shall be considered in the application of tolerances.

NOTE.—For machines in which the load-indicating device is actuated by a Bourdon pressure tube, a hydraulic (or steam-engine) indicator spring, or other device depending on the elastic properties of a material, the tolerances for the descending load shall be applied to differences in the indication for the corresponding ascending and descending readings. In such testing machines the test for ascending load shall be applied as rapidly as practicable after the testing machine has remained unloaded for at least six hours, and the time of applying the ascending series of loads shall not exceed 30 minutes. The descending load test shall be made after the maximum test load has remained on the machine for one hour, and the readings for descending loads shall be taken as rapidly as possible, and in any event the descending load test shall be completed within a period of 20 minutes.

3. For any loading range, the testing machine shall be verified by at least four test loads. It is recommended that the test loads used be 10, 20, 50, and 100 per cent of the maximum capacity at which the testing machine is to be used.

NOTE.—Where a large amount of testing of small specimens is to be done on a large capacity machine, it is recommended that the verification outlined above be supplemented by a series of smaller test loads selected in reference to the light work to be done on the testing machine.

(a) METHOD OF VERIFICATION BY STANDARD WEIGHTS

4. Standard metal weights of suitable design, finish, and adjustment shall be placed on the weighing platform of the testing machine or upon trays or other supports suspended from the load-measuring mechanism in place of the specimen. The weights shall be applied in increments and removed in the reverse order. They shall be arranged symmetrically with respect to the weighing platform, so that the center of gravity of the load lies in the vertical line through the center of the platform. The applied load and the indicated load shall be recorded for each test load applied, and the error computed from these data.

NOTE.—The method of verification by standard weights can be used only on vertical testing machines. The total load is limited by the size of the platform and the number of weights available. Often 20 weights of 50 pounds each are used, a total of 1,000 pounds. This method of verification is seldom used for any but small testing machines, as the loading range does not include the loads for which large machines are used.

(b) METHOD OF VERIFICATION BY STANDARDIZED PROVING LEVERS

5. The arrangement of proving levers for verifying testing machines is shown in Figure 44. The two levers rest on supports on the weighing platform of the testing machine. These supports shall move easily in a horizontal direction, which insures that the forces at each of the knife-edges shall be very nearly vertical. The inner knife-edges in each lever bear against a suitable block in the movable head of the testing machine. Weight trays or hangers are suspended from each of the outer knife-edges, and these trays or hangers are loaded with standard weights. The increment of load put on the testing machine by

the standard weights is the amount of standard weights multiplied by the lever ratio m/n , Figure 44.

6. The knife-edges, as well as their supports, shall be of hardened tool steel. The knife-edges shall be ground sharp with an angle of 90° . The load on any knife-edge shall not exceed 7,000 pounds per linear inch. The three knife-edges in each lever shall be parallel, and their edges shall lie in a plane. Each lever shall have machined surfaces in this or in some parallel plane, upon which a spirit level can be placed.

7. The lever ratio of a proving lever shall be determined by the use of at least three test loads; the amount of weights used shall not be less than the maximum load applied upon one of the arms of a lever in using the levers to verify testing machines. The proving lever shall be balanced over its center knife-edge with suitable weight trays suspended from the end knife-edges. Standard weights shall then be applied to the trays in three steps, corresponding approximately to 50, 75, and 100 per cent of the weights available, and the proving lever shall then be brought to a balance by the use of small weights and by observations of the freely swinging proving lever. From the weights in the two weight trays the lever ratio shall be computed.

8. The proving levers shall be placed symmetrically in the testing machine to be verified and both levers brought as near to a horizontal position as is

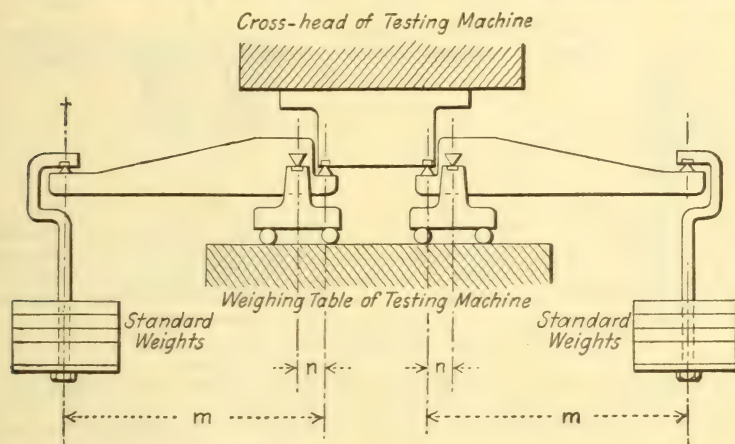


FIG. 44.—Proving levers

feasible, after applying each increment of load, by means of the movable head of the testing machine.³⁹ The testing machine shall be balanced with the levers in place and the weight trays empty. Standard weights shall be applied in increments, half an increment in each tray, and they shall be removed in the reverse order. The weights shall be placed symmetrically on the weight trays, with the center of gravity of the weights over the center of the tray. The applied load and the indicated load shall be recorded for each test load applied and the error computed from these data.

(c) METHOD OF VERIFICATION BY MEANS OF AN ELASTIC CALIBRATION DEVICE⁵¹

9. In this method the testing machine to be verified is tested by comparing its load indications with the amount of elastic deformation of a calibration device (2, 5), the deformation being measured with a delicate strainometer.

10. The calibration device may be a bar tested in tension or in compression or an elastic loop or other shape whose elastic deformation can be measured, and if a tension or compression bar is used it shall be fitted with spherical-

³⁹ The method of verification by standardized proving levers is used at the present time only on vertical testing machines. It is often used up to loads of 10,000 pounds.

⁵¹ The term "calibration device" shall be interpreted to include solid bars, hollow bars, elastic loops, and other members whose elastic deformation can be measured.

seated shackles or with a spherical-seated bearing block to insure as nearly axial loading as is possible. The arrangement shown in Figure 45 is suggested

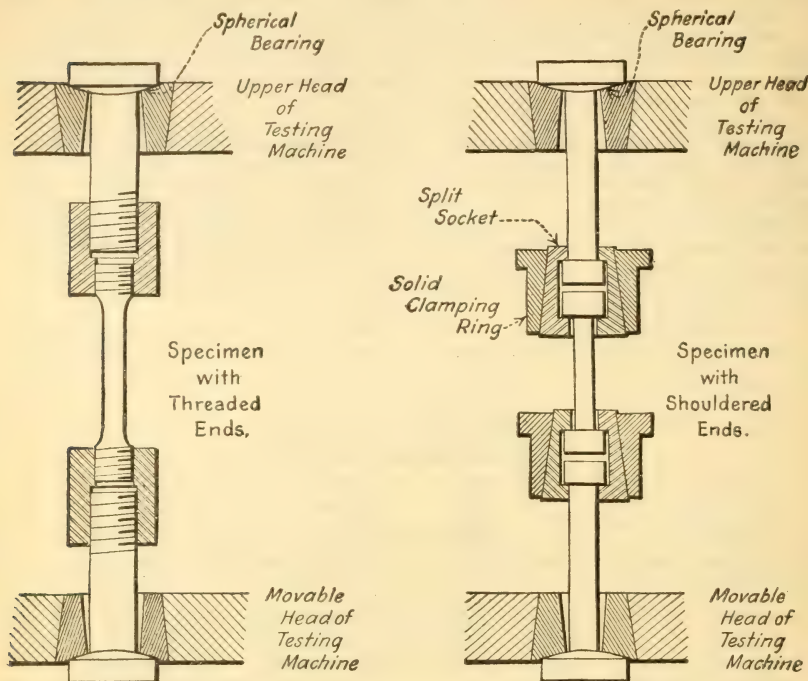


FIG. 45.—Testing arrangements for tension bars

for tension bars, and the arrangement shown in Figure 46 is suggested for compression bars.

11. (a) The calibration device shall be of such size that under the greatest load for which the device is to be used (called its full load) the stress on any cross-section will not be more than 80 per cent of the elastic limit of the material, as determined in accordance with Method II for the determination of elastic limit as described under the definition of the term "elastic limit" of the tentative definitions of terms relating to methods of testing (serial designation E6-25T) of the American Society for Testing Materials (2) on a test specimen cut from the bar, forging or casting for the device after its final heat treatment.

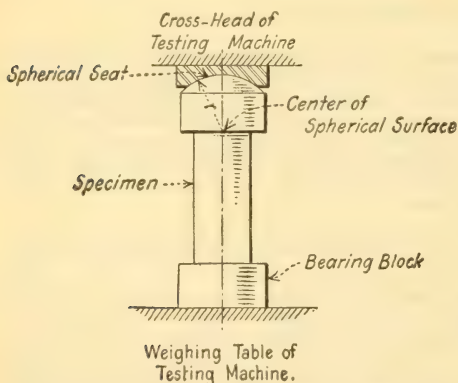


FIG. 46.—Testing arrangement for compression bars

(b) Before the calibration device is used for verification purposes, it shall be loaded to its full load at least six times, and between successive loadings it shall be heated to a temperature of 100° C. (212° F.) for a period of 30 minutes or shall be allowed to rest for a period of 24 hours. The object of this treatment is to adjust the device to its bearings and to relieve internal strain.

12. The loading range of a calibration device is the range of indicated loads for which the device gives results within specified tolerances. Obviously, the loading range may not include loads either greater than the largest load or less than the smallest load applied in verifying the calibration device.

13. (a) The loading range of a calibration device shall be determined by verifying its indications by the use of standard weights or by standard weights and proving levers. In verifying the indications of the device, an initial load of 1 per cent of its full load shall first be applied. All changes of strainometer reading and of applied load shall be reckoned from the readings at this initial load. Successive loads shall be applied, a recommended series of loads being 10, 20, 50, and 100 per cent of the full load of the device. Such a series shall be applied to the bar at least five times to determine the relation of applied load to strainometer reading, the shackles or bearing blocks being readjusted before each application of the series.

(b) The average of the five (or more) strainometer readings obtained in this way shall be taken for each load applied. The average of the differences (taken without regard to algebraic sign) between the individual strainometer readings for any load and the mean reading for that load constitutes the average deviation for that load. The lowest value of load for which the average deviation is not greater than 0.5 per cent of the strain for that load is the lower limit of the loading range of the calibration device.

NOTE.—If in taking five or more strainometer readings for any load the deviation of any one reading is more than 1 per cent from the mean reading, then this series of five or more readings shall be repeated.

14. The strainometer for elastic calibration devices which employ elastic bars in axial tension or axial compression shall be so designed as to indicate the average elongation or compression of the bar. It shall be sufficiently sensitive to indicate a change of 0.25 per cent of the minimum load in the loading range of the device. If the strainometer is not permanently attached to the calibration device, the strainometer shall be removed and replaced for each successive reading taken during the calibration of the device itself. This, of course, does not mean that the strainometer must be removed for each reading in the regular use of the instrument.

15. In using the calibration device it is important that just previous to taking any reading, including the initial reading of the series, there shall be no reversal in the direction of motion of the indicating mechanism of the strainometer, because such reversal may introduce error due to backlash in the instrument. That is, when taking readings for a series of increasing loads the load should be brought up to each predetermined test load, and when taking readings for a series of decreasing loads the load should be brought down to each predetermined test load.

16. In using a calibration device made of steel, correction for variation in mean temperature during any calibrations made on different occasions should be made by allowing a diminution of 0.015 per cent ($\frac{1}{133.3}$ of 1 per cent) in the modulus of elasticity of steel for each 1° F. increase in temperature. During any calibration with an elastic calibration device, temperature conditions should be kept as uniform as is possible.

(d) VERIFICATION BY COMPARISON METHOD

17. When the direct method of verification of a testing machine can not be carried out (as is the case at present with horizontal testing machines, for example), or when an elastic calibration device is not available, a comparison method of verification may be used. In this method the machine to be verified is tested by comparing its indications with the corresponding readings obtained under the same conditions by the use of another testing machine, especially tested and verified, as specified in section 18. The method of verification by comparison shall be carried out by the use of a series of companion specimens, half of which are to be tested in tension in the machine to be verified and half of which are to be tested in tension in the especially verified testing machine which serves as a standard machine. The general provisions respecting the application of the test loads and the loading range which are outlined in the

direct method of verifying testing machines shall apply to the verification of testing machines by the comparison method so far as is practicable.

NOTE.—The method of verification of a testing machine by standard weights (section 4) and the method of verification by standardized proving levers (sections 5 to 8) are both regarded as superior to the method of verification by the use of companion specimens. If the results of a verification test of a testing machine by the comparison method fail to agree with the results of a verification by either the standard-weight method or the proving-lever method, the results of the verification by the comparison method shall be discarded.

18. (a) A testing machine which is to be used as the standard for verifying other testing machines shall itself be verified by a special test, in which the verification is carried out twice. The loading range shall be determined by the tolerance for new testing machines, namely, ± 1 per cent (section 22). For the loading range established, the differences in indications found in the two tests of the standard machine shall not exceed 0.25 per cent.

(b) In verifying testing machines by the comparison method, corrections shall be applied to the indications of the standard machine and these corrections shall be based on the average correction obtained in the two tests used in its verification.

19. (a) For each test load, eight or more tension test specimens shall be cut from soft rolled or drawn steel and numbered consecutively, as in Figure 47, (a).

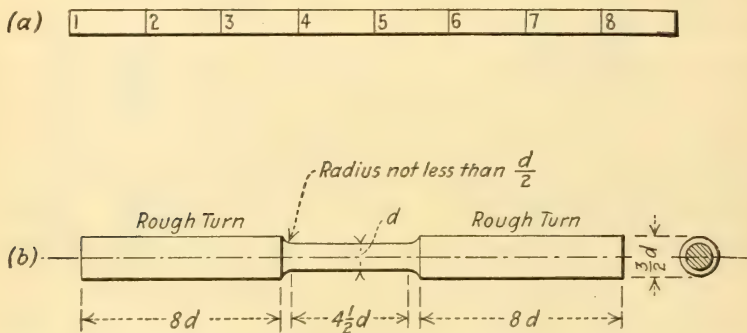


FIG. 47.—Comparison specimen: (a) Method of numbering specimens; (b) form of test specimens

(b) The tensile strength of the steel shall be determined by a preliminary test and the sets of specimens for the comparison test shall have such nominal cross-sectional areas as will give, approximately, the loads required. The form of the specimen shall be as shown in Figure 47, (b). The ends may be threaded, or otherwise machined, to fit holders, but it is recommended that those specimens shipped to another laboratory be left with cylindrical ends. The actual value of " d " (fig. 47) for each specimen shall be determined by means of a micrometer.

NOTE.—It should be noted that the standard tension test specimen, 0.5 inch in diameter and having a 2-inch gauge length, satisfies the requirements for a specimen for the verification of a testing machine by the comparison method. See 1924 Book of A.S.T.M. Standards (1, p. 42, fig. 1).

20. (a) The specimens having odd numbers shall be tested in the machine to be verified and those having even numbers in the standard testing machine. The tensile strength only shall be determined.

(b) The speed of the testing machine, by which is meant the speed of the pulling head when the machine is running idle with no specimen in the machine, when testing verification specimens shall be approximately the same for both the standard testing machine and the machine being verified. The speed shall be the slowest speed that can be obtained on both machines; in any case the speed shall not exceed 1 inch per minute per inch of diameter of comparison specimen.

(c) When testing verification specimens, especial care shall be taken to maintain the weighing beam of the testing machine in equilibrium as the tensile

strength of the specimen is approached. It is recommended that a mark be placed at the middle of the trig loop and that a pointer be placed on the beam for verification tests, and that the pointer be kept as nearly on the mark as possible.

21. (a) The average tensile strength (in pounds per square inch) of the odd-numbered specimens, multiplied by the nominal area of cross section, shall be considered a measure of the indicated load, and the average tensile strength of the even-numbered specimens, multiplied by the nominal area of cross section, shall be considered a measure of the applied load. The error shall be computed from these data.

(b) If the tensile strength (in pounds per square inch) of any verification specimen varies more than 1 per cent from the mean of the group with which it is tested, its tensile strength shall not be used in computing the error of the testing machine. If more than one specimen in the odd-numbered group or one in the even-numbered group exceeds the allowable variation in tensile strength, all the specimens shall be discarded and another set prepared.

TOLERANCES FOR TESTING MACHINES THAT MEASURE LOAD

22. All testing machines that measure load shall conform to the following tolerances:⁵²

(a) New machines.—The error for loads in the loading range shall not exceed ± 1.0 per cent for new machines or for those which have had any of the knife edges in the weighing mechanism repaired or renewed.

(b) Used machines.—The error for loads in the loading range shall not exceed ± 1.5 per cent.

23. The indicated load of a testing machine shall not be corrected either by computation or by the use of a calibration diagram to obtain values within the required tolerance.

TIME INTERVAL BETWEEN VERIFICATIONS

24. It is recommended that testing machines, when in constant use, be verified at intervals of six months and, when used intermittently, at intervals of two or three years. Testing machines shall, however, be verified immediately after making repairs or adjustments of the weighing mechanism, and whenever there is reason to doubt the accuracy of the results, without regard to the time interval since the last verification.

REPORTS AND CERTIFICATES

25. A clear and complete report shall be prepared of each verification of a testing machine. This report shall contain all data necessary to verify the accuracy of the report, and shall be filed for future reference. This report shall state clearly the method of verification used.

26. A certificate giving the maker's serial number and a brief description of the testing machine, the maker's name, the date of verification, and the loading range shall be signed by the person responsible for the maintenance of the testing machine, and this certificate shall be posted in plain view of a person operating the testing machine.

68. TENTATIVE METHODS OF TENSION TESTING OF METALLIC MATERIALS⁵³

(A. S. T. M. tentative standard method. (5) Serial designation E 8-27 T)

These methods deal with the form and dimensions, the machining, and the testing of tension test specimens of metallic materials. Such matters as methods

⁵² It is believed that the tolerances given in this section may be made much smaller when more perfect methods of verification become generally available; and that the tolerances might well be materially reduced if there is available equipment for verifying testing machines by the use of standard weights (sec. 4) or by the use of standardized proving levers (secs. 5 to 8).

⁵³ Criticisms of these tentative methods are solicited and should be directed to H. F. Moore, chairman of subcommittee E-1 on mechanical testing, University of Illinois, Urbana, Ill. These tentative methods are in effect a revision of the methods for tension tests of the standard methods of mechanical testing of metallic materials. The standard methods, which were last published under the serial designation E 1-18, have accordingly been withdrawn.

of taking samples from which to prepare test specimens are treated in the individual specifications for ferrous metals and for nonferrous metals.

TEST SPECIMENS

1. In recommending the following specimens for use in tension tests it is not intended to exclude entirely the use of other test specimens for special materials or for special forms of material; for example, in tests of tubing and pipe the form of specimen recommended for plate, shape, and flat material (fig. 48) is not generally suitable for use and a special form of specimen is specified. It is, however, recommended that wherever it is feasible the specimens here recommended be used.

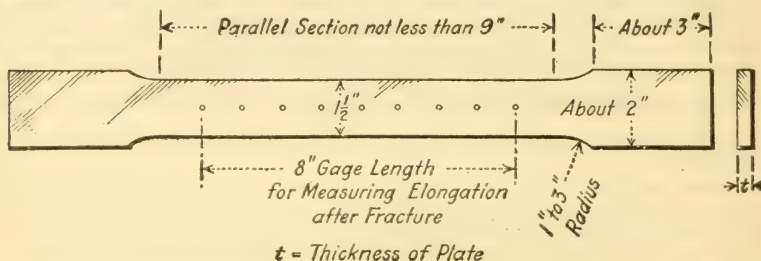


FIG. 48.—Tension test specimen for plate material

2. Tension test specimens of wire are of the full-size diameter as drawn, and tension test specimens of rods of ductile metal are often of the full-size diameter as rolled. Tension test specimens of pipe and of tubing are frequently of full size as manufactured, in which case the ends should be plugged with metal plugs which do not extend within the gauge marks.

3. The tension test specimen shown in Figure 48 is recommended for plate, shape, and flat material having a thickness of one-fourth inch or over.⁵⁴ The thickness of the specimen is that of the material tested.

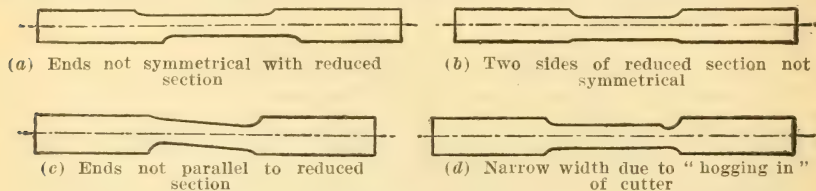


FIG. 49.—Common errors in preparation of plate or sheet-metal test specimens

NOTE.—When it is desired to use a specimen with a gauge length of less than 8 inches, the general proportions of Figure 48 should be followed. Specimens with a gauge length of 2 inches are occasionally used. When it is not convenient to machine specimens to the standard shape shown in Figure 48, specimens may be used with edges machined parallel for the entire length of the specimen. If such specimens are machined with a tool cutting across the edges of the specimen, it is important that the transverse tool marks be removed before the specimen is tested. For thick plate material it is usually preferable to use a machine specimen rather than to use a specimen the full thickness of the plate.

4. For tension tests of wrought ferrous and nonferrous metal in the form of plate, sheet, flat wire,⁵⁵ strip,⁵⁵ band,⁵⁵ and hoop⁶⁵ without respect to width,⁵⁵ length, grade, or method of manufacture, and having a thickness from 0.01 to 0.250 inch, the test specimen described in sections 5 and 6 is recommended.⁵⁶

⁵⁴ Attention is called to the fact that two types of specimen are applicable for material one-fourth inch in thickness, the plate specimen shown in Figure 48, and the thin sheet specimen shown in Figure 50.

⁵⁵ It is to be noted that the recommended specimen can not be used for flat wire, strip, band, or hoop material whose width is less than the end width of the specimen itself.

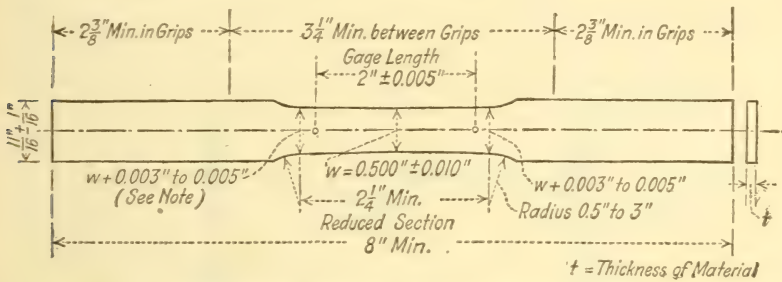
⁶⁵ This specimen may also be used in tests of material cut from tubing.

5. The specimen blank shall be cut from the material to be tested, having its length in such direction relative to the material as may be specified. The specimen blank shall conform to the dimensions shown in Table 9. Blanks cut with a torch shall not be used unless all metal affected by such cutting is removed by machining.

TABLE 9.—Dimensions for specimen blanks

Method of cutting long edges of blank	Nominal thickness of material	Minimum width of blank
	Inches	Inches
Sheared.....	Up to 1/8, inclusive.....	3/4
Sheared.....	Over 1/8 to 1/4, inclusive.....	1
Sawed ¹	Up to 1/4, inclusive.....	3/4

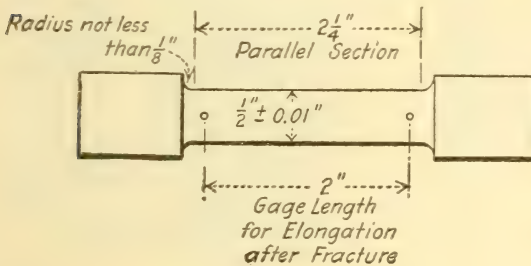
¹ Or otherwise machined without severely straining the metal.



Note: Gradual Taper from Ends of Reduced Section to Middle.
All Machining Dimensions are Shown below and Testing Dimensions above Specimen.

FIG 50.—Standard tension test specimen for thin sheet metals

6. Figure 50 shows the form of the finished specimen recommended for thin sheet metals. The reduced section of the specimen shall be machined in such a manner as to avoid leaving severe machining strains in the material. It is



Note:—The Gauge Length, Parallel Section, and Fillets shall be as Shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a Way that the Load shall be axial.

FIG. 51.—Standard 2-inch tension test specimen

essential that the reduced section and the ends of the specimen be symmetrical with respect to the longitudinal axis of the specimen.⁵⁷

In Figure 50 the 2-inch gauge length should be laid off with a tolerance in length of ±0.005 inch.

⁵⁷ For all specimens for flat metallic materials, both for thin sheet and for thick plate, it is important to so machine the specimen as to avoid the conditions shown in Figure 49.

7. The tension test specimen shown in Figure 51 is recommended for general use in testing metals.⁵⁸ The gauge length for measuring elongation after fracture is 2 inches.

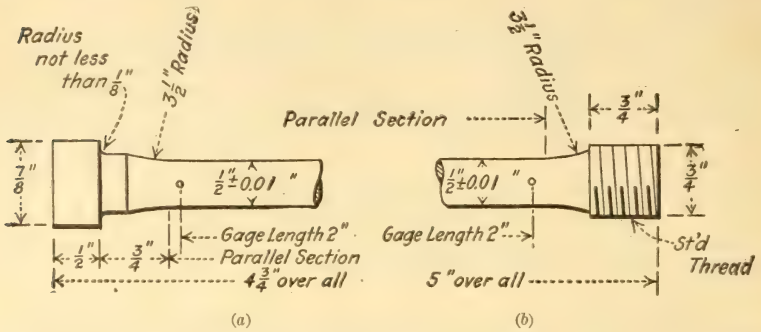


FIG. 52.—(a) Specimen with shouldered ends; (b) specimen with threaded ends

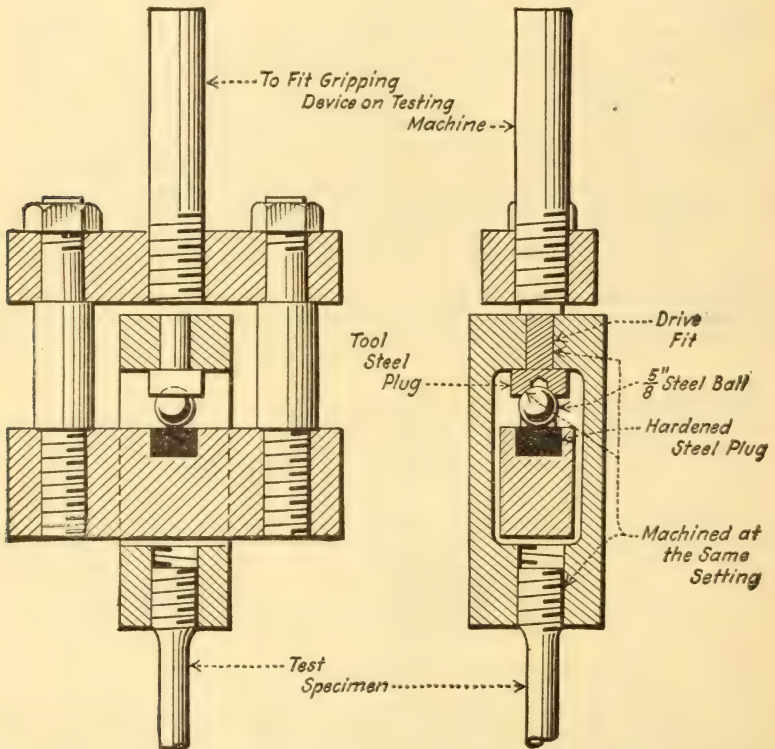


FIG. 53.—The gripping device is shown as used with threaded end specimens. A similar device fitted with split sockets would be used with shouldered specimens

8. When it is necessary to cut specimens from material (other than plate, shape, and flat material) which is of such size that the specimen shown in Figure 51 can not be used, it is recommended that a specimen with dimensions

⁵⁸Attention is called to section 10.

proportional to those shown in Figure 51 be used and that the specimen be made as large as feasible. In any such small-size specimen it is especially important that the gauge length for measuring elongation be four times the diameter of the specimen.

9. In all tension tests of metals the actual dimensions of cross section of the test specimens shall be measured with a micrometer reading to one-two hundredths of the dimension measured, and the stresses shall be computed on the basis of the measured cross section: it should never be assumed that the dimensions of the measured cross section are identical with the nominal dimensions.

NOTE.—It will be noted that the tolerance for diameter of specimens shown in Figure 51 permits the use of specimens 0.505 inches in diameter.

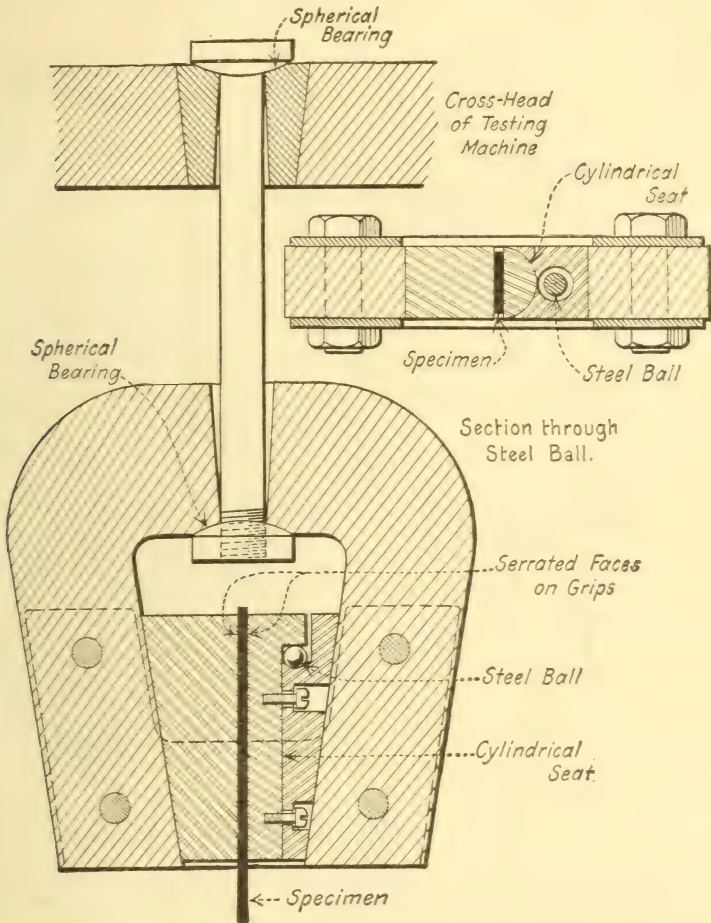


FIG. 54.—Self-adjusting grips for use with sheet metal

10. The shape of the ends of the specimen shown in Figure 51 are not specified above. For specimens of brittle metal it is desirable that the diameter of the specimen be changed gradually along its length from the minimum section at the gauge length to the diameter at the ends. Figure 52 shows two forms of end of specimen which have given satisfactory results in tests of brittle metals.

11. Specimens shall be finished so that the surfaces are smooth and free from nicks and tool marks. All ragged edges shall be smoothed.

GRIPPING DEVICES FOR TENSION-TEST SPECIMENS

12. For long bars of ductile metal and for the flat-plate test specimen shown in Figure 48, wedge grips, bearing directly on the end of the test specimen, generally furnish a satisfactory means of gripping the specimens.

13. For short specimens and for specimens of brittle material, wedge grips are not a satisfactory means of holding test specimens; it is necessary to use machined specimens and to use special means for insuring that the specimen, when under load, shall be as nearly as possible in pure axial tension. Figure 45 shows a device for gripping tension specimens with threaded ends and a device for gripping specimens with shouldered ends. Both these gripping devices are attached to the heads of the testing machine through spherical-seated bearings. The distance between spherical bearings should be as great as is feasible. For brittle materials, even the spherical-seated bearings shown in Figure 45 are not always effective in avoiding bending stress on the specimen. The gripping device shown in Figure 53 has proved satisfactory for testing such specimens, and is an excellent gripping device for any machined tension test specimen.

14. For specimens cut from thin plate material and from sheet material, ordinary wedge grips are liable to cut the specimens near the edges, giving a tearing stress rather than an axial tension. The self-adjusting grips shown in Figure 54 have proved satisfactory for tests of thin sheet copper and aluminum.

15. For tests of specimens of wire, ordinary wedge grips are liable to cut the wire at the edges of the grips. This cutting has been found to be largely eliminated by the use of grips of the form shown in Figure 55.

16. In making tension tests, care shall be taken to see that the axis of the test specimen is located in the center line of the heads of the testing machine and that the liners used behind wedge grips are of proper thickness. From time to time the openings in the heads of the testing machine shall be lined up and the heads tested for parallelism. Deviations from correct alignment and from parallelism should not exceed 0.01 inch for any position of the movable head within the range of movement during test. The heads of the machine

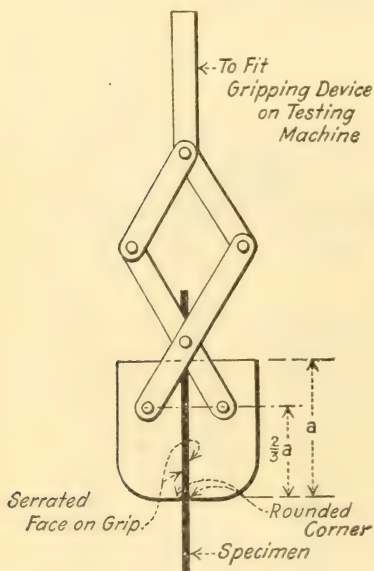


FIG. 55.—Grip for use with wire

should be free from sidewise motion that is visible to the unaided eye as the machine is run at its fastest speed.

SPEED OF OPERATION OF TESTING MACHINE

17. 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 specimens of metallic materials in gauge lengths of 2 and 8 inches, 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 speed of head of the testing machine shall be such that the load can be accurately weighed, but in no case shall the speed be greater than the values given in Table 10, which values represent the extreme of good laboratory practice for steel.⁶⁹

⁶⁹ The speeds given here are probably too high for satisfactory use in testing ferrous metals.

TABLE 10.—Limit of speed of testing head

Specified minimum tensile strength of material, pounds per square inch	Gauge length, inches	Maximum cross-head speed, inches per minute	
		Yield point	Tensile strength
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

18. In determining the elastic limit in accordance with method 1, section 20, the crosshead speed for the 2-inch gauge length shall not exceed 0.125 inch per minute.

19. In determining the elastic limit by method 2, section 20, the crosshead speed shall not exceed 0.025 inch per inch of gauge length per minute.

DETERMINATION OF ELASTIC LIMIT, YIELD POINT, AND TENSILE STRENGTH

20. The term "elastic limit" is defined as follows:

Elastic limit.—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

The determination of the elastic limit as thus defined would logically involve the application and release of a succession of increasing loads on a test specimen until there was observed deformation after release of a load. This procedure is very slow, and since for many materials experience does not indicate any appreciable difference between the elastic limit and the limit of proportionality of stress to strain (sometimes called the proportional limit), the determination of the stress at the limit of proportionality of stress to strain is regarded as an accurate determination of the elastic limit.

It is obvious that the values obtained in tests for determining the elastic limit will depend on the delicacy of methods and instruments used. It becomes necessary, therefore, that in any test the method used in obtaining the elastic limit be clearly stated. The following methods are in common use for determining a value designated as the elastic limit:

Method 1, proposed by committee A-1 on steel, and embodied in a number of specifications for steel. (For tension tests only.) The elastic limit shall be determined by an extensometer reading to 0.0002 inch. The extensometer shall be attached to the specimen at the gauge marks and not to the shoulders of the specimen nor to any part of the testing machine. When the specimen is in place and the extensometer attached, the testing machine shall be operated so as to increase the load on the specimen at a uniform rate. The observer shall watch the elongation of the specimen as shown by the extensometer and shall note, for this determination, the load at which the rate of elongation shows a sudden increase. The extensometer may then be removed from the specimen and the test continued to determine the tensile strength.

Method 2 (proposed by the late J. B. Johnson) is a somewhat more delicate method for locating the elastic limit than is method 1. In method 2 the elastic limit shall be taken as that stress at which the rate of deformation is 50 per cent greater than the initial rate of deformation. A stress-strain diagram is necessary for determining the elastic limit by this method. It is illustrated in

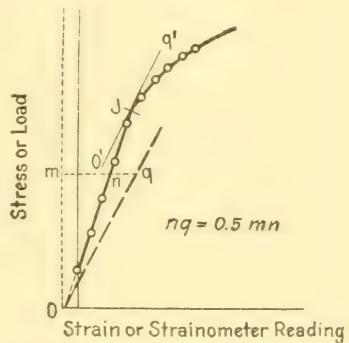


FIG. 56.—Illustrating method 2 for determining elastic limit

Figure 56. The initial rate of deformation is given by the ratio $mn:Om$. $nq=0.5mn$, $mq=1.5mn$, and the slope of Oq represents a rate of deformation 50 per cent greater than the initial rate. $O'q'$ is drawn parallel to Oq and tangent to the stress-strain diagram. The point of tangency J locates the elastic limit. In using method 2 the same precautions should be observed in attaching the strainometer as for the determination of the elastic limit by method 1. Method 2, in tension tests of metals, gives lower values for the elastic limit than does method 1. It is recommended that in using method 2 for tension tests the strainometer be of such sensitiveness that it will indicate a change of strain of 0.0001 inch per inch of gauge length (0.0001 millimeter per millimeter of gauge length).

Methods of high precision.—When it is desired to determine the elastic limit with a high degree of precision it is suggested that the experimenter devise his own procedure, and in reporting his results describe the procedure in detail, including a statement of the limits of sensitiveness of the apparatus used and of the procedure used in plotting the stress-strain diagram from which the determination of elastic limit is made. It is to be remembered that the accurate determination of the elastic limit requires the use of accurate and sensitive instruments and the use of accurate methods of plotting test data.

21. The term "yield point" is defined as follows:

Yield point.—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

Two methods are in use for determining the yield point: (1) The "drop of the beam" method, and (2) the method by use of dividers:

Method 1.—In method 1, load is applied to the specimen at a steady rate of increase and the operator keeps the beam in balance by running out the poise at a steady rate. At the yield point the increase of load stops (and for some metals there is an actual falling off of load), but the operator, running out the poise at a steady rate, runs it a trifle beyond the balance position, and the beam of the machine drops for a brief but appreciable interval of time. In a machine fitted with a self-indicating load-measuring device there is a sudden halt of the load-indicating pointer, corresponding to the drop of the beam. The load at the "halt" or the "drop" is recorded, and the corresponding stress is taken as the yield point. This method of determining the yield point requires only one man to conduct the test.

Method 2.—In method 2 an observer with a pair of dividers watches for visible elongation between two section marks on the specimens. When visible stretch is observed, the load at that instant is noted, and the stress corresponding to the load is taken as the yield point. When a strainometer⁶⁰ is used in place of dividers a deformation of 0.01 inch over a gauge length of 2 inches shall be regarded as indicating the yield point.

Some metals do not have a well-defined yield point.

22. The term "tensile strength" is defined as follows:

Tensile strength.—The maximum tensile stress which a material is capable of developing.

NOTE.—In practice, it is considered to be the maximum stress developed by a specimen representing the material in a tension test carried to rupture, under definite prescribed conditions. Tensile strength is computed from the maximum load carried during a tension test and the original cross-sectional area of the specimen.

The conditions that must be prescribed in a tension test of metallic materials are the form of specimen, the method of gripping the specimen, and the rate of application of load. These are covered in foregoing sections. No further detailed discussion of methods of determining tensile strength is necessary, as there is involved merely the observing and the recording of the maximum load carried by the specimen during the test.

THE MEASUREMENT, AFTER FRACTURE, OF THE ELONGATION AND THE REDUCTION OF CROSS SECTION OF TENSION-TEST SPECIMENS

23. The measurement of the elongation after fracture of tension-test specimens can be made with sufficient accuracy by means of a pair of dividers and

⁶⁰ The determination of yield point with a strainometer is sometimes made in tests on nonferrous metals.

a scale. The elongation should not be reported for any tension-test specimen which breaks outside the middle third of the gauge length.

NOTE.—If only a few specimens are furnished for testing it is recommended that in marking the gauge length for measuring elongation after fracture it be divided into eight parts, and that if a specimen breaks inside the gauge length but outside the middle third of the gauge length the requirement in the foregoing paragraph be waived, and the elongation be measured and reported as follows:

(1) If the specimen breaks in the middle half of the gauge length (between *C* and *G*), Figures 57 (a) and (b) the elongation is measured directly over the stretched gauge lengths (fig. 59 (b)).

(2) If the specimen breaks between *A* and *C* (or between *G* and *I*) but nearer *B* than *A* (or nearer *H* than *I*), as shown in Figure 59 (c) the elongation from *A* to *C* (or from *G* to *I*) is measured, and to this is added twice the measured elongation from *C* to *E* (or from *E* to *G*).

(3) If the specimen breaks within one-half a division of an end gauge mark (see fig. 59 (d)) the elongation from *A* to *E* (or from *E* to *I*) is measured and multiplied by 2.

The above method can be applied to specimens with any gauge length, and to turned specimens as well as flat specimens.

This method is not applicable to brass specimens.

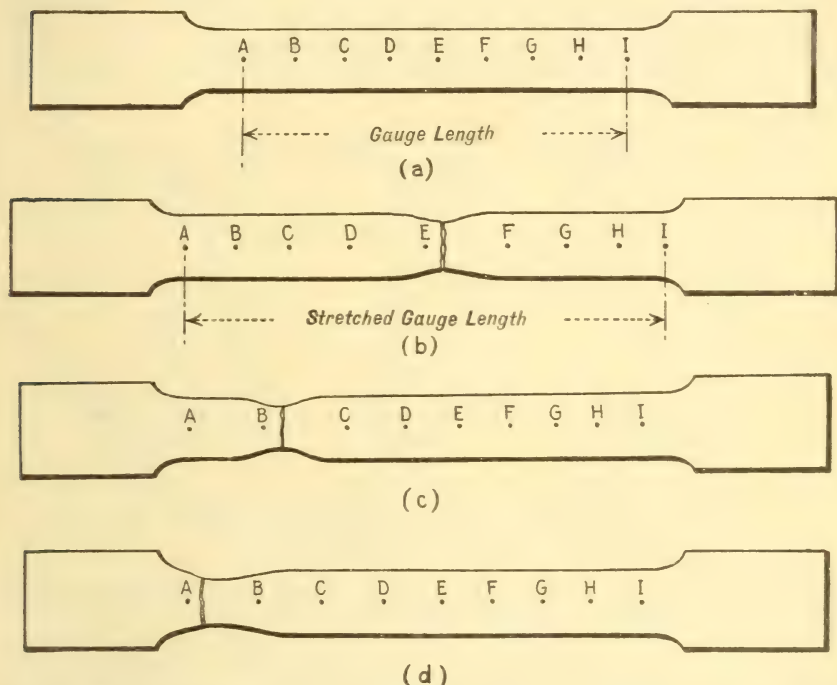


FIG. 57.—Method of placing gauge markings where only a few specimens are furnished for testing

24. The measurement of reduction of the dimensions of the cross section of a tension-test specimen may be made by the direct measurement with a micrometer of the smallest section of the fractured specimen. For round specimens this measurement can usually best be made by holding the broken pieces together in a vise or between centers and then measuring the average diameter of the smallest cross section by means of a micrometer fitted with points so shaped that they will come in contact with the specimen at its smallest diameter.

PLOTTING STRESS-STRAIN DIAGRAMS

25. A stress-strain diagram⁶¹ is a diagram plotted with values of stress as ordinates and values of strain as abscissas.

⁶¹The use of the term "stress-strain diagram" is frequently extended to cover diagrams plotted with values of applied load, or applied moment, as ordinates and with values of stretch, compression, deflection, or twist as abscissas.

Stress-strain diagrams are in some cases drawn directly by an autographic attachment to the testing machine. A more usual method of procedure consists in taking a series of load readings (from the balanced scale beam of the testing machine) with corresponding readings of the strain-indicating apparatus. The term "strainometer reading" will be used to denote the reading of the strain-measuring instrument. From these readings, or from values computed from them, there is plotted a diagram with stress-indicating values (load or stress) as ordinates, and strain-indicating values (elongation or strain) as abscissas. In planning such a test it is necessary to decide on the increment of load or the increment of reading of strainometer to be used between successive readings.

In Figure 58 are shown three typical stress-strain diagrams. The material for all three tests is the same, and the test specimens are all of the same size, so that the diagrams should be the same. The diagram shown in Figure 58 (*a*) is plotted from points determined by taking increments of load (*S*). In this diagram it is seen that, owing to the shape of the curve, data for locating points between *M* and *N* are lacking. That particular portion of the curve (the "knee" of the curve) is the part for which it is especially desirable to locate several points on the diagram.

In Figure 58 (*b*) is shown a diagram plotted from points determined by taking increments of strainometer reading (*e*). It will be noted that for this

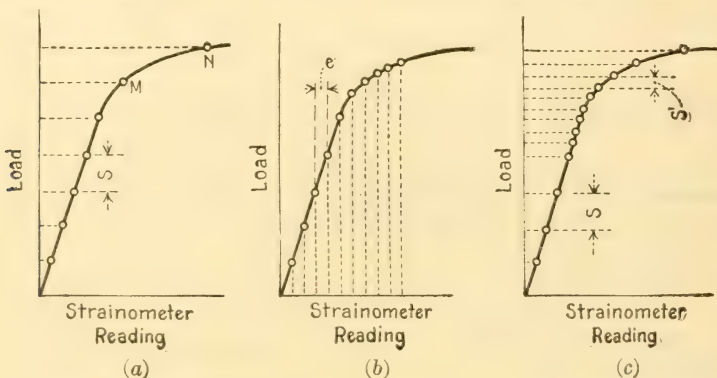


FIG. 58.—Illustrating three methods of plotting load-deformation curves. (*a*) With equal increments of stress; (*b*) with equal increments of strain; (*c*) with two values of increments of stress

diagram there are located several points near the knee of the curve, and the shape of the diagram in this important region is much more definitely determined than for the curve shown in Figure 58 (*a*).

The custom of choosing increments of load rather than increments of strainometer reading is quite common in tests of materials and is followed because, in general, it is easier to compute increments of load than it is to compute increments of strainometer reading. An estimate of the load necessary to stress the specimen up to the knee of the curve is made, and some fraction (frequently one-tenth) of this value is taken as an increment. The computation of the corresponding increment of strainometer reading is more complicated.

Sometimes the practice is followed of applying a few increments of load as determined above, and then applying load in much smaller increments (*S'*) until the knee of the curve is passed. Figure 58 (*c*) shows a diagram obtained in this manner. This method involves a marked increase in the number of readings necessary for a test, and with unknown material there is always some danger that the knee of the curve will be reached before the use of small increments of load is begun.

The following method of choosing increments for a test is suggested: Estimate the load corresponding to the knee of the stress-strain diagram and choose a value for increment of load about one-tenth of this value. Apply this increment of load (*S*) once and note the corresponding change of reading for the strainometer (*e*). Then for the remainder of the test use for the increment of strainometer reading a value which corresponds to some convenient interval on the scale of the strainometer and which is approximately equal to (*e*).

69. TENTATIVE METHODS OF COMPRESSION TESTING OF METALLIC MATERIALS⁶²

(A. S. T. M. tentative standard method (5), serial designation E 9-27 T)

These methods deal with the form and dimensions, the machining, and the testing of compression test specimens of metallic materials. Such matters as methods of taking samples from which to prepare test specimens are treated in the individual specifications for ferrous metals and for nonferrous metals.

TEST SPECIMENS

1. In recommending the following specimens for use in compression tests it is not intended to exclude entirely the use of other test specimens for special

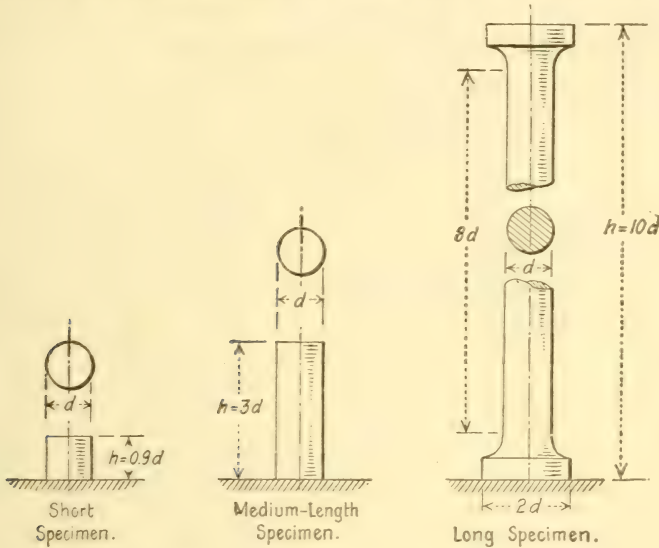


FIG. 59.—Proportions of three classes of compression specimens

materials or for special forms of material. It is, however, recommended that wherever it is feasible the specimens here recommended be used.

2. It is recommended that standard compression specimens be in the form of circular cylinders. The ends of a specimen should be plane, as nearly as can be determined by the use of a straightedge and the unaided eye. In most cases this requirement necessitates the turning or the grinding of the ends of the specimen.

3. There are recognized three classes of compression specimens for metallic materials, designated as short specimens, medium-length specimens, and long specimens, respectively. Short specimens are used for compression tests of such metals as bearing metals, which in service are used in the form of a thin plate or shell to carry load perpendicular to the surface. Medium-length specimens are used for determining the general compressive strength properties of metallic materials. Long specimens are best adapted for determining the modulus of elasticity in compression of metallic materials. In reporting the results of a compression test it is important that the dimensions of the test specimen be given.

⁶² Criticisms of these tentative methods are solicited and should be directed to H. F. Moore, chairman of subcommittee of committee E-1 on mechanical testing, University of Illinois, Urbana, Ill. These methods are in effect a revision of the methods for compression tests of the standard methods of mechanical testing of metallic materials. The standard methods, which were last published under the serial designation E 1-18, have accordingly been withdrawn.

4. Figure 59 shows the proportions of the three classes of compression specimens. Suggested dimensions for compression specimens for general use are:

Short specimens: $d=1\frac{1}{8}$ inches ± 0.01 inch (area of cross section is approximately 1 square inch), $h=1$ inch;

Medium-length specimens: $d=0.798$ inch ± 0.01 inch, $h=2\frac{3}{8}$ inches, or
 $d=1$ inch ± 0.01 inch, $h=3$ inches, or
 $d=1\frac{1}{8}$ inches ± 0.01 inch, $h=3\frac{3}{8}$ inches.

Long specimens: $d=1\frac{1}{4}$ inches ± 0.01 inch, $h=12\frac{1}{2}$ inches.

5. In all compression tests of metallic materials, the actual dimensions of cross section of the test specimens shall be measured with a micrometer reading to one-two hundredth of the dimension measured, and stresses shall be computed on the basis of the measured cross section. It should never be assumed that the dimensions of the cross section of the specimen are identical with the nominal dimensions.

6. Specimens shall be finished so that the surfaces are smooth and free from nicks and tools marks. All ragged edges shall be smoothed.

BEARING BLOCKS FOR COMPRESSION-TEST SPECIMENS

7. Both ends of a compression-test specimen shall bear on a carefully machined plane surface. The bearing blocks shall either be made of hardened steel or shall be faced with hardened steel.

8. One end of a compression test specimen shall bear on a spherical-seated compression block.⁶³ Figure 60 shows a satisfactory arrangement of compression specimen and bearing blocks. It is desirable that the spherical-seated bearing block be at the upper end of the test specimen (for specimens tested with the axis vertical). It is important that the center of the spherical surface of the spherical-seated bearing block should be in the flat face which bears on the specimen; otherwise lack of parallelism between faces of the specimen sets up a bending moment in the specimen (14).

9. It should be borne in mind that the object of a spherical-seated bearing block is to give the specimen as even a distribution

of initial load as is possible. Owing to friction, the spherical-seated bearing can not be relied on to adjust itself to bending action which may occur during the test.

10. In making compression tests, care should be taken to preserve the alignment of the heads of the testing machine, and to center the specimen in the testing machine. From time to time the heads of the testing machine shall be tested for parallelism. Deviations from parallelism should not exceed 0.01 inch. The movable head of the machine should be free from sidewise motion that is visible to the unaided eye as the machine is run at its fastest speed.

SPEED OF OPERATION OF TESTING MACHINE

11. In making compression tests of specimens of metallic materials the speed of the machine, by which is meant the speed of the cross head when the machine is running idle, shall be such that the load can be accurately weighed, but in no case shall the speed be greater than the values given in Table 11.

⁶³ The device shown in Figure 61 has been successfully used in compression tests of metallic specimens whose ends were machined with extreme care. It will be noted that this device does not involve a spherical-seated compression block bearing directly on the test specimen. This device or one similar has been successfully used in the British National Physical Laboratory and in the laboratories of the United States Air Service at McCook Field, Dayton, Ohio.

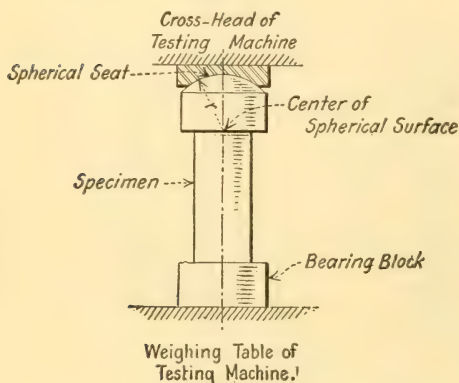


FIG. 60.—A satisfactory arrangement of compression specimen and bearing block

TABLE 11.—Speed of operation of testing machine

Length of specimen	Maximum cross-head speed
<i>Inches</i>	<i>Inches per minute</i>
1.00.....	0.05
3.00 or greater.....	0.10

12. In making tests to determine the elastic limit in compression of metallic materials, the crosshead speed of the testing machine shall in no case exceed the allowable speed of head for the corresponding tension test of the material, as given in sections 18 and 19 of the tentative methods of tension testing of metallic materials (serial designation E 8-27 T) of the American Society for Testing Materials (15). (See p. 111.)

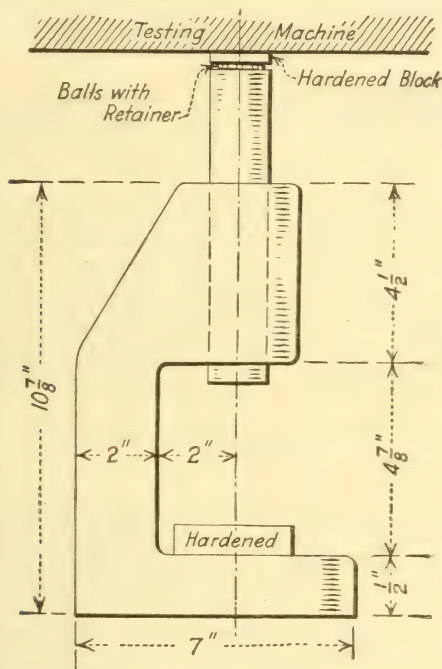


FIG. 61.—Compression tool

THE DETERMINATION OF ELASTIC LIMIT, YIELD POINT, AND COMPRESSIVE STRENGTH

13. (a) The term "elastic limit" in compression is defined the same as in tension, as follows:

Elastic limit.—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

(b) The same general methods for determining elastic limit, as given in section 20 of the tentative methods of tension testing of metallic materials (serial designation, E 8-27 T, see p. 111) of the American Society for Testing Materials (5) shall be used. For compression tests, method 2, or a still more precise method, seems generally suitable.

14. (a) The term "yield point" in compression is defined the same as in tension, as follows:

Yield point.—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

(b) The same methods for determining yield point as given in section 21 of the tentative methods of tension testing of metallic materials (serial designation, E 8-27 T, see p. 111) shall be used. The method by the use of dividers or by the use of a strainometer seems generally suitable for compression testing.

15. The term "compressive strength" is defined as follows:

Compressive strength.—The maximum compressive stress which a material is capable of developing.

NOTE.—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value, depending upon the degree of distortion which is regarded as indicating complete failure of the metal.

PLOTTING STRESS-STRAIN DIAGRAMS

16. The discussion and directions given in section 25 of the tentative methods of tension testing of metallic materials (serial designation, E 8-27 T, see p. 111 (5) apply to plotting stress-strain diagrams for compression tests.

70. STANDARD METHODS OF BRINELL HARDNESS TESTING OF METALLIC MATERIALS⁶⁴

(A. S. T. M. standard method (3) serial designation E 10-27)

The Brinell test for determining the hardness of metallic materials consists in applying a known load to the surface of the material to be tested through a hardened steel ball of known diameter. The diameter (or depth) of the resulting permanent impression in the metal is measured. The Brinell hardness number is taken as the quotient of the applied load divided by the area of the surface of the impression, which is assumed to be spherical. If P is the applied load (measured in kilograms), D is the diameter of the steel ball (measured in millimeters) and d is the diameter of the impression (measured in millimeters), then:

$$\text{B.H.N.} = \frac{P}{\frac{\pi D}{2} \left(D - \sqrt{D^2 - d^2} \right)}$$

in which B.H.N. is the Brinell hardness number in kilograms per square millimeter.

If the steel ball were not deformed under the applied load and if the impression were truly spherical, then the above formula would be a general one, and any combination of applied load and size of ball could be used. As the impression is not quite a spherical surface (since there must always be some deformation of the steel ball and some recovery of form of the metal in the impression), for a standard Brinell test the size and characteristics of the ball and the magnitude of the applied load must be standardized. (A standard ball 10 millimeters in diameter and a load of 3,000 kilograms for hard metals and 500 kilograms for soft metals is standard practice. See sections 3 and 6.)

In Table 12 is given the Brinell hardness number corresponding to various diameters of impression for both 500 and 3,000-kilogram load, making it unnecessary to compute for each test the value of the Brinell hardness number by the above formula.

⁶⁴ Under the standardization procedure of the society, these methods are under the jurisdiction of the A. S. T. M. committee E-1 on methods of testing. These methods are in effect a revision of the methods for Brinell hardness tests of the standard methods of mechanical testing of metallic materials. The standard methods, which were last published under the serial designation E 1-18, have accordingly been withdrawn.

TABLE 12.—Brinell hardness numbers

[Steel ball, 10 millimeters in diameter, pressures of 500 and 3,000 kilograms. Prepared by the engineering mechanics section, Bureau of Standards. The values given in this table for hardness numbers are merely solutions of the equation given in the introductory paragraphs. They do not imply that Brinell tests are feasible on materials of a hardness indicated by the highest values in the table]

Di- am- eter of inden- tation	Brinell hardness No.		Di- am- eter of inden- tation	Brinell hardness No.		Di- am- eter of inden- tation	Brinell hardness No.		Di- am- eter of inden- tation	Brinell hardness No.		Di- am- eter of inden- tation	Brinell hardness No.	
	500- load	3,000- load		500- load	3,000- load		500- load	3,000- load		500- load	3,000- load		500- load	3,000- load
Mm.	Kg.	Kg.	Mm.	Kg.	Kg.	Mm.	Kg.	Kg.	Mm.	Kg.	Kg.	Mm.	Kg.	Kg.
2.00	158	945	2.70	85.7	514	3.40	53.4	321	4.10	36.2	217	4.80	25.9	156
2.01	156	936	2.71	85.1	510	3.41	53.1	319	4.11	36.0	216	4.81	25.8	155
2.02	154	926	2.72	84.4	507	3.42	52.8	317	4.12	35.8	215	4.82	25.7	154
2.03	153	917	2.73	83.8	503	3.43	52.5	315	4.13	35.7	214	4.83	25.6	153
2.04	151	908	2.74	83.2	499	3.44	52.2	313	4.14	35.5	213	4.84	25.5	152
2.05	150	899	2.75	82.6	495	3.45	51.8	311	4.15	35.3	212	4.85	25.4	151
2.06	148	890	2.76	81.9	492	3.46	51.5	309	4.16	35.1	211	4.86	25.3	151
2.07	147	882	2.77	81.3	488	3.47	51.2	307	4.17	34.9	210	4.87	25.1	151
2.08	146	873	2.78	80.8	485	3.48	50.9	306	4.18	34.8	209	4.88	25.0	150
2.09	144	865	2.79	80.2	481	3.49	50.6	304	4.19	34.6	208	4.89	24.9	150
2.10	143	856	2.80	79.6	477	3.50	50.3	302	4.20	34.4	207	4.90	24.8	149
2.11	141	848	2.81	79.0	474	3.51	50.0	300	4.21	34.2	205	4.91	24.7	148
2.12	140	840	2.82	78.4	471	3.52	49.7	298	4.22	34.1	204	4.92	24.6	148
2.13	139	832	2.83	77.9	467	3.53	49.4	297	4.23	33.9	203	4.93	24.5	147
2.14	137	824	2.84	77.3	464	3.54	49.2	295	4.24	33.7	202	4.94	24.4	146
2.15	136	817	2.85	76.8	461	3.55	48.9	293	4.25	33.6	201	4.95	24.3	146
2.16	135	809	2.86	76.2	457	3.56	48.6	292	4.26	33.4	200	4.96	24.2	145
2.17	134	802	2.87	75.7	454	3.57	48.3	290	4.27	33.2	199	4.97	24.1	144
2.18	132	794	2.88	75.1	451	3.58	48.0	288	4.28	33.1	198	4.98	24.0	144
2.19	131	787	2.89	74.6	448	3.59	47.7	286	4.29	32.9	198	4.99	23.9	143
2.20	130	780	2.90	74.1	444	3.60	47.5	285	4.30	32.8	197	5.00	23.8	143
2.21	129	772	2.91	73.6	441	3.61	47.2	283	4.31	32.6	196	5.01	23.7	142
2.22	128	765	2.92	73.0	438	3.62	46.9	282	4.32	32.4	194	5.02	23.6	141
2.23	126	758	2.93	72.5	435	3.63	46.7	280	4.33	32.3	195	5.03	23.5	141
2.24	125	752	2.94	72.0	432	3.64	46.4	278	4.34	32.1	193	5.04	23.4	140
2.25	124	745	2.95	71.5	429	3.65	46.1	277	4.35	32.0	192	5.05	23.3	140
2.26	123	738	2.96	71.0	426	3.66	45.9	275	4.36	31.8	191	5.06	23.2	139
2.27	122	732	2.97	70.5	423	3.67	45.6	274	4.37	31.7	190	5.07	23.1	138
2.28	121	725	2.98	70.1	420	3.68	45.4	272	4.38	31.5	189	5.08	23.0	138
2.29	120	719	2.99	69.6	417	3.69	45.1	271	4.39	31.4	188	5.09	22.9	137
2.30	119	712	3.00	69.1	415	3.70	44.9	269	4.40	31.2	187	5.10	22.8	137
2.31	118	706	3.01	68.6	412	3.71	44.6	268	4.41	31.1	186	5.11	22.7	136
2.32	117	700	3.02	68.2	409	3.72	44.4	266	4.42	30.9	185	5.12	22.6	135
2.33	116	694	3.03	67.7	406	3.73	44.1	265	4.43	30.8	185	5.13	22.5	135
2.34	115	688	3.04	67.3	404	3.74	43.9	263	4.44	30.6	184	5.14	22.4	134
2.35	114	682	3.05	66.8	401	3.75	43.6	262	4.45	30.5	183	5.15	22.3	134
2.36	113	676	3.06	66.4	398	3.76	43.4	260	4.46	30.3	182	5.16	22.2	133
2.37	112	670	3.07	65.9	395	3.77	43.1	259	4.47	30.2	181	5.17	22.1	133
2.38	111	665	3.08	65.5	393	3.78	42.9	257	4.48	30.0	180	5.18	22.0	132
2.39	110	659	3.09	65.0	390	3.79	42.7	256	4.49	29.9	179	5.19	21.9	132
2.40	109	653	3.10	64.6	388	3.80	42.4	255	4.50	29.8	179	5.20	21.8	131
2.41	108	648	3.11	64.2	385	3.81	42.2	253	4.51	29.6	178	5.21	21.7	130
2.42	107	643	3.12	63.8	383	3.82	42.0	252	4.52	29.5	177	5.22	21.6	130
2.43	106	637	3.13	63.3	380	3.83	41.7	250	4.53	29.3	176	5.23	21.5	129
2.44	105	632	3.14	62.9	378	3.84	41.5	249	4.54	29.2	175	5.24	21.4	129
2.45	104	627	3.15	62.5	375	3.85	41.3	248	4.55	29.1	174	5.25	21.3	128
2.46	104	621	3.16	62.1	373	3.86	41.1	246	4.56	28.9	174	5.26	21.2	128
2.47	103	616	3.17	61.7	370	3.87	40.9	245	4.57	28.8	173	5.27	21.1	127
2.48	102	611	3.18	61.3	368	3.88	40.6	244	4.58	28.7	172	5.28	21.1	127
2.49	101	606	3.19	60.9	366	3.89	40.4	242	4.59	28.5	171	5.29	21.0	126
2.50	100	601	3.20	60.5	363	3.90	40.2	241	4.60	28.4	170	5.30	20.9	126
2.51	99.4	597	3.21	60.1	361	3.91	40.0	240	4.61	28.3	170	5.31	20.9	125
2.52	98.6	592	3.22	59.8	359	3.92	39.8	239	4.62	28.1	169	5.32	20.8	125
2.53	97.8	587	3.23	59.4	356	3.93	39.6	237	4.63	28.0	168	5.33	20.7	124
2.54	97.1	582	3.24	59.0	354	3.94	39.4	236	4.64	27.9	167	5.34	20.6	124
2.55	96.3	578	3.25	58.6	352	3.95	39.1	235	4.65	27.8	167	5.35	20.5	123
2.56	95.5	573	3.26	58.3	350	3.96	38.9	234	4.66	27.6	166	5.36	20.4	123
2.57	94.8	569	3.27	57.9	347	3.97	38.7	232	4.67	27.5	165	5.37	20.3	122
2.58	94.0	564	3.28	57.5	345	3.98	38.5	231	4.68	27.4	164	5.38	20.3	122
2.59	93.3	560	3.29	57.2	343	3.99	38.3	230	4.69	27.3	164	5.39	20.2	121
2.60	92.6	555	3.30	56.8	341	4.00	38.1	229	4.70	27.1	163	5.40	20.1	121
2.61	91.8	551	3.31	56.5	339	4.01	37.9	228	4.71	27.0	162	5.41	20.0	121
2.62	91.1	547	3.32	56.1	337	4.02	37.7	226	4.72	26.9	161	5.42	19.9	120
2.63	90.4	543	3.33	55.8	335	4.03	37.5	225	4.73	26.8	161	5.43	19.9	119
2.64	89.7	538	3.34	55.4	333	4.04	37.3	224	4.74	26.6	160	5.44	19.8	119
2.65	89.0	534	3.35	55.1	331	4.05	37.1	223	4.75	26.5	159	5.45	19.7	118
2.66	88.4	530	3.36	54.8	329	4.06	37.0	222	4.76	26.4	158	5.46	19.6	118
2.67	87.7	526	3.37	54.4	326	4.07	36.8	221	4.77	26.3	158	5.47	19.5	117
2.68	87.0	522	3.38	54.1	325	4.08	36.6	219	4.78	26.2	157	5.48	19.5	117
2.69	86.4	518	3.39	53.8	323	4.09	36.4	218	4.79	26.1	156	5.49	19.4	116

TABLE 12.—Brinell hardness numbers—Continued

Di- am- eter of inden- tation	Brinell hardness No.		Di- am- eter of inden- tation	Brinell hardness No.		Di- am- eter of inden- tation	Brinell hardness No.		Di- am- eter of inden- tation	Brinell hardness No.		Di- am- eter of inden- tation	Brinell hardness No.	
	500- load	3,000- load		500- load	3,000- load		500- load	3,000- load		500- load	3,000- load		500- load	3,000- load
<i>Mm.</i>	<i>Kg.</i>	<i>Kg.</i>	<i>Mm.</i>	<i>Kg.</i>	<i>Kg.</i>	<i>Mm.</i>	<i>Kg.</i>	<i>Kg.</i>	<i>Mm.</i>	<i>Kg.</i>	<i>Kg.</i>	<i>Mm.</i>	<i>Kg.</i>	<i>Kg.</i>
5.50	19.3	116	5.80	17.2	103	6.10	15.3	92.0	6.40	13.7	82.5	6.70	12.4	74.1
5.51	19.2	115	5.81	17.1	103	6.11	15.3	91.7	6.41	13.7	82.2	6.71	12.3	73.9
5.52	19.2	115	5.82	17.0	102	6.12	15.2	91.3	6.42	13.6	81.9	6.72	12.3	73.6
5.53	19.1	114	5.83	17.0	102	6.13	15.2	91.0	6.43	13.6	81.6	6.73	12.2	73.4
5.54	19.0	114	5.84	16.9	101	6.14	15.1	90.6	6.44	13.5	81.3	6.74	12.2	73.1
5.55	18.9	114	5.85	16.8	101	6.15	15.1	90.3	6.45	13.5	81.0	6.75	12.1	72.8
5.56	18.9	113	5.86	16.8	101	6.16	15.0	90.0	6.46	13.4	80.7	6.76	12.1	72.6
5.57	18.8	113	5.87	16.7	100	6.17	14.9	89.6	6.47	13.4	80.4	6.77	12.1	72.3
5.58	18.7	112	5.88	16.7	99.9	6.18	14.9	89.3	6.48	13.4	80.1	6.78	12.0	72.1
5.59	18.6	112	5.89	16.6	99.5	6.19	14.8	89.0	6.49	13.3	79.8	6.79	12.0	71.8
5.60	18.6	111	5.90	16.5	99.2	6.20	14.8	88.7	6.50	13.3	79.6	6.80	11.9	71.6
5.61	18.5	111	5.91	16.5	98.8	6.21	14.7	88.3	6.51	13.2	79.3	6.81	11.9	71.3
5.62	18.4	110	5.92	16.4	98.4	6.22	14.7	88.0	6.52	13.2	79.0	6.82	11.8	71.1
5.63	18.3	110	5.93	16.3	98.0	6.23	14.6	87.7	6.53	13.1	78.7	6.83	11.8	70.8
5.64	18.3	110	5.94	16.3	97.7	6.24	14.6	87.4	6.54	13.1	78.4	6.84	11.8	70.6
5.65	18.2	109	5.95	16.2	97.3	6.25	14.5	87.1	6.55	13.0	78.2	6.85	11.7	70.4
5.66	18.1	109	5.96	16.2	96.9	6.26	14.5	86.7	6.56	13.0	77.9	6.86	11.7	70.1
5.67	18.1	108	5.97	16.1	96.6	6.27	14.4	86.4	6.57	12.9	77.6	6.87	11.6	69.9
5.68	18.0	108	5.98	16.0	96.2	6.28	14.4	86.1	6.58	12.9	77.3	6.88	11.6	69.6
5.69	17.9	107	5.99	16.0	95.9	6.29	14.3	85.8	6.59	12.8	77.1	6.89	11.6	69.4
5.70	17.8	107	6.00	15.9	95.5	6.30	14.2	85.5	6.60	12.8	76.8	6.90	11.5	69.2
5.71	17.8	107	6.01	15.9	95.1	6.31	14.2	85.2	6.61	12.8	76.5	6.91	11.5	68.9
5.72	17.7	106	6.02	15.8	94.8	6.32	14.1	84.9	6.62	12.7	76.2	6.92	11.4	68.7
5.73	17.6	106	6.03	15.7	94.4	6.33	14.1	84.6	6.63	12.7	76.0	6.93	11.4	68.4
5.74	17.6	105	6.04	15.7	94.1	6.34	14.0	84.3	6.64	12.6	75.7	6.94	11.4	68.2
5.75	17.5	105	6.05	15.6	93.7	6.35	14.0	84.0	6.65	12.6	75.4	6.95	11.3	68.0
5.76	17.4	105	6.06	15.6	93.4	6.36	13.9	83.7	6.66	12.5	75.2	6.96	11.3	67.7
5.77	17.4	104	6.07	15.5	93.0	6.37	13.9	83.4	6.67	12.5	74.9	6.97	11.3	67.5
5.78	17.3	104	6.08	15.4	92.7	6.38	13.8	83.1	6.68	12.4	74.7	6.98	11.2	67.3
5.79	17.2	103	6.09	15.4	92.3	6.39	13.8	82.8	6.69	12.4	74.4	6.99	11.2	67.0

APPARATUS

1. The divisions on the micrometer scale of the microscope or other measuring device used for the measurement of the diameter of impression shall be such as to permit the direct measuring of the diameter to 0.1 millimeter and the estimation of the diameter to 0.01 millimeter.

2. (a) The balls used in Brinell hardness testing shall be of hardened steel or other equally hard material.

(b) A ball to be suitable for use shall not show a permanent change in diameter greater than 0.0025 millimeter (0.0001 inch) when pressed with a force of 3,000 kilograms against a piece of steel having a Brinell hardness number of 500 or greater.⁶⁵

(c) If a ball is used in a test of a specimen which shows a Brinell hardness number greater than 500, the ball shall be remeasured after the test. Should the ball show a permanent change in diameter greater than that permitted in paragraph (b), the ball shall be unsuitable for further use in a standard test.⁶⁶

3. The standard ball for Brinell hardness testing shall be 10 millimeters (0.3937 inch) in diameter with a deviation from this value of not more than 0.01 millimeter (0.0004 inch) in any diameter, but the diameter of any ball measured at various points shall be constant within a tolerance of ± 0.0025 millimeter (0.0001 inch).

For testing very small specimens or very thin specimens it is sometimes necessary to make Brinell hardness tests with a ball less than 10 millimeters in diameter. For such tests (which are not to be regarded as standard Brinell

⁶⁵ In testing softer metals, it is not, of course, necessary to have balls of this extreme hardness. However, a ball to be suitable for use should withstand pressure against the hardest material to be tested without showing a permanent change in diameter greater than 0.0025 millimeters.

⁶⁶ See footnote 64.

tests) the relation between applied load, P , measured in kilograms, and diameter of ball, D , measured in millimeters, should be

$$P=3,000 \frac{D^2}{10^2}=30 D^2 \text{ for iron and steel}$$

$$\text{and } P=500 \frac{D^2}{10^2}=5 D^2 \text{ for brass, bronze, and soft metals in general.}$$

For extremely soft metals the relation $P=D^2$ may be used.

Balls differing in size from the standard 10-millimeter ball shall conform to the requirements for the material and the tolerance in size of diameter as specified for the standard ball.

TEST SPECIMENS

Specimens used in Brinell hardness testing vary greatly in form since it is frequently desirable to make the impression upon a part to be used in the finished product rather than upon a sample test specimen.

4. The thickness of the piece tested shall be such that no bulge or other marking showing the effect of the load appears on the side of the piece opposite the impression. In any event, the thickness of the specimen shall be at least ten times the depth of impression.

5. The surface on which the impression is to be made shall be filed, ground, machined, or polished with emery paper so that the edge of the impression shall be clearly enough defined to permit of the measurement of the diameter within 0.01 millimeter (0.0004 inches).

APPLICATION AND MAGNITUDE OF LOAD

6. The load in the standard Brinell test shall be 3,000 kilograms for iron and steel and 500 kilograms for brass, bronze, and soft metals generally.

NOTE.—For extremely soft metals a load of 100 kilograms is sometimes used. If for any reason it is necessary to use loads differing from those specified above, the load used should be specifically stated in the test report, conveniently as a subscript of the hardness numeral.

7. (a) The load shall be applied steadily to the specimen without jerk.

(b) The load shall be applied for at least 10 seconds in the case of iron and steel and for at least 30 seconds in the case of other metals.⁶⁷

(c) The center line of the applied load shall be far enough from any edge of the specimen so that when a straightedge is applied to the edge after the load has been applied, the eye can detect no bulge in the edge due to the load and the resulting impression.

MEASUREMENT OF IMPRESSION

8. In the standard Brinell hardness test the diameter of the impression shall be measured.

9. When a micrometer microscope is used to measure the diameter of the impression the sharpness of definition of the edge of the impression can be increased by the use of a movable lamp for illuminating the specimen, placing the lamp so that the contrast of light and shade will bring first one edge of the impression, then the other into sharp definition. In testing very hard material, the sharpness of definition of the impression can be somewhat increased by the use of a ball lightly etched with nitric acid, or by the use of some pigment, such as Prussian blue, on the ball. In testing material in which there is considerable recovery of shape, the material may first be coated with a dull black pigment, such as drawing ink or a mixture of graphite and alcohol. The edge of the impression is rendered clear on a surface so coated.

BRINELL HARDNESS TESTS IN WHICH DEPTH OF IMPRESSION IS MEASURED IN PLACE OF DIAMETER

When it is necessary to make Brinell hardness tests very rapidly, as is sometimes the case when the Brinell test is used as a control test for the output

⁶⁷ It was suggested in discussion that for magnesium and magnesium alloys the minimum time of application be two minutes. It was also suggested that for any given soft metal a definite time for holding the load be specified rather than a minimum time. See discussion of the report of committee E-1, (16, p. 729-730).

of a heat-treating furnace, the depth of impression under load, or after the release of load, can be measured more expeditiously than the diameter of impression. The depth of impression is usually determined from the relative motion of the plunger carrying the ball and the specimen. This method of determining Brinell hardness is not to be regarded as a standard method, and when requirements of speed necessitate its use, the Brinell hardness number should not be determined from the geometrical relation of depth of impression to diameter, based on the assumption that the impression has a spherical surface. In such tests the relation between the Brinell hardness number and depth of impression may be determined with a fair degree of accuracy from tests on specimens of material of different degrees of hardness, several impressions being made on each specimen, and the depth under load, or the depth on released of load, and the diameter of impression after release of load being measured for each impression. By the use of a diagram or by interpolation, a table giving the Brinell hardness number for a given depth of impression can be prepared.

CALIBRATION OF BRINELL HARDNESS TESTING APPARATUS

10. A load-measuring device which is in common use for Brinell hardness-testing machines involves the measurement of fluid pressure on a piston of known diameter, working in a cylinder with so perfect a fit that packing is not necessary. To this piston is attached the ball. Other load-measuring devices use combinations of weights and levers. The use of dead weights acting on a small piston is to be preferred to the ordinary Bourdon gauge for measuring the intensity of fluid pressure.

11. The load-measuring device of a Brinell hardness-testing machine may be calibrated by the use of dead weights, or by the use of dead weights and proving levers or by an elastic calibration device or spring, in the manner described in the standard methods of verification of testing machines (serial designation (3. 13) E 4, see p. 105) of the American Society for Testing Materials.

12. The load-measuring device of a Brinell hardness-testing machine may be calibrated by making a series of impressions on specimens of different degrees of hardness, measuring the diameters of the impressions, making a second series of impressions by the use of any standardized testing machine and standard steel ball, and, using the same measuring device as for the first series of impressions, measuring the diameters of this second series of impressions. The error of the machine under calibration can be determined by the relative average values of the hardness numbers determined by its use and by the use of the standardized testing machine.

13. When the above companion-impression method of calibrating a Brinell hardness-testing machine is used it is recommended that the specimens used be approximately 1 by 1 by 12 inches in size, and that specimens with as wide a range of hardness as is feasible be used. It is recommended that impressions made by the machine under calibration be spaced about 2.5 inches apart along the specimen, and that the impressions made by the standardized testing machine be spaced alternately along the length of the specimen with the impressions made by the machine under calibration.

14. If any determination of hardness, either for the machine under calibration or for the standard machine differs by more than 2 per cent from the mean of the four determinations made with that machine it is recommended that such determination be discarded. If two determinations differ more than 2 per cent from the mean, the whole series should be discarded.

15. A Brinell hardness-testing machine is acceptable for use over a loading range within which its load-measuring device is correct within 3 per cent.

16. The micrometer microscope or other device for measuring diameter of impression may be calibrated by using it to measure lengths on a standard steel or glass scale. The eyepiece of a micrometer microscope should be adjusted so that throughout the range covered the error of reading does not exceed 0.01 millimeters.

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