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REPORT

ON

AN INVESTIGATION OF A BRIQUETTE STAIN NUMBER TEST FOR LABORATORY AND FIELD DETERMINATION OF THE ASPHALT CONTENT IN BITUMINOUS MIXTURES.

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INVESTIGATION OF A BRIQUETTE STAIN NUMBER TEST FOR LABORATORY AND FIELD DETERMINATION OF THE ASPHALT CONTENT IN BITUMINOUS MIXTURES.

INTRODUCTION

One of the most difficult tasks confronting the engineer in charge of an asphaltic concrete paving job is the determination of the optimum percentage of asphaltic binder. The need for careful regulation of the bitumen content is too well known to require further exposition here, but, in brief, too little asphalt will allow the pavement to ravel - too much will cause instability.

The problem naturally resolves itself into three parts; first, a Laboratory determination of the optimum ratio of asphalt to sand for the average gradations of the particular sand to be used on the job; second, field regulation of the asphalt to sand ratio to compensate for unavoidable changes in the aggregates; and third, accurate and thorough field control of the proportioning and mixing of the asphalt and aggregate.

The solution of the first part of the problem depends largely on the preliminary laboratory investigation made on the sand samples submitted from the field. This laboratory investigation determines the general qualities of the sand and the approximate asphalt ratio which will give the highest stability values, as measured by some laboratory stability test. It is the present practice of the California Division of Highways to use the Hubbard-Field method for this purpose.

The sand used on the job may vary considerably in grading and other characteristics from the sample furnished the laboratory, thus requiring different asphalt-sand ratios from those determined for the original sample.

This brings up the second part of the problem- when to alter the asphalt ratio and how much. If the variation is only in the grading, the experienced field engineer can readily vary the asphalt slightly to compensate, but if the character of the sand changes or new combinations of sand and filler dust are used, the field engineer must vary the proportions until in his judgment the asphalt ratio is properly readjusted. The only means available to him in the past for arriving at a decision in the field have been experience and the ability to guess right from the "feel", appearance, and the behavior of the mixture under the roller.

The third part of the problem - that of adjusting the operation of the plant scales, pug mill, etc., is usually solved in the first few days of operating, but a daily check

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of the plant operation is essential as insurance against trouble. Here again the engineer has been forced to rely on rough field tests and experience until the results of the laboratory tests are received. Several days usually elapse before the laboratory results are available, meanwhile the plant must be kept in operation, frequently turning out inferior mixtures while waiting for more accurate information.

PURPOSE OF THE PRESENT INVESTIGATION

The field engineer in charge of an asphaltic concrete mixing plant has long been in need of a simple but accurate test for determining the bitumen content of asphaltic concrete mixtures. To be practicable for field use, such a test should not only be accurate, but rapid and easy to make by men not particularly skilled in laboratory technique and should not require elaborate or expensive apparatus.

It is believed that the above requirements are met by the "Briquette Stain Test" as modified and amplified in the laboratory of the Materials and Research Department and described herein.

The "briquette stain number" test is based on the principles embodied in the well known "pat" stain test for determining the relative bitumen content of an asphaltic mixture.

The original crude "pat" stain test was improved by the Municipal Testing Laboratory of Rochester, N.Y., under the direction of Mr. Henry L. Howe*, who developed the compression stain print method, which consists of compressing the hot paving mixture to 3000 lbs. per sq. inch over a piece of brown kraft paper and examining the appearance of the stain.

A further improvement was reported by Mr. Donald M. Wilson in a paper read before the Chemical Engineering Group Society of Chemical Industry on October 9, 1931+.

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*Characteristics of Sheet Asphalt Paving Mixtures Proceedings Sixth Annual Asphalt Paving Conference 1927.

+The Manufacture and Testing of Asphalt Paving Materials. The Highway Engineer, January, 1932.

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This method which has been used successfully in England, consists essentially of compressing 200g. of asphaltic material heated to 200°C.(392°F.) and placed in a 2 in. internal diameter mold between wads of filter paper, to a pressure of 6000 pounds per square inch applied for 30 seconds. The papers of both wads are separated and examined and the average number showing a stain in the central portion to the nearest whole number is reported as the "pat stain number."

The original English procedure has been modified in this study, principally by using 110 g. per test so that the briquette, after separating from the papers, can be tested in the Hubbard-Field Machine*, thus securing additional information as to Hubbard-Field stability values at slight extra labor.

APPARATUS

The briquettes are fabricated in a mold (Fig. No.1) consisting of a hollow cylinder 4.5 in. long and 2.000 in. inside diameter, recessed at the bottom to a diameter of 2.8 in. for a depth of .25 in., a base plate, and a plunger having a thermometer well in the top.

A hydraulic jack bolted to the base of a framework and fitted with a special pump and a 20,000 pound gauge is used for compressing the briquette (Fig.No.2). A hot plate, several 5½ in. milk pans, a 100°-400°F.copper tipped thermometer, funnel, hammer, balance, ring for supporting the mold while removing the briquette, and a supply of 7.0 cm. filter paper discs complete the necessary equipment. Any suitable filter paper can be used. No. 4 Whatman filter paper was used in the tests described herein.

TEST PROCEDURE*

110 gm. of the minus 10 matrix material screened from the whole mix (or sand and asphalt proportioned and mixed in the laboratory) are placed in a small pan and heated on the hot plate to 300°F± 5°F.,stirring thoroughly meanwhile with the thermometer. The mold is heated to a temperature of 240°F± 10°F. as measured by a thermometer in

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*A Practical Method for Determining the Relative Stability of Fine Aggregate Paving Mixtures, by Prevost Hubbard and F. C. Field. Technical Papers, American Society for Testing Materials, Vol. 25, 1925, Part II, Page 335.

*See Appendix for further details.

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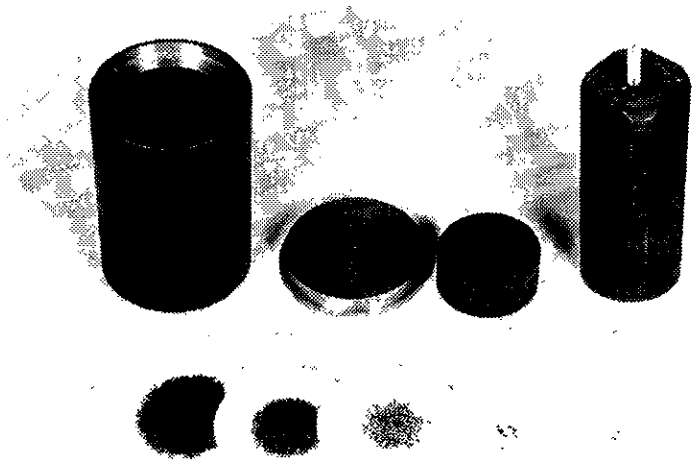

FIG. 1

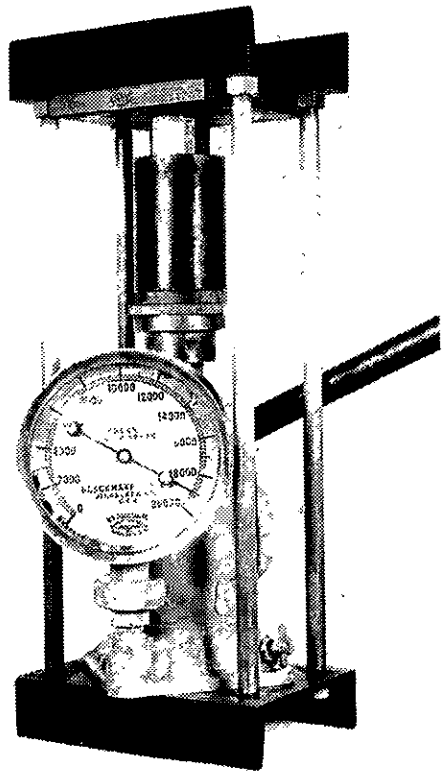

FIG. 2

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well of the plunger. Twenty filter paper discs are then placed in the enlarged bottom of the mold on top of the base plate. The hot mixture is poured into the mold, smoothed, and slightly compacted by several light blows with the plunger, after which the assembled mold is placed in the hydraulic press. The specimen is subjected to a pressure of 6000 pounds per square inch for a period of 60 seconds, applying the load at a uniform rate and taking 15 seconds to attain full pressure. The mold is then allowed to cool for 5 minutes, the briquette removed, the filter papers separated, and the number stained noted.

Pressure applied to the uncompressed material in the mold squeezes the sand grains closer together, displacing part of the asphalt film and forcing it into the voids between the grains and out into the layers of filter paper. The number of papers stained is a measure of the amount of asphalt forced out and is referred to as the "Briquette Stain Number".

The stain test may be considered a measure of the ability of the sand to carry asphalt, which property is a combination of several factors, such as voids, surface area, and surface texture of the sand. When mixtures of a given asphalt-sand ratio are compressed, a certain amount of asphalt will be forced into the filter paper, the quantity depending upon the above mentioned properties, and the test conditions, such as temperature and presssure.

The limits fixed for temperature, pressure, time, etc., are based on a series of tests in which these factors were varied until the desired degree of sensitivity, combined with accuracy was obtained; i.e., 300°F. was chosen as the highest temperature that could readily be reached without danger of overheating, 6000 pounds per suare inch was selected as the practical working pressure, 60 seconds compression to insure time enough for thorough compaction.

PRELIMINARY WORK AND TYPICAL RESULTS

Enough Briquette Stain Number tests, using the above described method, were made on sands from various parts of the state to indicate that the method is applicable to any sand. The data for one of these sands, included in this report, which are typical for any sand or sand mixtures, were obtained from a mixture of a crusher waste, a decomposed granite, and a filler dust, combined with varying percentages of asphalt. Fig. No. 3 shows graphically the values for the

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stain number, stain test briquette stability*, and apparent specific gravity.

It will be observed that the increase in the briquette stain number is gradual as more asphalt is added to the sand up to a certain critical point (A) or "break" in the slope of the stain number curve, after which the rise is quite rapid and at a fairly uniform rate.

The stability curve is of special interest, as the point of highest stability (B) has the same asphalt ratio as the break in the stain curve at (A). Attention is called to a second peak (C) in the stability curve. This second peak had not hitherto been suspected and was at first viewed with considerable skepticism. It was logical to assume that stabilities as measured in the Hubbard-Field machine would increase to a maximum and then continuously decrease as more asphalt was added. Such a simple relationship, however, does not appear to be the case.

Careful check tests, however, show the second peak occurring consistently in every sand tested. It is our belief that the condition influencing this second rise has no relation to the inherent stability of the mix, but in all probability is due to a lateral expansion of the higher asphalted and more plastic specimen with consequent friction against the side walls as the plunger descends. The resistance against the side walls retards the free movement and causes a slight rise in the pressure required to produce failure of the specimen.

The curves in Fig. No. 3 and their interrelations are typical for all sands, but numerical values, slope and relation to the bitumen ratio vary widely for different sands and sand filler combinations, as shown in Fig. No. 4. For this reason each sand or sand mixture must be tested at the headquarters laboratory to determine the optimum asphalt-sand ratio and the maximum stability obtainable.

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*The "Stain Test Briquette Stability", hereafter designated as S.T.B. Stability to differentiate it from the Hubbard-Field (H.F.) Stability Values for standard fabricated specimens is the "Stability Value" obtained by extruding the briquette fabricated during the stain test by the same procedure as is used for testing Standard H. F. Briquettes.

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The sands (Fig. 4) represent extreme types, No. 1 being a crushed granite having sharp, irregular and very rough particles, while No. 6 is a desert drift sand composed largely of smooth rounded quartz grains. The great difference in the characteristics of the sand is reflected particularly well in the stain number curves. Thus the "break" in the curve for crushed granite (No. 1) is sharp, but the "break" in the curve for the desert sand (No. 6) is more rounded and at a higher asphalt ratio. Stain curve No. 4 represents a half and half blend of the two sands and is located midway between the other two. Stain number curves Nos. 2,3, and 5 depict various blends having intermediate characteristics.

The stability value curves Nos. 1,4, and 6 also reflect the differences in the sands, however the maximum stability of each is located approximately over the break in its respective stain curve. Stability curves Nos. 2,3, and 5 show only the portions of the curves past the maximum, but indicate the relative positions of these blends with respect to the other stability curves. Sand No. 1 is much more critical as to the asphalt-sand ratio, as shown by the sharpness of the break in the stain curve and the comparative closeness of the two stability peaks.

The sharpness of these stability peaks and the high strength is usually found to be a characteristic of cru her waste and rough sands, while smoother, rounded sands as No. 6 have lower stabilities and the peaks are widely separated. The erratic results which would be obtained from variations in blending these two sands at the mixing plant are thus readily apparent.

Figures Nos. 5 and 6 show data for stain number, specific gravity, S.T.B. stability, and H.F. stability values obtained by adding 3.0% diatomaceous earth and 20.0% limestone dust,respectively, as a filler to a fifty-fifty blend of the two sands covered by Fig. 4. Stain tests were made over a wide range of asphalt ratios with the diatomaceous earth mixture and at intervals of 0.1% over the critical region, thus locating the curves with greater precision than would be necessary as a general rule. The accuracy of the stain test may be judged by the close alignment of most of the points. It may be noted that by adding diatomaceous earth to the sand, more asphalt was required to reach the maximum stability, while limestone dust reduced the required amount. Higher stabilities were, of course, obtained by adding fillers to the sands.

The results of the investigation with laboratory mixed sands and asphalts have shown that the stain test described herein is both sensitive and accurate and provides a rapid means for controlling the asphalt content at the optimum percentage required by the minus 10 material, based on the Hubbard-Field stability test.

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Asphaltic concrete under California Division of Highways specifications is of the "matrix" type; that is, the larger rocks are plums in the mortar mix of asphalt and fine material. The stability of this matrix is therefore of prime importance, if we assume that it determines the stability of the entire mix.

The quality of this matrix is determined by the Hubbard-Field stability test made on that portion of the whole mix, which is screened through a No. 10 mesh sieve. Stability value requirements have been fixed at a minimum of 1500 pounds for the base and leveling courses and 3000 pounds for the surface course when tested on the California Division of Highways' Hubbard-Field Asphalt Stability Testing Machine[x].

The optimum asphalt to fine aggregate ratio is obtained by first making a preliminary laboratory investigation to determine the ratio of asphalt to sand (sand and filler) which will produce the highest Hubbard-Field stability with the fine aggregate in question. The percentage of asphalt required for the whole mix is then calculated by assuming that the aggregate larger than 10 mesh will require 2.0 percent of asphalt by weight of the coarse aggregate. These determinations, together with stain number and stability curves are sent to the resident engineer for his guidance.

APPLICATION TO FIELD USE

Although the briquette Stain Number Test has proved its value for laboratory investigations of sands and filler dusts, its greatest usefulness is in the field. The test apparatus is so light and portable that it can be set up alongside the contractor's pug mill and used for making frequent checks on the operation of the mixing plant and to detect any variations in the character of the fine aggregate requiring a compensating change in the percentage of asphalt.

As previously pointed out and as illustrated in Fig. 4 each sand, or sand and filler dust blend, has its own characteristics which affect the shape of the stain number curve and the location (i.e. asphalt ratio and stain number) of the "break" in the curve. The grading of the sand, particularly the quantity of minus 200 mesh material, also affects the location of the "break" in the stain curve and slightly modifies its shape. Thus the field engineer should be provided with stain number and stability value curves for each blend of fine aggregate being used. These curves should also show the influence of any variations in the fine aggregate likely to occur during the progress of the work.

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[x] Standard Specifications, State of California, Department of Public Works, Division of Highways, January, 1935, Section 32, Article (c).

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In the event it is desired to obtain the highest possible H.F. stability values it is only necessary to keep the stain number within the limits predetermined by the Headquarters Laboratory. However, if it should be thought preferable to sacrifice H.F. stability values by increasing the asphalt ratio to obtain other benefits, the stain number of the ultimately adopted bitumen ratio may be chosen as the standard for field control purposes.

In either case the stain number test will foretell the approximate H.F. stability of the sample and its bitumen content. These values may be predicted with much greater accuracy if the surface area of the fine aggregate portion of the batch sampled for a stain number test is known. This factor may be readily obtained by sampling the fine aggregate from bin No. 1 and the whole mix simultaneously. If a grading analysis of the fine aggregate is thus determined the stability may be read from the curve of similar surface area as shown in Figures Nos. 7 and 8.

The values shown in Figures Nos. 7 and 8 were obtained by taking 60 simultaneous field samples of fine aggregate and matrix material and testing the former for sieve analysis and the latter for stain number, S.T.B. stability and H. F. stability. The stain number curve was obtained from preliminary laboratory tests made on an average blend of the sand being used. The family of stability curves were obtained by plotting the values for S.T.B. and H. F. stability values above the intersection of the ordinate of the stain number of the sample and designating the surface area of the aggregate by various shaped symbols as shown in Fig. No. 7. This method segregated the stability values according to surface area. Thus four portions of matrix material having stain numbers of 12½ and surface areas of 72, 77, 82, and 87 square feet would have S.T.B. stability values of 4175,4000,3725, and 3425 pounds respectively. It should be noted that the mixtures containing the most fines have the lowest stability values, which may account for the H.F. stability values for surface course samples containing filler dust frequently being lower than base and leveling course mixtures from the same job. This phenomenon can only occur when the asphalt ratio is too high and within certain limits. If the asphalt content were reduced so that the stain number would be from 3½ to 5½, the fine mixes would show the higher stability values. It should also be noted that the finer mixtures are more critical than the coarser mixtures regarding the asphalt content.

The same values have been plotted in Fig. No. 8, but the stability values and bitumen ratios have been plotted directly against the stain number. This method appears to be

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a more logical form for field use than the arrangement in Fig. 7, but either may be used. These charts will enable the field engineer to readily evaluate the stain number test in terms of stability and percent of asphalt.

In the event it should be necessary to make a change in the mixture the affect of the adjustments can be determined within a few minutes rather than the several days that must necessarily elapse before test results can be obtained from samples sent to the Headquarters Laboratory.

The stain test is also very useful to check on the operation of the plant; i.e., to determine if the asphalt and filler dust are being uniformly distributed by the pug mill; the time required for thorough mixing; and the ability of the asphalt weigh bucket to deliver a constant weight of asphalt.

At present the plant man is forced to rely upon a visual inspection and experience to make these adjustments, and then either wait several days until samples of the mix have been tested in the Headquarters Laboratory and reported to him, or trust to approximate extractions or other rough field tests. The field Briquette Stain Test affords an immediate and accurate check on the plant operations and so removes the guess work unavoidably connected with past procedure.

For example, the distribution of asphalt is of prime importance. The usual field method for determining this is to take samples of the mix from each end of the pug mill and compare their appearance. If there is a material difference in the quantity of asphalt at either end, it can be detected by this crude method and the asphalt chute adjusted until each appears to be alike. With the equipment for making a Briquette Stain Test a comparison of the asphalt-sand ratio in each sample may be determined as accurately as a laboratory extraction, and this information can be obtained within ten minutes from the time the samples are taken.

The same procedure will tell if the filler dust is being uniformly distributed, i.e., the addition of dust to the mix may change the magnitude of the stain, but it should not disturb the equality, providing the asphalt is being uniformly distributed throughout the length of the pug mill.

For preliminary tests after each adjustment of the asphalt chute, the matrix material may be screened immediately through a No. 6 sieve, and the stain test made at any

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convenient temperature over 200°F. This short method is more rapid, although less accurate than the standard method described herein in which the matrix material is screened through a No. 10 sieve and heated to 300°F. While the short method is sufficiently accurate for a comparison of two samples, the standard method should be used for a comparison with other stain numbers or to determine the absolute percentages of asphalt.

APPLICATION OF THE BRIQUETTE STAIN NUMBER TEST

The Briquette Stain Number Test has found many applications; in the laboratory, supplementing the present routine tests on asphalt sands and matrix material; in the field, serving as a guide for maintaining the optimum asphalt-sand ratio, for determining the distribution of asphalt and filler dust in the pug mill, and for detecting discrepancies in the quantities of asphalt or aggregate measured into the mill.

It is proposed that for field use three stain number curves with their corresponding Hubbard-Field stability values, be prepared by the Headquarters Laboratory for each asphaltic concrete contract. These curves will show the fluctuations, over the range of asphalt ratios expected for maximum, minimum, and average gradations in the sand. The resident engineer will then be supplied with a complete briquette stain number apparatus and accessories, a copy of these data, and recommendations from the Headquarters Laboratory as to the average optimum asphalt ratio, and the stain number limits. By keeping the stain number of his mix within the limits given by the Headquarters Laboratory, he will be assured that the asphalt ratio is being held close to the point which will give the maximum Hubbard-Field stability with the materials being used.

If he should want more complete information, the stain stability-bitumen ratio curves will be of assistance. For example if for some reason he should desire to use some other bitumen ratio than the one selected, he may determine, from the curve of the proper surface area, the stain number to be used and the probable Hubbard-Field stability of the matrix material.

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SUMMARY

1. The Briquette Stain Test gives a numerical measure of the quantity of asphalt squeezed out from a given mixture of asphalt and sand (and filler dust) while being compressed into a briquette under the following carefully controlled test procedure:

The asphalt mixture is heated to 300°F± 5°F., placed over a pad of filter paper discs in a modified Hubbard-Field mold, preheated to 225°-250°F. and compacted by a pressure of 6000 pounds per square inch applied for 60 seconds.

2. The Briquette Stain Number Test gives a sensitive and accurate measure of the ability of a sand to "carry" asphalt, and (as indicated by a "break" in the slope of the stain curve) the optimum ratio of asphalt to sand for the maximum S.T.B. or H.F. stability value.

3. The "break" in the stain curve occurs at different asphalt ratios for different sands and is dependent upon the percent of voids, surface area, and surface characteristics of the sand. With various gradings of sand, the shape of the curve and location of the "break" varies slightly with the surface area of the samples.

4. The Stain Test Briquette may be separated from the filter papers and tested for stability in the Hubbard-Field apparatus. The S.T.B. stability values are usually higher than the H.F. stability on the same sample, but the maximum stability values for each correspond to the same asphalt ratio.

5. As the asphalt to sand ratio is increased, the H.F. and S.T.B. stability values rise to a maximum, decrease, rise to a second peak of lower magnitude, and again decrease.

6. The accuracy and general applicability of the Briquette Stain Number Test and its relation to the maximum stability for any sand has been conclusively demonstrated by tests made on many sands of different types, from various parts of the State of California.

7. Stain number tests on field samples of the matrix material screened from the whole mix, give results similar to laboratory mixed samples.

8. A very satisfactory correlation was obtained by plotting the S.T.B. stability and H.F. stability values against the stain number curve, after the stability values had been differentiated according to the surface areas.

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9. The Briquette Stain Number Test was found to be at least as accurate as the extraction method for determining the bitumen ratio.

10. The usefulness of the Briquette Stain Number Test for plant control, particularly to determine and maintain the optimum asphalt-sand ratio, the distribution of asphalt and filler dust in the pug mill, and the uniformity of the operation of the plant was demonstrated by field observations and laboratory tests on an asphaltic concrete paving job.

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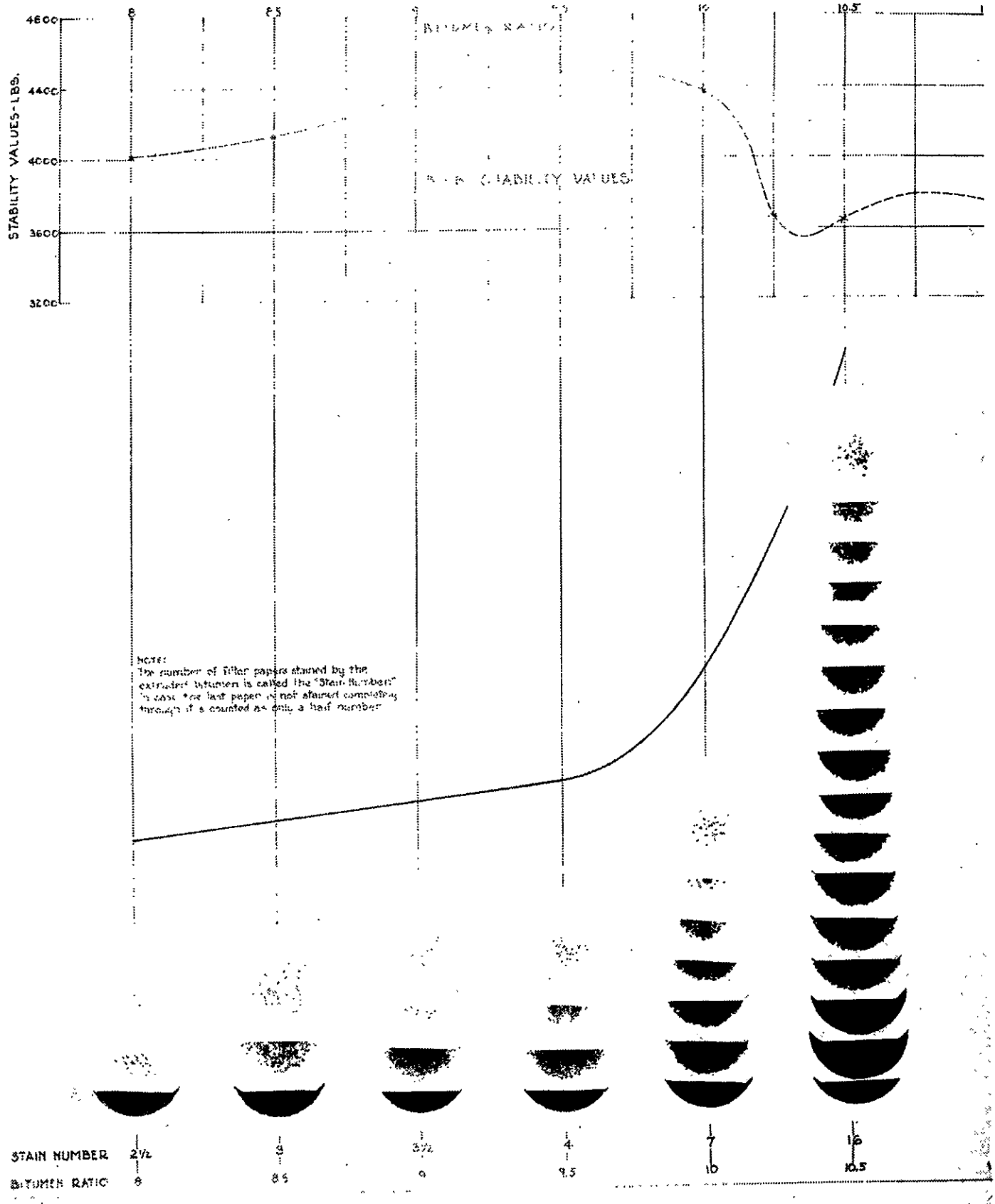
FIG. NO. 3.

SHOWING STAINED FILTER PAPERS OBTAINED BY USE OF VARIOUS BITUMINOUS RATIOS COMPARED TO S.T.B. STABILITY VALUES



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APPENDIX

STANDARD METHOD FOR THE DETERMINATION OF THE BRIQUETTE STAIN NUMBER OF FINE AGGREGATE AND BITUMINOUS BINDER MIXTURES.

This test is intended primarily for field use to determine the ratio of bituminous binder to fine aggregate and for checking upon the operations of the mixing plant.

APPARATUS

Forming Mold - A modified Hubbard-Field forming mold consisting of a 4.5 in. hollow cylinder 2.000 in. inside diameter recessed on the bottom end to a diameter of 2.8 in. and a depth of 0.25 in., a base plate, and a closely fitting plunger having a thermometer well in the top (See Figure 1).

Compression Machine - A compression machine consisting of a hydraulic jack bolted to the base of a framework and fitted with a special pump and a 20,000 pound gauge (See Figure 2).

Thermometers - Two coppered-tipped 100-400°F. thermometers with scale divided into 5°F.

Hot Plate - A solid top hot plate, either gas, electric or gasoline heated.

Mixing Pans - One dozen 5½ in. milk pans.

Funnel - A 6 in. metal funnel having a spout 1 7/8 in. outside diameter.

Hammer - A two pound hammer.

Protector Cap - A steel cap for protecting the top of the plunger.

Supporting Ring - A ring for supporting the mold while the briquette is being driven out.

Gloves - A pair of asbestos gloves.

Watch - A watch with a second-hand.

Marking Pencil - A white marking pencil for identifying briquettes.

Filter Papers - A supply of 7.0 cm. Whatman No. 4 filter papers.

Balance - A balance, capacity 1000 g., sensitive to 0.02 g., and a set of weights.

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Tongs - A pair of crucible tongs.

Tin Boxes - A supply of 3 oz. gill type tin boxes for heating asphalt.

PREPARATION OF SAMPLE

1. Sample Screened from Field Mix - The material to be tested for Briquette Stain Number shall be a representative sample of that portion of the whole mix which has been screened through a 10 mesh sieve (Similar to "minus 10 Hubbard-Field Stability samples").

2. Laboratory Mixed Samples - Fine aggregate passing a number 10 mesh sieve which has been intimately mixed with bituminous binder.

PROCEDURE

Fabrication of Briquette

1 - Weigh 110 grams of the prepared sample into a $5\frac{1}{2}$ in. milk pan. Heat on hot plate to 300°F.± 5°F. stirring thoroughly with a brass-tipped thermometer.

2 - Heat mold to a temperature of 240°F.± 10°F. as measured by a thermometer in the well of the plunger.

3 - Insert twenty filter papers into the recessed bottom end of the mold and place on base plate.

4 - Transfer the heated material to the mold by quickly inverting the milk pan over the funnel, previously inserted into the top of the mold.

5 - Remove funnel, insert plunger, cover with the protector cap and strike three light blows with the hammer. Remove cap and place the mold in hydraulic press and subject the specimen to a pressure of 6000 lbs. per sq. in. for a period of 60 seconds. The load should be applied at a uniform rate, taking 15 seconds to attain the full pressure.

6 - Release the pressure and cool for 5 to 10 minutes, place mold on supporting ring and drive the briquette out of the mold by hammering on the plunger.

Examination of Stained Papers - Separate the filter paper from briquette and note the number stained, counting each paper stained completely through as a whole number, and the last paper stained as one-half, in case the stain on the last paper was too light to penetrate to the back side.

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SUPPLEMENTAL LABORATORY TESTS

S.T.B. Stability - Test the briquette, fabricated while making the stain number test, for stability value in accordance with the procedure used for determining the Hubbard-Field stability values (See A.S.T.M. Technical Papers, Vol.25, 1925, Part II, Page 335).

In case the stain test is made in the field and additional information is desired, the stain test briquettes should be sent to the Headquarters Laboratory for the stability and other desired tests.

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