

PLAINTIFF'S EXHIBIT

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MANAGEMENT INFORMATION

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LABORATORY MANUAL OF TESTS

ASPHALTS, WAXES, TARS, PITCHES, ETC.

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SOFTENING POINT BY RING & BALL APPARATUS

Use: For Asphalts, Pitches, Tars, Waxes, and Formulations from same.

Apparatus:

The ring used shall be a brass-shouldered ring conforming to Specifications in A.S.T.M. Test E-28-51-T. A supporting wire is fastened to the side of the ring. The ball is of steel, 3/8" in diameter and weighs between 3.45 and 3.50 grams. If tests are run in duplicate, the average of the results is taken as the melting point. The results usually check within 1°F. Duplicate tests are desirable but not necessary unless requested by PBL.

Procedure:

A sample of the bitumen or other material to be tested is melted by a slow application of heat until sufficiently fluid to stir out air bubbles and to pour readily. Avoid overheating which causes appreciable volatilization. The ring is then placed upon an amalgamated brass plate and the heated bitumen poured into the ring until it slightly more than fills the ring. Burn the surface to remove air bubbles. Allow it to cool for 30 minutes, and remove the excess bitumen with a hot knife blade. The ball is then placed upon the surface of the bitumen in the center of the ring. For materials that have very hard surfaces, the ball may be heated by holding in the flame of a burner with a pair of tongs for approx. 5 seconds, then placing on the center of the sample in the ring. When the sample melts sufficiently to set the ball, the ring must be placed immediately in the beaker of water or glycerine.

The whole is suspended (with the ring in a horizontal position) inside and exactly one inch above the bottom of a 600 cc. beaker containing approx. 400 c.c. of liquid at 41°F. Distilled water shall be used for melting points below 176°F. and glycerine for higher melting points. The bulb of a 20 - 400°F. chemical thermometer is placed within 1/2" of the sample and at the same level. A piece of filter paper may be placed in the bottom of the beaker to catch the sample when it melts. Allow the sample to remain suspended 15 minutes before applying heat.

Heat is applied uniformly to the bottom of the beaker in quantity sufficient to raise the temperature of the liquid 90°F. per minute. Care should be exercised in rate of heating, as this is an important factor. The maximum permissible variation for any minute after the first three shall be 1°F.

SOFTENING POINT BY RING & BALL APPARATUS

All tests in which rate of raise exceeds these limits shall be rejected. The temperature is recorded at the start of the test and every minute thereafter. The melting point or softening point is that temperature at which the bitumen first touches the bottom of the beaker.

Precautions:

1. The use of freshly boiled distilled water is essential, as otherwise air bubbles form on the specimen and may affect the result.
2. Rigid adherence to the prescribed rate of heating is absolutely essential for reproducibility of results.
3. Owing to possible danger to health if mercury is handled carelessly, the following rules should be observed at all times:
 - (a) Store the mercury in a closed jug in a cool place.
 - (b) Strictly avoid spilling any mercury.
 - (c) Remove the mercury vapors by working under a suitable hood with good ventilation.
 - (d) Keep amalgamated brass plates and other apparatus at no higher than room temperature.

Bird & Son adoption of A.S.T.M. Test E28-51T.

JRL/jeh
June 1952

PENETRATION OF BITUMINOUS MATERIALS

(Adapted from ASTM Designation: B5-49)

Scope

1. This method covers the test for the penetration of semisolid and solid bituminous materials.

Definition

2. Penetration - Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle penetrates vertically into 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 g., 5 sec., and 25 C. (77 F.), respectively, and the units of penetration to indicate hundredths of a centimeter. The load is defined as the total moving weight of the needle and attachments.

Apparatus

3. The apparatus shall consist of the following:

- (a) Container - A container, in which the sample is tested, made of metal or glass, cylindrical in shape, and having a flat bottom. The container to be used for materials having a penetration of 200 or less shall have a nominal capacity of 3 oz.; its inside dimensions shall be substantially as follows: 55 mm. (2.17 in.) in diameter, and 35 mm. (1.38 in.) in depth. The container to be used for materials having a penetration over 200 shall have a nominal capacity of 6 oz.; its inside dimensions shall be substantially as follows: 70 mm. (2.75 in.) in diameter, and 45 mm. (1.77 in.) in depth.

Note: Containers known in the drug trade as seamless "ointment boxes" may be obtained in dimensions conforming to the above requirements.

- (b) Needle - A needle made from a cylindrical steel rod approximately 50.8 mm. (2 in.) in length and 1.00 to 1.02 mm. in diameter. It shall be symmetrically tapered at one end to a cone approximately 6.35 mm. (1/4 in.) in height and whose angle shall be within the range of 8 deg. 40 min. and 9 deg. 40 min. After tapering, the point shall be "blunted" by grinding off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm. in diameter. The finished needle shall be hardened and highly polished. The exposed length of the needle when mounted in the chuck of the penetration

PENETRATION OF BITUMINOUS MATERIALS

(Adapted from ASTM Designation: D5-49)

apparatus or in a ferrule shall be not less than 40 nor more than 43 mm.

- (c) Water Bath - A water bath maintained at a temperature not varying more than 0.1 C. from 25°C. The volume of water shall be not less than 10 liters. The sample shall be immersed in the bath to a depth of not less than 10 cm. (4 in.) and shall be supported on a perforated shelf not less than 5 cm. (2 in.) from the bottom of the bath.
- (d) Penetration Apparatus - 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.
- (e) Transfer Dish for Container - The transfer dish for container shall be a glass or metal cylinder with a flat bottom provided with some means which will insure a firm bearing and prevent rocking of the container. It shall have a minimum inside diameter of 90 mm. and a minimum depth above the bottom bearing of 55 mm.

Preparation of Sample

4. The sample shall be heated, with care to prevent local overheating and at the lowest possible temperature, until it has become sufficiently fluid for ready pouring. In no case shall the temperature of the sample exceed 200 F. above its softening point, determined in accordance with the Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (ASTM Designation: D-36). The sample shall be stirred until it is homogeneous and free from air bubbles. It shall then be poured into the sample container to a depth such that when cooled to the temperature of test the depth of the sample is at least 10 mm. greater than the depth to which the needle penetrates in the test. Separate samples shall be poured for each variation in test conditions. The container and its contents shall be loosely covered as a protection against dust and be allowed to cool in an atmosphere at a temperature not higher than 29.5 C (85 F.) and not lower than 21 C. (70 F.) for not less than 1 1/2 nor more than 2 hr. when the sample is in a 6-oz. container, and for not less than 1 nor more than 1 1/2 hr. when the sample is in a 3-oz. container. It shall then be placed in the water bath along with the transfer dish and be allowed to remain for not less than 1 1/2 nor more than 2 hr. when the sample is in a 6-oz. container, and for not less than 1 nor more than 1 1/2 hr. when the sample is in a 3-oz. container.

PENETRATION OF BITULINOUS MATERIALS

(Adapted from ASTM Designation: D5-49)

Procedure

5. (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 cover the container completely. The transfer dish containing the sample shall then be placed upon the stand of the penetration machine. The needle loaded with the 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 pointer brought to zero. The needle shall then be released for the specified period of time, after which the penetration machine shall be adjusted to measure the distance penetrated.
- (b) At least three tests shall be made at points on the surface of the sample not less than 1 cm. (3/8 in.) from the side of the container and not less than 1 cm. (3/8 in.) apart. After each test the sample and transfer dish shall be returned to the water bath while the needle is being carefully wiped toward its point, first with a clean cloth moistened with carbon tetrachloride to remove all adhering bitumen, and then with a clean, dry cloth. The reported penetration shall be the average, calculated to the nearest whole unit, of at least three tests whose values shall not differ from this average more than the tolerance calculated to the nearest whole unit for that penetration. The tolerance shall be calculated as follows:

$$\text{Tolerance} = \frac{\text{Penetration}}{100} \div 1$$

- (c) When desirable to vary the temperature and in order to provide for a uniform method of reporting results when variations are made, the following combinations of temperature, time, and weight are suggested:

At 0 C. (32 F.)..... 200-g. weight, 60 sec.
At 46.1 C. (115 F.) 50-g. weight, 5 sec.

When penetrations of 5 or less are obtained and more significant results are required, the test may be made using an increased load. Increments of 100 g. are suggested. When variations are made, the samples shall be prepared in accordance with Section 4, except that brine may be used in place of water in the water bath where necessary to maintain low temperatures.

PENETRATION OF BITUMINOUS MATERIALS

(Adapted from ASTM Designation: D5-49)

Reproductibility

6. Results of tests by this method should be reproducible between laboratories within a range above or below the average result determined as follows:

$$\text{Range} = \pm \frac{\text{Average Penetration}}{50} \pm 2$$

6/20/52
JRL/jeh

DUCTILITY

1. Definition:

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

2. Apparatus:

The mold shall be made of brass or aluminum. The ends of the mold are called clips, and the pieces which join the clips are called the sides. The dimensions of the mold shall be as follows:

Total length - Internal	7.45 - 7.55	cm.
Distance between Clips	2.97 - 3.03	"
Width at Mouth of Clip	1.98 - 2.02	"
Width at Min. Cross Section (halfway between clips)	.99 - 1.01	"
Thickness of mold throughout	.99 - 1.01	"

The water bath shall be maintained at a temperature of 77°F. (25°C.). 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 cm. and shall be supported on a perforated shelf not less than 5 cm. from the bottom of the bath.

Any apparatus may be used for pulling the mold of bitumen apart that is so constructed that the molded form will be continuously immersed in water at 77°F. and the two clips pulled apart at a uniform rate of speed of 5 cm. per minute. The Smith machine with a Dow mold is used at PBL.

3. Procedure:

Melting and Molding. The material to be tested shall be completely melted at such a temperature that it will be thoroughly fluid. Take the usual precautions not to over-heat the bitumen. It is then poured into the mold. Care should be taken to burn off the air bubbles from the asphalt. This can be done by running a burning match over the surface of the bitumen.

The mold shall be assembled on a brass or amalgamated tin plate, in order to prevent the material under test from sticking, the interior surfaces of the side pieces of the mold shall be thoroughly amalgamated.

DUCTILITY

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3. Procedure: (Cont'd)

In filling the mold, care shall be taken not to disarrange the parts and thus distort the form. In filling the mold, the material shall be poured in a thin stream back and forth from end to end of the mold until it is more than level full. It shall then be left to cool, after removing bubbles at room temperature and then placed in the water bath at 77°F. and kept at that temperature for 30 minutes. After this the excess of bitumen shall be removed by means of a hot knife in such a way that the mold shall be just level full. The plate, mold and all, shall then be replaced in the water bath and held at 77°F. for one hour and then the mold shall be removed from the plate and the side pieces detached.

The rings at the ends of the clips 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 cm. per minute. While the test is being made, the water in the tank of the ductility machine shall cover the sample by at least 2.5 cm. and shall be kept at the testing temperature of 77°F. When the specimen of bituminous material breaks the distance through which the clips have been pulled to produce rupture, shall be measured in centimeters, and that figure shall be known as the Ductility of the substance. The average of three tests shall be taken (two are sufficient for testing at PBL).

Make notation as to type of break, "sharp break", "necked down", etc.

Test at 32°F.

Bring the temperature of the water bath down to 32°F. by adding a sufficient amount of crushed ice and salt. Proceed with the test in the same manner as described above except the two clips shall be pulled apart at a uniform rate of one cm. per min.

This is a Bird & Son adoption of A.S.T.M. Test D113-44.

JRL/jeh
June 1952

FLASH POINT - OPEN CUP - FIRE POINT1. Definition

Flash Test or Flash Point is the lowest temperature at which under definite specified conditions a petroleum product vaporizes rapidly enough to form above its surface an air-vapor mixture which gives a flash or mild explosion when ignited by a small flame.

Fire Test, or Burning Point is the lowest temperature at which under definite specified conditions a petroleum product vaporizes rapidly enough to form above its surface an air-vapor mixture which burns continuously for at least 5 seconds when ignited by a small flame.

Self-Ignition Point is the lowest temperature at which under definite specified conditions, a petroleum product vaporizes rapidly enough to form above its surface an air-vapor mixture which is ignited by the heat of the petroleum product itself.

These definitions must not be misunderstood. The expression, "under definite specified conditions" is a vital part of the definition and must not be disregarded. While a given oil may have a "flash, open cup" of 300°F., under the definite specified conditions of the test, there is no assurance that the same oil may not flash at a lower temperature under other conditions such as those under which the material is actually used. Hence, it is extremely important that there be general recognition of the fact the "flash point", "fire point" and "self-ignition point" are merely empirical qualities determined by laboratory methods.

In all of the open-cup testers, the most volatile and inflammable vapors escape but not in sufficient quantity to be readily ignited. This defect of open-cups is increased by drafts and air currents. Hence, open-cup flash tests are usually 10 - 20°F. higher than those with a closed cup.

2. Apparatus

Standard Cleveland Open-Cup Tester. Standard A.S.T.M. Flash Point Thermometer. 1" immersion, accurate timer, suitable source of heat to heat the sample (electricity or gas), and a source of gas for the test flame.

3. Procedure

Clean the flash cup thoroughly so that there is no material left from previous determinations in the cup. Suspend the thermometer in a vertical position with bulb immersed to line approximately half way between the center and back of the cup.

FLASH POINT - OPEN CUP - FIRE POINT

3. Procedure: (Cont'd)

Fill the cup with the material to be tested in such a manner that the top of the meniscus is exactly at the filling line (inscribed on the inside of the cup) making sure that nothing is spilled on the cup above the line or any part of the apparatus outside. Failure to observe this is a frequent cause of the incorrect results.

The sample shall be heated at a rate not exceeding 300°F. per min. temp. rise, until a point is reached of approx. 100°F. below the probable flash point of the sample. Thereafter, the rate of heating shall be decreased for at least the last 50°F. before the flash point is reached. The rate during the last 50°F. shall be not less than 90°F. nor more than 110°F. per min.

Apply the test flame to the material being tested at each successive thermometer reading, which is to be made every 30 seconds. In doing this, observe the following instructions:

- a. Have the test flame approx. 5/32" in diameter.
- b. Move it across the center of cup in a straight line (or on the circumference of a circle having a radius of at least 6") at a right angle to the diameter of the cup passing through the thermometer.
- c. Make the sweep across the cup in 1 second, keeping the lower side of the flame exactly level with the upper edge of the cup.

When the approximate flash is known, application of the test flame need not be made until the temperature is 50°F. below the expected flash point.

4. Flash Point

Note and record as Flash C.O.C. (Cleveland Open Cup) the temperature read on the thermometer when a flash appears at any point on the surface of the material being tested. This flash must not be confused with a bluish halo which sometimes surrounds the test flame prior to the true flash.

5. Fire Point

If the fire point is also desired, continue heating without interruption at the same rate as before and applying the flame in exactly the same manner every 30 seconds. Note and record as Fire C.O.C. (Cleveland Open Cup) the temperature at which the material ignites and continues to burn for 5 seconds. The flame should then be extinguished by smothering with a sheet of metal.

FLASH POINT - OPEN CUP - FIRE POINT

6. Self-Ignition Point

This test is seldom used but is a continuance of the fire point with the same 10°F. rise per min. reading thermometer every 30 seconds. The flame is not used in this test. Read and record as self-ignition point that point at which the heat of the material being tested is sufficient to ignite the vapor driven off from the material.

Cements and Fibrated Roof Coating

A non-fibrous substance such as an oil or asphalt is not stirred while making this test, but substances such as fibrated lap cements must be stirred (with the thermometer bulb) continuously during the running of these tests.

Bird & Son adaptation from Day I and A.S.T.M. Test D92-46.

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ENGLER VISCOSITY DETERMINATIONS1. Apparatus

Engler Viscosimeter (Ubbelohde Modification)
 4-ounce seamless can
 Kodak Timer or Stopwatch
 Method of heating oil (ring burner)
 2 Thermometers

2. Method

Heat the oil bath of the viscosimeter to temperature at which test is to be run, stir frequently while heating. Bring temperature of sample to be tested to a point 10°F. above test temperature (for tests run at 200°F. or above) and pour into oil cup to level of projecting points, after wood plug used for closing outlet has been put in place. Replace cover and insert thermometer making sure bulb reaches into mass of substance being tested. With constant stirring and adjustment of flame under oil bath, bring temperature of test substance to desired temperature (oil bath usually 10 - 30°F. above desired).

Place 4-ounce seamless can beneath orifice outlet. Remove plug and start timer. When the top of the meniscus of the material in the 4-ounce can reaches the center of the groove near the top of the can, shut off timer and note time of efflux of 100 cc. in seconds.

PBL Rapid Method

Heat the oil bath with the gas flame and agitate until the following temperatures are reached, and adjust the flame so these temperatures will be maintained throughout the test. Then heat the sample on a hot plate, stirring and watching the temperature until it reaches the following desired temperature; then pour it into the viscosimeter and start the test immediately by pulling the wooden plug and starting the timer. It is not necessary to put a thermometer into the sample material when in the viscosimeter.

<u>Material</u>	<u>Oil Bath Temp.</u>	<u>Sample Temp.</u>
Asphalts	415°F.	405°F.
Base Wad Coating and Waxes	305°F.	302°F.
Asphalt Primer	130°F.	130°F.

3. Results

All viscosities are to be expressed in Engler degrees.

To convert to degrees Engler, multiply the time of efflux for 100 cc. in seconds by 2.35, divide this figure by the water factor of the viscosimeter, and report result as degrees Engler.

ENGLER VISCOSITY DETERMINATIONS

4. Cements

Following tests are run in the Engler:

- a. All Flux and Saturating Asphalts @ 400°F.
- b. Base Wad Coating @ 300°F.
- c. Asphalt Roof Primer @ 130°F.

5. Determination of "Water Factor"

Wash the inside of the oil cups first with ethyl or petroleum ether, then repeatedly with alcohol and finally with distilled water. Use a new wooden plug. Make sure the oil cup is level and that the room temperature is between 66 and 70°F. Fill the bath with water at 68°F. to the level of the oil cup cover. Insert the plug and fill the oil cup to slightly above the gage points with pure distilled water at 68°F. Manipulate the plug so that the orifice is filled with water and a drop of water is suspended from the bottom. Remove the excess of water from the oil cup with a pipette until the three gage points are exactly in the surface of the water. Place the clean dry 200 cc. receiving flask below the orifice. Raise the plug as far as possible starting the timer at exactly the same time. Hold the bath temperature at exactly 68°F., stirring occasionally. Stop the timer the instant the lower line of the meniscus reaches the 200 cc. mark on the receiving flask. Repeat the determination as before until six consecutive tests give results varying by not more than 0.5 second. The average of these six tests is the "water factor" of the viscosimeter. Any instrument whose factor is not between 50 and 52 is not standard.

The "water factor" should be checked at least every six months.

Bird & Son adaption from Day

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SAYBOLT VISCOSITY DETERMINATIONSApparatus:

1. Saybolt Viscosimeter
2. Universal Orifice and Furol Orifice
3. 60 cc Flask (special wide mouth type) or prepared 3-oz. Gill cans.
4. Kodak Timer or Stop Watch
5. 2 Thermometers with the proper range

Method

The oil bath surrounding the oil cup of the viscosimeter shall be held constant at such a temperature as will maintain the desired temperature in the standard oil cup.

Viscosity determinations should be made in a room free from draughts and rapid temperature changes. Strain all substances being placed in the oil cup.

Saybolt Viscosity

To make the test, start the oil bath agitator motor and plug in the electric heater cord. For tests below 200°F. use only one cord. For test temperatures over 200°F., plug in the two electric cords.

Viscosity Test for Oils and Light Liquids

Heat the oil to the desired temperature, clean out the standard oil cup, and screw in the desired orifice. Pour some of the oil through the cup. Insert the cork stopper in the lower end of the air chamber at the bottom of the oil cup, sufficiently to prevent escape of air, but not to touch the small outlet tube of the standard oil cup.

Heat the material to be tested outside the viscosimeter to slightly below the temperature at which the viscosity is to be determined and pour it into the standard oil cup until it ceases to overflow into the overflow cup. By means of the oil cup thermometer, keep the material being tested well stirred, also stir the oil in the surrounding oil bath. It is extremely important that the temperature of the bath be maintained constant during the entire test.

When the temperature of the bath and of the material in the standard oil cup are constant and the material in the standard cup is at the desired temperature, withdraw the oil cup thermometer; quickly remove the surplus material from the overflow cup by means of a pipette so that the level of the material in the overflow cup is below the level of the material in the standard

SAYBOLT VISCOSITY DETERMINATIONS

(Cont'd)

cup. Place the 60 cc. flask in position so that the stream of material from the outlet tube will strike the neck of the flask so as to avoid foam. Snap the cork from its position, and at the same time start the timer. Stop the watch when the bottom of the meniscus reaches the 60 cc. mark on the neck of the receiving flask.

The time in seconds for the delivery of the 60 cc. of material is the Saybolt viscosity of the material at the temperature at which the test was made. The result may be expressed as "Saybolt Universal Seconds" or "Saybolt Furol Seconds", depending, of course, on which orifice was used during the test. A Saybolt Universal figure is slightly more than 10 times a Saybolt Furol figure.

Viscosity Test for Asphalts and Asphaltic Formulations

Heat the agitated oil bath to the proper temperature, and screw in the desired orifice. Plug the small hole on the bottom of the viscosimeter with a small cork and place a 3-oz. scored Gill can on a level spot approximately 2" below the orifice opening. The 3-oz. Gill can should be previously scored with a line to mark the height when it contains exactly 60 ml. of water.

Heat the material to the proper temperature, and pour it into the viscosimeter until it just overflows over the oil tube shoulder; then place the cap over the oil tube, pull the stopper on the bottom of the viscosimeter and record in seconds the time required for 60 ml. of material to pass through the orifice.

Standard temperatures are as follows:

<u>Oil Bath</u>	<u>Sample</u>
305°F.	302°F.
410°F.	405°F.

Bird & Son adaption from Day
JRL/jeh
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FOAM TESTS

PURPOSE: To predetermine the amount of foam in petroleum asphalts.

1. Apparatus

1. Double beam balance and set of Metric Weights
2. Burette (10 ml.)
3. Electric hot-plate
4. Thermometer (400°)
5. Blotting paper (140% kerosene value as std.)
6. Stirring rod (glass)
7. Eastman Timer or a Stopwatch

2. Procedure

1. Heat about 300 grams of asphalt being tested to approximately 200°F. Any suitable container may be used.
2. Pour exactly 185 grams of the asphalt into test apparatus. Remove cylinder from balance gram scale and place on electric hot-plate. Bring temperature of asphalt in cylinder to exactly 394°F. The time required to soak blotting paper with water will permit the temperature to rise slowly to 400°F. (plus or minus 3 degrees F.) at which temperature the test shall be started. The cylinder must remain on the hot-plate for the duration of the test and the control set on "medium".
3. 3" x 3" samples of air-dried (3-5% moisture) blotting paper shall be available to all tests. For sake of standardization, blotting paper showing a kerosene value of 140% has been selected.

Exactly 3.3 ml. of water is run slowly from a 10 ml. burette onto the blotting paper. The complete operation should require approximately 35 seconds. The blotting paper surface should be thoroughly and uniformly covered with water. Samples not completely soaked, which expose dry portions of paper, shall not be used.

4. The water soaked blotting paper is immediately immersed in the hot asphalt described in No. 2 above and pushed to the bottom with a stirring rod.
5. Time the period from the operation of immersing the water soaked paper until the foam, in receding, passes lug "B" on wall of apparatus. The recorded period is the time in seconds mentioned under specification as "foam time".
6. Record the highest point the foam reached in inches.
7. As foam recedes, run stirring rod slowly around edge of foam in order to more closely detect the moment at which it passes lug "B".

FOAM TESTS

8. An average of three tests shall be recorded as the foam time.
9. Blotter paper is available from the Flora Blotter Company.
It is 120 lb. plain paper, and it may be purchased in
19" x 24" sheets.

Revised in June 1952
JRL/jeh

STAIN TEST (PAROID)

TEST: For stain from coating asphalts

1. Apparatus:

1. Thin metal sheets, 7" x 4"
2. Sheeting-out plate
3. Standard #170 Talc
4. Small hand roller
5. Coating to be tested at 350°F. to 400°F.
6. Constant temperature oven.

2. Preparation of Sample:

The asphalt shall be heated to the consistency of very thin molasses (about 350° to 400°F.) with care being taken to avoid local heating. The material shall then be stirred and sheeted out on the 7" x 4" metal sheets, using the sheeting-out plate, to a thickness of .015" to .020". Previously warmed talc shall be immediately sprinkled by means of a 28" mesh sieve onto the hot sheeted coating, care being exercised to have an even distribution over the entire surface and an excess of talc. The surfacing shall then be rolled into the coating with a small roller. The excess talc is removed by tipping and gently tapping the back of the sheet with the fingers; and then the talc surface shall be rolled again with the roller.

3. Test:

The sample being tested shall be prepared as above and placed in the constant temperature oven, together with a sample of standard coating prepared in exactly the same manner, at a temperature of 122°F. to 128°F. The test should continue at this temperature for sixty (60) hours. It is purely comparative, the standard coating being the reference and the samples being judged by the amount of stain present on the talc.

If a distinct difference is obtained between the standard and the unknown coating in a period shorter than 60 hours, the test may be discontinued and reported at such time.

Revised in June 1952
JRL/jeh

STAIN TEST - TEXAS

Method of Test for Stain Test on Asphalt.

1. Apparatus

1. A brass mold which will give a disc $3/4$ to 1" in diameter and $1/16$ to $1/8$ " thick.
2. Whatman No. 44 filter paper, 11 cm. in diameter.
3. A circular brass disc 40 mm. in diameter, ± 1 mm., weighing 64 g. ± 1 g.
4. A suitable drying oven.
5. A standard stain chart.

2. Procedure:

A sample of asphalt to be tested is heated to the lowest temperature at which it will pour into the mould. The disc made in this manner is allowed to cool thoroughly before slicing the excess asphalt from the top. It is then removed from the mold. This disc of asphalt is then placed between two pieces of Whatman filter paper No. 44, 11 cm. in diameter, in such a manner that the rough (screen) side of the filter paper faces the asphalt. The filter paper and asphalt are then placed on a stationary shelf in a drying oven on a smooth piece of card board and the brass disc placed on the paper over the sample. The oven is kept at a temperature 30°F . below the melting point of the sample of asphalt being tested for a period of 48 hours, after which the sample is removed from the oven and allowed to cool.

3. Grading of Asphalt Stain:

The asphalt paper sandwich is then placed behind the standard stain chart and the color comparison is made through the opening provided for this purpose. The sample shall then be reported in terms of grade and series. Series No. 3 and No. 4 should be used in grading stainless asphalts. Other samples should be graded by Series No. 1 and No. 2.

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JRL/jeh

NAPHTHA AND XYLENE SPOT TESTS FOR ASPHALTMaterial:

The naphtha solvent used shall be a straight run overhead distillate free from cracked products of any kind, and shall conform to the following requirements.

Gravity, A.P.I.	49 to 50
Distillation	
Initial Boiling Point	Above 300°F.
50 per cent over	335 - 355°F.
End Point	Below 410°F.
Aniline number	138 to 145°F.

The Xylene shall be C. P. grade.

Apparatus:

1 - 25 ml Erlenmeyer flask. Cork stopper for flask in which is fitted an 8" length of open 1/4" glass tubing.

1 - Glass stirring rod.

Filter Paper - #50 Whatman.

Procedure for Naphtha Spot Test:

A weight of the sample equivalent to 2 ml is placed in the Erlenmeyer Flask and if it does not flow readily at room temperature it is carefully heated in the flask on a hot plate until it flows out over the bottom. After cooling, 10.2 ml of the solvent are added; the stopper with its 8" length of tubing is quickly inserted into the neck of the flask; the contents of the flask are swirled with a rapid circular motion for five seconds and the flask is immediately immersed to its neck in a bath of gently boiling water (unless the sample is a thin liquid, in which case the heating bath is unnecessary). The contents of the flask shall again be swirled for five seconds at the end of each minute thereafter until complete dispersion takes place. If dispersion is not completed in from 6 to 8 minutes the test shall be repeated with the bath maintained at a temperature 25 deg. F. lower or higher (glycerine being substituted for the water bath in the latter case), until dispersion is effected within a stipulated time. After complete dispersion as judged by tilting the flask, the flask is allowed to cool to room temperature and if any loss in weight occurs it is made up with additional solvents.

After stirring, a drop of the asphalt solvent mixture is then placed on the filter paper. If the drop forms a brown or yellowish brown circular stain with a darker solid or annular nucleus in the center, the test shall be reported as positive.

If, however, the drop forms a uniformly brown circular stain, judgment shall be reserved and the asphalt solvent mixture shall be set aside in a tightly stoppered flask at room temperature in a subdued light for a further period of 24 hours. The mixture shall then be stirred until uniform and a drop from it shall again be placed on filter paper. If the drop from the 24 hour old mixture still forms a uniformly brown circular stain, the test shall be reported as "negative" but if a darker solid or annular nucleous as described in the preceding paragraph now forms in the center of the stain, the test shall be reported as "positive."

Procedure for Xylene Spot Test:

The Xylene test is used to determine whether the heterogeneity of an asphalt occurs among its bituminous fractions or whether it is due to finely divided non-bituminous matter. The Xylene test is run only if the asphalt shows a "positive" naphtha test.

Use exactly the same procedure for the Xylene test as was used for the Naphtha test except that Xylene must be used for a solvent instead of Naphtha.

If the Xylene Spot Test is "negative" it shows that the heterogeneity of the asphalt was within its bituminous components. If the Xylene Spot Test is "positive" it shows that the asphalt contains fine non-bituminous matter and therefore beyond the ordinary range of the spot test.

JRL/rp
April, 1959

CONTACT COMPATIBILITY OF BITUMINOUS MATERIALS

Scope: This method of test provides a means for evaluating contact compatibility between bituminous materials.

Outline

of Method: The method of test is generally used to determine compatibility between the saturant and coating used in the manufacture of prepared roofings. In the following description of the method, coating and saturant will be referred to, but it is understood that comparable bituminous materials may be substituted where this test procedure seems applicable.

A small drop of the saturant is placed on the freshly talced surface of the coating, and compatibility is judged by the degree to which an oily ring develops in the talc surrounding the drop.

Apparatus:

- a. Lid of 3-oz. tin or equivalent container
- b. Balance - an analytical balance having an accuracy of ± 1.0 mg.
- c. No. 325 U.S.S. sieve (ASTM E11-39)
- d. Dropping Device - wire 1.0 mm. (0.04 in.) in diameter.
- e. Oven - constant temperature oven capable of maintaining test temperature ± 1.0 C. (1.8 F.)
- f. Small Scale - graduated in 0.5 mm. divisions
- g. Magnifying Glass - approximately 4x
- h. Talc - a pure talc, variety soapstone, ground so that at least 70% passes the No. 325 sieve and oven-dried at 110 C.

Note: When this procedure is being used in purchase specifications, both buyer and seller shall use the same talc.

Procedure: Melt the coating at the minimum temperature required to render it fluid, stirring with a fine wire to eliminate bubbles, and pour it into a clean 3-oz. tin lid to a thickness of 3.0 to 6.0 mm. Pour carefully so as to provide a substantially smooth surface free of bubbles and other surface blemishes. Determine the weight and surface area of the filled tin after it has cooled.

Procedure: (Cont'd)

Make a preliminary dusting of talc by covering the coating surface with talc and removing excess, non-adherent dust by inverting the tin and dropping it approximately 2.5 centimeters onto the table top.

Make final application through a No. 325 sieve held 2.0 to 2.5 centimeters above the surface of the coating by gently shaking or tapping the sieve until a smooth uniform film of talc weighing 0.004 grams ($\pm 10\%$) per square centimeter has been applied.

Place three drops of saturant, each about 3.0 millimeters (± 0.5 mm.) in diameter, on the talced surface of the coating. Form the drops by plunging the wire into the molten saturant and, after the excess has drained off, allowing suitably sized drops to fall on the talc from a sufficient height to form substantially spherical drops.

Place the thus prepared specimen in the test oven so regulated as to maintain the specimen at the recommended test temperature (± 1.0 C.) for 72 hours. After cooling, examine the specimen and measure any oily ring formed around the periphery of the drop, recording its width to the nearest 0.1 millimeter. Measure the width from the line of contact of talc and drop to the outer limit of the oil ring.

Test Temperature: 100°F. above the Softening Point of the asphalt saturant used.

Results: All Bird & Son standard asphalts must not show an incompatibility greater than 0.5 mm.

This is a Bird & Son adaption of the Oliensis incompatibility Test.

June 1952
JRL/jeh

COMPATIBILITY TEST FOR ROOFING PRODUCTS

(3-M Co. - Colorquartz Laboratory - Standard Procedure No. 67)

This test for compatibility between coating and saturant asphalts in a shingle can be run on freshly made shingles, shingles having been in storage for a period of time, or on unexposed portions of exposed shingles.

There may be some objection to defining the results of this test in terms of compatibility. However, because of the similarity between the results of this test and that presented by Mr. G. L. Oliensis ("The Exudation Test for Bleeding in Bituminous Roofing" appearing in the "Industrial and Engineering Chemistry", Analytical Edition, Vol. 10, Page 199, April 15, 1938) (Colorquartz Laboratory Standard Procedure No. 59), we feel that we are justified, for the present, in our use of the terms "Compatibility and incompatibility". At any rate, the test gives data which may assist in interpreting the behavior of shingles on weathering, and consequently may be adaptable for use as a control in the manufacture of asphalt roofing.

Procedure:

On an area of about one inch square of the shingle to be tested, the mineral surfacing is removed by slicing through the center of the asphalt coating layer with an unheated spatula. After this has been accomplished, the remaining coating is pared down smoothly to as thin a layer as can be obtained without cutting into the felt. The surface of the felt may then be exposed in several selected small spots by means of the spatula. The total area of these spots should not represent more than about 15 per cent of the total area of surfacing removed.

The surface of the sample is dusted uniformly with roofer's talc by means of a camel hair brush, and the excess talc removed by gently tapping the sample on its edge. Care should be taken to prevent disturbing the talc surface from this time on.

The sample is then placed in an oven at about 130°F. (Note-1) and protected in such a way that air currents will not remove the talc. After about sixteen hours at 130°F. the sample is re-dusted with talc using the same procedure as for the first dusting. The sample is then returned to the oven at 130°F. At the end of two days, the sample is ready for observation.

Five different results have been observed which may be classified as follows:

Group 1 - The talc on the surface of the sample remains white or only slightly yellow.

Group 2 - The talc on the coating asphalt is blackened, and the talc on the spots of exposed felt remains white or only slightly yellow.

- Group 3 - The talc on the coating asphalt remains white or only slightly yellow, and the talc on the spots of exposed felt is blackened.
- Group 4 - The talc is blackened on both the coating asphalt and on the spots of exposed felt.
- Group 5 - The talc is colored by a brown spot or spots not existing generally on either the coating asphalt or on the felt, but seeming to indicate non-uniformity of asphalt or a contamination in the felt.

The results on samples tested should be classified, at present, according to the above grouping until such time as the meaning of each type is more fully understood.

Note-1: The temperature of 130°F. was chosen because it has given results which correlate closely with the bleeding produced on the back of shingles during outdoor exposure. A more rapid test, however, may be run with satisfactory results at temperatures higher than 130°F. if the properties of the asphalt saturant are such that it will not blacken the talc at the temperature of test.

Checked in June 1952
JRL/jeh

SPECIFIC GRAVITY OF ASPHALT
(By the Pycnometer Method)

This test is based on the principle that a sinking body displaces its own volume of water. The steps in the determination are as follows:

Procedure:

1. Dry and weigh a clean Pycnometer with its stopper.
2. Fill the Pycnometer with distilled water so that the excess water will come up through the hole in the stopper when it is put on. Place the stoppered Pycnometer in a water bath @ 60°F. for one hour, then remove, wipe off excess water, and weigh.
3. Dry the Pycnometer and stopper, and fill half full with asphalt. Cool to 60°F. and hold at that temperature for one hour, then weigh.
4. Pour distilled water in over the asphalt until the Pycnometer is filled, and put on the stopper as before. Weigh after it has been kept at 60°F. in a water bath for one hour.

Calculations:

- A. Subtract the value of Item 1. from Item 2.
This gives the weight of water.
- B. Subtract Item 1. from Item 3. This value is the weight of the asphalt sample.
- C. Subtract Item 3. from Item 4. This gives the weight of the added water.

The following formula represents the specific gravity which is the weight of the asphalt divided by the weight of an equal volume of water:

$$\text{Specific Gravity} = \frac{B}{A - C}$$

Manipulation:

When the Pycnometer is taken from the water, wipe off the top immediately, and then dry off the rest of the Pycnometer. Do not wipe off any water which comes up through the hole in the stopper thereafter. It is a result of expansion due to

SPECIFIC GRAVITY OF ASPHALT
(By the Pycnometer Method)

Manipulation: (Cont'd)

the heat from your hand. Weigh the Pycnometer as quickly as possible because the evaporation of the water will give a low result.

Results:

Two tests must be run on each sample, and the results should check within ± 0.005 specific gravity.

Multiply the specific Gravity by 8.34541 to obtain the weight in lbs./gal.

JRL/jeh

Revised June 1952

PLIABILITY OF COATING ASPHALTSApparatus:

1. Flat metal surface at least 6" x 14"
2. Mold - made of iron 1/8" thick 4" x 14" with slot cut in center 1" x 12".
3. Metal roller.
4. Supply of soapstone passing 35 mesh screen.
5. 35 mesh screen.
6. Mandril exactly 0.4" diameter.
7. Means of heating asphalt.

Procedure:

The asphalt to be tested is heated to thin molasses consistency (usual care being taken to avoid local heating) stirred and poured into the mold which is setting on the metal plate, both mold and plate having been previously either amalgamated or dusted with soapstone to prevent the asphalt from sticking to them. The surface of the asphalt is then dusted with soapstone by shaking the screen containing it over the mold, and the strip of coating rolled by running the roller over the surface of the mold several times. Rolling the long dimension of the mold, allow the sample to cool sufficiently to remove from the mold without distortion of the strip.

At least eight (8) strips of this sort must be prepared from each sample being tested and allowed to stand at room temperature not less than 12 hours nor more than 20 hours before proceeding with the test.

Immerse at least two of each of the strips in a water bath for 10 to 20 minutes at the following temperatures:

77°F.

63°F.

56°F.

50°F.

At each temperature bend the samples over the mandril double upon themselves (180°) in 2 seconds, noting signs of brittleness.

Results are noted at the different temperatures as:

1. Excellent - no cracks, no resistance to bend.
2. Good - no cracks, stiff to bend.
3. Fair - slight cracks, does not split through.
4. Slightly Brittle - slight cracks tending to break off.
5. Brittle - breaks off badly.

A sample testing 4 or worse at 56°F. is tested at 60°F. to determine whether it passes specifications.

Checked in June 1952
JRL/jeh

THE DETERMINATION OF PROPORTION OF BITUMEN SOLUBLE
IN CARBON TETRACHLORIDE

Apparatus:

1. Gooch Crucible.
2. Filtering Flask and Tubing and Pump.
3. Shredded Asbestos.
4. 2 - 150 cc. Erlenmeyer Flasks.
5. Bunsen Burner
6. Drying Oven
7. Analytical Balance
8. Desiccator

Procedure:Preparation of Gooch Crucible

Insert the filter tube in the stopper of the filtering flask, set the Gooch crucible in the filter tube, and connect the flask to the suction pump. Fill the crucible with some of the suspension of asbestos in water, allow it partly to settle in the crucible, and apply a light suction to draw off the water, leaving a firm mat of asbestos in the crucible. Add more suspended asbestos and repeat the process until a mat is built up that weighs 0.5 ± 0.1 g. after ignition. Wash the asbestos mat thoroughly with water, dry in the oven and ignite over a bunsen burner. Cool the crucible in the desiccator, weigh, and replace it in the filter tube supported in the clean, dry filtering flask.

Note: In the determination, the asbestos apparently adsorbs irreversibly a small amount of soluble bitumen (usually 1 to 5 mg. per gram of asbestos) which is not removed by subsequent washing with solvent. The weight of asbestos used, therefore, should be kept within the specified limits to ensure reproducible results.

Solubility Test

An amount of moisture-free material which shall contain approx. 1 gram of bitumen shall be weighed into a tared Erlenmeyer flask. 100 cc. of chemically pure C Cl_4 shall be added to the flask in small quantities with continued agitation until all lumps disappear and nothing adheres to the bottom. The flask shall be corked and set aside in subdued light for at least 12 hours.

THE DETERMINATION OF PROPORTION OF BITUMEN SOLUBLE
IN CARBON TETRACHLORIDE

Solubility Test (Cont'd)

The Gooch crucible shall then be set up again and the carbon tetrachloride solution carefully decanted through the crucible, with or without light suction. ~~From the solution to the crucible~~ A small amount of C Cl_4 shall be used to wash down the sides of the flask and then the precipitate shall be brought into the crucible and the flask scrubbed to remove all precipitate with a rubber policeman.

The contents of the crucible shall be washed with C Cl_4 until the washings are colorless. The crucible shall be dried in the oven at $212 - 250^\circ\text{F}$. for 20 minutes, cooled in the desiccator, and weighed.

Calculations:

$$\text{Proportion of Bitumen Soluble in } \text{C Cl}_4 = \frac{\text{Bitumen Soluble in } \text{C Cl}_4}{\text{Total Bitumen}}$$

Bird & Son adaption of ASTM Test D-165-42
JRL/jeh
June 1952

LOSS ON HEATING OF ASPHALTApparatus:

1. An electric oven fitted with a perforated revolving shelf. Oven capable of holding $325^{\circ}\text{F.} \pm 1.8^{\circ}\text{F.}$ for 5 hours.
2. Thermometer with range of $155 - 170^{\circ}\text{C.}$
3. 1 - 3-oz. Gill style seamless ointment box. (American Can)

Procedure:

Fifty grams of the water-free material to be tested shall be weighed into a tared 3-oz. can. The oven shall be brought to a temperature of 325°F. and can which contains the sample shall be placed on the revolving shelf. The oven shall then be closed and the shelf rotated 5 to 6 revolutions a minute during the entire test. The oven temperature shall be maintained at $325^{\circ}\text{F.} \pm 1.8^{\circ}\text{F.}$ for 5 hours after the sample has been introduced and the oven has again reached that temperature. The sample shall be removed from the oven, cooled and weighed, and the loss due to volatilization calculated.

During the 5-hour period of test, the temperature shall not vary more than the 1.8°F. All tests showing a greater variation in temperature shall be rejected.

Metric Equivalents

$325^{\circ}\text{F.} \pm 1.8^{\circ}\text{F.}$

$163^{\circ}\text{C.} \pm 1^{\circ}\text{C.}$

Bird & Son adaption of ASTM Test D-6-39-T

Revised June 1952

JRL/jeh

FRACTURE OF HARD ASPHALTS AND ROOFING

Apparatus: Hammer

Procedure:

The fracture of a hard asphalt is ascertained upon fracturing a specimen by subjecting it to a hard, sharp blow of a hammer and examining the cleavage surface.

Tests on Roofing Products (Slam Test)

Place a 1 sq. ft. Piece of Roll Roofing or one tab of a shingle in a refrigerator maintained at 0°F. for one hour. The sample shall then be removed from the refrigerator and immediately slammed, with the granule surface face down, against the top of a bench or similar object. One end of the sample shall be held in the hands and the slamming continued until a large portion of the granules drop off. A sample of our own product is usually tested simultaneously with a competitive product. The number of slams necessary to produce a substantial loss of granules is recorded.

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JRL/jeh

BARBER IMPACT TEST FOR BRITTLENESS

This test is applied to asphalts, asphaltic cements or any bitumen which is practically solid at ordinary temperatures; irrespective of the percentage of pure bitumen in the sample.

Apparatus:

The apparatus uses the same principle as Page Impact Machine. The cap weighs 100 grams, is 21 mm. diameter; and the bottom end is spherical of 21 mm. diameter. The hammer is 22 mm. diameter, weighs 425 grams, and slides freely in the guides. The cylinders to be tested are 32 mm. in diameter by 32 mm. high and are made in suitable, split, brass moulds. The cylinders are held in place on the anvil by a suitable spring loaded V shaped vise.

Method of Test:

Thoroughly coat mould with china clay slurry and allow to dry. Heat sample to 375 - 400°F. and stir well to insure uniformity. Pour at a temperature as close to 375°F. as possible, being sure to pour each mould a little more than full to allow for shrinkage. Allow to cool for one hour, then trim off the excess with a hot knife and coat the freshly exposed surface with china clay slurry as this will assist later in discovering the appearance of the break.

Remove the samples from the mould and immerse the samples in the 32°F. (brine) bath for one hour. The depth of the bath is 2-3/4" so that the sample when in position to run is one-half inch below the surface of the bath. The base of the tester shall rest directly on the bottom of the bath pan and the pan in turn shall rest on a 1-1/2" cork base.

Insert a test sample in the machine, so that it rests firmly on the anvil and adjust the vise to center it under the spherical cap. Lower the hammer and the cap in place, and adjust the scale to zero position. Raise the 425 gram hammer one cm. and let it fall freely. Increase the height of each blow one cm. until the sample is fractured, but be sure to allow the hammer to drop at the rate of one blow every three seconds. Disregard rebound of the hammer. Be sure that the slides are properly greased and that the instrument base is level and the slides are absolutely vertical.

Frequently the change in sound of the blow indicates a crack in the sample before the same is visible although this change in sound should not be relied on too greatly. Use at least six cylinders for the test and disregard any very high or low results in calculating the average. The average maximum drop required to fracture the cylinder is called the coefficient of brittleness.

Checked June 1952
JRL/jch

ASPHALT APPEARANCETest for - AsphaltsApparatus:

1. 3 ounce can (with cover)
2. Heater

Procedure:

Carefully melt a small quantity of the Asphalt to be tested to the temperature at which it can be poured, and pour into a 3 ounce can (penetration). At least one-half the surface shall be free from froth or bubbles. Allow the Asphalt to cool in a place free from draughts or dust. When cool examine the surface, then cover can and set aside for one week, after which period examine again.

Immediately after cooling, the surface should be glossy black and after standing 1 week the surface shall not show any dulling or frosting, or the separation of any greasy or oily material when compared with a freshly made sample.

Any sample carefully prepared for a penetration test is satisfactory for this test.

Checked June 1952
JRL/jeh

QUALITATIVE TEST FOR WATER IN ASPHALT

Test For: Asphalts, Tar, Pitches, Fuel Oil.

Apparatus:

1. Container - about 1 pint capacity
2. Heater
3. Thermometer

Procedure:

The substance to be tested is heated slowly till it can be poured. About one-half pint of the material is then poured into the test container.

Apply heat uniformly until a temperature of 350°F. has been attained. The presence of water will cause foaming and sputtering.

All traces of water are to be removed from the substance before the sample is submitted to further tests.

Checked June 1952
JRL/jeh

ADHESION OF ASPHALT TO ROOFING GRANULES

A test to determine the adhesion of Asphalt to colored granules.

Apparatus:

1. 400 cc beaker
2. Method of heating (gas burner or hot plate)
3. Spatula
4. 100 cc beaker

Procedure:

A small amount of Asphalt previously prepared having a melting point of 120°F. is heated slowly to a good pouring consistency.

4 grams of the granules being tested are weighed into a 100 cc beaker and 1.5 grams of the Asphalt poured on top of them, the Asphalt and granules are then worked into a ball with a spatula. 100 cc of water is placed in the 400 cc beaker and brought to a boil, when boiling, the ball of Asphalt and granules is dropped in and the boiling continued for 5 minutes.

The results of the test are reported as:

Excellent - No apparent change

Good - 1 or 2 granules showing through Asphalt

Fair - All outside granules show

Poor - Granules come out of Asphalt, ball disintegrates

Checked June 1952
JRL/jeh

PERCENT FILLER IN STABILIZED COATING

Object: A quick but satisfactory method of determining the percentage of mineral filler present in a stabilized coating.

Apparatus:

1. Porcelain crucible
2. Analytical balance
3. Bunsen burner
4. Dessicator

Procedure:

The sample of stabilized coating being tested is brought to a fluid condition by slowly heating, avoiding local heating. The sample is mixed thoroughly by stirring with a steel rod and a small portion of the sample poured onto an amalgamated tin plate. The sample is cooled at room temperature and a small sample (approximately one gram) weighed in a tared porcelain crucible. The crucible containing the sample is then placed on a triangle over the Bunsen burner and brought to a red heat and held there for one hour. At the end of this period, the crucible is cooled slightly and then placed in dessicator to cool to room temperature. When completely cool, weigh on analytical balance and compute percent filler.

$$\% \text{ Mineral Filler} = \frac{\text{weight mineral residue}}{\text{weight of original sample}} \times 100$$

Limitations:

Due to the excessive loss that occurs when limestone or dolomite are heated above 1000°F., the above test cannot successfully be used for coating asphalts that have been stabilized with these materials. A solubility in CCl_4 or benzene should be used if limestone or dolomite are found to be present. A fairly accurate figure may be obtained if the samples are ignited in a muffle furnace with a temperature of 1000 - 1050°F. for one hour.

Revised June 1952
JRL/jeh

PERCENT FILLER IN STABILIZED COATING
BY WESTPHAL BALANCE

Object: A method of determining the percentage of mineral filler present in a stabilized coating.

Apparatus:

1. Westphal balance
2. Pitch cube molds
3. Tared weight - 11.34 grams
4. Copper wire, 3 1/2" length of 0.012" diam, $\approx$ 0.05 g.

Procedure:

The sample being tested is brought to a fluid condition and is mixed thoroughly by stirring. The sample is poured into a pitch cube mold until filled to overflowing. The sample is cooled to room temperature and the top of the sample is cut off by using a hot knife flush with the top of the mold, thus giving you a perfect cube. The cube is now taken from the mold and a piece of wire is inserted into the sample cube. The wire with the sample cube is suspended from a tared weight on the Westphal balance into a graduate filled with water.

Weights are then placed on the arm of the balance to equally balance the sample.

The weights are recorded as follows:

The position of the largest weight is taken as the first number, the next largest is taken as the second number and so on.

The readings obtained are then compared with the corresponding percent fillers given by the following chart:

<u>Westphal Reading</u>	<u>% Filler</u>
53	- 50%
54	- 48
55	- 47
56	- 45
57	- 43
58	- 41
59	- 39
60	- 37
61	- 33
62	- 30
63	- 27
64	- 24
65	- 22

March 1956
EC/rp

FIXED CARBON IN ASPHALT

A test which is of use in the identification of an Asphalt.

Apparatus:

1. 20 gm. platinum crucible
2. Bunsen burner
3. Desiccator
4. Analytical balances

Procedure:

A $\frac{1}{2}$ gram sample of the bitumen being examined is weighed in a tared 20 gm. platinum crucible provided with a close fitting cover. This is heated for exactly seven minutes on a triangle in the full flame of a Bunsen burner which has been carefully regulated so as to give a flame 6-8 cm. high to the top of the inner cone, and fully 20 cm. high to the top. The crucible should be placed $\frac{1}{2}$ cm. above the top of the inner cone. Care should be taken to prevent drafts as a steady even flame is required.

The crucible while still covered is removed from the flame, cooled in a desiccator and weighed. The cover is then removed, the crucible returned to the flame, placed on its side to give access to air and ignited until only ash remains. The cover is also heated to remove any carbon that may have been deposited on its under side. After being cooled again the crucible and cover are weighed, this latter weight being subtracted from that obtained after the first ignition.

The loss in weight is that of the fixed carbon.

The remainder is the ash.

Checked June 1952
JRL/jeh

DETERMINATION OF MELTING POINT OF PARAFFIN WAXAmerican Method - English MethodApparatus:

1. Sheet metal boat of the following dimensions:

Length of Top	-	7-3/4"
Length of Bottom	-	6-1/2"
Width of Top	-	1-9/16"
Width of Bottom	-	7/8"
Depth	-	1-5/8"
2. Saybolt wax testing thermometer with range of 80°F. to 150°F. graduated into 1/4° divisions.
3. Stand clamp to support thermometer.

Procedure:

1. Adjust thermometer in the clamp so that the thermometer bulb is 1/4" to 3/8" from the bottom of the boat which is resting on the bench. Heat the wax to be tested 20° above the expected melting point in a clean glass beaker.
2. Pour the melted wax into the boat to a height of 1-1/8" corresponding to the immersion scale line.
3. Note and record the American Method melting point, the temperature when the film of solidified wax extends from the thermometer bulb to the edge of the boat.
4. Start moving the boat on the bench back and forth in a smooth uniform motion making 15 to 20 complete oscillations per minute. Note and record the temperature every thirty seconds.
5. As soon as three successive readings are the same, this temperature is the melting point by the English Method.
6. The most reliable method for obtaining the "American Method" melting point is by adding 3°F. to the melting point as obtained by the English Method.
7. Record results obtained by American Method.

Precautions:

Wax must not be overheated. Sample should be stirred during heating and removed from the heat as soon as melted.

Checked June 1952
JRL/jeh

SOFTENING POINT OF TAR PRODUCTS - CUBE IN WATER METHOD

A test to measure the softening point of a pitch or tar which has a softening point below 170°F.

Apparatus:

1. A mold suitable for forming a $\frac{1}{2}$ " cube
2. An L-shaped right angled hook made of #12 B & S copper wire - the foot of which shall be 1 inch long
3. A 600 ml. griffin low form beaker
4. Thermometer. 30 to 180°F. range
5. Heating apparatus

Procedure:

The mold is filled with the pitch being tested and cooled, all excess being removed. The cube of pitch is placed on the foot of the hook and the cube placed in the beaker filled 2/3 full of distilled water. The thermometer is suspended so that the bulb is in the water at the exact height of the cube. (1" from the bottom of the beaker).

Starting with the water at 60°F. apply heat so that the temperature will be raised. 9°F. a minute. The rate of rise of temperature shall be uniform and the maximum allowable variation after the first three minutes shall be 0.9°F. The temperature recorded by the thermometer at the instant the pitch touches the bottom of the beaker shall be reported as the "Softening Point".

The use of freshly distilled water is essential as air bubbles may otherwise form on the cube and retard its sinking. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results.

Bird & Son adaptation of A.S.T.M. Test D-61-38
June 1952
JRL/jeh

FLOAT TEST FOR COAL TAR SATURANT

(Modification of A.S.T.M. D-139-49)

Apparatus:

1. One aluminum float
2. Two brass collars
3. Thermometer; range -4 to 220°F.
4. Testing bath (a 2000 ml. beaker or a one gallon can)
5. Water bath @ 41°F. (a 2000 ml. beaker or a one gallon can)
6. Two small brass plates

Procedure:

Amalgamate the brass plates as follows:

1. Clean thoroughly with CCl_4 , then use soap and water
2. Rub with dilute HCl or with mercuric chloride solution
3. Pour on a small amount of mercury and rub it into the plate with a ductility side mold.

Bring the temperature of the 1/2 pint coal tar saturant sample to 125 F., then stir it thoroughly until it is homogeneous and free from air bubbles. Place the brass collars with the smaller end down on the amalgamated plates. Then pour the sample into each collar in any convenient manner until slightly more than level on top. Immediately immerse the samples for 5 minutes in the water bath @ 41°F. Then remove from the bath and trim the surplus material flush with the top of the collar by means of a spatula or steel knife that has been slightly heated. Then place the collars and plates in the water bath @ 41°F. and leave them in this bath not less than 15 nor more than 30 minutes.

Heat the water in the testing bath to 90°F. and then maintain this temperature $\pm 1^\circ\text{F}$. without stirring throughout the test.

After 20 minutes of immersion, take the test samples out of the 41 F. water bath. Remove the collar with the tar sample from the amalgamated plate and screw it into the aluminum float. Completely immerse the assembly for one minute in the water bath @ 41 F. Then remove the water from the inside of the float and immediately float the assembly on the testing bath. Lateral drift of the assembly shall be permitted, but no spinning motion

shall be intentionally imparted thereto. As the plug of tar 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.

The time in seconds, between placing the apparatus on the water and the water breaking through the material shall be determined by means of a stop watch, and shall be taken as a measure of the consistency of the tar. Always run two collars for each sample of tar to be tested.

PRECAUTIONS:

1. Be sure the collar fits tightly into the float and see that there is no seepage of water between the collar and the float during the test.
2. Owing to possible danger to health if mercury is handled carelessly, the following rules should be observed at all times:
 - a. Store mercury in a closed jug in a cool place.
 - b. Strictly avoid spilling any mercury.
 - c. Remove mercury vapors by working under a hood with good ventilation.
 - d. Keep amalgamated brass plates and other apparatus at not above normal room temperatures.

JRL/rp
April, 1959

MELTING POINT OVER MERCURY

Use: For Gilsonite.

Procedure:

A seamless 3-ounce tin or a glass dish of about the same size is filled with mercury to within $1/4$ " from the top and placed upon a suitable support over some heating device such as an electric heater or a Bunsen burner.

On the surface of the mercury are placed one or more microscope cover glasses upon each of which are placed from one to six particles of the gilsonite under examination. These particles are obtained by crushing the sample and selecting, with tweezers, specimens which are as near cubicle as possible and from $1/64$ " to $1/32$ " in size, preferably nearer the smaller size. The dish is then covered with an inverted glass funnel from which the stem has been removed, and a thermometer suspended above and entering the test system through the orifice of the funnel penetrating the mercury sufficiently to completely immerse the bulb.

The source of heat is regulated so that the temperature of the mercury will rise at the rate of 90°F. per minute.

The temperature at which the angles or edges of each particle round out sufficiently to leave no doubt in the operators mind as to a change from their original shape is to be considered the melting point of the particle. Twelve or more particles can be tested at a time if desired from as many lumps giving a fairly comprehensive survey of the shipment.

It is well to place the melting point apparatus in such a way between the operator and the source of light that the light will shine upon the surface of the cover glasses. The use of a hand magnifying glass which is held so as to get the greatest magnification is very useful in noting the change in the shape of the particles of the gilsonite being tested.

This method is secondary to the ball and ring method, and is seldom used.

Precautions when using Mercury:

See Test No. 3 in this Manual.

Checked June 1952
JRL/jeh

ACID VALUE TEST

Use: To determine the amount of "free acid" available in a sample of Pitch, Asphalt, Tar or Wax.

The acids measured are: Free fatty acids, free resin acids, and free asphaltous acids.

Procedure:

Boil 5.00 grams of the material being tested under a reflux condenser with 25 cc. of benzol until the material dissolves or is completely disintegrated; then add 100 cc. of carefully neutralized 95% ethyl alcohol and continue boiling for 20 minutes (Bituminous materials with high fusing-points should be fluxed to semi-liquid consistency with a given weight of neutral paraffin oil). The liquid is then decanted from the insoluble residue while hot, the latter boiled with another 50 cc. of ethyl alcohol and the process repeated, until the extract no longer reacts acid with alkali blue 6-B (or Phenolphthalein). The residue is then disregarded. To the combined extracts, add 10 cc. of 25% barium chloride solution, and 8 drops of a 3% alcoholic solution of alkali blue 6-B and 10 drops of a 1% alcoholic phenolphthalein solution, and titrate cold with standard N/10 caustic potash. As the free acids are neutralized by the alkali, the barium soaps are precipitated and the unsaponified substances are thrown out by the water contained in the N/10 caustic potash until at the close of the titration the solution becomes clear, rendering the blue-green to red end point fairly sharp.

The acid value is equivalent to the number of milligrams of potassium hydroxide required to neutralize the free acids in 1 gram of the substance.

The above test is of some help in determining the source and type of a substance.

The N/10 caustic potash is prepared by dissolving 5.612 grams of pure potassium hydroxide in 500 cc. of 95% alcohol, diluting to exactly 1 liter with water at 60°F. and carefully standardizing against sulfuric acid of known strength.

Checked June 1952
JRL/jeh

THE DETERMINATION OF ASPHALTIC BITUMEN WITH TAR

H. Mallison. Roads 11, 165 (1933); Bldg. Sci. Abstracts 6,334.

A sample consisting of 3-4 g. of the mixture is boiled under a reflux condenser for 15 min. with 10 times its wt. of CHCl_3 or CS_2 , filtered to sep. the "free C", this residue being washed with the solvent until colorless, and the solvent then distd. off. The bituminous residue is then treated with 6-8 ml. concd. H_2SO_4 on a boiling water bath, allowed to cool, washed, dild. with 500 ml. water, left to stand for 2 hrs., decanted through a weighed filter paper, washed with hot water, dried and weighed. The weight of the asphaltic bitumen so found is that present in the tar mixture.

From Chemical Abstracts, May 20, 1934, Volume 28, Number 10.

Checked June 1952
JRL/jeh

DIAZO REACTION FOR IDENTIFYING COAL TAR IN ASPHALT

Use: This test is used at PBL for distinguishing coal tar pitch from asphalt, or the identification of our asphalt roofing which has been painted with coal tar paint or mopped over with coal tar pitch instead of Neponset Compound.

The reaction was devised by Graefe in 1906, and is carried out as follows:

Procedure:

Boil 2 grams of the bituminous substance with 20 cc. of a normal aqueous solution of Na OH for approximately five minutes. After cooling, the liquid is filtered. If the filtrate is dark, add some finely powdered Na Cl. Then cool the solution as low as possible with ice in a CaCl solution. Add a few drops of freshly prepared diazobenzolchloride solution. If this solution is added slowly and phenols are present, a red coloration will result providing the whole solution is ice cold. A red coloration will result in the absence of phenols if the mixture gets above 32°F.

Preparation of Reagents:

Normal aqueous Na OH is prepared by adding 40 grams of solid Na OH to 500 cc. of distilled water, and when complete solution results, diluting the solution to 1 liter with more distilled water.

Preparation of Diazobenzolchloride (Gattermann):

Freshly distill enough aniline to yield 6 grams. Dissolve 5 grams of it in 15 grams of concentrated H Cl and 30 grams of distilled water; cool the solution in ice and treat with a cold solution of 5 grams of sodium nitrite in 15 grams of distilled water until free nitrous acid which is evolved is shown by turning starch Potassium Iodide paper blue.

Checked June 1952
JRL/jeh

LUBERMAN-STORCH REACTION FOR ROSIN, ROSIN OIL,
AND CHOLESTEROL - RAPID METHOD

Use: This is a rapid qualitative test for the detection of Rosin, Rosin Oil, or Cholesterol in our roofing. The first is evident if roofing is laid over pitchy lumber.

Procedure:

A one-gram sample is dissolved in 3 cc. of Acetic anhydride at a gentle heat, cooled and the clear liquid decanted into a porcelain crucible. Add 1 or 2 drops of H_2SO_4 (1.53 sp. gr.). Rosin and Rosin Oil will produce a fugitive violet coloration turning to a brown. Cholesterol will produce a fugitive rose color rapidly turning to a dark green.

Linseed, cotton seed, china-wood and corn oils give a permanent greenish brown coloration.

1.53 sp. gr. H_2SO_4 is made by diluting 34.7 cc. of concentrated acid with 35.7 cc. of distilled water.

Checked June 1952
JRL/jeh

TENTATIVE METHOD OF TESTING BITUMINOUS MATERIALS
FOR SETTLING OF INSOLUBLE MATTER

Use: This is a method for the determination of the amount of mineral or other insoluble matter in bituminous materials, which settle out when the material is maintained in the liquid state.

Apparatus:

- a. 400 cc. tall form beaker approx. 12 cm. high, or similar receptical
- b. 2 - 250 cc. beakers
- c. Cary-Curr extraction apparatus
- d. Paper filtering thimbles
- e. Steam oven
- f. Analytical Balance
- g. Desiccator
- h. Constant temperature oven (400°)

Procedure:

A representative sample of approx. 400 cc. shall be heated to 400°F. in the tall form beaker and treated as follows:

1. Stir thoroughly and decant approximately 2.5 grams into a tared 250 cc. beaker and weigh; add 150 cc. pure carbon tetrachloride slowly with continued agitation until all lumps disappear and nothing adheres to the beaker. Decant into tared filter thimble and wash any residue remaining in beaker into the thimble. Place thimble in the extraction apparatus and extract with carbon tetrachloride until drippings are colorless. Remove filter thimble and after drying in steam oven, cool in desiccator and weigh. The beaker shall be dried and reweighed; the difference between the original and final weights shall be added to the weight of sediment in filter.

$$\% \text{ Sediment} = \frac{\text{Wt. of final sediment}}{\text{Original weight of sample}} \times 100$$

2. Hold at 400°F. in oven for one-half hour without agitation and decant approx. 2.5 grams into tared 250 cc. beaker, weigh and treat as in Step 1.

Percentage Settling:

Percent sediment in Step 1. minus percent sediment in Step 2.
divided by percent in Step 1.

Note: Gooch Crucible Method for testing insoluble material may be used in place of Cary-Curr Method. In the case of very fine mineral matter, the Gooch Method is to be preferred.

Checked June 1952
JRL/jeh

COLOR TEST FOR PITCHProcedure:

Carefully weigh 1/2 gram pitch and place in a closed container with exactly four ounces, or 118.4 c.c., of benzol.. Allow pitch to dissolve cold (this will require about two hours). When pitch has completely dissolved, pour the solution through a fine strainer to make sure that no skins or undissolved particles remain. If all the solution goes through the strainer, then sufficient of the solution to fill the flat vial is poured into same and the vial is then compared with the color standards by holding to a light, preferably daylight.

Checked June 1952
JRL/jeh

SOLIDS IN PITCH EMULSION

A test in which the liquids of an emulsion are removed by drying.

The sample being tested is stirred brickly and a large drop placed on a tared small flat container (the cover to a 3 ounce seamless tin is satisfactory). This is weighed and should be between 0.5 and 1.0 gm. The sample is then placed in a 220 F. oven for four hours, removed and cooled and reweighed.

$$\text{Percent solids} = \frac{\text{weight of dried sample}}{\text{weight of original sample}} \times 100$$

Checked June 1952
JRL/jeh

TOTAL SOLIDS IN ASPHALT EMULSIONS

Weigh out about 3 grams of well stirred emulsions into a small 4-oz. can cover or beaker which has been previously weighed. Place in steam oven for 1-1/2 to 2 hours or until dry. Weigh again and calculate as follows:

$$\frac{\text{Weight of dry sample}}{\text{Weight of wet sample}} \times 100 = \% \text{ Solids}$$

Solubility test on residue from above test determines the type and amount of filler or fibre if present.

Checked June 1952
JRL/jeh

STRENGTH TEST FOR ASPHALT EMULSIONS
Standard Procedure

The following is a method for determining the strength, or stability, of an asphalt emulsion, by measuring its resistance to an electrolyte, such as sodium chloride, in terms of cc. of 10% of NaCl solution. These electrolytes, when added to an emulsion in the proper amounts, neutralize the negative, repellent charges on the asphalt particles, thus allowing them to join up, or coalesce, forming large particles which do not pass through a filter paper.

1. Make up a 10% sodium chloride solution by dissolving 10 parts of C. P. NaCl in 100 parts of distilled water. The sodium chloride should be weighed accurately on an analytical balance and placed in an empty bottle. The water is then weighed into the bottle on the larger laboratory balance.
2. Dilute the asphalt emulsion to be tested by pouring 20 parts of the emulsion into 100 parts of distilled water. This weighing may be done on an ordinary balance. Above figures are for a 60% solids-content emulsion, or $20 \times .60 = 12$ gms. of asphalt. If solids content is 65%, then $\frac{12}{.65}$, or 18.5 gms. of emulsion should be used, etc.
Add water to bring total weight to 120 gms.

The emulsion must be thoroughly stirred before taking a sample out for dilution.

3. Pour some of the 10% sodium chloride solution and the diluted emulsion into two separate burettes.
4. The next step is to decide upon the number of cc. of 10% NaCl solution to be tried as a starter. This may not be difficult for an experienced operator, but for the inexperienced, it might be well to start with 1 cc. Measure into a graduate enough distilled water to bring the total of salt solution plus water to 15 cc. (thus, 14 cc. in this case). Note: NaCl is not yet added, but amount only decided upon. Run this distilled water into a test tube of about 30 - 35 cc. capacity.
5. Run 5 cc. of the diluted emulsion from the burette into the water in the test tube.
6. Run the previously determined cc. of salt solution (1 cc. as an example) from the burette into the emulsion and water in the test tube.
7. Test tube should now be corked and placed in some form of horizontal shaking machine, preferably hand-driven, so that test tube will receive 90 complete shakes; that is, back and forth, in one minute. This effect may be duplicated fairly close by holding the test tube in hand, in a horizontal position, and shaking uniformly and smoothly, 90 times in one minute.

8. Contents of this test tube are now filtered through an E&D #617 filter paper which has been previously folded into a 3" funnel.
9. The strength number of any given emulsion is the lowest number of cc. of 10% salt solution which will give a practically clear filtrate. 1 cc. less than the above gives a dark brown filtrate. Thus, in the example given above, where 1 cc. of salt solution was used, if filtrate is dark brown it means that another trial must be made with 2 cc. of salt solution and 13 cc. of water using, of course, 5 cc. of fresh emulsion. These trials must be continued until the filtrate shows a sudden decrease in turbidity. Every further addition of NaCl solution will produce a fairly clear filtrate, but as stated above, lowest number of cc., or point where filtrate first clears, is reported as the strength number. Thus, if filtrate showed a break from a turbid to a clear color upon the addition of 5 cc. of salt solution, strength number is 5. It is possible that emulsion may be so weak that it will break with the addition of 15 cc. of water only (without NaCl), and in that case stability would be zero.

Based on our experience, a #50, #30, or #10 emulsion should have a strength number, or stability, of about 6.

Checked June 1952
JRL/jeh

DEMULSIBILITY OF EMULSIFIED ASPHALT

This method conforms with the demulsibility test for emulsified asphalt as required by the various State Highway Departments.

Apparatus:

- (a) A 600 cc. beaker, a glass stirring rod, and a 50 cc. graduate (graduated in 1 cc. divisions).
- (b) A No. 14 mesh screen

Solution Required:

A 0.02 N calcium chloride solution: Dissolve 2.02 grams $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ in one liter of freshly distilled water.

Procedure:

One hundred grams of the emulsified asphalt shall be placed in a 600 cc. glass beaker, the weight of which has been previously determined, and 35 cc. of 0.02 N calcium chloride solution shall be added during a period of approximately two (2) minutes, being continually stirred with a weighed glass rod. The emulsion and the solution of 0.02 N calcium chloride shall be maintained at a temperature of 77°F (plus or minus 2°F.) during the two minutes required for adding the reagent and stirring. The contents of the beaker shall then be drained through a weighed No. 14 sieve and the unbroken emulsified asphalt in the breaker and on the rod shall be rinsed through the sieve with distilled water until there is no appreciable discoloration of the rinsing water. The beaker, rod and sieve shall then be dried in an oven at 325°F. for two hours, or until completely dehydrated, and weighed. The weight of the asphaltic residue deposited in the beaker, and on the rod and on the sieve shall be ascertained by subtracting from this weight, the weight of the beaker, rod and sieve. The weight of this asphaltic residue, divided by the weight of the emulsion taken, gives the percentage of the asphaltic residue deposited. This percentage of asphaltic residue divided by the percentage of the residue obtained in the distillation test, gives the percentage of the residue obtained in the demulsibility test on the basis of the distillation residue and will be known as the demulsibility of the emulsion.

Above is for quick-breaking type. For slow-breaking emulsions, use 50 cc. of .10 N CaCl_2 per 100 gms. emulsion.

Accuracy:

Duplicate determinations should not differ by more than 2 units of the percentage found.

Checked June 1952
JRL/jeh

TOTAL SOLIDS IN ROSIN SIZE

Weigh out about 3 grams of rosin size into a small 4-ounce can cover or beaker which has been previously weighed. Place in steam oven for 1-1/2 to 2 hours or until dry. Weigh again and calculate as follows:

$$\frac{\text{Weight of dry sample}}{\text{Weight of wet sample}} \times 100 = \% \text{ Solids in Rosin Size.}$$

Checked June 1952
JRL/jeh

SEPARATION OF ASPHALT TO ITS CONSTITUENT PARTS

Note: The following test method was adapted from the work done by Dr. O. G. Strieter, of the Bureau of Standards. Dr. Strieter's early method is described in N. B. S. Research Paper R. P. 1387, dated May 1941. In the following method we have incorporated all the changes made by Dr. Strieter up to September 1947.

Apparatus:

- 1 - 500 ml. Wide Mouth Erlenmeyer[®] Flask
- 1 - Small Spatula (3" blade)
- 1 - Buechner Funnel (for 11 cm. diam. filter papers)
- 1 - Large Evaporating Dish
- 1 - Side-arm Vacuum Flask
- 2 - 250 ml. Beakers
- 1 - Soxhlet Extractor with ground glass joints capable of holding thimbles 43 x 123 mms.
- 1 - Steam Bath or an Electric Hot Plate
- 1 - Gooch Crucible and Vacuum Apparatus
- No. 42 Filter Papers (11.0 cm. diam.)
- Soxhlet Thimbles (43 x 123 mms.)

Chemicals

- 1. N-Petane - Technical Grade, 94% distilling between 95° and 97°F.
- 2. Ethyl Ether - Analytical Grade, meeting A. C. S. Standard Specification.
- 3. Florex XXF, 100% passing through a No. 100 Screen, Floriden Co., Quincy, Florida
- 4. Attapulugus Fullers Earth. Regular VM, 100% passing through No. 100^U Screen.

Procedure:

Chill the asphalt to approximately 40°F., cut into small pieces, and weigh 5 grams $\pm$ 0.5 gm. on a tared watch glass. Then transfer to a 400 ml. wide mouth Erlenmeyer flask. It may be more expedient to weigh flux asphalt directly in the 400 ml. flask by pouring in the melted asphalt.

Add 100 ml. normal pentane to the flask and shake lightly or rotate to dissolve the asphalt; then add 75 ml. pentane, agitate again; then add another 50 ml. pentane, lightly cork the flask, and allow it to stand overnight.

- A. Asphaltenes: To collect the asphaltenes, filter the pentane solution through a Gooch crucible with slight suction. Collect the filtrate in a 600 ml. beaker. Wash the asphaltenes with pentane until the latter runs clear. Dry the Gooch crucible in the steam oven (220 - 250°F.) for one hour, cool in a desiccator, and weigh.

If there is evidence of asphaltenes adhering to the walls of the flask, wash them down with carbon tetrachloride, dry the flask in the steam oven (220 - 250°F.) for one hour, cool in a desiccator, and weigh. The combined weights of asphaltenes divided by the original weight of asphalt, multiplied by 100 gives the percentage of asphaltenes.

- B. Oily Constituents: Add Fullers Earth or Florex slowly to the pentane filtrate in the 600 ml. beaker. Agitate the solution thoroughly while adding, and do not add more than one to two grams at one time at the beginning. The end point is reached when there is a marked change in color of the solution, from blackish brown to deep red or orange - red when examined by northern daylight. Approach of the end point can be determined by observing the added Fullers Earth. The first portions are darkened decidedly, due to absorbed resins, but when practically all of the resins are absorbed, the earth is darkened only slightly, and the solution at the top becomes lighter in color and usually takes on a slight fluorescence.

At the approach of the end point, it may be more helpful to filter 50 mls. of the solution through a Buechner funnel after each additional of Fullers Earth to better observe the color.

When the end point is reached, allow the Fullers Earth to settle, and pour the solution through a Buechner funnel with a No. 42 Filter Paper. Transfer the Fullers Earth to the Buechner funnel, and wash with approximately 50 ml. of pentane.

B. Oily Constituents: (Cont'd)

Transfer the filtrate, which contains the oily constituents, to a large evaporating dish, and evaporate the solution at room temperature under the hood until the volume is approximately 50 ml., then transfer to a tared 250 ml. beaker, and wash the last traces from the evaporating dish with pentane. Continue to evaporate the solution in the 250 ml. beaker until most of the pentane has evaporated; then heat the beaker at 220°F. in a steam oven or over a steam bath for one hour, cool in a desiccator, and weigh. This portion of the oily constituents is known as the "cold extracted oils".

Transfer the Fullers Earth, while still damp, to a Soxhlet Extraction thimble (43 x 123 mms.), and plug the end with cotton. Place the filled thimble in the Soxhlet extraction apparatus, cover with pentane, and let stand overnight.

Extract for 7 hours with pentane, allow to stand overnight again covered with Pentane, and extract an additional 5 hours. The extractor should be heated on a steam bath or on an electric hot plate on "low" with 2 layers of asbestos paper covering the stove surface.

Concentrate the extraction liquid as described for the "cold extracted oils". Combine this portion of the oily constituents, designated as "hot extracted oils" with the cold extracted oils. The weight of the combined portions divided by the weight of the original asphalt sample multiplied by 100 gives the percent oily constituents.

- C. Asphaltic Resins: Air dry the extraction thimbles and contents from the determination of the hot extracted oils until free from the odor of pentane, replace them in the extraction apparatus, and extract with ethyl ether in the manner described for the oily constituents until the extract is water white. This extraction should not take more than 5 hours. Filter the ether extract through a Gooch crucible, and concentrate it in the manner described for the oil-pentane solutions. Water often present in ethyl ether in small amounts or condensed water may appear on the surface of the solution when ethyl ether is evaporated. It can be removed by adding small amounts of absolute alcohol during the last stages of the evaporation. Remove the last traces of ether alcohol and water by heating the residue to not over 176°F. and cooling in a vacuum desiccator. The weight of the residue divided by the weight of the original asphalt sample multiplied by 100 gives the percent Asphaltic Resins.

Note: Samples as large as 50 gms. of asphalt may be used if corresponding changes are made with the other apparatus and solvents. With larger asphalt samples, the color change is more distinct when adding Fullers Earth.

JRL/hem
Sept. 1954

DETERMINATION OF THE SULPHUR CONTENT OF COAL,
OIL, AND OTHER COMBUSTIBLES (from bomb washings)

The sulphur in coal, oils, and other combustible materials may be determined from the washings in the bomb after the same are burned in the calorimeter. During combustion the sulphur is converted into sulphurous or sulphuric acids which condense upon the inside of the bomb.

Upon opening the bomb, these acids may be washed out with distilled water in a wash bottle into a beaker. Add 5 ml. of a saturated Na_2CO_3 solution to the washings, heat to boiling on a hot plate and boil slowly for 10 min. Filter the hot solution through a No. 42 filter paper using light suction if necessary. Wash the precipitate six times with boiling distilled water, and then discard the precipitate.

To the filtrate, add 10 ml. of Bromine water and then acidify with dilute HCl . (Use a 10 ml. burette and alkacid paper.) Boil to expel the bromine (clear solution), then add 10 ml. of 10% BaCl_2 solution with a pipette. Boil for 15 min., then let stand overnight at approximately 200°F .

Filter on an ashless filter paper (No. 42) and wash with hot distilled water until the washings show no chloride (white ppt. of Ag Cl) when a few drops of a dilute solution of Ag NO_3 are added to the filtrate. (Need approximately 600 ml. hot distilled water). Fold the wet precipitate in the filter paper and ash in a weighed platinum crucible in the muffle furnace.

Calculations:

$\text{Wt. of Ba SO}_4 \text{ (gms.)} \times \text{Factor } 0.1373 = \text{Wt. of Sulphur in gms.}$

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Checked June 1952
JRL/jeh

DETERMINATION OF HEAT OF COMBUSTION OF FUELS
AND OTHER COMBUSTIBLES

1. COAL:

Manipulation:

Place the lower half of the bomb in the iron plate holder, and adjust the fuel pan support in proper position. This support is held by a taper pin which fits into the inner end of the insulation plug, which plug is at the side of the lower cup of the bomb. The taper pin holding the fuel pan support should be entered firmly into the insulation plug in order that the support will not permit tipping which would likely result in the upsetting of the charge of fuel at the time of ignition.

The fuse wire is connected to the binding post on the fuel pan support and extends across the bomb to the binding post at the opposite side of the bomb, sufficient length being allowed that the wire will dip down enough to be in contact with the fuel which is afterwards placed in the pan.

Care must be taken that the wire does not touch the fuel pan.

The fuel used is sampled, crushed, and powdered according to directions given below.

Fill a test tube or convenient weighing vial with the prepared sample and weigh the same accurately to one tenth of a milligram. Pour from this into the pan in the bomb until the pan is approximately half full. Weigh the vial again, and the difference of the above weighings gives us the net quantity of fuel in the bomb. (Alternate Method) Fill a 3-oz. Gill can or a convenient vessel with the prepared sample, and weigh the desired amount directly onto the calorimeter ignition pan with the use of a small spatula. This weight should be greater than five tenths of a gram and not more than one and two tenths grams. For hard coal, the maximum charge should be not greater than one gram. Hard coal should not be as finely divided as soft coal, (through a 60-mesh sieve is sufficient.)

CAUTION!

All interior walls of the passages leading from oxygen supply to bomb including valves and gauges must be kept absolutely free from oil and also the interior wall of THE BOMB as the presence of small quantities of oil in a stream of oxygen will sometimes cause a violent spontaneous explosion.

DETERMINATION OF HEAT OF COMBUSTION OF FUELS
AND OTHER COMBUSTIBLES

1. COAL: (Cont'd)Oiling Calorimeter Bomb:

A sufficient amount of oil should be placed on the threads of the lower cup to the bomb of this Calorimeter to give the same slight lubrication. The bomb contains oxygen under pressure, and no oil should be allowed to run inside the bomb. Neglect to properly oil the threads above referred to may do the instrument damage beyond repair.

2. COKE, HARD COAL, etc.:

The determination of the heat of combustion of coke, and extremely hard coals, is best made by burning the same mixed with a ready burning combustible, such as a high grade bituminous coal. This auxiliary combustible facilitates the complete combustion of the whole mixture of coke or hard coal.

A known weight of the auxiliary combustible should be placed at the bottom of the pan and the coke or hard coal sprinkled over it. The auxiliary combustible should be dried and carefully standardized as to its rise in temperature per gram in the calorimeter when the same is completely burned.

3. LIQUID FUELS:

Calorimeter outfits having bombs with acidproof linings are preferable in testing liquid fuels.

HEAVY OILS:

In the handling of liquid fuel, the most suitable method of preventing the evaporation of the samples is to hold same in a small weighing bottle with a stopper. A dropper is used for the purpose of transferring the liquid fuel from the bottle into the bomb. After introducing the oil into the fuel pan of the bomb, the stopper is replaced in the weighing bottle. The weight of the bottle with the stopper is taken before and after the oil is removed. The difference in the two weighings gives the net weight of the liquid fuel in the bomb. With heavy oils, the oil can be dropped directly onto a small quantity of ignited asbestos in the pan. As soon as the oil is placed in the fuel pan, the upper half of the bomb should be immediately placed in position to prevent any vaporization of the sample. Use approx. 0.6 gm. sample.

LIGHT FUEL OILS, GASOLINE, ALCOHOL, etc.:

Because of the rapid evaporation of the lighter fuel oils, it is not advisable to pour the same directly into the fuel pan. Small gelatine capsules can be obtained which may be filled with ignited

DETERMINATION OF HEAT OF COMBUSTION OF FUELS
AND OTHER COMBUSTIBLES

(Cont'd)

LIGHT FUEL OILS, GASOLINE, ALCOHOL, etc.:

asbestos and into which the light oils may be poured, the latter being absorbed by the asbestos. The filled capsule is sealed, placed in the fuel pan and burned in the usual manner, using iron wire for ignition. The dry weight of the capsule and asbestos must be known and after filling capsule with charge and sealing same, the weight of the whole is taken. Care should be used that no air bubbles are enclosed with the charge in the capsule, as the fuel will otherwise ignite with explosive violence.

The upper half of the bomb is placed in position and the nut screwed down as far as may be by hand, care being taken not to cross the threads. The shoulder on the upper half of the bomb, over which the nut makes bearing contact, should be lubricated with oil. Extreme care should be taken that no oil or grease is deposited on the lead gasket, as the bomb when working properly, closes without the upper half turning on the gasket due to the contact friction of the nut. Any oil on the lead gasket would tend to hinder the proper action in this respect.

The large wrench is used to make the joint tight, and operator should apply the same very nearly to his full strength. (If the thread of the large nut is kept free from dirt, the same will turn into place freely by hand.)

The bomb is now ready to be filled with oxygen, and this is accomplished by means of the spindle valve at the top of the bomb. The nipple is coupled to the oxygen piping by means of the attached hand union. (The oxygen piping should be properly located and screwed fast to the bench.) IN HANDLING THE BOMB, CARE SHOULD BE TAKEN NOT TO TIP OR JAR THE SAME, AS FUEL MAY BE THROWN FROM THE PAN.

Close the valve (on the bomb) that is not connected to the oxygen, and open the one that is connected to the oxygen supply one revolution. Also open the valve on the pressure gauge line one revolution.

Then the valve on the oxygen supply tank is very cautiously opened. The pressure gauge should be carefully watched and the tank valve so regulated that the pressure in the system shall rise very gradually. When the pressure reaches 300 pounds per square inch, the tank valve is closed, and the spindle valve on the bomb immediately after, and then the pressure gauge line valve.

Note: Use ~~600~~⁵⁰⁰ lbs. pressure when making sulphur determinations.

The bomb should be immersed in water immediately to detect any possible leakages. (Preferably a glass jar, as slight leaks are detected by looking from all sides.)

DETERMINATION OF HEAT OF COMBUSTION OF FUELS
AND OTHER COMBUSTIBLES

(Cont'd)

LIGHT FUEL OILS, GASOLINE, ALCOHOL, etc.:

The bomb is now ready for the calorimeter, which is prepared as follows:

Nineteen hundred and fifty grams of water are placed in the calorimeter can. The bomb is then placed in the bucket and the stirrer and thermometer are lowered into position. The large thermometer is then immersed about three inches in the water and the thermometer bulb should be at least one-half inch from the bomb. Care should be taken that the bomb does not touch the sides of the can or that the STIRRER DOES NOT TOUCH THE BOMB.

Ignition Wiring: One terminal of the electric circuit for igniting the charge is connected to the bomb by means of the taper binding post which fits into the spindle at the top; the other is connected to the outer end of the insulation plug on the lower cup of the bomb. A short length of insulated wire carrying the taper pins fitting into the outer end of the insulation plug and the valve spindle is included with the apparatus. The other ends of these insulated wires are fitted with connectors for the terminals from the switchboard. One 100 watt lamp and one 150 watt lamp in parallel are placed in series with the fuse wire when 110-volt circuit is used for firing. Four 100 watt lamps in parallel are placed in series with the fuse wire when 220-volt circuit is used.

The temperature of the calorimeter is noted and the water in the jacket is brought to this same temperature. The jacket water must be originally at a lower temperature than the calorimeter water and is brought to the calorimeter temperature by causing an electric current to flow through the same. Connections are made from the supply circuit to the binding posts on the jacket with suitable switch in circuit.

In making the first run of a day's testing, the temperature of the calorimeter can be adjusted to the lowest practical working temperature of the calorimeter. This temperature of the calorimeter is usually the lowest temperature which can be conveniently read on the thermometer stem just above the thermometer holder, CARE BEING THAT THE THERMOMETER BULB IS PROPERLY IMMERSED (the bottom of bulb 4" from surface of water and not less than 1/2" from any part of the bomb). The jacket is filled with water at a temperature lower than that of the calorimeter water and is brought to this latter temperature by throwing in the switch on the jacket circuit. The jacket thermometer is closely watched and when the temperature is approaching that of the calorimeter, the circuit is opened. The temperature of the jacket is held within approximately one-tenth of a degree from that of the

DETERMINATION OF HEAT OF COMBUSTION OF FUELS
AND OTHER COMBUSTIBLES

(Cont'd)

LIGHT FUEL OILS, GASOLINE, ALCOHOL, etc.:

calorimeter; with the stirrer running for two or three minutes, the temperatures throughout the calorimeter are allowed to equalize. One or two preliminary readings at one-minute intervals are then taken to determine the initial temperature. The charge is then ignited by snapping the switch below the 100 watt and 150 watt bulbs on the panel. When the two lights go out, it indicates that the charge has been fired (0-3 sec.) and the circuit broken inside the bomb by the burning out of the firing wire. The panel switch is then shut off.

As the temperature rises in the calorimeter, the temperature of the jacket is carried along in close proximity to the same. This is accomplished by closing the switch in the jacket circuit and permitting the current to flow as long as the jacket temperature does not exceed the calorimeter temperature. The jacket temperature is carried along with the calorimeter temperature until it approaches its maximum, and then the jacket circuit is opened. With reasonable care on the part of the operator in watching the temperature, he will soon become proficient in maintaining equal temperatures in the jacket and calorimeter.

When the calorimeter temperature is approaching its maximum, readings of the calorimeter thermometer are taken at one-minute intervals, the maximum temperature thereby being accurately determined. When this is done, no further readings are necessary and the run is finished.

The second run can be made without changing either calorimeter or jacket water when the regular calorimetric thermometers, with twelve or more degrees range, are used. The bomb is refilled with charge of combustible and oxygen; and after checking the weight of the calorimeter water against losses when removing the bomb after the first run, the second run is conducted in like manner to the first. Only one or two minutes of stirring will be found necessary to establish complete temperature equilibrium; and after taking initial temperatures, the charge is at once ignited.

DETERMINATION OF HEAT OF COMBUSTION OF FUELS
AND OTHER COMBUSTIBLES

Heat of Combustion Calculations

For Fuel Oil

Sample No.

Date

Run No.

Wt. of Ign. dish and oil ----

Wt. of Ign. dish ----

Wt. of Sample ----

Wt. of Water 1950 gms.

Temp. readings-

1. 6. 11.

2. 7. 12.

3. 8. 13.

4. 9. 14.

5. 10. 15.

Temp. at Firing ---- $\div$ (Corr. Factor) =

Temp. at Max. ---- $\div$ (Corr. Factor) =

Rise in temp. corrected for errors in therm. ----

Rise per gm. of sample ----

The water equivalent of bomb, calorimeter can,
stirrer, etc. is 578.

Gram calories of fuel oil = $(1950 \div 578) \times$ rise in temp.
in $^{\circ}\text{C}$ per gm. of sample.

B.T.U. per pound of Fuel Oil = Gram calories per gm. $\times 1.8$.

Note: Whether running sulphur content or not the bomb
must be washed out with distilled water and dried
after using.

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SAMPLING BITUMINOUS MATERIALS

(ASTM Designation: D 140-52T)

Scope

1. These methods apply to the sampling of liquid, semisolid, or solid bituminous materials at the point of manufacture, and from bulk storage, tank cars, distributors, barrels, drums, or cakes.

Purpose

2. (a) Sampling is equally as important as the testing, and every precaution shall be taken to obtain samples that will show the true nature and condition of the materials they represent. Test results are valuable only when the tests are performed on a sample truly representative of the material under consideration.

(b) Samples may be taken for either of two purposes:

- 1.) To represent as nearly as possible an average of the bulk of the material sampled.
- 2.) To ascertain the maximum variation in characteristics which the material may possess.

In either case, the samples shall be obtained by methods hereinafter described.

Time and Place

3. (a) Whenever practicable, 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 shall be taken from the shipment immediately upon delivery.

Size of samples

4. The size of samples to be submitted for testing shall be as follows:

(a) Liquid Materials:

- 1.) For routine laboratory examination, 1 qt.
- 2.) From bulk storage, during loading or filling distributors, tank cars, or trucks, 1 gal.

SAMPLING BITUMINOUS MATERIALS

(ASTM Designation: D 140-52T)

(a) Liquid Materials: (Cont'd)

3.) From barrels or drums, 1 qt.

(b) Semisolid or Solid Materials:

1.) From barrels, drums or cakes, 2 to 3 lb.

2.) From crushed or powdered material in bulk or bags, 2 to 3 lb.

Containers

5. Containers for liquid bituminous materials shall be small-mouth cans with cork-lined screw caps, except for emulsions, in which case they shall be wide-mouth glass jars or bottles. Containers for semisolid and solid materials shall be friction-top cans.

Protection and Preservation of Samples

6. (a) Care shall be taken that the samples are not contaminated with dirt or 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.
- (c) Samples of emulsions shall be packaged in such a manner as to protect them from freezing.

SAMPLING AT PLACE OF MANUFACTURE

From Bulk Storage

7. (a) Liquid Materials and Materials Made Liquid by Heating - The inlet and outlet to the storage tank shall be closed and a 1-gal. 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, and when so taken, enough material shall be allowed to flow through the drain cocks and discarded to ensure a representative sample from the contents of the tank. Otherwise, samples may be taken by lowering into the material a suitable weighted bottle or can.

SAMPLING BITUMINOUS MATERIALS

(ASTM Designation: D 140-52T)

From Bulk Storage (Cont'd)

7. (a) The bottle or can shall be fitted with a stopper which shall 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 may be tested separately for consistency in order to detect stratification. They may then be combined, thoroughly mixed, and a one-gallon sample taken therefrom for other tests that may be required for determining average characteristics of the material.
- (b) Crushed or Powdered Materials in Bulk -
Solid bituminous materials in crushed fragments or powder shall be sampled in accordance with the Standard Method of Sampling Coke for Analysis (ASTM Designation: D 346). The gross sample shall be not less than 50-lb. from which shall be selected the 2- to 3-lb. sample for test.

From Barrels, Drums, Cakes, and Bags

8. (a) Liquid Materials -
Samples of liquid bituminous material from barrels or drums shall be secured by taking one quart of material from packages selected at random according to the cube root method described in Paragraph (b). 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 not possible to differentiate between the various batches or runs, each sample shall be examined separately.
- (b). Semisolid or Uncrushed 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 below. 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, the following table is given, showing the number of samples to be selected for shipments of various sizes:

SAMPLING BITUMINOUS MATERIALS

(ASTM Designation: D 140-52T)

From Barrels, Drums, Cakes, and Bags (Cont'd)

8. (b)

<u>Packages or Cakes in Shipment</u>			<u>Packages or Cakes Selected</u>
2	to	8	2
9	to	27	3
28	to	64	4
65	to	125	5
126	to	216	6
217	to	343	7
344	to	512	8
513	to	729	9
730	to	1000	10
1001	to	1331	11

Samples shall be taken from at least 3 in. below the surface and at least 3 in. 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 3/4 in. 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 1/4 lb. 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 one-gallon 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 one-gallon 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.

(c) Crushed or Powdered Materials -

Where the crushed or powdered material is in barrels, drums, or bags, a number of packages shall be selected at random as described in Paragraph (b). A sample at least 1 1/2 lb. in weight shall be selected from near the center of each container to yield a gross sample weighing not less than 50 lb., from which the 2- to 3-lb. sample for test shall be selected as prescribed in the Standard Method of Sampling Coke for Analysis, (ASTM Designation: D 346).

During Loading or Filling

9. When tank cars, distributors, barrels, or packages are being filled, samples may conveniently be taken from the pipe line through which the material is flowing, as described in Paragraphs (a) and (b) (Note).

SAMPLING BITUMINOUS MATERIALS

(ASTM Designation: D 140-52T)

During Loading or Filling (Cont'd)

9. (a) Preferred Procedure - Continuous Sampling, Applicable Only When Filling Line is Full and Under Pressure -
The sampling pipe shall be inserted into a rising section of the pipe line on the discharge side of the pump or in a completely filled line in which the material is flowing by gravity. The sampling pipe shall be not more than one-eighth the diameter of the pipe line and its opening should be turned to face the flow of the liquid. This pipe shall be provided with a plug cock and shall discharge into a sample receiver. The plug cock shall be so adjusted that there is a steady continuous flow of bituminous material through it and so regulated that the sample receiver will fill to the required amount in the same time that is required to make the entire loading. In the case of semisolid materials, the receiver shall be provided with a closed steam coil which shall keep the contents at a temperature just above the liquefying point. The sampling shall be so regulated that, for each 1000 gallons of material loaded, at least one 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 loading. At completion of the loading, the receiver shall be thoroughly agitated and a one-gallon sample taken therefrom.
- (b) Alternative Procedure -
If continuous sampling equipment is not available, or in case the material flows by gravity through pipe lines that are not completely filled, samples shall be obtained by taking not less than ten dipperfuls at the outlet at uniform intervals throughout the entire loading. In no case shall the sample total less than 4 gallons. At completion of the loading, the receiver shall be thoroughly agitated, and a one-gallon sample taken therefrom.

SAMPLING AT POINT OF DELIVERY

Tank Cars or Trucks

10. (a) A careful observation of the condition of the material in regard to the presence of foam, sediment, or free water on top or at the bottom of the material in the car shall be made immediately before sampling, and, if such extraneous materials are present in measurable quantities, their volume shall be estimated and samples of each shall be taken and properly labeled. If possible, the foreign material shall be removed and discarded before sampling.

SAMPLING BITUMINOUS MATERIALS

(ASTM Designation: D 140-52T)

Tank Cars or Trucks (Cont'd)

10. (b) Liquid bituminous materials shall, if practicable, be sampled before heating, but if heating is necessary, at no time shall the temperature exceed that required for satisfactory sampling. Semisolid or solid bituminous material shall be rendered fluid by heating.
- (c) Samples shall be taken by means of a weighted bottle or can, as described in Section 7 (a), from the top, middle, and bottom of the material in the car or from the unloading pipe line as described in Section 9 (a) or (b).

Distributors

11. The contents of distributors shall be thoroughly mixed by circulation of the contents with the pump (see Section 10 (b)). Samples shall then be taken by means of a weighted bottle or can, or from drain cocks, as described in Section 7 (a). One sample shall be sufficient.

Ships and Barges

12. If sampling is required before unloading, top, middle, and bottom samples should be taken by means of a weighted bottle or can, as described in Section 7 (a). If sampling is not required before unloading, or if an average sample is satisfactory to the recipient, it shall be taken while unloading as described in Section 9 (a) or (b). In case of separate tanks or partitioned holds, each compartment or tank should be sampled individually.

Barrels, Drums, Cakes, and Bags

13. Sample as described in Section 8.

Crushed or Powdered Material in Bulk

14. Sample as described in Section 7 (b).

Note: (Indicated on Page 4)

JRL/jeh
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The methods of sampling at place of manufacture presuppose that the vessels into which the material is to be placed for shipment are clean and free from any form of contamination. Should they not be, or should there be any question regarding their condition, the sampler shall make appropriate comments to accompany the sample, and may take additional samples of the loaded vessels before leaving the place of manufacture, in accordance with the methods of sampling prescribed in Sections 10 to 13 for sampling at point of delivery.

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INDEX - Laboratory Manual of Tests

BLISTER TESTS

Test No.

1. Roofing and Shingles
2. Insulating Siding
3. Strieter Blister Test
4. Flintkote Insulating Siding Blister Test

BLISTERING TEST FOR ROOFING & SHINGLES

The samples to be tested shall be approximately 4"x8".

The samples are soaked in water for 72 hours. At the end of this time, the samples are removed from the water and allowed to drain by suspending in 100° oven for 1/2 hour. This drying may be hastened by removing the excess of moisture by means of compressed air.

After the excess moisture is removed, the samples are placed in an oven heated to 176° F. for two hours, removed and inspected.

Results shall be graded as follows:

Traces - One or two very small blisters.

Slightly Blistered - A few small blisters.

Blistered- Surface covered with small blisters.

Badly Blistered- Surface covered with large blisters.

Any other variations than mentioned above should be noted.

This test has proven effective in detecting material that will blister under natural weathering.

Checked June 1952

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AP

BLISTERING TEST FOR INSULATING SIDING

Samples to be tested shall be approximately 4" x 8". Cut edges of the samples shall be given a heavy coat of quick setting or plastic cements and allowed to dry before testing.

The samples are soaked in water for 72 hours. At the end of this time, the samples are removed from the water and allowed to drain by standing on edge for about 1/2 hour. This drying may be hastened by removing the excess of moisture by means of compressed air.

After the excess moisture is removed, the samples are placed in an oven heated to 170° F. for two hours, removed and inspected.

Results shall be graded as follows:

Traces - One or two very small blisters.

Slightly Blistered - A few small blisters.

Blistered - Surface covered with small blisters.

Badly Blistered - Surface covered with large blisters.

Any other variations than mentioned above should be noted.

This test has proven effective in detecting material that will blister under natural weathering.

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STREITER BLISTER TEST

Object - To test prepared roofing for the tendency of that material to blister.

Apparatus:

Constant temperature oven capable of holding 175° F.

Pan for water.

Procedure:

Samples of the prepared roofing to be tested are cut to any convenient size. (We use a 5"x9" sample) holes are punched near the top and hooks or clips inserted. The samples are then hung in the 175° F. oven for two hours. At the end of the 2 hour period the samples are taken from the oven and quickly submerged in water at room temperature, left for ten minutes in the water, removed, dried on the surfaces with a damp cloth and replaced in 175° oven for a repetition of the cycle. After 3 complete wet and heat cycles the samples are left for 16 hours at the 175° F. and the whole is considered one cycle.

All samples are closely inspected at the end of each complete cycle.

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FLINTKOTE INSUL SIDING BLISTER TEST

Accelerated blistering test for brick siding based on Ford Company test.

A sample, six-inches wide, shall be cut across the full width of each master panel tested. Both long edges of this piece shall expose the unsaturated fiber of the panel.

Samples, so cut, shall be fully immersed in a water bath maintained at $160^{\circ}\text{F.} \pm 3^{\circ}\text{F.}$ and kept immersed for one hour. The specimens shall be arranged in a vertical position, approximately one inch apart with the top of the specimens one to two inches beneath the surface of the water. This is best done by means of a simple rack in which the specimens are placed. The specimens shall be prevented from floating out of position by a suitably weighted bar laid across their top edges. To aid in maintaining a constant and uniform temperature, the bath shall be provided with mechanical agitation.

At the determination of the one-hour immersion period, the samples shall be removed and their surface superficially dried by mopping with a paper towel. Samples shall be placed immediately, granule face up, on the shelf of an air recirculation oven maintained at $240^{\circ}\text{F.} \pm 3^{\circ}\text{F.}$ At the end of a two-hour period, the specimens shall be removed from this oven and allowed to cool, granule surface up.

When at approximately room temperature, the degree of blistering shall be observed and recorded. The following scale, based upon comparison with standard panels, may be used.

0	No blister
0/	Trace of blisters
1	Very slight blister
2	Moderate blisters
3	Heavy blisters
4	Excessive blisters

Weights of specimens taken before and after water soaking may be of interest.

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A.P.
8/19/54



INDEX - Laboratory Manual of Tests

CEMENT ASBESTOS PRODUCTS

Test No.

1. Strength Test of Asbestos - Cement Products
2. PBL Water Penetration Test - Cement-Asbestos Products
3. Blooming of Asbestos-Cement Products
4. Percentage Moisture in Cement - Asbestos
5. Water Absorption Test - Cement=Asbestos Products
6. Consistency Test for Lap Cement, Liquid Roof Coating, Cold Cement, and Plant Band Adhesive
7. Testing Consistency of Plastic Cement
8. Workability of Cements
9. Open Cup Flash Point for Lap Cement and Liquid Roof Coatings
10. Sliding of Cements
11. Specific Gravity and Weight per Gallon
12. Specific Gravity of Oils and Solvents (Hydrometer)

STRENGTH OF ASBESTOS-CEMENT PRODUCTS

To test the breaking strength of Cement-Asbestos Siding six (6) strips, 3 with the grain and 3 across the grain of the sample are cut with an Asbestos Siding cutter. These samples are $1\frac{1}{2}$ " x 9" in size.

The testing apparatus used at PBL is a Scott Tensil Strength Tester, which shows the breaking strength in pounds, and has a ratchet arrangement holding the needle at the maximum strain reached.

PBL has had a pair of frames made which fit in the clamps of the tester. The frame fitting the top clamp is a bar of $\frac{3}{8}$ " square steel with two triangular knife edge bearings welded to it seven inches apart, so the $1\frac{1}{2}$ " wide sample rests on knife edges exactly seven inches apart. The frame fitting the bottom clamp is a single knife edge applying pressure in the center of the seven inch span. The pointer on the dial registers pounds required on this single knife edge to break the strip of siding being tested.

This reading is the breaking strength and is used directly in figuring the Modulus of Rupture.

The formula for finding the Mod. for these dimensions is:

$$\text{Mod. } x = \frac{\text{Breaking strength } x 7}{\text{Caliper }^2}$$

The caliper is the average of 4 measurements taken along the line of fracture.

The embossed graining is not ground from the sample but the samples are tested just as they are made, giving a caliper slightly higher than that of the actual fracture.

The manually operated pulley applying pressure to the samples is turned at the rate of 80 rev. per min.

June 1952

PBL WATER PENETRATION TEST - CEMENT-ASBESTOS PRODUCTS

The sample to be tested is cut into 3 sections (in the case of siding, giving 3, 8"x12" pieces). A putty ring about $\frac{1}{2}$ " high is built-up on each piece, enclosing at least 60% of the surface.

The samples are then placed (ring side up) on dry papers over which penescope powder has been sprinkled.

The area within the ring is then filled with water and the time elapsing between the placing of the water and the appearance of the first spot about 1" in diameter in the penescope powder is the "water penetration time".

The average of the penetration times of the three parts will be taken as the penetration time of the sample.

Penescope powder is a powdered sugar and methyl violet mixture. 2% methyl violet, 98% sugar, (by weight) which is very sensitive to moisture.

June 1952

BLOOMING OF ASBESTOS - CEMENT PRODUCTS

A test to determine the tendency of Cement-Asbestos products to effloresce.

Samples of Cement-Asbestos products being tested for bloom are cut into $2\frac{1}{2}$ "x8" pieces. A hole is put in each sample about 1" from one end in the center of the panel. The samples are then placed in water to a depth of about 4 inches and left soaking for 16 hours (a pint cement can is satisfactory for each sample). At the end of the soaking period the samples are suspended above the water for four hours, the water removed and the samples left to dry in the air. (The samples should be suspended in a place relatively free from drafts and air currents).

When the samples are dry they are inspected visually and rated according to amount of bloom present. Occasional microscopic examinations should be made of bloomed samples to note type of bloom crystals.

Checked June 1952

PERCENTAGE MOISTURE IN CEMENT-ASBESTOS

A test to determine the amount of moisture present in Cement-Asbestos products.

Samples being tested are cut to a convenient size about 8"x12" weighed and placed in the 220°F. oven for 24 hours. At the end of this period the samples are taken from the oven and cooled to room temperature. When the samples are cooled they are again weighed.

$$\text{Percent Moisture} = \frac{\text{loss in weight}}{\text{weight of dried sample}} \times 100$$

Checked June 1952

WATER ABSORPTION - CEMENT-ASBESTOS PRODUCTS

Test to determine the tendency of Cement-Asbestos products to absorb moisture.

Samples being tested for this quality are dried by the method used in percent moisture test, or the samples measured in that test may be used. The dried sample is weighed, submerged in tap water at room temperature and left for 24 hours. At the end of this period the samples are removed, the surfaces dried with a damp cloth and weighed.

$$\text{Percent water absorption} = \frac{\text{gain in weight of sample}}{\text{weight of dried sample}} \times 100$$

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CONSISTENCY TEST FOR LAP CEMENT, LIQUID ROOF COATING,
COLD CEMENT, AND PLANT BAND ADHESIVE

Apparatus:

The apparatus for controlling the consistency is a special PBL Viscosimeter No. 2, similar in principle to the Saybolt Viscosimeter. It consists of a cylindrical vessel 3-3/4" diameter and 7" in height with a removable bottom for cleaning. Four metal lugs are secured on the inside, two 1" from the bottom and two 1" from the top. These serve to measure a constant volume of the material expelled through the orifice. A blueprint of the apparatus is available at PBL.

Materials with varying consistencies may be tested by means of the removable plugs with different sized orifices. The orifice is sealed with a tight fitting cork stopper. Use 3/4" orifice for Liquid Roof Coating, Lap Cement, Cold Cement, and Plant Band Adhesive.

Preparation of Sample:

A sample of at least 1200 cc. of material should be brought to a temperature of 90 - 215°F. by means of a water bath.

When used for control testing at FBC, the sample of material to be tested may be taken directly from the top of the mixing kettle after the proper mixing time.

Method of Testing:

The viscosimeter is filled to the level of the upper lugs with the material to be tested. The temperature is then recorded. The time of out-flow of the volume between the upper and lower lugs is determined by means of a stop watch. This reading should be compared with the value as plotted on the standard Time Temperature curves which give the allowable limits for approval for each type of material being tested.

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TESTING CONSISTENCY OF PLASTIC CEMENTApparatus:

1. A Schutte Consistency Tester, consisting of a Cylindrical tube 1-7/8" diameter and 10-1/8" long and a brass collar 5/8" in diameter and 1" in height which screws into the base.
2. Timer.
3. Amalgamated tin, or brass plate.
4. Thermometer

Preparation of Sample

The material to be tested is first introduced into the brass collar, care being taken that the collar is completely filled. The collar is then immersed in water at 77°F. for 10 minutes while standing on the amalgamated tin or brass plate.

Method of Test

Screw collar into tube while immersed in the water and set combined tube and collar down in the 77°F. water so that the water just covers the lower shoulder of the tube. Hold the piece of metal against the bottom of the collar and fill the tube with water at 77°F. Quickly remove the metal from the bottom of the collar, starting at the same time the timer does. The interval between the removing of the metal plate and the displacement of the cement from the collar is recorded as the "Shutte" of the material.

Checked June 1952

WORKABILITY OF CEMENTS

Object: To test the workability of cements at 77°F. and 10°F.

Procedure:

- a. At 77°F. spread a sample of the cement being tested with a spatula to a thickness of about one-eighth of an inch on a piece of saturated felt. The cement should spread easily without drawing or pulling.
- b. Apply a small amount of the cement about one-half inch thick on a piece of saturated felt and place in refrigerator for 30 minutes at 10°F. The cement should remain in a plastic form and spread easily at this temperature.

Checked June 1952

OPEN CUP FLASH POINT FOR LAP CEMENT AND LIQUID ROOF COATINGS

1. Definition: See open cup- Flash and Fire points.
2. Apparatus: Standard Cleveland open cup tester, suitable thermometer, stop watch, Bunsen burner as heating source.
3. Procedure: Clean the flash cup thoroughly so that there is nothing from previous determinations left in the cup. Suspend the thermometer in a vertical position and approximately half-way between the back and center of the cup.

Fill the cup with the material to be tested in such a manner that the top of the meniscus is exactly at the filling line (inscribed on the inside of the cup) Making sure that nothing is spilled on the cup above the filling line or on any part of the apparatus outside. Failure to observe this is a frequent cause of in-correct results.

Place the heating burner under the center of the cup and regulate the flame so that the temperature goes up at the uniform rate of about 3°F per minute. It is essential to constantly stir the material under test, thus maintaining an even temperature throughout the liquid.

Apply the test flame to the material being tested at each successive thermometer reading, which is to be made every 30 seconds. In doing this, observe the following instructions:

- a. Have the test flame approximately 1/8" long.
- b. Move it across the center of the cup in a straight line at a right angle to the diameter of the cup passing through the thermometer.
- c. Make the sweep across the cup in one second, keeping the lower side of the flame exactly level with the upper edge of the cup.

When the approximate flash is known, application of the test flame need not be made until the temperature is 50°F below the expected flash point.

Note the record as flash O.C. (open cup) the temperature read on the thermometer when a flash appears at any point on the surface of the material being tested. This flash must not be confused with a bluish halo which sometimes surrounds the test flame prior to the true flash.

Copied 10/22/45

Copied 8/11/54

Checked June 1952

SLIDING OF CEMENTS

A test to determine the tendency of a cement to slide when subjected to roof conditions.

A sample of the cement is spread with a converted 2 inch cement spreader (filed to leave a $1/8$ " deep band the width of the spreader).

Three types of surfaces are tried for each cement tested, by placing the band about one inch from the top of a 4" x 6" sample of saturated felt, 75# smooth surface roofing, and smooth steel. A piece of string is embedded along the center of the band exactly 2" down from the top of the sample.

The test pieces are then placed in a horizontal position for 1 hour in a well ventilated room at a temperature not lower than 59°F. not in the direct rays of the sun. Suspend the samples for 5 hours in an oven having a temperature of 140°F. The degree of sliding is directly measurable by measuring the distance between the top of the panel and the string, and subtracting the original measurement.

Checked June 1952

SPECIFIC GRAVITY AND WEIGHT PER GALLON

DIRECTIONS FOR USING WEIGHT PER GALLON CUP (132.0840 g.)

This cup may be used for determining the weight per gallon and specific gravities of asphalt cements, roof coatings, and paints.

Directions for Using:

Clean cup and lid thoroughly with carbon tetrachloride or with soap and water or both. Dry in an oven at 220°F. for one hour, place in a desiccator until it cools to room temperature, then weigh it on an analytical balance. Fill the cup to approximately 1/16" below the top making sure that no air bubbles are buried under the material. Then carefully place the lid on the cup and press down so all excess material is extruded through the small hole in the center of the lid. Remove all the extruded material and wipe off the lid with a rag, then weigh the filled cup and lid.

Calculations:

The cup holds exactly 41.66 ml. Therefore, the weight of the contents in grams divided by five, is the weight in pounds per gallon. The weight of the contents of the cup multiplied by 0.024 gives the specific gravity of the material. Calculations may be made at various temperatures, but unless otherwise specified the weight per gallon should be calculated at room temperature for asphalt cements and roof coating.

Note: This weight per gallon cup is a standard of the "Institute of Paint and Varnish Research, Henry A. Gardner Laboratory", 1500 Rhode Island Ave., N.W., Washington, D. C.

JRL/jeh
7/28/50

JRL/cal
8/11/54

Checked June 1952

SPECIFIC GRAVITY OF OILS AND SOLVENTS

For Oils and Solvents.

Place the material being tested in a hydrometer jar about 5 cm. in diameter and 35 cm. high, a 100 cc. graduate will answer the purpose. Suspend a thermometer so that the bulb is entirely immersed and bring the temperature of the whole to 68°F. $\pm 0.5^\circ$ as rapidly as possible. When temperature is at 68°F. insert hydrometer and take the reading when the hydrometer becomes constant.

A hydrometer should be selected which has the proper range for the material being tested. An accuracy of at least $1/2^\circ\text{B.}$ is expected. Readings may be reported in degrees Baume' or the ratio of density to water. When it is not convenient to make a determination at 68°F. a correction table may be used. (See Mark's hand book or Day)

Checked June 1952



INDEX - Laboratory Manual of Tests

FELT AND PAPER

Test No.

1. Xylol Test for Determining Speed of Saturation
2. Procedure for Determining Per Cent Saturation of Asphalt Saturated Felt
3. Standard Procedure for Determining Kerosene Value of Dry Felts and Papers
4. Mullen Test
5. The Elmendorf Tearing Test
6. Tensile Strength of Felt and Paper
7. Determination of Moisture in Felt
8. Determination of Pliability of Saturated Felt
9. Oil Penetration Felt
10. Determination of Entrapped Air
11. Hertzberg Stain Test for Felt Analysis (Microscopic)
12. Moisture Loss - Vapor Seal Tests

XYLOL TEST FOR DETERMINING SPEED OF SATURATION

Reagent: Water-white Xylol

Test Specimen: 15 mm. wide strips in the machine direction of the felt. Each strip is marked with pencil lines drawn horizontally across it at points exactly 1 cm. and 4 cm. from the lower end.

Procedure:

Clamp the strip in a vertical position by its upper end well above the surface of the Xylene. Quickly lower the clamp and strip until the surface of the liquid coincides with the 1 cm. mark and measure with the stop-watch the time in seconds required for the Xylene to raise from the 1 cm. mark to the 4 cm. mark.

Run separate tests on at least 5 strips and average the results.

Note: Small variations in the depth of dunking the dry felt will affect the result considerably.

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PROCEDURE FOR DETERMINING PERCENT SATURATION
OF ASPHALT SATURATED FELT

Object: This method covers only the determination of percent saturation on a dry felt basis of asphalt saturated felt.

Apparatus:

1. A 1000 cc. wide mouth Erlenmeyer Flask.
2. An Analytical Balance.
3. Three large size Weighing Bottles.
4. A Dessicator.
5. An extraction condenser coil.
6. An electric hot plate.
7. Oven at 220°F.

Test Specimen:

A sample, 2" x 4", is cut from the sheet under test and treated as follows: The reported results shall be the average of the results obtained by the desaturation of three samples from different parts of the sheet.

Procedure:

After cutting the test specimens from the saturated sheet, place them in the dessicator for one hour. Then, using the analytical balance, weigh the samples separately, and return to the dessicator. Place about 175 cc. of carbon tetrachloride in the Erlenmeyer flask, and place it on the hot plate. The weighed samples are suspended by paper clips from the condensing coil which forms the cover of the extraction flask. Water is circulated through the coil in sufficient quantity to condense the solvent and prevent its escape from the flask. The desaturation is continued for one-half hour after the drippings from the felt samples first show clear. Remove samples from flask and place in 220°F. oven for one-half hour. Place in dessicator to cool. When cool, weigh samples and compute the percentage of saturation by the following formula:

$$\% \text{ Saturation} = \frac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Dry Weight}} \times 100$$

Desaturate Coal Tar Felt by method given under A.S.T.M. D-1146-27.

Checked June 1952
JRL-FHC/jeh

STANDARD PROCEDURE FOR DETERMINING KEROSENE VALUE
OF DRY FELTS AND PAPERS

Object: To cover the determination of the theoretical maximum possible saturation on dry felts and papers.

Apparatus:

1. A 50 cc. Burette
2. A grain scale (1000 grain capacity)
3. A 220°F. Oven

Test Specimen:-

Three samples, 2" x 5", are cut from the dry felt sheets, and tests run on each sample separately. The results reported, however, are the average of these three samples.

Procedure:

The 2" x 5" test specimens are first placed in the 220°F. oven for a period of 20 minutes taking care not to compress, bend or crack these samples in any way. When the test is being conducted for control purposes, it may be necessary to reduce this 20-minute period somewhat, but this will not affect the results to any great extent.

When ready to run the test, remove the test specimens one at a time, and weigh as accurately as possible, yet fairly rapidly on the grain scale. Immediately upon reaching room temperature, each sample is saturated with kerosene from an ordinary 50 cc. burette as follows: Hold the sample with a pair of tongs or forceps in a position slightly inclined from the horizontal. Run the kerosene from the burette over the entire surface, finally retarding the flow to a slow dropping so that even distribution can be readily obtained. The end point is reached when the entire surface has an unbroken, glossy and distinctly wet appearance. At this point, the sample is turned so that its diagonal is vertical and allowed to drain for exactly 30 seconds. The drops falling from it, as well as any lost in the saturating operation, are counted and given a value of 0.03 cc. That part of a drop left suspended, when the 30-second period is reached may also be estimated. The total excess kerosene in the form of these drops is subtracted from the burette reading. The corrected burette reading is then used in the formula for kerosene value listed below:

$$\text{Kerosene Value} = \frac{1605 \times \text{cc. of Kerosene Used}}{\text{Weight in grains of sample (Bone Dry)}}$$

In order that the test may be standardized as much as possible, the kerosene used for saturating the dry felt should be of a water white variety with a specific gravity between .73 and .80.

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JRL-PHC/jeh

MULLEN TEST

OBJECT

To determine the bursting strength of dry and saturated felts and papers.

PROCEDURE

Obtain a 6" strip across the width of the roll being tested. Five readings shall be made on each side of the sheet alternately staggered across the sheet.

A standard Mullen Testing Machine shall be used, and run at a uniform rate of 120 revolutions per minute, which increases the pressure at a rate of three pounds per second. Average the readings obtained, and record as pounds per square inch.

*CAUTION: Care should be taken not to attempt to test a material which will not burst at the maximum reading of the tester. This would injure the instrument.

FHC/rp
4/13/61

THE ELMENDORF TEARING TEST

Description:

The Elmendorf Tester is a heavy pendulum mounted on ball bearings. The bob or Sector A carries a clamp N and a dial graduated from 0 to 100. On the same axis, a pointer is mounted which has a constant friction just sufficient to stop at the highest point reached by the swing of the sector.

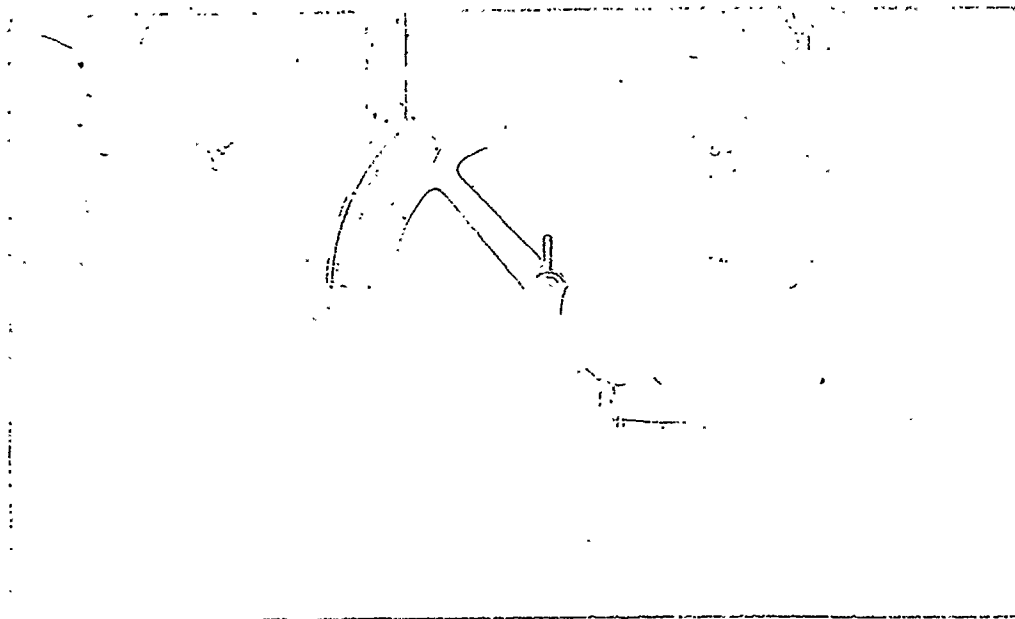
If the sector is raised 70 degrees from the vertical and allowed to rest against the stop, it will, when the stop is depressed, swing to a height of 70 degrees (less the small loss by friction which is compensated) on the opposite side and the pointer will rest at zero. This is in accordance with the laws of mechanics, since the work done in lifting the section 70 degrees to the left is spent in carrying it to the same height on the right.

If we now lift the sector and clamp several strips of paper in the jaws N, set the pointer and depress the cutter handle to start the tear and define its length, we are ready to make a test.

We now depress the stop and the sector will swing to the right but will not rise 70 degrees because part of the energy of the lifted sector has been spent in tearing the paper. The reading on the dial shows the amount of work done in tearing the paper.

If four sheets were torn, giving a certain value, eight sheets would give a value twice as great, or two sheets half as great.

The number of sheets used varies, with the strength of the material, tested, from 1 to 16. When values are to be reduced to grams for a common standard, the base number of sheets is 16, and the eight-sheet reading must be multiplied by 2 and the two-sheet reading by 8 to get the value in grams.



Procedure:

First make sure the instrument is level. Do this with the leveling screw C in the base D. When the instrument is level, the white mark E on the sector A will be opposite the end of the spring stop H when the spring stop is depressed, and the sector is free to swing.

To test for zero, push the sector A to the left until the spring stop H engages the right hand edge of the sector. Set the pointer K against the pointer stop L. This pointer stop is fastened to the base by screw M. Release the sector by depressing the spring stop H as far as it will go and hold it down until the sector has swung once in each direction. Release the spring stop to clamp the swing of the sector. Note the reading of the pointer K, if not at zero, adjust the position of the pointer stop L by means of screw M until the pointer on further trial comes to zero. The accuracy of the tests is the same as the accuracy of the zero test.

To test the Paper:

Prepare several samples ($2\frac{1}{2} \times 2\frac{1}{2}$) using the Elmendorf Test Cutter. Set sector A and pointer K as in zero test. Open both jaws of paper clamps N and insert 2, 4 or 8 sheets, seeing that all sheets reach way down into both jaws. Clamp both jaws tight and depress cutter lever O to low position and release.

Depress the spring stop H as far as it will go and hold it down until the sector has swung once in each direction. The sector will swing a distance indicated by the position of the pointer. If eight sheets are used, the reading should be multiplied by 2 to express the force in grams. If four sheets, multiply by 4, and if two, multiply by 8.

Take tear tests both with and across the grain, unless otherwise requested.

June 1952

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TENSILE STRENGTH OF FELT & PAPER

OBJECT

To determine the tensile strength of felt and paper.

PROCEDURE

Cut 10 strips of felt or paper, 1" x 6", with the fiber grain.

Both sets of test strips will be tested at 70°F. in a Scott tester or similar machine having a capacity between 100 and 150 lbs. The test strip shall be gripped 1 1/2" on each end, leaving 3" between the clamps, and the lower jaw shall travel at the rate of 12" per minute. If any strip breaks nearer than 1/2" to either clamp, the reading shall be disregarded and another strip tested in its place. The average of the 10 readings from the strips shall be taken as the breaking strength of the sample.

FHC/rp
4/13/61

DETERMINATION OF MOISTURE IN FELT

OBJECT

To determine the percentage of moisture in felt.

PROCEDURE

A sample of approximately 500 grains or 32.4 grams is taken from the roll about 9" out from the core. This sample is immediately weighed and heated in a constant temperature oven which is maintained between 220° and 225°F. for one hour. The sample is removed from the oven and quickly weighed.

The moisture content is calculated as follows:

$$\frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Wet Weight}} \times 100 = \text{Per Cent Moisture}$$

FHC/rp
4/13/61

DETERMINATION OF PLIABILITY OF SATURATED FELT

Object: To determine the pliability of saturated felt.

Procedure:

Cut five strips 1" wide and 6" long in the direction of the fiber grain, and immerse in water @ 77°F. for 10 to 15 minutes. Remove the strips from the water, and immediately bend each strip over a mandrel 1/16" in diameter through an arc 180° at a uniform rate in approximately 2 seconds. Dry thoroughly and examine.

If one or more of these five strips crack on this test, cut ten strips from another part of the roll, and repeat the test. If one or more of these strips crack, the material is reported as failing on the pliability test.

Test for pliability of saturated felt given under federal specification HH-F-191.

Checked June 1952
JRL-FHC/jeh

THE "OIL PENETRATION" FELT

Object: To obtain a gauge of the rate at which roofing felt may be expected to saturate. From a manufacturing standpoint, the saturating rate is of first importance as it is a controlling factor in machine speeds obtainable upon the saturator. The present test, considered in conjunction with the kerosene test, gives the information needed regarding the saturating qualities..

Apparatus:

No elaborate apparatus is required to carry out this test. A glass pipette viscosimeter and an accurate thermometer are the chief requirements in addition to the oil, containers, and stop watch. A pipette viscosimeter is used to check up the viscosity of the oil from time to time, and this may be easily made from an ordinary 60 cc. glass pipette. An accurate thermometer is needed to continually check up the temperature of the oil, as the viscosity changes appreciably with a few degrees difference in temperature.

Description of the Oil:

A clear light-bodied petroleum oil, of carefully controlled viscosity, is used for the testing; for control work it is often advisable to have several mixtures of oil at hand which may be used according to variations in room temperatures. The oil is a mixture of a clear light engine oil and of clear paraffin oil; the exact proportions vary according to the temperature at which the mixture is used. In order that the test may be standardized, we have determined the oil to be used at 72°F. to be one that has a Saybolt Universal Viscosity of 171.6 seconds when tested at 100°F. A mixture of about one part Red Engine Oil and five parts paraffin oil may be made up and used as a guide in obtaining the proper viscosity.

Description of the Test:

The test consists in floating a small sample of dry felt upon an oil bath of definitely controlled viscosity and noting the time in seconds required for the oil to strike through by capillary attraction and darken practically the entire uppermost surface. The test is carried out from both sides of the sheet and both results are recorded.

THE "OIL PENETRATION" FELT

Procedure:

The viscosity of the oil is determined with the control pipette, and if not correct, is adjusted either by changing the temperature or by changing the proportions of the oil mixture. When the correct viscosity has been obtained, a sample 2" x 2" is laid upon its surface, and the stop watch is started simultaneously. The sample is then continuously watched, and the time when the oil has struck up through the sheet and is judged to have darkened 90% of the uppermost surface is noted, and the elapsed time in seconds is recorded. A sample is first tested with the wire side up and then an adjacent sample is again tested with the felt side up, or the two may be carried out simultaneously. The number of seconds required for the oil to penetrate is called the Oil Penetration Test.

Notes Upon Test:

The pipette viscosimeter is easily made by drawing down the narrow lower portion of a 50 cc. glass pipette to a rounded hole of the proper diameter in a gas flame. These pipettes cannot be expected to read exactly the same, and each must be standardized. However, we find that an instrument which delivers water in 26 seconds, by taking the reading to the upper point in the restricted tip delivers the proper oil in very close to 60 seconds in the usual case.

In laboratory control work, it is far more convenient to have several pans of oil ready mixed, of slightly different composition, and in case one is not exactly correct, use another judging the same according to the temperature and viscosity; it is even often found convenient to change from one oil to another as the room temperatures vary during the day. Otherwise, a temperature control apparatus must be provided or else the composition of the mixture continually changed.

It is considered that the sheet is thoroughly saturated with oil when the end point reading is taken. 90% of the area covered is taken as the end point due to the fact that felt very often contains particles of unbeaten stock which tends to resist the penetration of the oil and to produce a light spot on the surface; in order to get representative figures upon the real penetrating quality of the sheet it is advisable to ignore these spots, and we have therefore set 90% of the area covered as the most reasonable end point.

THE "OIL PENETRATION" FELT

Notes Upon Test:

Interpretation of Results:

When this test is carried out as described above, tests upon usable felt vary from 2 seconds up to about 30 seconds according to the weight and character of the felt; felt is occasionally found that will run up to 40, 50, and even 60 seconds in test, but such felt is almost sure to be slow saturating and unsatisfactory in practice.

There is a certain difference in the test from the two sides of the sheet which may in certain cases run up to 25% difference. We find that the test is usually the quickest with the felt side uppermost and are in the habit of placing most weight upon the test from this side.

There is a considerable difference in tests obtained upon bone dry samples and upon those in air dry condition. We are in the habit of reporting tests in our control work direct from the machine which, of course, is an intermediate condition. The condition of the sample, whether bone dry or air dry, should be reported along with the result of the test.

Checked June 1952
JRL/FHC-jeh

DETERMINATION OF ENTRAPPED AIR

The voids in asphalt-saturated felts, prepared roofing and any asphalt-filler mixtures are filled with air. A satisfactory method for determining this air content has been developed as follows:

Procedure:

1. Fill a 500 cc. graduate cylinder with carbon tetrachloride.
2. Place the asphalt sample in the reservoir A (Fig. IV) of a tar acid funnel. The copper wire gauze is then placed at the bottom to prevent the asphalt sample from falling from the funnel. The tar acid funnel shown in Fig. IV has the constricted part of the bulb or reservoir cut off. This is convenient for adding the sample, but is not necessary.
3. Immerse the funnel (reservoir A first) with open stop-cock into the carbon tetrachloride contained in the 500 cc. graduated cylinder. When the funnel is full and the tetrachloride has passed the stop-cock, the latter is quickly closed.
4. The 500 cc. cylinder containing the tar acid funnel is then placed in a water bath and kept at 50°C. A glass jar is suitable for this purpose. The air evolved rises to the top and displaces the carbon tetrachloride.
5. After all the air from the sample has been evolved (bubbling having ceased), the carbon tetrachloride in the 500 cc. cylinder is cooled to 20°C. and the volume of air in the burette is then read. This volume divided by the volume of the sample gives the volume percentage of the entrapped air or voids. The volume of the sample of roofing is readily determined from the linear measure of the sample in centimeters. In case of asphalt the specific gravity must be known.

The method as described required no special apparatus, as suitable equipment can be found in any laboratory. Furthermore, the method is rapid and a complete determination can be made in less than one hour. Owing to the simplicity of the method and the inexpensiveness of the apparatus, it is possible for every laboratory to be equipped with several units, thus making testing on the large scale possible.

DETERMINATION OF ENTRAPPED AIR

The following table illustrates some of the results obtained with this method:

<u>Sample</u>	<u>Volume of Sample cc.</u>	<u>Volume of Air Evolved cc.</u>	<u>Volume Percentage of Air</u>
Experimental Roofing	28	5.2	18
" "	28	3.8	13
Commercial Roofing	26.4	3.6	13
" "	28.0	0.8	3
" "	28.0	0.6	2
Straight Asphalt	24	0.5	2
Asphalt plus Filler	24	1.5	6

The above results show that the percentage of voids varies for a given sample.

Checked June 1952
JRL/jeh

HERTZBERG STAIN TEST FOR FELT ANALYSIS (MICROSCOPIC)

1. Make a saturated $ZnCl_2$ solution @ $70^{\circ}F$.
2. Make a second solution of .25 gr I_2 and 5.25 gr KI in 12.5 c.c. distilled water.
3. Mix 25 c.c. of first solution in total solution #2. Put in a narrow glass cylinder and allow to stand until clear.
4. Stain sample with finished solution and observe under microscope.

Chemical wood stains blue to purple.

Cotton and linen " red.

Ground Wood " yellow.

Bagging " yellow brown to red.

Wool does not change color. That is if wool fibre had been dyed it retains that color.

Bagging can be distinguished from ground wood by the shape of fibre. Bagging fibres are long and thin while ground wood is short with square ends.

June 1952

MOISTURE LOSS - VAPOR SEAL TESTS

A test to measure the amount of moisture passing through a known surface of paper in a known time. A test method to test vapor seal papers.

Into an 8 ounce seamless can is placed about 50 gm. of water with wicks rising from the water and reaching almost to the top of the can. A round hole 2" in diameter is cut in the cover of this can and over the opening, by means of a ring and soldered bolts, is fastened and sealed a piece of the paper being tested. Sufficient asphalt is used when sealing the paper so that when the cover is slid onto the can, it too is sealed.

The test specimen so prepared was placed in a desiccator containing dry calcium chloride which produces a humidity approximating zero percent. After a short time (2 hours) in the desiccator, the sample is removed and weighed. This figure is assumed to be the original weight. The sample is weighed daily for 15 days, the loss of weight each day and for the whole time is recorded and reported as loss per day, or for 15 days for the area of paper under test.

In order to approximate the humidity specified by the Forest Products Laboratory we modify this test somewhat by placing a saturated solution of calcium chloride in the desiccator. Such a solution at 76°F. gives a humidity of 31%.

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INDEX - Laboratory Manual of Tests

NAILS AND WIRE

Test No.

1. Zinc Coating on Nails
2. Priest Copper Sulphate Test for Roofing Nails
3. Nails - Tests for Brittleness of Heads
4. Test of Tin Plating on Nails
5. Shingle Tying Wire

ZINC COATING ON NAILSStrip TestControl TestTest:

The sample, consisting of ten nails selected at random from the quartered sample should be weighed and then immersed in 100 c.c. of hydrochloric acid (specific gravity 1.20) to which has been added 5 cubic centimeters of an antimony chloride prepared by dissolving 20 grams of antimony trioxide or 32g Sb-Cl₃ in 100 cc. of hydrochloric acid (specific gravity 1.20), until the zinc coating has completely dissolved. The solution of the average zinc coating rarely requires more than 15 to 20 seconds and the end point is evidenced by an almost total cessation of bubbling. The same 100 cc. of hydrochloric acid can be used for at least five samples. Five cubic centimeters of the antimony chloride solution, however, should be added for each sample on account of the antimony being removed from solution by the iron.

The samples are washed and scrubbed under, running water, dried with a towel and laid in a warm place for a few seconds. The samples are weighed and the loss in weight figured to ounces of coating per square foot of nail area by the following formulas:

Nail Area:

D - diameter of nail head in inches.

L - Average length of shank in inches.

T - Thickness of shank in inches.

$$\text{Area of nail in sq. in.} = \left(\frac{11}{7} \times \frac{D^2}{4} \right) + \left(\frac{22}{7} \times LT \right)$$

Oz. of Zinc/ sq. ft.

W (L) - Wt. in grains lost by dissolving nails in caustic solution.

A - Area of nail sq. in. $\frac{W(L)}{AN} \times .33 = \text{oz. of zinc per sq. ft.}$

N - No. of nails used in analysis.

Gauges	=	10	=	.1350"
		10½	=	.127"
		11	=	.1205"
		11½	=	.113"
		12	=	.1055"

June 1952

PRIEST COPPER SULPHATE TEST FOR ROOFING NAILSAPPARATUS

The apparatus to consist of a beaker, a chemical all glass thermometer with Fahrenheit scale, a watch with a second hand, a standard solution of copper sulphate, cheesecloth, and a solvent such as carbon tetrachloride or chloroform. It is desirable that running water be available.

TEST SOLUTION

The copper sulphate solution to be used is made of chemically pure copper sulphate crystals and distilled water. It is prepared by dissolving a sufficient amount of the crystals in hot water (contained in a copper bottle or tank) to insure solution of slightly above the specific gravity of 1.186 after the solution has been cooled to 65°F. (18.3°C). Any free acid in the solution is neutralized by the addition of oxide of copper (about 1 g of oxide of copper per liter of solution). The solution is diluted with distilled water to a specific gravity of 1.136 ($\pm$ 0.003) at 65 F (18.3 C) and then filtered.

REACTION OF TEST SOLUTION

The zinc coating of the test sample is dissolved by the standard copper sulphate solution, and copper is precipitated and deposited on the test piece in its place. So long as the zinc remains on a given portion of the test sample, the copper which is deposited on that portion is spongy and can be easily removed by washing and wiping. When the zinc has been entirely dissolved at a given portion of the sample so as to expose the steel or iron, the copper which is deposited on that portion (usually of a bright copper color) ordinarily adheres to the steel or iron of the sample sufficiently to prevent its removal by washing and wiping.

METHOD

A sample nail is thoroughly cleaned with carbon tetrachloride or chloroform to remove grease, paraffin, etc. The solvent should be allowed to evaporate before the test sample is immersed in the copper sulphate.

A glass vessel having a diameter approximately twice the diameter of the nailhead is filled with the standard solution to a sufficient depth to immerse the nail to be tested. The temperature of the solution shall be 65 F plus or minus 2F. The nail is suspended in the center of the standard solution and allowed to remain 60 sec. During the period of immersion the nail is not removed nor the solution stirred. At the end of 60 sec. the nail is removed from the vessel, rinsed in clear water, lightly wiped with wet cheesecloth until loosely adhering deposits of copper are removed, and dried with clean cloth to remove surface water. The condition of the immersed surface is then observed to see whether or not all or any part of the portion which was immersed has a bright deposit of closely adhering metallic copper.

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If the nails being tested show a copper deposit on the steel wire shank after four or fewer dips, they will be considered to have failed.

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BB-5/19/42

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CL-7/30/54

June 1952

NAILS

TEST FOR BRITTLENESS OF HEADS

Test:

Ten representative nails are selected for this test. In testing, the nail is placed with the edge of the head resting on a metal or hard surface. The other edge is struck with blows of a hammer until the head either bends flat with the shank or breaks off.

The head should not be so soft that it bends readily without at least three firm blows.

If, when the head is bent so that it touches the shank, the head breaks away from the shank, the nail is considered as having failed to pass the test.

June 1952

TEST OF TIN PLATING ON NAILS

A test to determine quickly the amount of tin plating on tin plated nails.

This test is a method of roughly determining whether or not the plating on a nail is satisfactory. The test is most satisfactory when run against a standard.

Ten samples of nails are taken at random from the shipment being tested and ten from a standard lot. The two groups are divided into 10 pairs of one standard and one unknown each and each pair dipped into 10% nitric acid for one minute, removed, dried and examined and if no base stock is noted the cycle is repeated until the unknown nail shows base stock due to removal of plating, or until 7 complete dip and dry cycles have been completed.

If at the end of seven cycles the nail does not show loss of plating or more discoloration than the standard nail it is considered satisfactory.

June 1952

SHINGLE TYING WIRE

MATERIAL

A soft basic galvanized wire used in tying bundles of roofing.

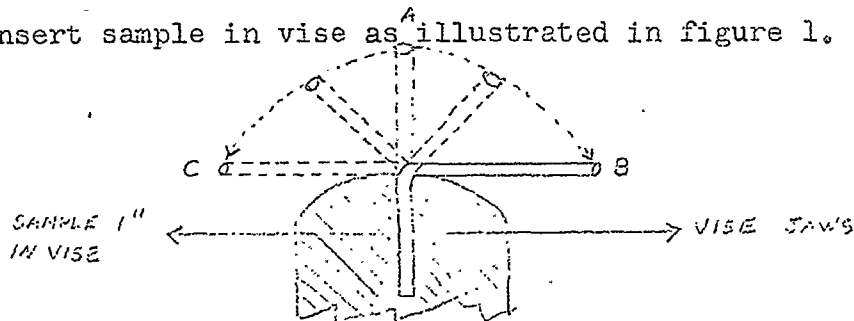
SPECIFICATIONS (17 gauge only)

1. Caliper: .050" minimum
2. Bending Strength: Minimum - 7 bends
Maximum - 17 bends
3. Twisting Strength: Minimum - 30 twists
4. Stiffness Tests - deflection from horizontal
Maximum deflection - 30°
Maximum release deflection - 15°
5. Linear ft./lb.: Minimum 1250'

DESCRIPTION OF TEST

Bending:

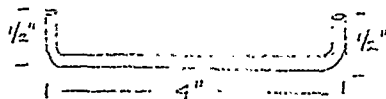
1. Select three pieces 4" long and test individually as follows:
2. Insert sample in vise as illustrated in figure 1.



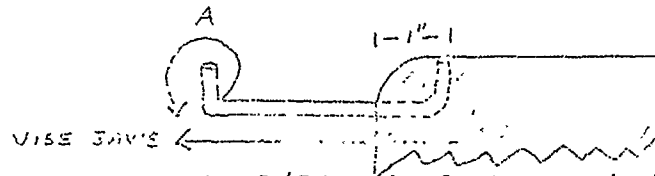
3. Bend as illustrated above - A to B, B to C, C to B, and repeat until wire breaks.
4. Record the number of bends; B to C is one bend, and C to B one bend. Take the average number of bends before breaking of the three samples.

Twisting Strength:

1. Select three pieces 5" long and bend as illustrated in figure 2.



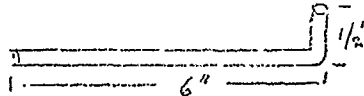
2. Insert sample in vise for a distance of 1" as illustrated in figure 3.



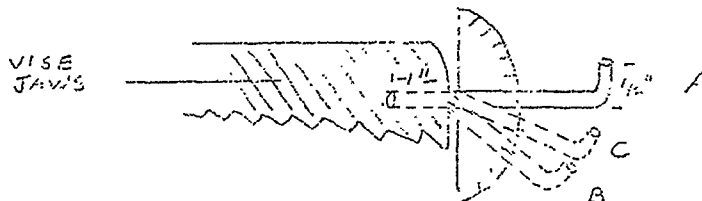
3. Clasp with pliers the 1/2" end of wire marked "A" and twist in clockwise direction until the wire breaks.
4. Record the number of complete turns before breaking. Average the result from the test of three samples.

STIFFNESS

1. Select 3 pieces 6 1/2" long and bend as illustrated in figure 4.



2. Insert sample in vise for a distance of 1" as illustrated in figure 5.



3. Add a 150 gram weight on end of wire and let hang for 30 seconds. Take reading on the protractor which subtracted from 90° will be recorded as deflection from horizontal. Remove weight and let wire return. Take this reading, which subtracted from 90° will be the release deflection.
4. Average the result from the test of three samples.

FHClark/rp
1/24/62

SECRET

INDEX - Laboratory Manual of Tests

PIGMENTS

Test No.

1. Standard Test Procedure for Mass Tone and Tinting Strength
2. Laboratory Procedure for Pigment Tests
3. Firing Test of Pigments

STANDARD TEST PROCEDURE FOR MASS TONE AND
TINTING STRENGTH OF PIGMENTS

Apparatus:

The apparatus shall consist of the following:

- (a) Regular Laboratory balance, sensitive to 1 mg.
- (b) A Brown burette, which consists of a small burette with reservoir.
- (c) Glass mullers shall be standard type as specified by ASTM with 2-3/4 to 3 inch diameter grinding face.
- (d) Rubbing surface shall be a ground glass plate.
- (e) 2 Spatulas having a 3 inch blade.
- (f) Microscope Slides.
- (g) Counterpoised watch glasses.

Materials:

- (a) A standard color (sample submitted by the Mfg.).
- (b) Reduction pastes (zinc oxide base).
- (c) A quantity of Lindol (as supplied by PFL).

Mass Tone Procedure:

Carefully weigh out on separate watch glasses 2 gr. of standard color and 2 gr. of the sample being tested. Transfer these samples to the ground glass plate adding the same measured amount of lindol to each. Enough lindol is added to work up a paste with the spatula. When all the dry pigment is worked up by means of the spatula, rub up the paste with the glass muller. This is done by giving the paste 100 rubs and picking it up with the spatula after every 25 rubs. Use a stroke 3 or 4 inches wide and 12 to 15 inches in length. In counting the rubs, one stroke up and one stroke back is considered one complete rub.

When the mulling is completed place a daub of each paste in juxtaposition on a microscope slide and smooth them off. Then compare the color by looking at the pastes on the top and not thru the glass. The mass color of the standard and the sample must be substantially the same in order for the sample to pass this test.

Tinting Strength Procedure:

Weigh as before on the counterpoised watch glasses the standard color and the sample to be tested. This time weigh out only .2 gram of each. Thoroughly wet up these pigments on the ground glass plate with lindol

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using the spatulas. When this is done add equal amounts of the reduction paste (usually 2 grams) to each of the pastes and again mix with the spatulas until no streaking is noticed. Then rub each paste up with the mullers giving each 100 rubs and pick it up with the spatulas after each 25 rubs. When mulling is completed place a daub of each paste in juxtaposition on a microscope slide and smooth them off. Judge the color immediately. If the sample is not as dark as the standard sample then it does not pass this test.

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LABORATORY PROCEDURE FOR PIGMENT TESTS1. Fineness:

50 grams of the pigment are washed with water through a 200 mesh screen. Very slight pressure is used in the washing, no attempt being made to force particles through the screen with high water pressure. The screen with the residue is dried in an oven at 220 deg. F. After the residue is completely dried, it is weighed on an analytical balance and the percentage residue calculated.

2. Oil Absorption:

The sample to be tested should be first placed in a suitable container (a small wide mouth bottle is adaptable) and well shaken, so as to assure the elimination of any packed particles or lumps. This is especially important if the pigment has been packed in barrels or otherwise kept in a manner that is liable to cause it to pack together. If excessively moist, the sample should then be air dried at room temperature.

Twenty (20) grams of the pigment are placed in a 50 cc beaker. Aged linseed oil is then run in from a burette, either drop by drop or with successive small additions. Either of these methods will give check results. If the drop method is used, the rate of flow at the start should be about one drop per second. If the small addition method is used, quantities of 1/2 cc. should be added. As the absorption of the oil increases, the rate of flow or quantity of the additions is decreased. The oil is run in so as to strike the dry pigment in the center. As the oil comes in contact with the pigment, the dry pigment that has not been wet should be lifted from the outer edge and placed over the oil so as to bring all the oil surface in contact with the pigment. This is best accomplished either by lifting it up on the spatula and dumping it off on the oil, or by very lightly throwing it over the oil.

When the pigment particles become wet with the oil, they tend to coalesce and form small lumps of paste. These lumps should be kept distributed throughout the mass. This is done by lightly stirring the mixture of these paste lumps and dry pigments with a finger and wrist motion, being careful not to use pressure in the mixing, as this factor will tend to vary the results. If the drop method of oil addition is used, this stirring should be carried on continuously with the operation of bringing the dry pigment in contact with the oil surface. If the successive addition method is used, this stirring should be done after each addition. As the absorption of oil progresses these lumps of paste, by taking up more pigment and matting together, form larger lumps which when stirred around form balls. When this point is reached the rate and quantity of the oil addition should be very much decreased and only a few drops added at a time. In adding the oil at this point, it should be allowed to strike on these lumps and not on the remaining dry pigment. After each oil addition, these lumps are lightly stirred around so as to bring the oily surface into contact with the remaining dry pigment.

With further oil addition and stirring these balls will, with most pigments, join together and form one large lump with but little dry pigment remaining. With certain pigments, however, the lumps or balls of paste do not join together but remain separate and continue to collect more pigment. At this point, which is close to the end point, the oil is added very carefully, one or two drops at a time. With many pigments one drop is all that is necessary to establish the end point. At this stage, the oil should be allowed to strike on the surface of the lump or lumps. They should then be worked around to pick up more of the pigment. When all of the remaining dry pigment has been picked up and wet, the end point is reached. This is indicated by the paste lump becoming much softer and easier to spread with the spatula. When stirred around it smears the sides and bottom of the glass. With most pigments only one or two drops of oil is required to change the relatively hard dry lumps which do not smear the walls of the container into a fairly soft paste which will smear when stirred around. If when the end point is reached, the paste lumps are broken down or spread with the spatula, it will be noted that the oil is uniformly distributed throughout the mass and no dry pigment remains.

Different pigments have certain individual characteristics in absorbing oil. These must be given consideration, and it is sometimes necessary to slightly modify the procedure in order to conform with these peculiarities. For example, some pigments absorb the oil readily and in a uniform manner, while others take up the oil slowly. The paste lumps of some are smooth and free from hard agglomerates and are easily kept uniformly mixed. Others tend to form hard lumps. With such it is difficult to keep the mass uniform. When the end point is reached with certain pigments, the paste lumps will be quite firm. Other pigments will be firm up to the end point and then suddenly become very soft; in some instances of semi-paste consistency. These differences are largely due to certain surface tension relations of the pigment and oil. For ordinary purposes, a variation of 0.2 cc. is sufficiently close. A variation of more than this amount on check tests can only be considered as an error.

Summing up the test should be carried out under the following conditions:

- a. The pigment must not be moist.
- b. It should be well shaken before making the test.
- c. It should not be of a temperature lower than 70 deg. or higher than 100 deg. Fahrenheit.
- d. The oil should be well settled and of a standard acid value.
- e. The oil should not be added too fast nor should it be added slower than necessary.
- f. The mixture should only be stirred enough to keep the mass uniform and bring the pigment in contact with the oil.
- g. Excessive stirring tends to lower the result.

- h. The stirring should be done lightly and care taken not to use pressure or endeavor to mix the pigment and oil by this means.
- i. When nearing the end point, the oil should be added slowly and in small amounts (one or two drops at a time). (Circular #85, Paint Manufacturers' Assn.) This test is of value in determining the following:
 1. Relative amount of surface in the pigment mass.
 2. State of sub-division of pigment particles.
 3. Comparative specific surface of various pigments.
 4. Variation in fineness of particle sub-divisions of various lots of the same pigment.

Gardner Method-Paint Manufacturers' Assn.

3. Color & Tinting Strength of Slate Flour:

Weigh accurately two grams of the sample and standard slates and two grams of zinc oxide. Add enough aged linseed oil to form a stiff paste with both the sample and standard using the same amount in both cases. Both pastes are thoroughly mixed on a glass plate using considerable pressure to break up all pigment agglomerates. Care should be taken that both samples are mixed an equal amount, using the same pressure for both. Transfer portions of each paste to a glass plate, quite close together, and then draw a spatula across both samples so as to make them meet in a line and examine for strength of color and tint.

June 1952
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FIRING TEST OF PIGMENTS

A test to determine the appearance and color of a pigment when fired under conditions similar to actual production. Used to test pre-shipment and protested samples of pigments.

The sample being tested is mixed with clay and silicate in the same proportions used on the granules. Enough water is added to make a paste of a very thin "Milky" consistency. The mixture is then ground with a spatula in circular motions on a smooth glass plate until smooth and free from lumps. The paste is then spread in a thin even film on a piece of double thick window glass 5"x2" in size. The prepared slide is then air dried by placing in a horizontal position on a bench away from current of air at a temperature of 65°F. for one hour or until the surface is dried. The samples are then placed in an oven at 220°F. for 10 minutes to completely desiccate the film.

At the end of this period the sample is placed on a piece of cast iron 1 inch thick and slightly larger than the size of the slide, and the iron plate and glass slide with pigment under test are placed in a "Muffle" furnace which has previously been brought to the desired temperature, which is in the usual instance that temperature at which the pigment will be fired when applying it to the granules. (1000°F.)

After 10 minutes in the furnace the sample and base plate are removed and placed on a bench to cool slowly. The iron plate causing the glass slide to cool slowly avoiding cracking and curling of the glass. When cool the slide is examined and compared with a slide made with the standard pigment similarly handled and fired with the test pigment.

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AP - 7/26/54



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ROCK, GRANULES, SURFACINGS, ASBESTOS, AND DUST

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3. Procedure for Washing Natural Colored Slate
4. Procedure for Matching Colored Granules
5. Embedding of Granules
6. Wet Embedding of Granules
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9. Oil Absorption Test on Granules
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- 22. Kerosene Absorption of Asbestos Fibre
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SCREEN TESTS FOR TALC, ASBESTOS, AND DUST

Use: A test to determine the grading by particle size of surfacings, fillers, and asbestos.

Apparatus:

1. Ro-Tap Shaker
2. Tyler Standard 8-inch Screens
3. Riffle - for dividing sample
4. Gram Scales

Procedure:

Take sample being tested and pour it through the riffle, pour the portion of the sample landing in one riffle pan through again. Weigh 100 grams from pan containing approximately one-quarter of original sample of slate, soapstone or talc; 25 grams of asbestos and place in top screen. Replace cover and put screens with pan on bottom in the Ro-Tap shaker and start shaker. Shake for 5 minutes (granules, #36 M.Tal (Soapstone), #37 M.Tal (Talc) 3 minutes (asbestos), 20 minutes (#154 Soapstone).

When shaken for the prescribed period of time, weigh material retained on each screen. Record as percent retained on each screen.

Checked June 1952 - JRL/rp

SIEVE ANALYSIS OF ROOFING GRANULES1. SCOPE

This method is intended for the sieve analysis of granular mineral surfacing materials, such as crushed slate, stone, etc., used on the weather surface of asphalt roofing and shingles.

Where this procedure is adopted, the following instructions shall apply.

2. APPARATUS

- a. A set of consecutive sieves conforming with ASTM Designation E-11, 8" in diameter. These shall include the following:

<u>Sieve Designation</u>	<u>Sieve Opening</u> (microns)
No. 8	2380
No. 12	1630
No. 16	1190
No. 20	840
No. 30	590
No. 40	420
No. 50	297

- b. A pan with a solid bottom, to fit at the bottom of the stack of sieves, to catch all material passing through the finest sieve.
- c. Sieve Shaker: A mechanically operated sieve shaker which automatically imparts to the set of sieves a rotary motion and tapping action of uniform speed. A device known as a Ro-Tap machine is satisfactory.
- d. Scales: Sensitive to 0.1 gram.

3. SAMPLING

A gross sample shall be reduced by hand-quartering to not less than 100 grams. This shall be weighed within an accuracy of $\pm$ 0.1 gram.

4. PROCEDURE

- a. The sieves shall be assembled in the same consecutive order as listed above and the solid pan shall be added below the bottom sieve. The weighed test sample shall be placed on the top sieve and this sieve closed with a cover. The sieve assembly shall then be securely fastened in the Ro-Tap shaking device, and the shaker operated for a period of five minutes.

4. PROCEDURE (Cont'd)

- b. The portion of the samples retained on each of the sieves and on the pan shall be carefully removed and weighed to an accuracy of not less than 0.1 gram.

5. REPRODUCIBILITY

Duplicate determinations by the same operator, using the same sieves, shall check within 1% of the total weight of the sample, for the portion of the sample retained on each sieve and on the pan.

6. REFERENCE

ASTM D-451.

2/1/54

PROCEDURE FOR WASHING NATURAL COLORED SLATE

Method of preparing samples of natural colored slate granules for color matching and other tests.

Procedure:

Take approximately 200 grams of the sample being prepared, and place in a 400 cc. beaker; cover with carbon tetrachloride, bring to a boil on a hot plate, and boil for three minutes, decant the liquid and repeat twice. Dry granules in air. This process will remove all oil from the granules.

When carbon tetrachloride has dried, place the granules in an 8-ounce bottle, and fill the bottle with water; put on cover and shake gently for ten seconds by repeatedly inverting the bottle. Decant off the water, and fill with clean water from the top; repeat until ten washings in the bottle have been made. Spread the wet granules on paper and dry in oven at 200°F.

If the granules have not been oiled, only the second part of the process need be performed.

Checked June 1952
LWW/jeh

PROCEDURE FOR MATCHING COLORED GRANULES

Object: To keep a constant check on colored granules produced by Bird & Son and to control colors of natural slate granules purchased outside of Bird & Son by matching all colors against standard color samples.

Procedure:

A sample, approximately 200 grams in weight, is taken from the sample being tested and subjected to the standard procedure for washing granules, using both the carbon tetrachloride and water if the original sample was oiled, and only the water if the sample was dry. When the sample is thoroughly dried, it is screened to approximately the same grading as the standard sample and compared for color directly against the standard. Unoiled granule samples directly from FBK do not need washing.

The color comparison is carried out by placing about 10 grams of both the standard and the test sample on a piece of saturated felt, the surface of both piles being flattened with some flat object (the bottom of a 4-ounce ointment tin) to give a somewhat roughened circle composed of half standard and half test sample granules, both having the same type of surface with the line of demarkation between the two samples forming a diameter of the circle. By rotating the sample and getting light reflected from it from all angles, any difference in color between the two segments of the circle can be noted.

Care must be taken to match a deoiled and washed sample against a deoiled and washed standard, and a sample which has been subjected only to the water washing to a standard which has been similarly treated.

Checked June 1952
LWW/jeh

EMBEDDING OF GRANULES.

Use: To assist in control of slate embedding in the manufacture of slate surfaced roofing products.

Procedure:

1. Take samples every half hour, from center of cooling rack.
 - a. Flat shingles - 6" to 10" from each edge of sheet.
(Back and front of rack).
 - b. Slate surfaced rolls. One sample from center of sheet.
 - c. Thick Butt Shingles - one sample from center of each overlay lane, one sample from each outside base lane.
2. Cut sample to $8\frac{1}{4}" \times 1\frac{3}{4}"$.
3. Knock off all loose slate, particularly at edges of sample.
4. Weigh sample to the nearest half grain and record the weight in column headed "weight before".
5. Run scrub test which consists of 50 complete cycles of the brush on either the Funkhouser or the 3M Tester.
6. Knock off loose granules, and dust and weigh again, recording under "weight after".
7. Difference between these two weights is recorded under "Loss".
8. In addition to the above, the product, color and date should also be noted on form provided.

The brush on the control tester should be checked daily by the routine tester and checked against a standard brush once a week.

Operating Limits

#11 Granules (FBK)	2.0 - 4.0 grains loss
#11 Natural Slate	3.0 - 5.0 grains loss
#9 Granules	3.0 - 5.0 grains loss

Checked June 1952
LFW/jeh

WET EMBEDDING OF GRANULES

Samples being tested for adhesion of granules while wet should be machine made to get the most satisfactory results.

Procedure:

Samples of roofing being tested are cut to required size and weighed, care being taken to wipe all loose granules from surface and edges.

Samples are then immersed in water at room temperature and left for 15 hours. At the end of this period, the samples are removed from the bath and placed in the tester tray which has previously been half filled with water at room temperature.

The test is then run in the standard manner having a rubbing time of 50 complete cycles while under water. The sample is then dried to constant weight at 100°F. and the loss recorded as follows: Wet Embedding Loss.

Checked June 1952
LWW/jeh

DETERMINATION OF OPACITY OF ROOFING GRANULES
TO ULTRA VIOLET LIGHT

Apparatus:

1. Dark room with safe-light, Wratten Series 1.
2. Opaque lamp housing with means of directing rays through filter.
3. G. E. Mercury Vapor sunlight bulb, type S1 and transformer.
4. Corning Glass Filter #597, 3" x 3".
5. Eastman Process Plates, 5" x 7".
6. Timer, accurate to 1 sec.
7. Photographic developing equipment and chemicals as specified in literature accompanying plates.
8. Opaque template 5" x 6 21/32" x .075" $\pm$.025", in which has been cut four equally spaced round holes, 2" in diam. Smaller holes, 3/16" in diam. - cut beside larger holes - serve as means of identification, (0, 1, 2, 3 small holes arranged in order). Template must be perfectly flat, and may be of any rigid material, such as sheet brass or aluminum.
9. Analytical balance, sensitive to 0.01 g.
10. Tyler testing sieves, 10 mesh and 14 mesh.
11. Opaque mask, with 2" diam. hole for measuring light transmission through developed plate.
12. Weston Master Exposure Meter.
13. Source of white light for measuring density of developed plate.
14. 1-ounce glass bottles with screw tops.

Procedure:

1. Source of Ultra Violet Light

The source of ultra violet light is suspended in a dark room six feet above the location selected for the exposure of the photographic plate. The entire illumination of the ultra violet lamp is designed to pass through the filter and reach the plate in substantially parallel rays.

DETERMINATION OF OPACITY OF ROOFING GRANULES
TO ULTRA VIOLET LIGHT

Procedure: (Cont'd)

2. Preparation of Granules

The granules are screened so that a portion passes a 10-mesh sieve and is retained on a 14-mesh sieve. This portion is washed and dried and 2.3 grams are weighed out on the analytical balance and stored in a 1-ounce bottle until ready for use. A granule of known safe opacity, and a granule of a known poor opacity are prepared in similar fashion to serve as standards.

3. Preparation of Photographic Plate for Exposure

The plate is prepared for exposure in the dark room using the safe-light for illumination. The protecting slide is removed from the plate holder and the template is placed directly on the glass plate. Each of the four 2" holes in the template is designed to hold one 2.3 gram sample of granules. The granules are placed directly on that part of the plate exposed by the hole. The granules are then spread out evenly by any means that is practical without scratching the emulsion. A small piece of stiff paper works satisfactorily to manipulate the granules into position. Care must be taken that the template is lying perfectly flat on the photographic plate. Otherwise, some of the granules will slip between the template and the plate, giving unsatisfactory results.

4. Exposure of the Photographic plate

The prepared plate is now placed in horizontal position directly below the source of ultra violet light. The ultra violet light is directed on the plate for 2 minutes and then turned off. The template is then removed, the granules shaken off the plate, and the protective slide replaced. The plate is then developed by ordinary photographic methods. All photographic plates must be used before the expiration date of the emulsion, and developed in identical manner, according to directions prescribed by the manufacturer of the plates.

5. Reading of Developed Negative-visual method

The developed plate will now appear totally clear in the portions unaffected by light. The four circular areas representing the 2" holes in the template will appear black, speckled with lighter areas, which are the images of the granules. Opaque granules will appear clear, with a slight halo, as no light will have affected the area beneath them other than a small amount reflected obliquely from adjacent granules to points just under the edges of each granule.

DETERMINATION OF OPACITY OF ROOFING GRANULES
TO ULTRA VIOLET LIGHT

Procedure:

5. (Cont'd)

Less opaque granules will be represented by the degree to which the plate has been affected by the light. Pure quartz granules, being practically transparent, will have nearly invisible images on the developed plate.

Once a granule of known safe opacity has been established, a visual examination of the plate will be adequate for an on-the-spot determination of opacity. For matter of record, however, a quantitative measurement should be made by means of the Weston Exposure Meter.

6. Reading of Developed Negative-quantitative Method

A quantitative determination of the opacity of a type of granule may be determined by means of the Exposure Meter. The source of white light is directed through the 2" hole in the mask upon the exposure meter. The developed plate is held against the mask so that the 2" diameter exposed area coincides with the hole in the mask. The scale reading is recorded in Weston units for each type of granule tested.

The Weston meter is calibrated so that the units are scaled in direct proportion to the amount of light affecting it. Therefore, the measurement of the opacity may be stated in per cent of a know satisfactory granule by dividing the figures obtained for the unknown granule by that obtained for the known.

Superseded May 5, 1953
LWW/jeh

PIGMENT FIXATION - ARTIFICIALLY COLORED GRANULES

Object: To determine the solubility of the pigmented coating on a granule in boiling water.

Procedure:

Twenty-five grams of the granules retained on a 65-mesh screen, previously washed in petroleum naphtha and dried, are weighed into a 250 cc. Erlenmeyer flask with ground-glass neck to connect with reflux condenser; and 100 cc. of filtered tap water is added. The flask is then put on a hot plate under reflux and boiled for four hours.

A gooch crucible is prepared, dried, and weighed, and the water in the flask containing the test granules is, after stirring vigorously by rotating the beaker, decanted through the gooch. The granules are then washed twice and this water decanted through the gooch.

The crucible is then dried and weighed and the gain in weight recorded as "color loss". The amount of material recovered from 25 grams shall not be more than .050 grams.

Reflux at 20 drops/min.

Checked June 1952
LWW/jeh

OIL ABSORPTION TEST ON GRANULES*

Porous granules or granules with a soft or porous coating show tendencies to blister asphalt roofing and to become stained. These properties, and the resistance to these tendencies, can be measured with fairly good accuracy by the amount of slate oil or the like, which the granules will absorb, according to the following procedure.

Reagent:

Slate oil containing an oil soluble dye, Dupont Oil Red will give satisfactory results as a dye. (Various other oil-soluble dyes, especially red or bright colored dyes, can be used.) Make a paste by adding a small amount of slate oil to 10 grams of dye. Put the paste in one liter of slate oil, mix thoroughly and let stand over night. After most of the residue has settled, filter off the clear colored solution which is now ready for use.

The residue can be used again when making more dyed slate oil by mixing in with the new paste.

Procedure:

Weigh out 88 g. of thru 14 on 20 mesh granules (viz. through "14-mesh" screen and retained on "20-mesh" screen) when testing the No. 11 grade or 44 g. of thru 8 on 10 and 44 g. of thru 10 on 14 when testing the No. 9 grade, and transfer to a 500 ml. Erlenmeyer flask. In testing raw granules be sure they are dry and reasonably free of dust. Add the colored oil solution a few drops at a time, shaking vigorously after each addition. Test for saturation by placing a piece of filter paper (Whatman #4) over the mouth of the flask and tipping so that the granules rest against the paper for 5 seconds. The formation of a definite stain on the paper indicates the end point which is fairly sharp. The number of ml. of oil used is multiplied by 20 and reported as the "oil absorption". The figure is the number of pounds of oil required to saturate one ton of granules. With a little experience, results should check within .05 ml. or 1.0 lb. per ton.

*Minnesota Mining Procedure
From U. S. Pat. 2,417,058

Copied January 1956
ink/rp

SULPHUR DIOXIDE TEST

Object: A test of synthetically colored granules to ascertain their reaction when in an oxidizing atmosphere.

The granules submitted to the test shall have been applied to roofing, preferably on a commercial roofing machine.

Procedure:

Samples for this test shall be cut approximately 1" x 6" in size. The test is a modification of the standard accelerated weathering cycle and is as follows:

	Rain	Light
*SO ₂	10:15-11:30	11:45-8:00

Periodic rain with consequent high humidity during long light exposure 2 days a week.

*For exposure to sulphur dioxide, the test samples are moistened and placed in a one-liter wide mouth Erlenmeyer flask fitted with a two-hole rubber stopper. Sulphur dioxide is introduced into the flask from the usual type of generator or supply tanks, by means of a glass tube extending to the bottom of the flask. A glass tube from top of flask to a water seal permits the air in the flask to be forced out and replaced with sulphur dioxide. The excess SO₂ is collected in a 600 ml. beaker containing water. The flow of SO₂ and the moving of the samples from the Erlenmeyer flask occurs when the water is saturated with SO₂.

This test shows the permanency of color on the granules. Sulphur dioxide in the presence of water is an oxidizing agent, and pigments which change in color due to oxidation are particularly susceptible to this test. Sulphur dioxide is, of course, present in all industrial and residential atmospheres.

Granules which show no change after 3 cycles are considered to be permanent.

Checked June 1952
JRL-ARA/jeh

ALKALINITY OF SYNTHETICALLY COLORED GRANULES

Object: A test to determine the alkalinity of granules.

Procedure:

The filtrate from the pigment fixation test is titrated with .025N hydrochloric acid using phenolphthalein as an indicator.

Calculate and report as percent Na₂ O.

Calculations:

$$\% \text{ Na}_2 \text{ O} = 100 \times \frac{\text{cc. of .025N. H Cl} \times 0.000775 \text{ (grams Na}_2 \text{ O equivalent)}}{25 \text{ (grams of granules used in test)}}$$

(to 1 cc. .025N H Cl)

Checked June 1952
JRL/jeh

HEAT TEST - COLORED GRANULES

Object: A test to determine the result of heat on synthetically colored granules.

Procedure:

About 5 grams of granules which have been previously washed in petroleum naphtha to remove possible oil or wax, are placed in a porcelain crucible without cover and heated over a bunsen burner at a dull red heat for approximately 10 minutes.

From time to time during the ignition, examine the sample for charring, evolution of acrid fumes, the presence of oily distillates or organic decomposition products.

The granules after ignition are cooled and then agitated with boiling water in order to ascertain whether the pigment is incorporated in a glaze or only attached imperfectly as a result of the removal of an organic binder.

- a. Synthetically colored granules which lose their pigment or respond unfavorably in the manner indicated are classed as "painted" or dyed", and are considered of inferior quality. Fading of the color and loss of surface covering due to the well known destructive effect of the weather on paints, granules of this type may be expected to last from one to two years.
- b. Synthetically colored granules which suffer no substantial loss of color and respond favorably to this test are classed as: Ceramic, silicated or having an inorganic binder.

Granules of this class are considered as of satisfactory quality, provided they also react favorably when subjected to the other tests.

Checked June 1952
LWW/jeh

SOOT RETENTION BY ROOFING GRANULES1. SCOPE

This procedure is applicable to roofing granules of all types, but is suitable only for comparison within a laboratory. Where this procedure is adopted, the following instructions shall apply.

2. APPARATUS

- a. 8 oz. round wide mouth glass jar, cork stoppered.
- b. Ro-Tap machine.
- c. ASTM Standard sieves as follows:

<u>Designation</u>	<u>Sieve Opening</u> (microns)
No. 325	.44
No. 100	149
No. 20	840
No. 12	1680

- d. Buchner Funnel to serve as a water spray.
- e. Tumbler.

3. MATERIALS

- a. At least 10 grams of a suitable soot. It can be prepared as follows:

39% - graphite #0574.*

58% - R-600 Slate Flour**, which sieves as follows: Passing No. 325 mesh - 98% minimum.

3% - #140 Carbon Black***

- b. At least 50 grams of granules to be tested.

4. PROCEDURE

- a. Screen the granules to be tested using Nos. 12 and 20 sieves. Place 50 grams of the material retained on the No. 20 sieve in the wide mouth bottle with 10 grams of the soot, shake by hand for 10 minutes, rotating so that the dust and granules become thoroughly mixed.
- b. Transfer the dust and granules to a 20 mesh screen and shake in the Ro-Tap machine for 10 minutes to separate the dust from the granules.

4. PROCEDURE (Cont'd)

- c. Place about 25 grams of the dusted granules on a No. 100 sieve, and locate it 6" below an inverted Buchner Funnel, set up to serve as a water spray. Adjust the water pressure so that the flow is approximately 5 gallons per minute, and wash the granules for 5 minutes while playing the spray uniformly over them. Dry the granules.

5. OBSERVATIONS AND REPORT

Compare the dried granules with a sample of original granules and report the color change as one of the following:

- a. no change
- b. slightly darker
- c. darker
- d. very much darker

*Made by Joseph Dixon Crucible Co., 167 Wayne St., Jersey City 3,
New Jersey

**This material can be obtained from the Central Commercial Co.,
332 South Madison Avenue, Chicago 4, Illinois

***Produced by Binney & Smith Co., 41 East 42nd Street,
New York, N. Y.

2/1/54

QUANTITY OF OIL ON GRANULES

A control test measuring the amount of oil put on roofing granules during commercial processing.

The sample of granules procured for this test should be representative of the lot being tested and should come from below the surface which may have dried somewhat and not be representative.

A 50 gm. sample is taken representative of the larger sample and weighed into a 250 cc. beaker and filled with carbon tetrachloride, allowed to stand for one hour at 100°F., removed the carbon tetrachloride which has not evaporated, filter through a weighed qualitative filter paper and washed with a stream of carbon tetrachloride and the beaker rinsed clean and poured into the filter. The oil free granules are then dried in an oven held at 220°F. and weighed. The loss of weight is regarded as the amount of oil and the figures are converted to the amount on a ton, and reported as amount of oil per ton.

June 1952

PERCENT MOISTURE - RAW AND COLORED GRANULES

A test to determine the amount of moisture on granules.

The sample being tested is to be a representative sample taken from stock.

Procedure:

A 4 ounce seamless ointment tin is tared and about 100 gms. of the sample weighed into the tared tin. The tin and granules are then placed in a constant temperature oven holding a temperature about 230°F.. The sample is left at this temperature for 5 hours, removed and cooled in a desiccator. When cool the sample is carefully weighed.

$$\% \text{ moisture} = \frac{\text{loss in weight of granules}}{\text{weight of moisture free granules}} \times 100$$

June 1952

TESTING OF ROCK BASES FOR GRANULES

Use: Tests to which rocks are submitted to ascertain their suitability as a base for colored granules.

In order for a base rock to be satisfactory for colored granules, it should have a reasonably constant color before and after firing, be free from objectionable amounts of impurities, and have a satisfactory hardness index.

Procedure:

1. Hardness: The rock being tested is compared by scratching the surface with a series of minerals of known hardness ranging from Talc to Diamond. To be satisfactory for use as colored granules, the rock should test between 5.0 and 7.0. These numbers are represented by 5.0, Apatite; 6.0, Microcline; 7.0, Quartz.

(A Cenco-Ward series of testing standards used)

2. Color: The color of the rock should at all times match a standard sample. Rock which shows any marked change in color during the firing process is particularly undesirable, as it makes for poor color control on the finished granules.
3. Heating: All granules should be heated to about 1,000°F. and watched carefully for "popping" or fracturing. White and light colored granules should be watched particularly in this respect, and also for any tendency to turn gray.

4. Chemical Tests:

- a. Iron - Heat to boiling 2 gm. of granules (P-14, R-28) with 5 cc. of conc. H Cl. Dilute with 20 cc. water.
 - I. To 10 cc. of solution, add 3 drops of 0.1% Potassium Ferricyanide solution. Blue color denotes the presence of Iron in the ferrous state.
 - II. To 10 cc. of solution, add 3 drops of 0.1% Potassium Ferrocyanide solution. Blue color denotes the presence of Iron in the ferric state.
- b. Iron - Heat a quantity of granules for about 15 minutes in a crucible at 1400-1600°F. Compare color after cooling with the color of the original granules. Considerable reddening or darkening will indicate considerable iron. Light color on roasting is not usually objectionable.

Procedure: (Cont'd)

4. c. Iron - To a 10 cc. portion of a solution like (a), add NH_4OH until distinctly basic. A flocculent precipitate of greenish or reddish brown will indicate iron. Dark greenish may be chromium.
- d. Carbonate - Observe any effervescence on addition of acid to granules or dust. Much effervescence indicates a probable objectionable amount of carbonate.
- e. Sulphide - Odor noticeable on addition of acid to granules.
- f. Calcium - Neutralize about 10 cc. of solution (a) with NH_4OH , filter and add 3 drops of 10% ammonium oxylate. A fine white precipitate shows the presence of calcium. Calcium oxylate precipitated.
- g. Magnesium - Filter solution from (f), and add a few drops of sodium phosphate, warm solution if necessary. A precipitate will show the presence of magnesium.
- h. Sulphate, Chloride, etc., tests may be made by extracting roasted granules with hot water and testing with the proper reagents, e. g.

Ba Cl_2 for SO_3

Ag NO_3 for Cl_2

BB-4/29/40
Rev. June 1952
LWW/jeh

ALKALINITY TEST FOR BASE GRANULES

Twenty-five grams of unwashed raw granules passing 10 mesh and retained on 14 mesh are placed in a small beaker containing 100 cc of distilled water, previously heated to 100°F. The beaker and contents are then placed in an oven at 100°F., and held at that temperature for 30 minutes. At the end of that time, remove from oven, add distilled water to make up to original volume, filter and take 75 cc of filtrate for sample.

Titrate sample with 0.025 Normal Hydrochloric Acid using methyl orange as indicator.

Alkalinity number or milli-equivalents of alkali are calculated as follows:-

$\frac{4}{3} \times \text{cc of acid (required to neutralize)} \times 0.025 = \text{Alkalinity No.}$

A satisfactory granule should not exceed 0.020 and a lower alkalinity number is to be preferred.

JRH/jp
8/20/46

JRH/cl
7/19/54

June 1952

OIL ABSORPTION OF FBW DUST FOR FF
Adapted from ASTM Test D281-31

Apparatus: Balance
Tared Watch Glass
Glass Plate
Raw Linseed Oil, clear, acid value of from 1 to 3
Graduated burette
Steel spatula

Procedure:

Weigh exactly 1g., or any multiple thereof, of the air dried dust, and place on the glass plate. Record the level of the linseed oil in the burette. Add the linseed oil drop by drop to the dust, thoroughly incorporating the oil with the dust by rubbing up with a sharp edged steel spatula after the addition of each drop. The test is completed when exactly enough oil has been incorporated with the dust to produce a very stiff, putty-like paste, which does not break or separate. Record the new level of linseed oil in the burette.

Calculation:

Calculate the volume of oil used by the difference in the burette readings. Convert to weight by multiplying by the specific gravity of the oil. From the weights of the oil and dust used in the test, calculate the number of pounds of oil required to exactly "wet" 100 lb. of pigment.

LWW/jeh
8/21/50

LWW/cal
7/15/52

June 1952

DENSITY OF MINERAL FILLERS BY THE PYKNOMETER METHOD

Apparatus: Hubbard-Carmick Pyknometer or Specific Gravity Bottle.

Procedure:

1. Weigh the Pyknometer.
2. Weigh the Pyknometer plus Sample.
3. Weigh the Pyknometer plus Distilled water @ 60°F.

Weight of Pyknometer plus Sample minus Weight of Pyknometer
 = Weight of the Sample

Weight of Pyknometer plus Water minus Weight of Pyknometer
 = Weight of Water @ 60°F.

$\frac{\text{Weight of Sample}}{\text{Weight of Water @ 60°F.}} = \text{Relative Density.}$

Relative Density x 62.3 = Weight per Cu. Ft. or Actual Den.

Maximum Density = 48 lbs. per cu. ft.

Note in filling the bottle with Slate Dust the Slate Dust must not be compressed or packed in. Pyknometer should be tapped slightly with spatula or pencil to settle dust. Fill even with top of Pyknometer and then carefully fit in glass stopper, do not force stopper on, but allow the Slate Dust, displaced by stopper, to overflow through hole in center. The overflow or excess can then be wiped off.

In filling the bottle with water at 60°F. the Pyknometer should be filled and then immersed in distilled water @ 60°F. for 15 minutes. When the Pyknometer is taken from the water wipe off the top immediately and then dry off the rest of the Pyknometer. Do not wipe off any water which comes up through hole in stopper thereafter as this is result of expansion due to heat from hand. Weigh Pyknometer as quickly as possible because the evaporation of water will give an inaccurate result.

June 1952

PROCEDURE FOR MAKING DUST CONCENTRATION SURVEYPROCEDURE USING SMITH GREENBURG IMPINGER APPARATUS

All the glass impinger nozzles and sample bottles should be thoroughly cleaned before any sampling can be done. When all the glass equipment has been thoroughly cleaned, the sample bottles should be filled to approximately the 75 cc. mark with distilled water, or in cold weather a mixture of distilled water and alcohol. The distilled water is obtained by redistilling water furnished by FBE and using glass throughout the laboratory distilling apparatus. In order that this redistilled water may be reasonably free from dust particles a small amount of potassium permanganate and aluminium hydroxide should be added to the water before it is redistilled. To obtain a sample of the dusty air the suction pump is set up at a point adjacent to the location where the sample is to be taken and connected to the outlet of the impinger bottle by means of the white rubber suction hose. In setting up the impinger bottle it is important that the impinger nozzle should be near the center of the bottle and not farther away from the bottom than the indicator line which is approximately $1/4$ " from the bottom of the impinger bottle. Additional care must be taken in setting up the impinger bottle that no dust can fall into the open end of the impinger nozzle during sampling. This is best done by locating the bottle in such a manner that no dust can fall off of rafters or flooring immediately above the bottle. If this is not possible, some means should be provided to protect the bottle from such a mishap. Before starting up the suction pump for obtaining the dust sample, be sure and connect up the U tube which measures the pressure difference across the impinger nozzle. When everything is in readiness for sampling, the suction pump motor is turned on and immediately the column of mercury in the U tube is adjusted by means of the bleeding valve to a point where one cubic foot of air is being sampled per minute. See calibration marks on U-Tube. The length of the sampling period may vary considerably but is usually between 10 and 20 minutes. There is no need to sample an atmosphere longer than is necessary to cloud up the water in the sample bottle. When the sample has been obtained, the suction pump is shut off and the total time of sampling recorded. The impinger nozzle is next removed from the impinger bottle and washed off so that any dusty water adhering to it will become part of the sample. The bottle is then stoppered and labeled with the total time of sample, location and any other data necessary for identification of the sample.

DUST COUNTING OF SMITH GREENBURG IMPINGER SAMPLE

When the sample bottles are brought to the laboratory, each sample is measured carefully and the volume recorded. The samples are then stirred and 10 cc. of the sample fluid is diluted to a given quantity. It is desirable to dilute the sample sufficiently to limit the count to below 100 particles per quarter field under the microscope. Usually the dilution of five or ten times is sufficient for this. The water used for diluting the sample is, of course, the dust-free redistilled water used in the original sample. Before preparing the counting cells the microscope is prepared by placing the Whipple disc in the 10 power eye-piece of the Bausch & Lomb laboratory microscope and the 10 power objective rotated to position. The tube length of the microscope is then adjusted so that one side of the entire ruled area of the Whipple

disc is equivalent to one millimeter on the stage of the microscope. A stage micrometer is used for this calibrating. When the microscope has been calibrated, a clean Hatch counting cell is filled with distilled water from a sample bottle that has been carried in the sample case during the dust sampling procedure but into which no sample of dust has been introduced. This will give us a blank count which can be subtracted from the actual count of the various samples. This blank sample count is obtained by counting five quarter fields and averaging the results. Next the actual samples are counted by counting five quarter fields on two different cells prepared from each of the diluted samples. In counting these samples and blank it is necessary to rack down the microscope condenser and to have the condenser iris open wide so that light conditions do not change and affect the results. A sub-stage lamp is also used to help eliminate light intensity variances. If the counts are made immediately after the cells are prepared, it is often necessary to focus the microscope up and down in order to bring in all the visible particles. Using the Hatch cell if five minutes is allowed for the particles to settle, it will be found that most of the visible particles under light field illumination will have settled to the bottom of the cell and little or no focusing of the microscope will be necessary. The dust counts obtained by the counting of samples in the Hatch cell are reported as number of particles per cubic foot of air samples. The following formula is used to obtain this calculation:

Formula #1

$$\text{Count/cu.ft.} = \frac{4 A D S}{.00025 T}$$

Where A = Average count per quarter field
Minus the blank count.

D = Dilution factor.

S = Volume of Sample in CC.

T = Time in minutes

DUST SAMPLING WITH THE BAUSCH & LOMB DUST COUNTER

This apparatus is a portable dust sampling equipment and contains besides the sampling apparatus a microscope for counting the samples immediately after sampling. To operate the sampler, the three winged nuts are loosened and the bottom chamber removed from the equipment. A circular sample slide is then inserted in place in such a way as to fit the slide locating screw head into the notch cut into the glass sample slide. The sample slide carries a sand blasted circle on which one can mark the date or place of the sampling. The bottom dust chamber is then placed back in position, being sure to fit the locating pin in the locating hole and tighten down the winged nuts as evenly as possible but not with unusual force. After placing the sample slide into this and securely fastening the bottom of dust chamber, the cover to the sample tube is removed and a piece of slightly moistened blotting paper is introduced into the tube, taking care not to block off the opening of the aperture to the atomizer bulb. The instrument is

then ready to take twelve consecutive samples but before doing so it is desirable to inspect the cleanliness of the slide and count the number of particles already in the field. This amount is then deducted from the final dust count. When a sample is to be taken, the cover of the dust chamber is removed and the rubber atomizer bulb is squeezed two or three times in order to bring a new air sample into the tube. In order to obtain a specimen of this air sample, it is necessary to make a quick backward pull of the piston handle. In heavy dust concentration it is desirable to reduce the number of particles impinged on the sample slide and, therefore, the piston is slowly moved out to a notch at approximately the half way position. The piston is then withdrawn the remaining distance with a quick backward pull as previously. The number of the sample being taken at any particular time is the number appearing in that window marked "dust". In order to view this sample it is necessary to rotate the slide so that the number will appear in the window marked "Microscope". The dust ribbon of the sample taken can then be examined by placing the small sub-stage lamp under the condenser of the microscope and the dust ribbon will appear as an illuminated streak across the field of the microscope. The eyepiece of the microscope is provided with a counting graticule similar to a Whipple disc. Each square of this graticule measures 30 microns so that two rows of squares measure 60 microns, or exactly 1/100 of the total length of the dust ribbon. All the particles should be counted that are located within the boundary line of this double row of squares which appear on the counting graticule. Counts should be made at four different positions along the ribbon of dust and the counts then averaged to get an average count across the ribbon. In order to convert this count to particles per cubic foot it is necessary to point off one place if a full stroke of the plunger was used in obtaining the sample. For instance, if 24 particles are counted within the boundary line, it means that there are 2.4 million particles per cubic foot of air in the air sampled. If, however, only half of the pump capacity has been used, the counter's result will, of course, have to be multiplied by two after pointing off. The two lines parallel to the rows of squares in the counting graticule are 5 microns away from the outside edge of the squares and provide a means for estimating particle size. Particles appearing in this space should not be counted in on the sample.

June 1952
ap

BLOOMING CABINET - DIRECTIONS FOR USE

To test granules for tendency to bloom:

- Granules:
1. Weigh 25 gm. granules into 9 cm. Petri dish and tap edge of dish until granules are evenly distributed over area of dish.
 2. Place 7 cc distilled water on granules without disturbing even arrangement of granules in dish. See that granules are uniformly wet.
 3. Place dish with wet granules in refrigerator at 0°F. or lower and allow to freeze. Dish should be level.

Blooming Cabinet:

1. Be sure drip pan under refrigerator coils is empty.
2. Turn thermostat to lowest point until the temperature of the cold side is below 0°F. Leave thermostat at lowest point so that refrigerator unit operates continuously throughout test. When test is completed, turn thermostat to 35° adjustment.
3. When temperature on the cold side is below 0°F, turn on blower and intermittent heater. Heater thermostat control should be at 50°. Allow to operate until Relative Humidity indicator is at minimum point and begins to fluctuate through standard cycle as heaters turn off and on.
4. Place Petri dish with frozen granules in cabinet below stream of air issuing from slot in pipe in upper compartment. Place where air blast on granules will be at a maximum.

This is with the near edge of the dish about 1 1/2" from near edge of the air discharge pipe. Leave until all water is evaporated. This should be about 2 1/2 to 3 hrs. in the case of ordinary granules. With granules specially treated, the time may be longer, depending on the type of treatment.

Evaluation of amount of bloom:- The amount of bloom is an estimated figure expressed in per cent based on the amount of white bloom appearing as contrasted with a totally white granule.

Precautions

- I. Granules: 1. When granules are being frozen, they should not be left more than a few hours in the refrigerator, or a portion of the original 7 cc water will evaporate.

BLOOMING CABINET - DIRECTIONS FOR USE

-2-

Precautions (continued)

2. After the test is completed, if the weather is excessively warm and humid, the dishes should be put in a dessicator at the blooming cabinet and allowed to remain until their temperature is the same as the surrounding air. Otherwise, condensation of moisture on the cooler granules will cause the bloom to disappear.

II. Blooming cabinet:A. Refrigerator

1. Drip pan should be empty before starting test.
2. Refrigerator thermostat should be set at 35° to 40° while tests are not being run. This will keep coils defrosted but will turn refrigerator over often enough to keep it from losing compression. It should not be disconnected for any period of time.
3. If the motor on the refrigerator vibrates excessively, it is an indication that the belt is worn. This should be replaced with a Gates 2420 V belt. At least one new spare should be on hand at all times.
4. About once a year the pressure of the refrigerant should be checked by a refrigerator repair man and returned, if necessary, to the standard 175 to 180 lbs. pressure.

- B. Blower- 1. The set-screw fastening the fan to the shaft sometimes becomes loose. This should be checked periodically.

2. The outlet slot on the pipe should be directed downward at an angle of approximately 30° with the horizontal.

C. Intermittent Heaters

1. These are controlled by a thermostat mounted on the outside of the cabinet. The setting for the blooming test is 50°F. The setting may be changed by turning the screw on the top of the thermostat housing.
2. The heaters should not be turned on without turning on the blower.

BLOOMING CABINET - DIRECTIONS FOR USE

-3-

- D. Humidity Control:-
1. Clock is electrically operated and does not need winding.
 2. The charts are changed weekly. To remove chart, lift pen-arm, and pull out fastening device at hub of clock. Pen holds about three days' supply of ink when full.
 3. Relay in clock shuts off refrigerator when pen arm drops below setting of control arm. Control arm is key operated. This should be at 0% R.H. (below minimum obtainable) when running blooming tests.

III. General-

- a. Keep cabinet clean at all times.
- b. Shut off blower and heaters and return refrigerator thermostat to 35° when not conducting test.
- c. If no tests are being run for sometime, it is advisable to empty drip pan every 3 or 4 days.

8/25/49

7/19/54

June 1952

KEROSENE ABSORPTION OF ASBESTOS FIBRE1. Definition:

The kerosene value of a fiber is the number of cubic centimeters of kerosene which will be absorbed by one pound of the fiber. The length of time required to wet the sample is also recorded.

2. Apparatus:

- 1 - 250 cc graduate
- 1 - 25 cc Screen Bottom Tube.
(This tube should be eight inches long and three quarters of an inch in diameter. It should have a 60 mesh copper screen bottom. A test tube with the bottom cut off will be satisfactory.)
- 1 - Tube Clamp
- 1 - Stop Watch

3. Procedure:

Weigh out 11 grams of a representative sample of the asbestos to be tested. Put the 11 gram sample into the tube tapping the sides of the tube until the asbestos is quite compact. Do not pack the sample tightly. Next, fill the graduate to the 250 cc mark, with water white kerosene. Clamp the top of the tube in a clamp on a ring stand and slowly lower the tube into the kerosene, avoiding breaks in the continuity of the Asbestos if possible, and suspend the tube so that the level of the kerosene inside the tube is equal to the level of the kerosene in the graduate. Record the length of time required to wet the asbestos fibre. Allow it to soak thus for 5 minutes and then raise the tube and suspend it so that the kerosene can drip back into the graduate. After the sample has drained for 20 minutes, take the reading on the graduate.

4. Calculations:

- a. Subtract from the initial graduate reading the final reading. This is the volume of kerosene absorbed by the sample in cubic centimeters.
- b. The constant weight of 11 grams must be converted into pounds. This will always be .0242 pounds.
- c. Divide the result in (a) by (b) .0242. The result will be the number of cubic centimeters absorbed by one pound of fiber.

Revised January 1953

JRL/JEH

Revised July 19, 1954

JRL/CAL

June 1952

MEASURING THE DIFFUSE REFLECTION OF ROOFING GRANULES

Apparatus:

1. Photovolt Photoelectric Reflection, Model 610 with search unit 610-Y.
2. Glass color filter, tristimulus amber
3. Glass color filter, tristimulus blue
4. Glass color filter, tristimulus green
5. Steel plaque with white porcelain enamel coating having a luminous reflectance of 72 for tristimulus amber filter, 74 for tristimulus green filter, and 76 for tristimulus blue filter. This is the working standard.

Scope:

The reflection meter is used to measure the diffuse reflection of granules as an indication of color or degree of whiteness. It can also be used to test raw material pigments.

Depending on the color filter being used, the instrument furnishes either readings of reflectance in specific spectral bands or readings in terms of total luminous apparent reflectance (whiteness, lightness), or tristimulus values in terms of the I.C.I. Standard Observer for Colorimetry for the purpose of color specification and color matching.

The reflection meter consists of two units: The instrument proper which contains the indicating meter, power supply, and controls; the "search unit" which comprises the light source and photo cells.

Procedure:

1. All reflectance measurements shall be made on granules which have not been oiled.
2. The grading of the granules does not affect the measurement.
3. All measurements of reflectance shall be made in comparison to the working standard (A white porcelain enamel coating on a steel plaque having a luminous reflectance of 72 for tristimulus amber filter).
4. The sample of granules is poured in a conical pile into a 3" x 3" box. This cone of granules is flattened so that the granules are flush with the top of the box.
5. The lamp should be warmed for 30 minutes prior to taking readings.

6. With the switch on "Off" set galvanometer on zero. This setting must be checked from time to time and readjusted if necessary.
7. Turn the two zero suppressor control knobs on the left all the way clockwise and leave in this position.
8. Insert a tristimulus filter and turn switch to "On."
9. The search unit is first placed on the calibrated enamel plaque which serves as a working standard, and the galvanometer needle is set on the calibration value of the plaque by using the coarse sensitivity control knob which is the second knob from the right. Then the exact adjustment is made by using the fine sensitivity control knob which is the first knob on the right.
10. Then the search unit is placed on the flattened granule sample to be tested, being careful to avoid disturbing the smooth surface, and its reflectance value is read after the needle has come to rest.
11. Place the search unit on the working standard and check whether the needle again indicates the rated value. If necessary, readjust setting and get new reflectance value.
12. Repeat this process using all three tristimulus filters.
13. It will mostly be found that one of the filters gives a wider spread of readings than the other two. This filter should then be used for grading of the surfaces under test as it will permit detection of the smallest differences in shade.
14. When the instrument is to be used for complete color specification and color matching, three readings are taken on each sample using the three tristimulus filters. From the readings obtained, the tristimulus values x, y, and z are computed according to the following equations:

$$x = 0.8 \text{ amber} + 0.18 \text{ blue}$$

$$y = \text{green}$$

$$z = 1.18 \text{ blue}$$

The green filter is identical with the y value. This green filter is referred to as the y filter or luminosity filter, the y function of the I.C.I. system is defined as representing the luminous effect. Measurements with the green tristimulus filter alone serves for determining "luminous apparent reflectance" or "lightness" or "whiteness."

Recording Data:

The readings shall be numerically recorded in comparison with the working standard and the type of filter used.

CES/rp

MEASURING THE TOTAL WATER SOLUBLE IONIC
CONCENTRATION OF HIGH TEMPERATURE FIRED
ROOFING GRANULES

CONDUCTANCE TEST

OBJECT:

To determine if the granules have been fired at a high enough temperature to insolublize the water soluble ions.

APPARATUS:

Betz Conducto Bridge

PROCEDURE:

1. Weigh 25 grams of granules and place in 250 ml extraction flask.
2. Pour 100 ml of boiling distilled water into extraction flask.
3. Swirl contents of the extraction flask for 60 seconds.
4. Pour 70 ml of decant into 100 ml graduated cylinder.
5. Cool cylinder until contents are below 100°F.
6. Place dip cell of conducto-bridge into solution to be tested. Move cell up and down under the solution level, once or twice to assure removal of air bubbles inside cell casing. Immerse cell to a point above the air vents.
7. Measure the temperature of the solution, then set the pointer of the lower "temperature" dial to the corresponding value.
8. Rotate the pointer of the upper "concentration" dial until the black segment of the cathode ray tube reaches its widest opening.
9. The conducto-bridge is now in balance as indicated by the cathode ray tube.
10. The setting is now read directly in mocromhos of specific conductance.

REPORTING:

Record result in micromhos of specific conductance.

CES/rp
2/1/61

Test No. 25

MEASURING THE E.M.F. OF LOW TEMPERATURE
FIRED ROOFING GRANULES

pH TEST

OBJECT:

To find the E.M.F. of low temperature granules

APPARATUS:

E.M.F. Meter - (pH meter)

PROCEDURE:

1. Weigh 25 grams of granules to be tested and place into 125 ml Erlenmeyer flask.
2. Pour 70 ml of boiling distilled water over granules
3. Place flask of granules and water on hot plate and bring to boil.
4. Remove flask from hot plate and place in cold water, allowing temperature of granules and water to cool to 20°C or 68°F.
5. Pour liquid from flask to 50 ml Griffin beaker and test liquid for pH according to instructions that are furnished with E.M.F. meter.

REPORTING:

Record result in pH units.

CS/rp
2/1/61

M
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INDEX - Laboratory Manual of Tests

MISCELLANEOUS TESTS

Test No.

1. Asphalt Removing Liquid
2. Testing and Analysis of Roll Roofing
3. Distribution of Paroid Coating
4. Per Cent Na_2O in Sodium Silicate
5. A 0.025 NORMAL Hydrochloric Acid (for weak caustic)
6. Modified Methyl Orange Indicator
7. Per Cent Rubars in Wind Seal Anti-Stick Solution
8. Testing of Wind Seal Adhesive

ASPHALT REMOVING LIQUID

For a 2000 Gram Batch:

920 Grams Butyl Stearate
784 Grams Sulphonated Pine Oil
189 Grams Oleic Acid
47 Grams Triethanolamine
2000 Grams

- A. Mix Butyl Stearate and Oleic Acid
- B. Mix Sulphonated Pine Oil and Triethanolamine

Then mix together A and B.

About 100 grams of water can be added to the above mixture to extend it. At times it may be possible to add more water without the mixture becoming cloudy.

The triethanolamine oleate soap can be prepared separately, if desired, and stirred into a mixture of Sulphonated Pine Oil and Butyl Stearate.

Viscosity will vary at times because of changes in the character of the ingredients.

Large batches made with fresh materials are preferable to making small batches both from the standpoint of cost but also of uniformity.

The cost of the mixture may be cut down by a little experimentation, the Butyl Stearate being the most expensive material yet is the solvent for the asphalt. Although some trials have been made by adding glycerine and/or lanolin to get a mixture which might be more soothing, they have not progressed far enough to be able to make a definite decision.

Sulphonated Castor Oil can be used in place of Sulphonated Pine Oil, but is more expensive and at least at present is not necessary.

Checked June 1952

JRH/jeh
1/15/51

AP
8/19/54

METHODS OF TESTING ASPHALT ROLL ROOFING
SURFACED WITH FINE MINERAL GRANULES OR WITH
POWDERED TALC

Scope - Section 1

These methods cover procedures for the physical testing and chemical examination of roll roofing composed of Asphalt Saturated Roofing Felt coated with asphalt and the coated portion surfaced with mineral powders or granules.

Types of Roofing - Section 2

Asphalt Roll Roofings shall be divided into two types, as follows:

- a. A single thickness of Asphalt Saturated Felt coated with asphalt, compounded with finely powdered mineral filler and surfaced with powdered mineral matter such as talc.
- b. Similar to type A but with the asphalt coating on the weather side surfaced with granules.

Sampling - Section 3

From each shipment or a portion thereof representing a product of the same kind, class and weight, a number of rolls shall be selected at random equivalent to one-half the cube root of the total number of rolls included in the lot, except that in lots of 1,000 or less five rolls shall be taken. If the cube root as calculated proves to be a fractional number, it shall be expressed as the nearest higher whole number.

METHOD OF PHYSICAL TESTING

Packing Materials, Nails and Cement - Section 4

Each roll selected shall be weighed to the nearest $1/4$ pound. The wrappers, packing materials and cement from each roll shall be weighed together to the nearest $1/4$ pound and the average weight thereof per square shall be recorded. The quantity and character of nails and cement shall be recorded.

Weight of Loose Surfacing - Section 5

Each roll selected shall be unwound and the workmanship and finish of the roofing observed. The weight of any loose surfacing that may fall off during this operation shall be recorded.

Width, Length and Area - Section 6

The width and length of each roll selected shall be measured to the nearest $1/4$ " and the area of material contained in each roll shall be calculated in square feet. The average area of the rolls examined shall be calculated from these values.

Minimum Weight per 103 Square Feet - Section 7

From the information obtained under Sections 5 and 6, the net weight, exclusive of loose surfacing of roofing per 103 square feet contained in each roll selected shall be calculated. The minimum weight per 103 square feet of the rolls examined shall be recorded. This shall be regarded as the minimum weight per 103 sq. ft. of the lot.

Average Weight per 103 Square Feet - Section 8

The average weight per 103 square feet for the rolls examined shall be calculated. This shall be regarded as the average weight per 103 square feet of the lot.

Selecting Average Weight Rolls - Section 9

From the rolls examined the one whose weight per 103 square feet is nearest the average weight of the lot shall be selected. The first convolution or two shall be unrolled and with a knife and straight-edge the sheet shall be cut across at right angles to the edges. A sample measuring exactly 30" in the direction of the rolls length shall then be removed. The width of this sample shall be measured to the nearest $1/32$ " and the weight of the sample in ounces determined, neglecting any loose surfacing. The weight in pounds per 103 sq. ft. shall then be calculated from the following formula. Weight in pounds per 103 square feet equals the weight of the 30" sample in ounces divided by the width of the 30" sample in inches, and this result multiplied by 32.4. The weight so determined shall check within 1.5% the average weight per 103 square feet of the laps (section 3). If this sample fails to check, then additional samples shall be cut from the same roll until one is obtained which does and this sample shall be reserved for further examination, as described in sections 10 to 19.

Pliability - Section 10

From the sample selected in accordance with section 9, 10 strips 1" in width by 8" in length shall be cut, five in the direction of and five across the fibre grain. They shall be immersed in water at 77°F. for 10-15 minutes, then removed and each strip immediately bent with the weather side up 90° over a rounded edge of a block at a uniform speed in approximately two seconds time. The blocks shall be 3" square by 2" thick with rounded corners of $1/2$ " radius for Type A Roofing and $3/4$ " for Type B Roofing. In bending the strips shall be held by hand tightly against the upper 2" face of the block and the projecting end of the strip shall be bent over the rounded corner without exerting any strain other than that required to keep the strip in contact with the block and to avoid kinking. Any surface ruptures exceeding $1/8$ " in length shall be considered failures.

Failure on Heating - Section 11

Two strips each approximately 4 x 4" shall be cut from the sample selected in accordance with section 9. The strips shall be weighed and then suspended vertically in the same direction as the material would be applied to the roof in the center of an air oven maintained at 175°F. plus or minus 5°F. by means of a thin wire fastened through

hole punctured near one edge. The strips shall be maintained at the prescribed temperature for exactly two hours, then cooled in a desiccator and each specimen weighed. The average loss of volatile matter shall be calculated as a percentage. Any change in appearance of the specimen shall be recorded, such as flowing, sagging, blistering or absorption of the asphalt coating, also sliding of granular surfacing. The internal dimensions of the oven used for this test shall be not less than 12x12x12" and the thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimen.

METHODS OF CHEMICAL ANALYSIS

Separation of Roofing into Component Parts

Analysis of Roofing Composition - Section 12

The composition of the roofing shall be analyzed as follows:

- a. Weight of Dry Felt per 108 sq. ft.
- b. Weight of Saturant (soluble in carbon disulphide) per 108 sq. ft.
- c. Weight of weather side coating (soluble in carbon disulphide) per 108 sq. ft.
- d. Weight of reverse side coating (soluble in carbon disulphide) per 108 sq. ft.
- e. Weight of mineral matter per 108 sq. ft. passing a #14 sieve and retained on a #100 sieve.
- f. Weight of mineral matter per 108 sq. ft. passing a #100 sieve.

Preparation of Specimens for Analysis - Section 13

From the sample representing the average weight of the lot of roofing as obtained in section 9, specimens shall be cut each measuring approximately 2" in width by 3" in length. These specimens shall be weighed, the length and width measured to within 1/32" inch, and the weight calculated per 108 square feet. Any of the 2x3 specimens whose weight varies more than 1.5% from the average weight of the lot as determined in section 8, shall be rejected. This process shall be continued until eight acceptable specimens are obtained which shall be used for determining the composition of the roofing as described in sections 14 to 19.

Two of the weighed 2"x3" specimens shall each be separated into three horizontal sections. The asphalt coatings with attached mineral surfacing shall be removed in such a manner that some of the saturated felt adheres to them leaving a central section of saturated felt free from coating. Any mineral surfacing detached during this operation shall be added to the section to which it belongs.

Saturant in Dry Felt - Section 14

The saturated central horizontal sections from the two test specimens, section 13-B, shall be weighed and then extracted with carbon disulphide in a suitable extractor or centrifuge until the washings are colorless.

(benzol having a boiling point of 80 to 82°C. - carbon tetrachloride or Chloroform may be used, if desired, but in case of dispute carbon disulphide shall be used). The desaturated felt shall be dried in air then placed in a tared weighing bottle and further dried at 221 to 230°F. for thirty minutes and weighed. From this weight and the original weight of the saturated felt specimen the percentage of asphalt saturant in the dry felt shall be calculated. This figure shall be used later for calculating the weight of saturants (soluble in carbon disulphide) per 108 square feet. The dry felt shall be preserved for the determination of ash, section 17.

Weight of Weather Side Coating (Soluble in Carbon Disulphide) - Section 15

The horizontal sections of the weather side containing the asphalt coating and mineral surfacing, section 13-B, shall now be extracted with carbon disulphide in a suitable extractor until the washings are colorless. The insoluble materials shall be removed and dried in air and the pieces of felt picked up and brushed free of adhering mineral matter. This felt shall then be placed in a tared weighing bottle, further dried at 221 to 230°F. for thirty minutes and weighed. From this weight, corrected for excess ash, as in section 17, and the percentage of asphalt saturant determined in section 14, calculate the weight of asphalt in this felt. The mineral matter shall be dried in air until free from carbon disulphide, then placed in a tared weighing bottle, further dried at 221 to 230°F. for thirty minutes, cooled in a desiccator and weighed. This mineral matter shall be retained for sieve analysis (section 19).

Calculations - From the original weight of the sample, subtract the combined weights of felt, asphalt, saturant in the felt and mineral matter. The difference is the weight of weather side coating soluble in carbon disulphide per 108 square feet.

Weight of Reverse Side Coating (Soluble in Carbon Disulphide) - Section 16

The horizontal sections of the reverse side (which will be the side opposite to that selected under section 15) containing the coating and mineral surfacing (section 13-B) shall now be extracted with carbon disulphide and the weight of coating soluble in carbon disulphide calculated as in section 15. The mineral matter recovered shall be combined with that obtained from the weather side (section 15) and retained for sieve analysis. The felt shall be ashed as in section 17 in order to determine the true weight of felt.

ASH OF DESATURATED FELT - Section 17

The desaturated felt obtained in sections 14, 15 and 16 from the center and weather and reverse sides shall be ashed separately in weighed crucibles either over an open flame or in a muffle furnace until all carbon has been consumed. The weight shall be determined. The percentage of ash in the center portion will be assumed to be the true percentage of ash of the entire felt. The excess ash in the weather side portion will be added to the mineral matter passing a #100 sieve, as determined in section 19. This weight will also be used for correcting the total weight of desaturated felt and weight of saturant soluble in carbon disulphide. In like manner the excess ash in the

reverse side portion will be added to the mineral matter passing a #100 sieve and for correcting the total weight for felt and saturant soluble in carbon disulphide.

Weight of Dry Desaturated Felt per 108 Square Feet - Section 18

The combined weights of the felt from the center portion obtained in section 14 and the weights of the felt from the weather side and reverse side portions obtained in sections 15 and 16 respectively corrected for excess ash, shall be the total weight of dried felt in the two 2x8" specimens. From this weight calculate the weight of dry felt per 108 sq. ft.

Weight of Mineral Matter - Section 19

The total mineral matter recovered in sections 15 and 16, plus the excess ash (section 17) shall be moistened with a few drops of alcohol, boiled with 100 cc. of water and washed successively through a #14 and a #100 sieve with sufficient hot water to remove the fine mineral matter. The mineral matter retained on each sieve shall be collected separately, dried at 221 to 230°F. for thirty minutes and weighed. From these weights the weight of mineral matter shall be calculated and reported as weights of mineral matter per 108 sq. ft. passing a #14 sieve and retained on a #100 sieve and passing a #100 sieve (by difference). To check the weight of mineral matter passing a #100 sieve, the total aqueous suspension of mineral matter passes through the #100 sieve shall be recovered by filtering through a weighed Gooch crucible, dried at 221 to 230°F. and weighed.

Checked June 1952

TEST FOR DISTRIBUTION OF PAROID COATING

Object:

This test is devised principally to determine the relative amounts of front and back coating in a comparatively short time without stopping the machine. Operators making this test should follow the instructions below very carefully.

Procedure:

Slow machine down to its minimum speed. Stick a piece of Scotch Tape, approximately 12" in length so that the folded strip covers about 6" of the face and 6" of the back saturated sheet before it passes into the coating rolls. Apply tape with cotton gloves to the saturated felt and pat quickly but firmly to insure thorough adhesion of the tape to the surface. After coating and surfacing this area, remove the Scotch Tape very carefully with the adhering coating and surfacing and cut into a 2"x4" sample. Remove any saturated felt on the back of the sample, and weigh on grain scale, and by reference to the table determine the amount of front or back coating.

Net Weights have been calculated in whole numbers. For example: If sample weighs 45.5 grains, compute results by referring to the nearest whole number, which would be 46 grains.

Note

The Scotch Masking type used in these tests is purchased from the Minnesota Mining and Mfg. Co. It is 3" wide with a semi-corrugated back. It is essential that each new roll or shipment conform with the standard tape in all specifications.

Net per 108 square ft.	=	5.0 lbs.
Caliper	=	.008 lbs.
Length	=	72 yards roll
Width	=	3"
Type	=	Semi-Corrugated back

Checked June 1952

EZ:hc
7/2/41

AP - 8/19/54

PERCENT OF Na₂O IN SODIUM SILICATE

Weigh out a sample of a half gram to a gram of the silicate to be tested in a covered vessel. Dissolve the sample in about 80 cc of distilled water and determine the alkali as Na₂O by titration with standard hydrochloric acid (tenth normal) with modified methyl orange.

$$\% \text{ Na}_2\text{O} = \frac{\text{cc} \times \text{N} \times \text{milliequivalent wt. of Na}_2\text{O}}{\text{weight of sample}}$$

where cc = cc of HCl

N = Normality of HCl

.031 = Milliequivalent wt. of Na₂O

Checked June 1952

JPK/jp
10/22/47

AP
8/19/54

A .025 NORMAL HYDROCHLORIC ACID (FOR WEAK CAUSTIC)

1. Measure out about 4.03 cc. of concentrated H Cl (37-38% H Cl. Sp.Gr. 1.19) into a 2 liter (or larger) bottle, dilute with distilled water to 2000 cc., and mix thoroughly.
2. Weigh, accurately, .3313 grams of Sodium carbonate - C.P. - powdered - into a 250 cc volumetric flask and dilute to mark with distilled water. Be sure that all of carbonate is dissolved before using solution.
3. Measure out 10 cc of carbonate solution, by means of a burette or pipette, into a beaker, and add 2 or 3 drops of methyl orange indicator. Titrate this solution with the hydrochloric acid solution made up as under (1.) above, using a burette. If acid is exactly .025 Normal, 10.0 cc will be required to turn indicator from yellow to pink. If more than 10.0 cc is required, it indicates that acid is too weak and that more H Cl must be added to solution. Care should be taken at this point to add only a small amount of H Cl, as solution is very weak. Usually 1 or 2 drops is sufficient. If less than 10 cc of acid is required to neutralize the sodium carbonate, it indicates that acid is too strong, and more water must be added.

NOTE: A normal solution of hydrochloric acid contains one gram molecular weight of H Cl, or 36.45 grams, per 1000 c.c. of solution. Thus, the 4.03 cc of H Cl, in (1), above, was obtained as follows:

$36.45 \times 2 = 72.90$ gms for 2000 c.c. of 1 N solution
 $72.90 \times .025 = 1.8225$ gms for 2000 c.c. of .025 N solution
 acid used, however, contains only 38% H Cl; thus:

$$\frac{1.8225}{.38} = 4.79 \text{ gms of } 38\% \text{ acid}$$

Also, specific gravity of acid is 1.19; thus

$$4.79 \text{ gms} = \frac{4.79}{1.19} \text{ cc} = 4.03 \text{ c.c.}$$

B. 2.5 Normal Hydrochloric Acid (for strong caustic)

1. Weigh 1.575 gms of C.P. oxalic acid into a beaker, add about 15 - 20 cc of distilled water, and let dissolve.
2. Weigh 20 gms of C.P. Sodium (Na OH) hydroxide and dilute to 200 cc with distilled water.
3. Titrate sodium hydroxide solution against oxalic acid solution, using phenolphthalein as in indicator. If 10 cc of Na OH is required to neutralize the 1.575 gms. of oxalic acid, the solution is exactly 2.5 N. A 2.5 N solution of Na OH is desirable, in that it simplifies later titration against 2.5 N H Cl. This may be obtained by adding more Na OH, if solution is less than 2.5 N, or more water if it is stronger than 2.5 N. If it is not thought

- 2 -

necessary to have the N aOH solution exactly 2.5 N, the following formula may be used.

$A \times B \times C = X$, where

A = cc of N aOH solution used

B = Normality of N aOH

C = Milliequivalent wt. of oxalic acid - .063

X = Grams of oxalic acid

Thus, the normality, B, is found as follows:

$$B = \frac{X}{A \times C} = \frac{1.575}{A \times .063} = \text{Normality}$$

4. Measure 403 cc of concentrated hydrochloric acid into a 2 liter (or larger) bottle, dilute with about 1600 cc of distilled water, and mix thoroughly.
5. Titrate this hydrochloric acid solution against the standard sodium hydroxide solution by one of following two methods, using methyl orange as indicator in either case:
 - a. When N aOH solution is exactly 2.5 normal, 10 cc of H Cl solution should exactly neutralize 10 cc of N aOH solution. If this is true, acid is 2.5 N, but if more or less than 10 cc of H Cl solution is required, it should be corrected by adding either more acid or more water.
 - b. When N aOH solution is not exactly 2.5 normal, following formula should be used:

$A \times B = C \times D$, where

A = CC of N aOH

B = Normality of N aOH

C = CC of H Cl

D = Normality of H Cl

Thus, normality of H Cl = $\frac{\text{Normality of N aOH} \times \text{cc of Na OH}}{\text{cc of H Cl}}$

To obtain a normality of exactly 2.5 normal, water or acid should be added, as above.

(continued)

PHENOLTHALEIN SOLUTION

1 part Phenolphthalein.
50 parts distilled water.
50 parts Ethyl Alcohol.

Add dilute NaOH to turning point of indicator, slightly pink.

Checked June 1952

MODIFIED METHYL ORANGE INDICATOR

1 part methyl orange

1.4 parts xylene cyanole FF

Dissolve 1.0 gm methyl orange in 1 liter of hot distilled water, filter if necessary and cool. Add 1.4 gm xylene cyanole dye. Basic color - green; end point - gray; acid color - pink.

Note: Indicator solution, or dye, may be obtained from Eastman (D.P.I.) in Rochester, New York.

JWJ/rp
6/10/58

PER CENT RUBARS IN WIND SEAL ANTI-STICK SOLUTION

This test is a rapid titration method for determining the amount of Rubars, as received, present in the solution. The sample to be tested should be representative of the tank, after thorough mixing.

Procedure:

An exact 5 ml aliquot portion of the tank sample is placed in a 125 ml Erlenmeyer flask and diluted with approximately 50 ml of distilled water. Three drops of Modified Methyl Orange indicator is added and the solution is titrated with one tenth normal hydrochloric acid. Titration is carried on until the clear green solution reaches a very cloudy gray end point, almost on the verge of turning pink.

Concentration by weight of the solution is then determined by using the attached chart.

JJJ/rp
7/16/58

TESTING OF WIND SEAL ADHESIVEFBB Batches in Special Melt Kettle

Every new batch made at FBB shall be tested. A one pint sample will be brought down to PBL by an FBB man.

Wind Seal Adhesive in FBPM#9 Kettle

A one pint sample shall be drawn off and tested by PBL on days when Wind Seal Shingles are being run on either FBPM#1 or FBPM#9. Approximately 1/2 gallon of Wind Seal Adhesive shall be drawn off into the waste drum before taking the PBL sample.

Tests to be Run

1. Softening Point (in water)
2. Pen. @ 115°F. (50 gms - 5 sec.)
3. Pen @ 77°F. (100 gms. - 5 sec.)

Specifications

	<u>Min.</u>	<u>Std.</u>	<u>Max.</u>
Softening Point, °F.	140	145	150
Pen @ 77°F.	--	--	15.0
Pen @ 115°F.	--	--	80.0

Preparation Before Testing

1. Cut the top one-half inch from a 3 oz. Gill type can with the laboratory can opener. Use this "cut down" can for the pen @ 115°F. Use the lid from a 3 oz. can for the pen @ 77°F.
2. Have two softening point rings cleaned and set up on a glycerine coated brass plate.
3. Have a softening point apparatus ready. Also have a 0 to 400°F. range thermometer and a low temperature gas torch.
4. Place the one pint sample of Wind Seal Adhesive on a hot plate and stir it slowly with the 400°F. thermometer until the temperature reaches 250°F. Then quickly place the two penetration tins on the same hot plate, remove the thermometer from the pint can and wipe it off on a rag (using caution not to burn the fingers). With a pair of tongs, place the two hot pen tins on a level spot on the laboratory bench. Then remove the pint sample of Wind Seal Adhesive from the hot plate with a pair of tongs; pour off the top of the sample until about one-half pint remains; then pour the 115° pen, the 77° pen, and the two softening point rings in the foregoing order. The penetration tins shall be filled until the material is level with the top. Then remove the air bubbles from the pen samples by playing a low temperature flame such as a match or gas torch over the surface. Avoid excessive firing of the surface.

Penetration Tests

Let the poured samples remain at room temperature for one hour plus or minus 5 minutes. During this time, set the ~~50°F. bath~~ bath at 77°F. $\pm 0.1^\circ\text{F}$. When the hour has past, ~~place each pen tin in its respective water bath and hold them in the water bath for one hour ± 5 minutes.~~ ~~When the hour is up, run the 115°F. pen first.~~ Place the water vessel that holds the pen sample into the water bath for a few minutes; set the penetrometer dial to zero; remove any of the weights and install the needle. Then quickly remove the transfer dish with the sample from the bath and place it on the penetrometer. Adjust the needle and shadow until they just touch each other and drop the needle for 5 sec. Then quickly loosen the needle and place the transfer dish with the sample back into the 115°F. bath. Always return the transfer dish with the sample back to the bath while cleaning the needle and getting set for the next test. Clean the needle by wiping on a rag. The needle may be dipped in an asphalt solvent before wiping. Never place the needle in a flame as the temperature will be ruined in a matter of seconds.

Run three penetration tests and take an average of the three. If, however, the first two tests are identical, it is not necessary to run a third.

Softening Point

Let the two sample rings cool for one hour on the brass plate then cut the excess adhesive off the top with a shoe knife that has been heated on the face of a laboratory hot plate. Then center a steel ball on the top of each sample and place in a 600 ml beaker filled to 3 1/4" of distilled water. After ten minutes run the regular softening point test by placing the beaker and contents on the hot plate assembly. Suspend the softening point rings so the bottom of the ring is exactly 1" from the bottom of the beaker. Place the thermometer so that the bottom of the bulb is level with the bottom of the ring. Apply heat from the hot plate to the beaker so that the temperature rises at the rate of $9^\circ\text{F.} \pm 1^\circ\text{F.}$ per minute. Record the temperature when the adhesive covered ball touches the bottom of the beaker. For final result take an average of the two softening points.

JRLukko/rp
July, 1961

PBL