

# Standard Test Methods for Sampling, Testing, and Analysis of Asphalt Roll Roofing, Cap Sheets, and Shingles Used in Roofing and Waterproofing<sup>1</sup>

This standard is issued under the fixed designation D228/D228M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

#### 1. Scope

1.1 These test methods cover procedures for sampling, examination, physical testing, and analyses of asphalt-containing materials used in roofing and waterproofing. These materials include but are not limited to roll roofing, cap sheets, and shingles. Any of these materials are allowed to be partially or fully coated, surfaced, or laminated, or a combination thereof.

1.2 The test methods and procedures in this standard appear in the following order:

Section	Content
6	Types of Roofing
7	Sampling
8	Mass and Area Determination
9	Selection of Representative Specimens
10	Moisture
11	Pliability
12	Mass Loss and Behavior on Heating
13	Tear Strength
14	Fastener Pull-Through Resistance
15	Preparation and Selection of Small Test Specimens for Analyses
16	Analysis of Glass Felt Products
17	Analysis of Roofing Products with Organic Felts
18	Ash of Desaturated Felt
19	Calculation
20	Adjusting Back Coating Fine Mineral Matter and Back Surfacing
21	Report
22	Precision and Bias

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the

responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation

D146/D146M Test Methods for Sampling and Testing Bitumen-Saturated Felts and Woven Fabrics for Roofing and Waterproofing

D225 Specification for Asphalt Shingles (Organic Felt) Surfaced With Mineral Granules (Withdrawn 2012)<sup>3</sup>

D1079 Terminology Relating to Roofing and Waterproofing

D1922 Test Method for Propagation Tear Resistance of Plastic Film and Thin Sheeting by Pendulum Method

D2178/D2178M Specification for Asphalt Glass Felt Used in Roofing and Waterproofing

D2626/D2626M Specification for Asphalt-Saturated and Coated Organic Felt Base Sheet Used in Roofing

D3462/D3462M Specification for Asphalt Shingles Made from Glass Felt and Surfaced with Mineral Granules

D3909/D3909M Specification for Asphalt Roll Roofing (Glass Felt) Surfaced With Mineral Granules

D4601/D4601M Specification for Asphalt-Coated Glass Fiber Base Sheet Used in Roofing

D4897/D4897M Specification for Asphalt-Coated Glass-Fiber Venting Base Sheet Used in Roofing

D4932/D4932M Test Method for Fastener Rupture and Tear Resistance of Roofing and Waterproofing Sheets, Roll Roofing, and Shingles

D6380/D6380M Specification for Asphalt Roll Roofing (Organic Felt)

F1667 Specification for Driven Fasteners: Nails, Spikes, and Staples

<sup>&</sup>lt;sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D08 on Roofing and Waterproofing and are the direct responsibility of Subcommittee D08.02 on Steep Roofing Products and Assemblies.

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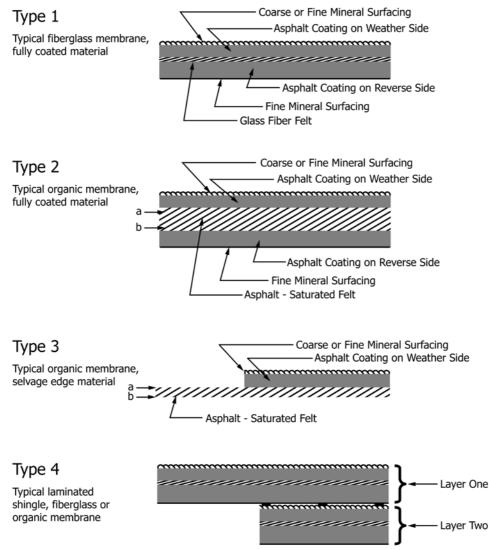
<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

### 3. Terminology

- 3.1 Definitions:
- 3.1.1 For definitions of terms used in these test methods, see Terminology D1079.
- 3.1.2 As referenced in 15.1, "Materials of Uniform Composition" designates products that are manufactured to be the same thickness, ply count, and mass per unit area in all areas of the material. This would include traditional mineral surfaced rolls, "3 Tab" shingles, and other shingles that have areas of material removed for appearance purposes (cut-outs) but the rest of the shingle is of uniform composition. See Fig. 1, Type 1 or Type 2.
- 3.1.3 As referenced in 15.2, "Materials of Nonuniform Composition" designates products that are intentionally manufactured to have different thickness or mass per unit area within different areas of the product. Examples of materials of nonuniform composition are selvage edge rolls and overlaid or

- embossed shingles (where not all of the product has the same thickness). See Fig. 1, Type 3.
- 3.1.4 As referenced in 15.4, "Materials of Multiple Layer Composition" designates products that are fabricated in multiple layers. See Fig. 1, Type 4.
- 3.1.5 As referenced in these test methods, "Machine Direction" (indicated as MD) is the direction running the long dimension of a roll product (before samples or specimens are cut) or the long dimension of a shingle, unless otherwise known and agreed to between supplier and purchaser. "Cross Direction" (indicated as CD) designates the direction perpendicular to the MD.
- 3.1.6 A production lot is defined as all material produced within one production shift of the same type, composition, and color (where applicable). A delivery lot is defined as a shipment or fraction thereof representing a product of the same type, composition, and color (where applicable).



Note 1—Not to scale.

Note 2—Type 3 has been illustrated without asphalt coating or fine mineral surfacing on the reverse side. It is not prohibited to fabricate this product with asphalt coating or fine material surfacing on the reverse side.

FIG. 1 Types of Asphalt-Coated Roll Roofing and Shingles

# 4. Significance and Use

- 4.1 These test methods include procedures for sampling, examination, physical testing, and analyses of asphalt roll roofing, cap sheets, and shingles used in roofing and water-proofing. Other components of these materials are allowed to include, but are not limited to, felts, mats, films, foils, mineral stabilizers, papers, and mineral surfacing.
- 4.2 These test methods include tests that are not required by every product standard that references Test Methods D228/D228M. The individual product standards are the authority for which tests are required for compliance. It is not prohibited to run tests in addition to those required in the product standards, but these test methods make no claim to their suitability or significance.
- 4.3 Five random samples are required from lots equal to or less than 1000 packages. The number of samples required for lots greater than 1000 packages is dependent on the variation in the unit mass within the lot and is determined by the two-step sampling plan in 7.4.
- 4.4 The results of a visual examination, physical testing, and compositional analysis are required for each sample. The analytical data are further used to compute the probable minimum and the probable range for the average mass of each of the components.

#### 5. Materials

5.1 Filter Paper—The extractions for analysis of glass felt products (Section 16) and analysis of roofing products with organic felt (Section 17) require the use of filter paper with a particle retention of 2.7  $\mu$ m (see Note 1), This filter paper is dried for 60 min  $\pm$  10 % in an 80°C [176°F] oven and stored until needed in a desiccator.

Note 1—Whitman No. 50 filter paper [185 mm] has been found satisfactory for this use.

#### 6. Types of Roofing

- 6.1 Asphalt-coated roll roofing and shingles are divided into the following types for the purposes of these test methods (see Fig. 1).
- 6.1.1 *Type 1*—A single thickness of glass felt, coated with asphalt and mineral surfacing such as in Specifications D2178/D2178M, D3462/D3462M, D3909/D3909M, D4601/D4601M, and D4897/D4897M. The backing material (designated "Fine Mineral Surfacing" in Fig. 1) shall be permitted to be any suitable material that prevents these products from sticking together while packaged.
- 6.1.2 *Type* 2—A single thickness of asphalt-saturated felt coated with asphalt and mineral surfacing such as in Specifications D225, D2626/D2626M, and D6380/D6380M, Class M. The backing material (designated "Fine Mineral Surfacing" in Fig. 1) shall be permitted to be any suitable material that prevents these products from sticking together while packaged.
- 6.1.3 *Type 3*—Similar to Type 2, but asphalt coated and surfaced with mineral granules for part of one side of the saturated felt such as in Specification D6380/D6380M, Class WS. This type also includes products similar to Type 1 or Type

- 2 that have overlay or embossed areas. This does not include products that are fabricated in multiple layers.
- 6.1.4 Type 4—This material is comprised of multiple layers bonded with a suitable adhesive, typically a bituminous material. Evaluation of the nature of the adhesive or quality of the bond, or both, is outside the scope of this test method. Fig. 1 illustrates a typical configuration for Type 4. This "Type" description is provided to aid the user in understanding how to match a given product composition and assembly to its corresponding Type. It is not intended to limit or exclude products with similar, but not identical constructions. Examples of known variations, which shall not be prohibited include (but are not limited to):
  - 6.1.4.1 Materials using other adhesives,
  - 6.1.4.2 Materials fabricated with more than two layers,
- 6.1.4.3 Materials that use different relative proportions for the layers, and
  - 6.1.4.4 Materials with other core compositions

## 7. Sampling

- 7.1 The rolls or packages selected in accordance with this section constitute the representative sample used in Sections 8 and 9.
- 7.2 Select five rolls or packages from the lot at random. Do not select any material that shows visual indications of damage from shipping or handling. Determine the average net mass in  $g/m^2$  [lb/100 ft<sup>2</sup>] and the standard deviation in accordance with 8.1 8.8.
- 7.3 If the lot is 1000 or fewer rolls or packages, proceed to Section 8. If the lot is 1001 or greater in number, follow the second part of the sampling plan in 7.4.
- 7.4 Calculate the required number of samples based on the standard deviation (*s*) of the preliminary sampling by:

$$n = \frac{t^2 s^2}{d^2} \tag{1}$$

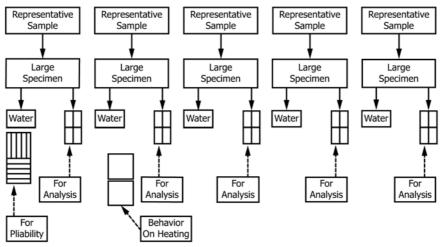
where:

- n = total number of samples required (n 5 more rolls or packages must be selected at random as samples),
- t= test statistic for number of samples in the preliminary test series for  $4^{\circ}$  and a 95 % confidence that the calculated average mass will not exceed d (t=2.776), and
- $d = 100 \text{ g/m}^2 \text{ [2 lb/100 ft}^2 \text{]}$  (the mean mass obtained from the analysis should be within  $\pm 100 \text{ g/m}^2$  of the true value, with 95 % confidence).
  - 7.5 See Fig. 2.

# 8. As Received Mass and Area Determinations, All Types of Roofing

- 8.1 *Gross Mass*—Determine and record the mass of each representative sample to the nearest 0.1 kg [0.2 lb].
- 8.2 Net Mass—Disassemble each package or unroll each roll of the representative sample; shake off any loose surfacing and determine and record the net mass of all the shingles or the

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In Section 7, initial "Representative Samples" are selected (typically five per lot). These are full bundles or rolls. These are the samples used in Section 8.

In Section 9, one large "Specimen" is taken from each representative sample (full shingles from 8.5 or a portion of each roll selected in Section 7). These are confirmed to be within 1.5 % of the mass per area (from 8.6) for the corresponding representative sample.

In Section 10 (where product standards require measurement of water content per Test Method D95), 50-g small test specimens are taken from each "Large Specimen" for determination of water.

In Section 11, five MD and five CD small test specimens are cut from one of the "Large Specimens" for evaluation of pliability.

In Section 12, two small test specimens are cut from one of the "Large Specimens" for evaluation of behavior on heating.

In Section 15, small test specimens (three for glass felt, four for organic felt) are cut from each of the "Large Specimens" for analysis by Section 16 (for glass felt) or by Section 17 (for organic felt).

FIG. 2 Sample Selection Summary and Flow Diagram (See Individual Sections for Sample Selection Details)

entire roll to the nearest 0.1 kg [0.2 lb]. Where a product standard requires it, the loose surfacing is to be collected and the mass determined.

- 8.3 Packaging and Fixture Mass—Determine and record the mass to the nearest 0.1 kg [0.2 lb] of the packaging and all associated fixtures shipped with each roll or package of the representative sample, such as nails and adhesive.
- 8.4 *Dimensions of Roll Products*—Measure and record the length and width of each roll of roofing and the selvage width to the nearest 3 mm [1/8 in.].
- 8.5 Shingle Count and Dimensions—From each of the representative sample packages generated in Section 7, count and record the number of shingles in each package. Select one shingle randomly from each package. Measure the width, length, and cutout dimensions for each of these selected shingles to the nearest 1 mm [ $\frac{1}{32}$  in.]. Calculate and record the average for each of these measurements.
- 8.6 Calculate the area of the roofing and the net mass per unit area of the roofing. Report the net mass per unit area in  $g/m^2$  [lb/100 ft<sup>2</sup>] for each representative sample (without packaging, cut outs or loose surfacing).
- 8.7 Calculate and record the average net mass per unit area for the combined representative samples (without packaging, cut outs, or loose surfacing) in g/m<sup>2</sup> [lb/100 ft<sup>2</sup>].
- 8.8 Calculate and record the estimate of the standard deviation by:

$$s = \sqrt{\frac{\sum X^2}{n-1} - \frac{(\sum X)^2}{n(n-1)}}$$
 (2)

where:

s = an unbiased estimate of the standard deviation,

 $\sum X^2$  = the sum of the squares of the individual mass determinations.

 $(\sum X)^2$  = the square of the sum of the individual mass determinations, and

n = the number of rolls or packages in the representative sample.

8.9 Calculate the 95 % confidence interval for the average mass by:

$$\frac{\sum X}{n} \pm \frac{ts}{\sqrt{n}} \tag{3}$$

where:

 $\sum X$  = sum of the individual mass determinations, and

= appropriate t statistic for 95 % confidence and n-1 dF (t = 2.776 for n = 5; consult standard reference table for other values of n).

# 9. Selection of Representative Specimens, All Roofing Types

9.1 Several product standards reference sampling as designated in Test Methods D228/D228M for tests that are not specifically covered by Test Methods D228/D228M. Unless otherwise specified in these test methods, the large specimens and small test specimens shall all be selected from the representative sample (roll or package selected in Section 7) that has the individual net mass per unit area closest to the average net mass per unit area for the combined representative samples as determined in Section 8. If more than one roll or

package needs to be selected to provide sufficient specimens for all the tests dictated within the product standard, any additional specimens shall be selected from the representative sample that has the net mass per unit area that is next closest to the average net mass per unit area of the combined representative samples.

Note 2—It is the intent of these test methods that whenever not specifically directed to select specimens in some other manner, specimens shall be selected from the representative sample that is the closest in mass per unit area to the average mass per unit area of the combined representative samples.

- 9.2 For roll products, select a specimen of roofing, the full sheet width and at least 1 m [3 ft] in length from each roll, starting a minimum of three wraps into the roll. For shingle products, the shingles from 8.5 shall be used.
- 9.3 Determine the mass of each specimen and calculate the mass in  $g/m^2$  [lb/100 ft<sup>2</sup>].
- 9.4 For Types 1, 2, or 3, discard all specimens that differ by more than 1.5 % from the net mass determined in 8.6, select replacements, and determine the mass as in 9.3.
- 9.5 Continue this process until five representative specimens are obtained, no more than one from each package or roll, that reflect that roll or package's average net mass as determined in 8.6.
- 9.6 If fewer than five specimens are available, use all the available specimens and adjust the final calculations to reflect the lower number of samples tested.

9.7 See Fig. 2.

#### 10. Moisture

10.1 Determine the water in each sample in accordance with Test Method D95; use 50 g [0.11 lb] of product for each determination, cut up to fit in the flask. Report the water content as a percent of the dry (water-free) product mass.

10.2 See Fig. 2.

# 11. Pliability

11.1 From one of the large specimens selected in Section 9, cut ten small test specimens  $25.4 \pm 3$  mm  $[1 \pm \frac{1}{8}$  in.] in width by 200  $\pm$  50 mm [8  $\pm$  2 in.] in length, five MD and five CD. For Type 3 materials, these small test specimens are to be taken from the area that is asphalt-coated and surfaced with mineral granules. For Type 4 (laminated) materials, unless the product standard to be applied directs otherwise, the small test specimens are to be cut from a single layer. It is not prohibited to use single layer specimens from a multiple layer section of the shingle that has been separated into individual layers with suitable care. Any material damaged or significantly bent or creased during the separation process is to be discarded. Unless the product standard being evaluated directs testing at another temperature, condition the small specimens and the block at 23  $\pm$  2°C [73  $\pm$  4°F] for 2  $\pm$  0.1 h and perform the test at 23  $\pm$  $2^{\circ}$ C [73  $\pm$  4°F]. Perform the test with the weather side up, at a uniform speed through 90° in approximately 2 s over the rounded edge of a block. If a water bath is needed to hold the designated temperature, the samples are to be placed in a plastic bag before placing them in the water bath so that they can be tested dry. Evaluation of the sample for cracking is much clearer when the sample does not have a wet surface.

11.1.1 The corner radius over which the small specimens are to be tested is typically specified in the individual product standards. If not otherwise specified, the block will be 75 mm minimum [3 in. minimum] square by 50 mm minimum [2 in. minimum] thick with rounded corners of  $13 \pm 1$  mm [ $\frac{1}{2} \pm 0.04$  in.] radius for Type 2 roofing and  $19 \pm 1$  mm [ $\frac{3}{4} \pm 0.04$  in.] radius for Types 1 and 3 roofing. When bending, hold the specimens by hand tightly against the upper face of the block and bend the projecting end of the specimen over the rounded corner without exerting any stress other than that required to keep the specimen in contact with the block and avoid kinking.

11.1.2 For coated products, failure of a specimen in this test is defined as cracking of the coating asphalt that exposes the reinforcement of the specimen (organic or fiberglass). The cracking shall be visible to the naked eye when the specimen is viewed in the bent condition on the mandrel block. Separation of granules or other superficial fissures that do not extend through the coating asphalt surface to the reinforcement do not constitute cracking. Fracture through the specimen is also considered a failure. Report the number of specimens passing.

Note 3—Some products require testing with granules surfacing up and granule surfacing down. Those products shall have that additional requirement clearly stated in their product standard. The additional samples are to be selected in a manner consistent with the instructions above. This test method is intended for use only with coated products. See Test Methods D146/D146M for pliability testing on non-coated products.

11.2 See Fig. 2.

# 12. Mass Loss and Behavior on Heating

12.1 Cut two test specimens, each approximately 100 by 100 mm [4 by 4 in.] from a large specimen selected in accordance with Section 9. Condition the smaller specimens for 24 h in a desiccator, determine the mass to the nearest 0.1 g, and then by means of a thin wire fastened through holes punctured near one edge, suspend them vertically in the center of an air oven maintained at  $80 \pm 3^{\circ}$ C [176  $\pm 5^{\circ}$ F] with the cross machine direction vertical. The internal dimensions of the oven shall be not less than 305 by 305 by 305 mm [12 by 12 by 12 in.]. The oven shall be electrically heated with forced draft. Insert a thermometer in the center of the oven to such a depth that its bulb is in line with the center of the specimens. Maintain the specimens at the prescribed temperature for 2 h  $\pm$ 5 min, then cool in a desiccator and determine the mass of each specimen. Calculate the average loss of volatile matter as a percentage of the final specimen mass. Record any change in appearance of the specimen such as blistering, absorption of the asphalt coatings, or sliding of coating or granular surfacing. Record the extent of the latter to the nearest 2 mm [1/16 in.].

12.2 See Fig. 2.

## 13. Tear Strength

- 13.1 *Tear Strength*—Use Test Method D1922 as modified here.
- 13.1.1 Specimens shall be rectangular, 76 by 63 mm [3 by 2.5 in.]  $\pm 3$  %.

- 13.1.2 Condition specimens at  $23 \pm 2^{\circ}$ C [73  $\pm 4^{\circ}$ F] for at least 2 h prior to testing and conduct tests at  $23 \pm 2^{\circ}$ C [73  $\pm 4^{\circ}$ F].
- 13.1.3 Each specimen will be composed of a single layer. Cut specimens from shingles in areas free of sealing resin and release tape. The 76-mm edges of the specimens shall be parallel to the long dimension (machine direction) of the shingles so that the tears will run in the short dimension (cross machine direction) of the shingle. Enough specimens shall be prepared so that ten results can be recorded after excluding any that must be rejected as prescribed in 13.1.4.
- 13.1.4 Use an Elmendorf Tear Strength Tester with 3200 or 6400 g [31 or 63 N] full scale capacity. Make all tests with granule surface of specimens facing away from the knife blade. Do not reject the results from specimens that tear through a side edge as opposed to the top edge. Reject results of specimens that tear in such a way that the portion of the specimen that is in the stationary jaw rubs against the pendulum
- 13.1.5 Report the average tear resistance of ten specimens to the nearest 0.1 N [10 g].
- 13.1.6 The following criteria shall be used to judge the acceptability of the results at the 95 % confidence level:
- 13.1.6.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 17 %.
- 13.1.6.2 *Reproducibility*—The results submitted by each of two laboratories should be considered suspect if they differ by more than 28 %.

# 14. Fastener Pull-Through Resistance

- 14.1 *Scope*—This test method measures the force required to pull a fastener head through a specimen of material under defined conditions in a specified test apparatus. See the individual product standards for the significance and use of this test.
- 14.2 Fasteners—Various fasteners suitable for application of asphalt roofing materials shall not be prohibited in this test. The following instructions and procedures are based on a standard galvanized roofing nail with 9.5 mm [3/8 in.] diameter head (as specified in Specification F1667, Table 29). See Note 4. The specific fastener(s) required or allowed is specified in the product standard where this test method is applied. When this test method is not used for determining compliance to a product standard, the fastener employed must be reported with the results and the orientation of any prominent geometric features of the fastener with respect to the roofing product orientation shall also be reported (for any fastener other than the standard nail referenced above).

Note 4—When other fasteners, for example staples, are used with this test method and apparatus, the effects of varying orientation of the staple crown with the orientation of the specimen (parallel to machine direction or at some angle to the machine direction), or the effects of the staple crown not being flat and flush relative to the specimen surface, will generally cause greater variability in the results than when using a roofing nail.

14.3 Specimens—For single layer materials, prepare ten specimens 98  $\pm$  3 mm [3 $\frac{7}{8}$   $\pm$   $\frac{1}{8}$  in.] square for each test

condition. If specimens include areas containing sealant (factory-applied adhesive) or release tape, or both, because it is inherently located in the nailing area when the material is applied in accordance with the application instructions, then it shall be noted in the report since it could affect the result. When testing materials with cutout areas, any specimen where the cutout is visible through the opening in the plate (see 14.6) will have the effect of increasing the variability of the results.

14.4 For materials that include multiple layers, prepare ten specimens 98  $\pm$  3 mm [37/8  $\pm$  1/8 in.] square using the following procedure: These specimens shall be cut from the manufacturer's specified fastening position on the material. First, determine the fastener placement position from the manufacturer's application instructions. Once this position is determined, cut a 98  $\pm$  3 mm [3\%  $\pm$  \\dots in.] wide strip of material centered on this fastening position. Typically, this strip will be cut from the long dimension, or length, of the shingle or roll product. Use this strip to cut consecutive specimens 98  $\pm$  3 mm [37/8  $\pm$  1/8 in.] in length. These strips are cut from multiple large specimens such that no less than three specimens in a row nor more than four specimens in a row are cut from one large specimen when generating the ten specimens required for testing. If specimens include areas containing sealant (factory-applied adhesive) or release tape, or both, because it is inherently located in the nailing area, then it shall be noted in the report since it could affect the result.

Note 5—Specimens shall be permitted to include areas containing factory-applied adhesive (sealant) or release tape, or both, if this is expected to be in the nailing area when the shingles are applied in accordance with the manufacturers'/sellers' instructions. If this is the case, it shall be noted in the report since it could affect the result. For normal testing, the central area of the specimen where the nail penetrates shall be typical of the single thickness exposed area of the shingle under test. For multi-layered shingles, the test shall be performed in the area specified for fastening in the manufacturers'/sellers' instructions.

- 14.5 Conditioning—Condition specimens at the temperatures prescribed in the appropriate product standard for at least two hours prior to testing, and conduct the test at the prescribed temperatures. Other test conditions shall be permitted to give indications of fastener pull-through resistance at different temperatures, provided that the specimens are conditioned for at least 2 h and test at the desired temperature, and that the temperature used is noted in the report.
- 14.6 Nail Placement—Push a 38 mm [ $1\frac{1}{2}$  in.] long galvanized roofing nail with a 9.5 mm [ $\frac{3}{8}$  in.] diameter head through the center of the specimen within  $\pm 5$  mm [ $\pm \frac{1}{4}$  in.] of the intersection of the diagonals of the square specimen (entering from the granule side and exiting at the backsurfacing side as in normal application) such that the head of the nail rests against the granule surface and the shank protrudes from the back surface. The use of a fixture to locate the center of the specimen or to draw diagonals with chalk to facilitate central positioning of the nail is not prohibited.
  - 14.7 Fixture and Sample Assembly:
- 14.7.1 Prepare the specified number of specimens, each with a new nail in position, and condition them to the test temperature as directed by the product standard under investigation as a set prior to testing. When this test method is not

used for determining compliance to a product standard, the test temperature must be reported.

14.7.2 Clamp the base part of the apparatus shown in Fig. 3 into the lower jaws of a constant rate of extension test machine capable of applying a force of at least 450 N [100 lbf] at an extension rate of 100 mm [4 in.]/min.

14.7.3 Position the plate part of the apparatus shown in Fig. 3 over the specimen (with the weather side down) so that the plate is centered over the specimen and with the nail shank protruding through the center of the 64 mm [2½ in.] diameter hole. Place the assembled specimen, nail, and plate into the base part of the apparatus with the nail pointing upwards so that it can be gripped by the upper jaw of the test machine. The arrangement of the assembly ready for test is shown in Fig. 4.

14.7.4 Clamp the nail shank in the upper jaw of the test machine and pull the nail through the specimen at a rate of 100 mm [4 in.]/min. Record the maximum force in N [lbf] to the nearest 0.5 N [0.1 lbf] required to completely pull the head of the nail through the specimen. If a strip-chart recorder is used, choose a scale where the maximum pull-through force is at least 50 % of the full-scale reading.

14.8 *Report*—Report the number of layers and the type of material tested. Report the average and standard deviation of the pull-through force for the ten specimens tested. Report the test conditions and note any special circumstances, for example if the pull-through was performed in an area containing sealant or release tape, or both. Report also the type of fastener used in the test.

14.9 *Precision and Bias*—The following criteria shall be used to judge the acceptability of results at the 95 % confidence level:

#### 14.9.1 Repeatability:

14.9.1.1 *Single-Layer Specimens*—Duplicate results by the same operator on the same sample shall be considered suspect if they differ by more than 15 %.

14.9.1.2 *Multi-Layer Specimens*—Duplicate results by the same operator on the same sample shall be considered suspect if they differ by more than 15 %.

14.9.2 Reproducibility:

14.9.2.1 *Single-Layer Specimens*—The results submitted by each of two laboratories shall be considered suspect if they differ by more than 20 %.

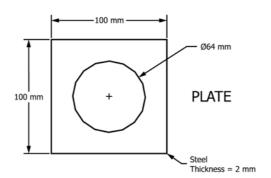
14.9.2.2 *Multi-Layer Specimens*—The results submitted by each of two laboratories shall be considered suspect if they differ by more than 25 %.

14.9.3 *Bias*—This test method for measuring fastener pull-through resistance has no known bias. Test Method D4932/D4932M is testing material in a different manner. The differences in results between these tests have not been investigated.

Note 6—This precision and bias was generated by pulling nails, as described in 14.2, through a fiberglass mat-based shingle and may vary when other materials are tested.

# 15. Preparation and Selection of Small Specimens for Analyses

15.1 Materials of Uniform Composition (Type 1 and Type 2)—Each of the five large specimens selected in Section 9 is to have small test specimens cut for composition analysis. Cut 50-by  $100-\pm 1$ -mm [2- by  $4-\pm \frac{1}{32}$ -in.] small test specimens from each of the large specimens. Compare the equivalent mass per unit area of the small test specimens to the mass per unit area previously generated for the large specimens (Section 9). For products that contain organic felt, four small test specimens must be cut from each large specimen that are within 1.5 % of



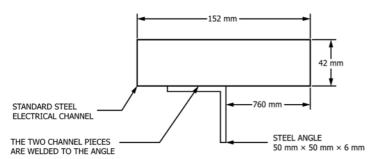
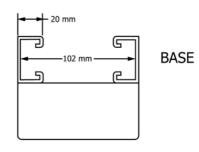


FIG. 3 Base and Plate Parts of Apparatus





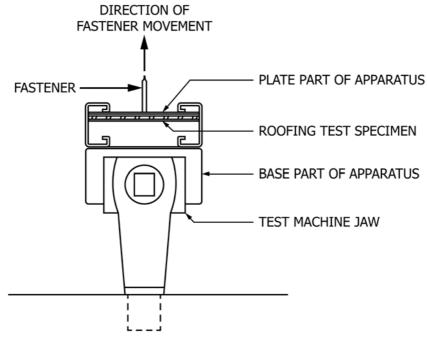


FIG. 4 Bottom Clamp with Sample Installed in Apparatus

the equivalent mass per unit area of the large specimen. For products that contain only glass felt, three small test specimens must be cut from each of the corresponding large specimens that are within 1.5 % of the equivalent mass per unit area of the large specimen. The small test specimens from each large specimen are to be kept together as a unit, separate from the small test specimens cut from the other large specimens. The "top" of each product is the surface that is applied toward the weather.

15.2 Materials of Nonuniform Composition (Type 3)—Cut 50- by 100-  $\pm$  1-mm [2- by 4-  $\pm$   $\frac{1}{32}$ -in.] small test specimens that are representative of the different materials, types of surfacing or thickness present in the five large specimens from Section 9. By proportion, select small specimens that are within 1.5 % of the equivalent mass per unit area of each of the large specimens. Some product standards direct the analysis of the mineral surfaced area only, or the different areas of composition to be analyzed separately. The product standard is to be the final authority on which portions of this material are to be analyzed. The balance of this analytical method assumes that a roofing product of uniform composition is being tested. Use the same procedures for the small specimen representative samples of the nonuniform products. At least five sets of four small representative specimens are required for each composition of products containing organic felts; five sets of three small specimens are required for each composition containing glass felts.

15.3 Materials of Multiple Layer Composition (Type 4)—In the case of multiple layer shingles, determine the mass per unit area and the relative area proportion for each layer configuration (proportion of each configuration for products that have additional combinations of single, double, triple, and so forth, layers). This relative proportion is to be determined from the average surface area measured on four consecutive shingles

from one of the representative samples, measuring only the portion of the shingle that is exposed to the weather when applied in conformance with the manufacturer's application instructions. Cut 50 by  $100 \pm 1$  mm [2 by  $4 \pm \frac{1}{32}$  in.] small test specimens so that they represent the same proportion of each layer configuration as the entire exposed area of that large specimen. At least five sets of four small representative specimens are required for each composition of products containing organic felts; five sets of three small specimens are required for each composition containing glass felts.

Note 7—As an example, if a laminated shingle has some areas that are single thick and some that are double thick, determine what the relative percentages of single thickness and double thickness are for the exposed area of the shingle. The small test specimens shall be cut to represent the same proportion.

15.4 See Fig. 2.

# 16. Analysis of Glass Felt Products

16.1 Total Net Mass—Identify the specimen from each set of three small specimens cut in Section 15 that is the closest to representing the unit mass of the large specimen from which it was obtained. Record the mass of each specimen so selected, to the nearest 0.01 g, as "Total Net Mass." The top section of Table 1 shows the selection process for the five representative specimens for a fiberglass-based product (note the masses for three small specimens).

### 16.2 Total Asphalt:

16.2.1 Wrap each of the small specimens identified in 16.1 in two layers of pre-dried filter paper and secure each wrapped small specimen with a soft copper wire. It is not prohibited to cut the specimens into smaller pieces so that the wrapped specimens fit into the extraction glassware. Use suitable care to ensure that all specimen pieces, fragments, or particles, or combination thereof, as generated by the cutting process, are

TABLE 1 Analysis of Roofing Products that Contain Glass Felts Work Sheet

#### Selection of Representative Specimens (Section 15)

	Small Specimen: Sample	1	2	3	Large Specimen Mass From Section 9				
Α	grams,	22.64	22.59	22.70	22.65				
	percent of mean	100.0	99.7	100.2					
В	grams,	22.84	22.68	22.73	22.74				
	percent of mean	100.4	99.7	100.0					
С	grams,	22.67	22.63	22.60	22.64				
	percent of mean	100.1	100.0	99.8					
D	grams,	22.36	22.31	22.47	22.36				
	percent of mean	100.0	99.8	100.5					
Ε	grams,	22.78	22.89	23.01	22.87				
	percent of mean	99.6	100.1	100.6					
	Note that these are examples of typical data  Analysis (Section 16)								
	Analysis (Section 10)								
	Selected Small Specimen	A1	B3	C2	D1	E2			
Tota	I net mass (16.1)	22.64	22.73	22.63	22.36	22.89			
Total mass + tare (16.2.1)		24.65	25.61	25.00	25.08	24.86			
Extract, residue and tare (16.2.2)		20.61	21.37	20.98	20.94	20.60			
Total asphalt (16.2.3)		4.04	4.24	4.02	4.14	4.26			
Coarse mineral matter (16.3.2)		9.89	9.99	10.16	9.74	10.06			
Fine mineral matter (16.3.2)		7.29	7.90	7.38	7.71	7.06			
Perd	ent FMM in FMM + total asphalt	64.3	65.1	64.7	65.1	62.4			
Glas	s felt (16.4.2)	0.44	0.47	0.46	0.51	0.49			

collected and included in the wrapped specimen. Mark and record the mass of each wrapped specimen, to the nearest 0.01 g [ $2 \times 10^{-5}$  lb], as "Total Mass + Tare."

16.2.2 Extract the asphalt from each specimen from 16.2.1 in a Soxhlet or similar extractor with any suitable solvent (Note 8) until the extract is clear. Dry the extracted specimens in a hood at room temperature. Final dry each specimen in a  $105^{\circ}$ C [221°F]  $\pm$  5% forced draft oven for 60 min  $\pm$  10%. Cool the specimens in a desiccator to room temperature and record the mass of each, to the nearest 0.01 g, as "Extraction Residue + Tare."

Note 8—A "suitable solvent" is any solvent that effectively separates the asphalt from the mineral matter and leaves less than 0.01 g of solvent residue (nonvolatile components) behind when the extracted specimens are dried. Extraction time varies with the solvent used and the composition of the material being extracted. Extraction times in excess of 30 hours are possible.

16.2.3 Record the difference in the mass measured in 16.2.1 and the mass measured in 16.2.2 as the "Total Asphalt."

16.3 Mineral Matter in the Extraction Residue:

16.3.1 One at a time, open each package from 16.2.2 over a nest of No. 6 [3.25-mm], No. 70 [212- $\mu$ m] sieves, and a pan. Retain the glass felt after removing, and putting in the sieve nest, as much of the fine mineral matter that is in or on the glass felt. Dust all mineral matter off the filter papers into the sieve nest and discard the cleaned papers and the wire.

16.3.2 Tap and shake the sieve nest until no change is noted in the contents of each sieve. Record the mass of the material retained on the No. 70 [212-µm] sieve, to the nearest 0.01 g, as the "Coarse Mineral Matter." Record the mass of the material in the pan of the nest, to the nearest 0.01 g, as the "Unadjusted Fine Mineral Matter."

16.4 Felt in Glass Felt Products (Table 2):

**TABLE 2 Report Glass Felt Roofing Shingles** 

Physical Tests	Mean	Standard	95 % Confidence Limits			
		Deviation	Samples		Mean	
Behavior on heating (Section 12)						
Mass loss, %	0.12	0.02	0.07	0.17	0.10	0.14
Appearance change	none					
Composition, Pounds per 100 ft <sup>2</sup>						
Glass felt	1.84	0.096	1.59	2.09	1.72	1.96
Total asphalt	16.43	0.392	15.42	17.44	15.94	16.92
Coarse mineral matter	39.56	0.572	38.09	41.03	38.85	40.27
Fine mineral matter	29.64	1.192	26.57	32.70	28.16	31.11
Fine mineral matter as a percent based on the total asphalt and fine minerals	64.3	1.01	61.7	66.9	63.1	65.6
Moisture, percentage of dry mass	0.0	0.0	0.0	0.0	0.0	0.0

16.4.1 Determine the unadjusted mass of the glass felt from 16.3.1 to the nearest 0.01 g.

16.4.2 Clean the felt from 16.4.1 in an ultrasonic cleaning bath (Note 9), dry the cleaned felt in a forced draft  $105^{\circ}$ C [221°F] oven for 60 min  $\pm$  10 %, and determine its mass to the nearest 0.01 g.

16.4.3 Add the difference between the masses determined in 16.4.1 and 16.4.2 to the mass of the fine mineral matter in 16.3.2.

Note 9—Steps 16.4.2 and 16.4.3 may be omitted if ultrasonic cleaning equipment is unavailable or was not used.

# 17. Analysis of Roofing Products with Organic Felts

17.1 Total Net Mass—Identify the specimen from each set of four small specimens cut in Section 15 that is the closest to representing the unit mass of the large specimen from which it was obtained. Record the mass of each specimen, to the nearest 0.01 g, as "Total Net Mass." The top section of Table 3 shows the selection process for the five representative specimens for an organic-based product (note the masses for four small specimens).

#### 17.2 Total Asphalt:

17.2.1 Wrap each of the small specimens identified in 17.1 in two layers of predried filter paper, and secure each wrapped small specimen with a soft copper wire. Mark and record the mass of each wrapped specimen, to the nearest 0.01 g, as "Total Mass + Tare."

17.2.2 Extract the asphalt from each specimen from 17.2.1 in a Soxhlet or similar extractor with any suitable solvent until the extract is clear. (See Note 8, subsection 16.2.2.) Dry the extracted specimens in a hood at room temperature. Final dry each specimen in a 105°C [221°F]  $\pm$  5 % forced draft oven for 60 min  $\pm$  10 %. Cool the specimens in a desiccator to room temperature and record the mass of each, to the nearest 0.01 g, as "Extraction Residue + Tare."

17.2.3 Record the difference in the mass measured in 17.2.1 and the mass measured in 17.2.2 as the "Total Asphalt."

#### 17.3 Mineral Matter in the Extraction Residue:

17.3.1 One at a time, open and test each package from 17.2.2 over a nest of No. 6 [3.25-mm], No. 70 [212-μm] sieves, and a pan. Dust off into the sieve nest the fine mineral matter that is on the felt. Save the felt recovered for the dry felt determination in 17.4 and the ash of the desaturated felt in Section 18. Dust all mineral matter off the filter papers into the sieve nest and discard the cleaned papers and the wire.

17.3.2 Tap and shake the sieve nest until no change is noted in the controls of each sieve. Record the mass of the material retained on the No. 70 [212- $\mu$ m] sieve, to the nearest 0.01 g, as the "Total Coarse Mineral Matter." Record the mass of the material in the pan of the nest, to the nearest 0.01 g, as the "Total Unadjusted Fine Mineral Matter."

17.4 Unadjusted Total Dry Felt Mass—Dry each felt from 17.3.1 in a  $105^{\circ}$ C [221°F]  $\pm$  5 % forced draft oven for 60 min  $\pm$  10 %, cool in a desiccator, and record the mass as the "Unadjusted Dry Felt." Save the felt for work in Section 18.

17.5 Top Coating Analysis:

17.5.1 *Total Top Mass*—Identify the small specimen of the four cut in Section 15 that is the second closest to representing the unit mass of the large specimen from which it was obtained. (The first was used in 17.1.) Warm the small specimen for not more than 5 min at a temperature of not more than 65°C [150°F], and with a sharp knife or spatula, pull off the back coating and part of the saturated felt in the horizontal plane indicated by the arrow b in Fig. 1.

17.5.2 Repeat all the steps in 17.2 – 17.4 to record the "Total Top Mass," "Total Top Asphalt," "Top Coarse Mineral Matter," "Top Unadjusted Fine Mineral Matter," and "Unadjusted Top Felt."

#### 17.6 Percent Saturation:

17.6.1 Warm the last two of the small specimens of each small specimen set from Section 15 for not more than 5 min at a temperature of not more than  $65^{\circ}$ C [ $150^{\circ}$ F], and with a sharp knife or spatula separate them into three horizontal sections at approximately the planes indicated by the arrows a and b in Fig. 1. Remove both the top and back coatings, with the attached surfacing, and a thin layer of felt, so that a thin layer of asphalt saturated felt core is obtained free of other materials. Discard the top and back coating sections.

17.6.2 Wrap each specimen in one layer of predried filter paper secured with a copper wire. Record the mass of the wrapped felt, to the nearest 0.01 g, as "Saturated Felt + Tare."

17.6.3 Extract each specimen from 17.6.2 in a Soxhlet or similar extractor with any suitable solvent until the extract is clear. (See Note 8, subsection 16.2.2.) Dry each package in a hood to remove most of the solvent, finish the drying in a forced draft vented oven at  $105^{\circ}$ C [221°F]  $\pm$  5% for 60 min  $\pm$  10%, cool in a desiccator, and record the mass, to the nearest 0.01 g, as "Desaturated Felt + Tare."

17.6.4 Carefully unwrap the felt pieces from 17.6.2, redry, cool in a desiccator, and record the mass, to the nearest 0.01 g, as the "Desaturated Felt." Save the felts for the work in Section 18.

#### 18. Ash of Desaturated Felt

18.1 Ash the desaturated felts obtained in 17.4, 17.5.2, and 17.6.4 separately in predried tared crucibles, either over an open flame or in a muffle furnace, until all carbon has been consumed. After cooling, add to each ash approximately five times its mass of saturated ammonium carbonate solution, let digest for 60 min  $\pm$  10 % at room temperature in a covered beaker or crucible, dry in an oven at 105 to 110°C [221 to 230°F] to constant mass, and record the net mass as "ash."

18.2 The percentage of ash in the center portion (from 17.6.4) is assumed to be the true percentage of ash of the felt. The difference between this ash and the percentage of ash of the felts recovered in 17.4 and 17.5.2 is presumed to be included mineral matter from the coating. This percentage difference is converted to mass and added to the mass of fine mineral matter to obtain the "Total Dry Felt" and "Top Dry Felt." The corresponding correction is made to the mass of extracted felt from extraction of total and the top coating analyses.

# TABLE 3 Analysis of Organic Felt Shingle Products Worksheet

Selection of	of Representative	Specimens (Sect	ion <mark>15</mark> )		
					Large Specimen Mass From Sectio
Small Specimen	_1_	_2_	_3_	_4_	9
A grams,	26.05	25.29	25.88	25.55	25.69
percent of mean	101.44	98.4	100.73	99.5	
B grams,	26.56	26.45	24.94	24.36	25.58
percent of mean	103.83	103.4	97.5	95.2	:::
C grams,	23.56	23.64	23.54	24.36	23.78
percent of mean	99.1	99.4	99.0	102.4	
D grams,	26.00	23.45	22.98	26.17	24.65
percent of mean	105.47	95.1	93.2	106.2	
E grams, percent of mean	24.94 97.3	26.03 101.5	26.05 101.59	25.55 99.6	25.64
Analys	sis Unadjusted To	otal Specimen (17	.2)		
Selected Small Specimen, g/5000 mm <sup>2</sup>	_A4	_B3_	C2	_D2_	_E4_
T. 1				·	
Total net mass + tare (17.2.1)	28.55	27.94	26.64	26.45	28.55
Extract, residue + tare (17.2.2)	20.70	20.21	19.49	18.47	20.62
Total asphalt (17.2.3)	7.85	7.73	7.15	7.98	7.93
Coarse mineral matter (17.3.2)	8.71	8.25	8.01	6.93	8.34
Unadjusted fine mineral matter	6.32	6.17	5.94	5.77	6.56
Unadjusted dry felt (17.4)	2.67	2.79	2.54	2.77	2.72
Anal	ysis Unadjusted	Top Coating (17.5	)		
Selected Small Specimen With Back Coating and Attached Felt Removed, g/5000 mm <sup>2</sup>	A	<u> </u>	C	D	E
Top mass + tare	18.51	18.54	17.99	18.47	19.1
Extract, residue + tare	12.99	13.52	13.27	13.37	14.01
Top specimen asphalt	5.52	5.02	4.72	5.10	5.09
Coarse mineral matter	5.66	6.16	6.20	5.93	6.55
Unadjusted fine mineral matter	2.30	2.61	2.38	2.65	2.76
Unadjusted top dry felt	2.03	1.75	1.69	1.78	1.70
	Percent Satur	ation (17.6)			
Selected Small Specimen With Front and Back Coating	A	B	C	D	E
Removed, g/5000 mm <sup>2</sup>					
Saturated felt + tare	7.07	7.94	8.42	9.6	10.57
Felt + tare	4.72	4.70	4.86	5.07	5.81
Dry Felt	1.72	1.70	1.86	2.07	2.81
Percent Saturation	136.62	190.5	191.39	218.8	169.39
Saturated felt + tare	8.04	8.08	7.35	8.89	10.09
Felt + tare	4.68	4.84	4.45	5.58	5.63
Dry felt	1.68	1.84	1.45	2.58	2.63
Percent saturation	200.0	176.1	200.0	128.3	169.5
Mean percent saturation	168.31	183.3	195.69	173.5	169.48
Percent	Ash in the Extra	cted Felt (Section	18)		
Specimen	A	B	C	D	E
Total specimen	13.35	11.04	13.41	11.41	10.37
Top specimen	9.37	11.43	10.72	21.08	11.86
Percent saturations	6.63	5.15	5.21	8.66	8.11
	Correction				
Total specimen	6.72	6.88	8.2	2.75	2.75
Top openings	2.74	6.00	E E 1	10.40	2.05

# 19. Calculation

Top specimen

19.1 Use the adjusted mass of the dry felt from each total sample to calculate the mass of the dry felt in  $g/m^2$  [lb/100 ft<sup>2</sup>] (Note 10).

Note 10—Multiply the mass in grams by 3.97 to obtain the mass in lb/100 ft<sup>2</sup> for a 2- by 4-in. sample. Use 200 as the factor to obtain  $g/m^2$ .

12.42

3.85

5.51

6.28

2.74

- 19.2 Calculate the percent saturation in each small sample as 100 times the difference between the masses in 17.6.2 and 17.6.3 divided by the mass in 17.6.4. Use the mean of at least two determinations in all further calculations.
- 19.3 Calculate the mass of the saturant by multiplying the dry felt mass in 19.1 by the mean percent saturation from 18.2 divided by 100.
- 19.4 Calculate the mass of the saturant in the top coating analysis by multiplying the adjusted mass of the top dry felt by the mean percent saturation divided by 100. Convert the mass calculated to the mass in g/m<sup>2</sup> [lb/100 ft<sup>2</sup>].
- 19.5 Calculate the mass of asphalt in each total sample by converting the mass in 17.2.3 to g/m<sup>2</sup> [lb/100 ft<sup>2</sup>].
- 19.6 Calculate the mass of asphalt in each top sample by converting the mass in 17.5.2 to g/m<sup>2</sup> [lb/100 ft<sup>2</sup>].
- 19.7 Deduct the saturant mass in 19.3 from the asphalt mass in the total sample in 19.5 to obtain the total unfilled asphalt coating.
- 19.8 Deduct each saturant mass in 19.4 from each top asphalt mass in 19.6 to obtain each total unfilled top coating.
- 19.9 Deduct each top coating mass in 19.8 from each total unfilled coating mass in 19.7 to obtain each back unfilled coating mass.
- 19.10 Convert each mass of the total coarse mineral matter and the total unadjusted fine mineral matter from 17.3.2 to  $g/m^2$  [lb/100 ft<sup>2</sup>].
- 19.11 Convert each mass of the top coarse mineral matter and the top unadjusted fine mineral matter from 17.5.2 to  $g/m^2$  [lb/100 ft<sup>2</sup>].
- 19.12 Deduct each mass of the top coarse mineral matter in 19.11 from the total coarse mineral matter in 19.10 to obtain the unadjusted mass of the bottom surfacing.
- 19.13 Deduct each mass of the top unadjusted fine mineral matter in 19.11 from each total unadjusted fine mineral matter in 19.10 to obtain the unadjusted mass of the fine mineral matter in the back coating.

# 20. Adjusting Back Coating Fine Mineral Matter and **Back Surfacing**

20.1 Fine mineral matter from the back surfacing frequently includes fine mineral matter from the back coating. To adjust both the back coating fine mineral matter and the back surfacing, assume that the percent of the fine mineral matter, based on the combined masses of the top asphalt coating and the top fine mineral matter, is a constant. Adjust the fine mineral matter in the back coating by reducing it to the same percentage of fine mineral matter as found in the top filled coating. Add the excess fine mineral matter to the "Total Coarse Mineral Matter" less the "Top Coarse Mineral Matter" to obtain the "Back Surfacing" (Note 11).

Note 11-The accuracy of this procedure depends on the screen

grading of the back surfacing used on the product, and a negative quantity of back surfacing is sometimes determined with these test methods as a result of normal mass variations between specimens.

## 21. Report

- 21.1 Report the data on a form similar to Tables 1 and 2 for Type 1 products and Tables 3 and 4 for Types 2 and 3 products or in any manner convenient to the user. Report all percentages to the nearest 0.1 %. Report all mass per unit area data to the nearest 0.5 g/m<sup>2</sup> [0.01 lb/100 ft<sup>2</sup>].
- 21.2 Tables 1-4 show the sources of the data and some of the steps in the calculations from the raw data.

#### 22. Precision and Bias

- 22.1 *Precision*—Interlaboratory round robin tests show that the variation within a product can be greater than the variation between laboratories. Data from two laboratories can be compared statistically using the following procedure:
  - 22.1.1 Calculate the mean variance of each set of data using:

$$V = \frac{s^2}{n} \tag{4}$$

where:

V = mean variance.

s = estimated standard deviation, and

= number of samples.

22.1.2 Calculate the "effective number of degrees of freedom" using:

$$f = \frac{(V_A + V_B)^2}{\frac{(V_A)^2}{n_A + 1} + \frac{(V_B)^2}{n_B + 1}} - 2$$
 (5)

where:

= effective number of degrees of freedom,

f = effective number of degrees of freedom,  $V_A$  and  $V_B$  = mean variance of each data set as calculated in 21.1, and

 $n_A$  and  $n_B$  = number of samples in each set.

- 22.1.3 Look up the value for t in Table 5, the student's t-distribution for the effective number of degrees of freedom at a significance level of 0.05.
- 22.1.4 Compute the maximum probable difference between the averages of each set using:

$$u = {}^{t}0.975 \times (V_{A} + V_{B})^{0.5} \tag{6}$$

- 22.1.5 There is no significant difference between the data sets if the difference between the averages of each set is less
- 22.1.6 In case of dispute, if the difference between the data averages is significant, retesting by both laboratories, or testing by an independent referee laboratory, is recommended.
  - 22.2 Bias—There is no known bias in these test methods.

#### 23. Keywords

23.1 analysis; asphalt; cap sheets; composition; physical testing; roll roofing; sampling; shingles

#### **TABLE 4 Total Analysis (With All Adjustments)**

Small Specimen					
Pounds per 100 Square Feet	Α	В	C	D	E
Dry felt	9.88	10.42	9.25	10.69	10.55
Saturant	16.64	19.10	18.11	18.55	17.88
Top surfacing	22.46	24.44	24.60	23.53	25.98
Top coating	18.07	18.78	16.14	20.84	20.42
Top coating asphalt	8.72	7.99	6.33	9.44	9.21
Top coating filler	9.35	10.78	9.81	11.40	11.21
Back coating	12.02	8.43	10.04	8.11	9.72
Back coating asphalt	5.80	3.58	3.94	3.67	4.38
Back coating filler	6.22	4.84	6.10	4.43	5.34
Back surfacing	22.33	17.80	15.66	11.34	16.83
Total Net mass	101.39	98.97	93.81	93.06	101.38
Average Total Analysis					
	<u>Mean</u>	Sample Range	p = 0.05	Range of Mean	ns, $p = 0.05$
Pounds per 100 Square Feet		min	max	min	max
Dry felt	10.16	8.80	11.52	9.50	10.82
Saturant	18.06	15.94	20.18	17.03	19.08
Top surfacing	24.21	21.18	27.23	22.75	25.67
Top coating	18.85	14.49	23.21	16.75	20.95
Top coating asphalt	8.34	5.46	11.22	6.95	9.73
Top coating filler	10.51	8.46	12.57	9.52	11.51
Back coating	9.66	6.08	13.23	7.94	11.38
Back coating asphalt	4.27	2.19	6.36	3.27	5.28
Back coating filler	5.39	3.60	7.17	4.52	6.25
Back surfacing	16.79	7.68	25.90	12.39	21.18
Total Net Weight	97.723	88.42	107.03	93.23	102.22

TABLE 5 Student's t-Distribution, 0.05 Significance Level

f	<sup>t</sup> 0.975	f	<sup>t</sup> 0.975	f	<sup>t</sup> 0.975
6	2.447	11	2.201	16	2.120
7	2.365	12	2.179	17	2.110
8	2.306	13	2.160	18	2.101
9	2.262	14	2.145	19	2.093
10	2.228	15	2.131	20	2.086

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