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(14 Pages)

BITUMINOUS MATERIAL CONTENT OF ASPHALTIC CONCRETE MIXTURES BY THE NUCLEAR METHOD

(An Arizona Method)

SCOPE

1. (a) This procedure describes a method for determining the percent bituminous material content of asphaltic concrete mixtures, by testing a sample with a device that utilizes neutron thermalization techniques.

(b) Modifications to this procedure for Asphaltic Concrete Friction Course, Asphaltic Concrete (Asphalt-Rubber), and Asphaltic Concrete Friction Course (Asphalt-Rubber) are given in Section 7.

(c) This test method involves hazardous material, operations, and equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

(d) Safety procedures for operation, transport, and storage of nuclear gauges shall be in accordance with the manufacturer's recommendations and the applicable regulations of the Arizona Radiation Regulatory Agency (ARRA).

(e) If recommended by the manufacturer, equipment operators should wear an approved form of radiation dosimetry film badge capable of monitoring the occupational radiation exposure.

(f) See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

(g) Metric (SI) units and values are shown in this test method with English units and values following in parentheses. Values given for metric and English units may be numerically equivalent (soft converted) for the associated units, or they may be given as rounded or rationalized values (hard converted). Either the metric or English units along with their corresponding values shall be used in accordance with

applicable specifications. See Appendix A2 of the Materials Testing Manual for additional information on the metric system.

APPARATUS

2. Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus for this test procedure shall consist of the following:

(a) Nuclear asphalt content gauge and manufacturer's instruction manual. While exact details of the manufacture of the apparatus may vary, the system shall consist of the following items and meet the criteria for variability given below. The equipment shall be so constructed as to be licensable in accordance with applicable health and safety regulations.

1) Neutron Source - An encapsulated and sealed radioactive source such as "Americium-241: Beryllium", or "Californium-252".

2) Detectors - Any type of thermal neutron detector, such as helium-3 or boron tri-floride.

3) Read-Out Instrument, such as a scaler or a direct-reading digital device calibrated in percent asphalt cement.

4) A minimum of four standard stainless steel sample pans [approximately 230 mm long by 185 mm wide by 100 mm deep (9" x 7-1/4" x 4")].

5) The variability of the gauge shall be no greater than $\pm$ 0.05 percent asphalt for a four-minute count. The variability shall be determined utilizing a three point calibration for a conventional asphaltic concrete mix with a design asphalt content of approximately five percent. The variability of the gauge is determined from the slope of the calibration curve and the standard deviation of the measured counts of the calibration sample at the mix design percent asphalt. If the gauge fails to meet the variability requirement shown above for a four-minute count, the variability may be determined utilizing either 8-minute or 16-minute counts as necessary. If the variability is determined using an increased count time, that count time is the minimum that is to be used for testing subsequent field samples [See paragraphs 6(g) and 6(i)]. In lieu of an increased count time, a new set of calibration samples may be prepared and tested with greater care. If the gauge fails to meet the variability requirement after increased count times or preparation of additional calibration samples, the gauge must be returned to the manufacturer for necessary repair and recalibration. Variability is

calculated as shown below. The variablity of the gauge should be checked at minimum 12 month intervals. Documentation of the variability determination shall be kept on file.

$$V = \frac{S.D.}{S}$$

Where: V = apparatus variability, in percent asphalt.
S = slope of the calibration curve, in counts (y-axis) per percent asphalt cement (x-axis). [See note below.]
S.D. = standard deviation, in counts. The standard deviation is calculated from 20 individual four-minute readings (8-minute or 16-minute counts if used) taken on the calibration sample at the mix design percent asphalt content.

NOTE: The slope of the calibration curve, as calculated and given by the gauge may <u>not</u> be the actual numerical value for the slope, and in addition, the slope may <u>not</u> be given in counts (y-axis) per percent asphalt (x-axis). For instance, the Troxler Nuclear Asphalt Content Gauge (Model 3241-C) gives a numerical value for slope which is actually 1000 times the actual slope, and in addition, the value given by the Troxler gauge is given for percent asphalt (y-axis) per counts (x-axis). The following is an example of the slope of the calibration curve as given by the Troxler gauge and the determination of the corrected slope value to be used in the Apparatus Variability equation given above:

Troxler gauge reported slope = 5.220075
[Slope based on percent asphalt (y-axis) per counts (x-axis).]

Determination of corrected slope:

$$\frac{5.220075}{1000} =$$

0.005220075 (percent asphalt/counts)

Reciprocal of slope to obtain slope based on counts (y-axis) per percent asphalt (x-axis):

$$\frac{1}{0.005220075} =$$

191.568 (counts/percent asphalt)

In this case, the value of 191.568 is the slope of the calibration curve and is the value which is to be used for "S" in the equation for Apparatus Variability given above.

(b) Scale(s) or balance(s) capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except the readability and sensitivity of any balance or scale utilized shall be as shown below:

1) The scale or balance to be used for preparing calibration samples shall have a readability and sensitivity of at least 1 gram.

2) The scale or balance to be used for testing field samples shall have a readability and sensitivity of at least 5 grams.

(c) Oven(s), capable of heating and maintaining temperatures to 177 °C (350 °F). Two ovens may be required.

(d) Steel straightedge, approximately 300 mm (12 inches) in length.

(e) Either a piece of plywood of at least 20 mm (3/4 inch) nominal thickness, or a metal plate of at least 6 mm (1/4 inch) nominal thickness, having an area slightly larger than the top of the sample pans.

(f) Mixing apparatus - Mechanical mixing is recommended; 19 liter (20 quart) capacity mixer is required. (Hand mixing may be performed if desired.)

(g) Miscellaneous pans, bowls, spoons, and spatulas.

(h) Thermometer with temperature range 10 to 260 °C (50 to 500 °F).

(i) Hot plate(s) capable of heating and maintaining temperatures to 177 °C (350 °F).

(j) Flat bottom metal scoop at least 50 mm wide by 150 mm long (2 inches x 6 inches).

PRECAUTIONS

3. (a) Since nuclear equipment measures the total amount of hydrogen in the sample, this procedure is sensitive to changes in moisture content. It must be remembered that both bituminous material and water contain hydrogen.

(b) Keep any other source of neutron radiation at least 10 meters (30 feet) from the equipment.

(c) Do not place the equipment where the amount of hydrogenous material may change during the calibration or testing procedures. Hydrogenous materials are those containing hydrogen, for example, water or plastic materials.

(d) Moving the gauge to a new location may have an effect on the results. Therefore, a new background count and/or calibration is required if the gauge is moved. If the original calibration specimens are retested, they shall be heated in an oven at 121 to 149 °C (250 to 300 °F) for at least one hour to drive out any condensation or absorbed moisture and to bring them to testing temperature.

SAMPLING

4. (a) For preparing calibration samples, aggregate samples are obtained in accordance with Arizona Test Method 105. Samples shall be adequately dried, if necessary, to a free-flowing condition in the portion passing the 4.75 mm (No. 4) sieve. A maximum of approximately 45 kg (100 pounds) of aggregate, representative of the mix design gradation, will normally be required.

(b) When testing field samples of Asphaltic Concrete, a sample of the freshly produced mix is obtained in accordance with Arizona Test Method 104.

CALIBRATION

5. (a) A calibration must be performed for each asphaltic concrete mixture that is to be used on the project. Test results on asphaltic concrete mixtures may vary with changes in the type and gradation of aggregate, (percentage and source of

mineral admixture, if used), and percentage and source of bituminous material. Accordingly, a calibration curve must be developed for each mix design. The calibration curve shall be established with three or more points. The calibration procedure consists of preparing and testing one dry aggregate blank sample, and a minimum of three asphaltic concrete calibration samples at varying bituminous material contents.

(b) Before a calibration is performed, a 16-minute background count shall be taken and recorded.

(c) For the dry aggregate blank sample, weigh up one approximate 10 kg (22 pound) aggregate sample representative of the mix design aggregate gradation. Thoroughly blend the aggregate and dry to constant weight at 121 to 149 °C (250 to 300 °F), and then place in a tared sample pan in at least two approximately equal depth layers. After placing each layer in the pan, gently spade the aggregate with the scoop or wide blade-like tool. The coarse and fine material shall be uniformly distributed. Too much manipulation of the material can result in segregation of the material, causing the fines to migrate to the bottom. Fill the pan slightly above the top rim. Place the straightedge firmly across the rim and, using a sawing motion, strike off the surface of the sample so that it is flush with the rim. Gaps between the straightedge and the sample shall be filled with fine aggregate and the sample leveled. Do not further compact the sample. Determine and record the weight of the dry aggregate blank sample to the nearest 5 grams.

(d) Place the dry aggregate blank sample in an oven at 121 to 149 °C (250 to 300 °F). A count will be determined on this sample, as described in paragraph 5(l), at the completion of the testing of the asphaltic concrete calibration samples.

(e) For the asphaltic concrete calibration samples, weigh up a minimum of three aggregate samples representative of the mix design aggregate gradation. These aggregate samples shall weigh at least 500 grams more than the dry aggregate blank sample weight determined in paragraph 5(c). If mineral admixture is used, the appropriate type and amount shall be added to the aggregate and thoroughly blended. Dry the samples to constant weight at the mixing temperature required as shown in paragraph (g) below.

(f) Bituminous material of the same source, grade, and type as that which will be used in production of the asphalt concrete mixture shall be used in the calibration samples. Normally three samples are prepared; one at the design bituminous material content, one at 1.0% above, and one at 1.0% below. The percent of bituminous material is based on the weight of total mix. The weight of bituminous material to be added for each percent of bituminous material is determined by the following:

$$\left[\begin{array}{c}\text{Weight of}\\ \text{Bituminous}\\ \text{Material}\end{array}\right] = \frac{\left[\begin{array}{c}\text{Weight of Aggregate}\\ \text{(and mineral admixture when used)}\end{array}\right]}{100 - \left[\begin{array}{c}\text{Percent of}\\ \text{Bituminous}\\ \text{Material}\end{array}\right]} \times \left[\begin{array}{c}\text{Percent of}\\ \text{Bituminous}\\ \text{Material}\end{array}\right]$$

(g) All bowls, sample pans, and mixing tools shall be heated to approximately 149 °C (300 °F). It is recommended that a "butter mix" be utilized to condition the mixing equipment. If a "butter mix" is utilized, the mixing equipment shall be scraped consistently clean with a spatula after each batch. As an alternate to a "butter mix", the mixing equipment shall be scraped as clean as possible with a spatula after each batch. All samples shall be mixed at the same mixing temperature, ± 6 °C (10 °F). During mixing, a hot plate shall be placed under the mixing bowl to reduce loss of heat while mixing. The sample shall be sufficiently mixed to ensure thorough coating. Unless otherwise indicated on the mix design, the temperature of the bituminous material and aggregate (and mineral admixture when used) at the time mixing begins shall be in accordance with the following:

BITUMINOUS MATERIAL	TEMPERATURE RANGE
AC 10, AC-20	149 ± 6 °C (300 ± 10 °F)
AC-30, AC-40	152 ± 6 °C (305 ± 10 °F)
PG 58-XX, PG 64-XX	149 ± 6 °C (300 ± 10 °F)
PG 70-XX, PG 76-XX	152 ± 6 °C (305 ± 10 °F)
Polymer Modified Asphalt	160 ± 6 °C (320 ± 10 °F)
Asphalt-Rubber	163 ± 6 °C (325 ± 10 °F)

(h) At the time of beginning the fabrication of each asphaltic concrete calibration sample, the mixture shall be 121 to 149 °C (250 to 300 °F). The mix with the lowest bituminous material content shall be prepared first. Fill the tared sample pan in approximately equal depth layers (two layers minimum), uniformly distributing the sample in the pan. After placing each layer in the pan, gently spade the mix with a spatula, wide blade-like tool, or scoop and lightly press the mix down. Add mix until the top of the material is mounded approximately 25 to 38 mm (1 to 1-1/2 inches) above the top of the pan. Using a spatula or trowel, round the top of the mixture so that it is at the interior edges of the pan but not on the top rim of the pan. Use the metal plate or plywood to consolidate the asphaltic concrete mixture. If necessary, this can be done by placing the pan on a clean floor, placing the metal plate or plywood on top of the sample pan and standing on the plate. If desired, a piece of waxed paper or other suitable material may be placed on the top of the mix to avoid sticking. If necessary, fill any voids and consolidate the mix with the metal plate of plywood until the pan is

completely full and the mix is even with the top of the pan. Determine and record the weight of this asphaltic concrete calibration sample to the nearest gram. This weight is identified as the asphaltic concrete calibration sample weight and is used (± 5 grams) for the remaining calibration samples.

(i) Place the sample in the gauge and take a 16-minute count. Record the measured count.

(j) Repeat steps in paragraphs 5(h) and 5(i) to fabricate and test the remaining calibration samples. These samples shall be fabricated in the same manner as the first calibration sample and shall be within ± 5 grams of the asphaltic concrete calibration sample weight. If samples are not able to be tested immediately after fabrication, they shall be placed in an oven at 121 to 149 °C (250 to 300 °F) until they can be tested.

(k) Prepare a calibration curve in accordance with the manufacturer's calibration instructions. On some gauges this curve may be generated internal to the gauge. To be considered acceptable, a calibration curve should have a correlation coefficient greater than or equal to 0.995.

$$\text{Correlation Coefficient} = \sqrt{1 - \frac{\Sigma (y - y')^2}{\Sigma (y - \overline{y})^2}}$$

Where: y = Actual percent bituminous material value for each sample.
y' = Calculated percent bituminous material value for each sample, from curve.
$\overline{y}$ = Mean of the actual percent bituminous material values for all samples.

(l) Remove the dry aggregate blank sample from the oven. Place in the gauge take a minimum four-minute count. This count is recorded and may be used to determine if significant changes occur in the aggregate during asphaltic concrete production. [See paragraph 6(i).]

(m) Once a calibration has been performed for the asphaltic concrete mixture, other gauges from the same manufacturer may use a cross calibration for testing of the same asphaltic concrete mixture. Cross calibrations shall be performed in accordance with the gauge manufacturer's recommendations, and shall have been done within the preceding 12 months. In cases of dispute, the gauge derived calibration shall be the referee method. A new cross calibration is required after any gauge repair.

PROCEDURE (FIELD TEST SAMPLE)

6. (a) A 16-minute background count shall be taken and recorded each day before any test samples are run.

(b) Obtain a representative sample for determining the bituminous material content. Generally 8 kg (18 pounds) should be adequate.

(c) If desired, the entire sample for determining the bituminous material content may be dried to constant weight in an oven at 143 ± 6 °C (290 ± 10 °F) in lieu of obtaining a moisture content sample as described in paragraph (d) or (h) below. If a moisture sample is obtained, the percent moisture is determined in accordance with Arizona Test Method 406. The percent moisture is subtracted from the gauge percent bituminous material to obtain the corrected (actual) percent bituminous material.

(d) If the entire sample for determining the bituminous material content has not been dried to constant weight as described in paragraph (c) above, or the moisture content sample is not to be taken at the completion of testing of the bituminous material content sample as described in paragraph (h) below, a moisture content sample is taken immediately after the fabrication of the bituminous material content test sample as described in paragraph (e) below.

(e) If the field sample is at fabrication temperature at the time of obtaining the bituminous material content test sample, the bituminous material content test sample is fabricated immediately. If the field sample is not at fabrication temperature, enough material for both the bituminous material content test sample, and a moisture content sample when necessary, shall be obtained and placed in a single pan and brought to fabrication temperature.

(f) At the time of beginning the fabrication of the bituminous material test sample, the mixture shall be at a temperature of 121 to 149 °C (250 to 300 °F). Fill the tared sample pan in approximately equal depth layers, using the same number of layers as was used in fabrication of the calibration samples. Uniformly distribute each layer of the sample in the pan. After placing each layer in the pan, gently spade the mix with a spatula, wide blade-like tool, or scoop and lightly press the mix down. When the pan is nearly full, weigh the sample. Add enough mix until the sample weight is within ± 10 grams of the asphaltic concrete calibration sample weight as determined in paragraph 5(h). If necessary, the mix may be pressed down using the metal plate or plywood during addition of material. Using a spatula or trowel, round the top of the mixture so that it is at the interior edges of the pan but not on the top rim of the pan. Use the metal plate or plywood to consolidate the asphaltic concrete mixture until it is even with the top rim of the pan. If necessary, this can be done by placing the pan on a

clean floor, placing the metal plate or plywood on top of the sample pan and standing on the plate. If desired, a piece of waxed paper or other suitable material may be placed on the top of the mix to avoid sticking. Reweigh the sample, and assure the weight is within ± 10 grams of the asphaltic concrete calibration sample weight. Record the weight of the test sample to at least the nearest 5 grams.

(g) Immediately after fabrication place the sample in the gauge. Take and record a minimum four-minute count, and determine the gauge percent bituminous material. If an increased count time (8-minute or 16-minute) was used to determine the variability of the gauge [paragraph 2(a)(5)], that count time is the minimum that is to be used.

(h) If the bituminous material content sample has not been dried to constant weight prior to testing, or if the moisture content sample has not been taken at the time of fabrication of the bituminous material content test sample, a representative 1000 ± 50 gram moisture content sample shall be immediately taken from the bituminous material content test sample upon determination of the bituminous material percent in the gauge.

NOTE: Generally the samples taken for bituminous material content and moisture content are obtained from a larger field sample from which material for other tests, e.g., Marshall and Rice is also split out. In case there is a need for additional testing, it may be desirable to save material from the bituminous material content test sample along with excess material from the field sample.

(i) If desired during asphaltic concrete production, a dry aggregate blank sample may be prepared and tested to ensure that changes in aggregate do not occur unnoticed. Testing may be performed at any time that a change in the aggregate is suspected. This sample shall be dried to constant weight and fabricated in the manner described in paragraph 5(c). The weight of this sample shall be within ± 10 grams of the weight of the dry aggregate blank sample as determined in paragraph 5(c). Place sample in the gauge and take and record a minimum four-minute count. If an increased count time (8-minute or 16-minute) was used to determine the variability of the gauge [paragraph 2(a)(5)], that count time is the minimum that is to be used. If a significant change is noted in this count [greater than ± 1.0% of the calibration blank count determined in paragraph 5(l)], a new calibration may be warranted. The project supervisor should be notified immediately of a possible change in aggregate, pit conditions, and/or moisture retained in the aggregate.

MODIFICATIONS FOR ASPHALTIC CONCRETE FRICTION COURSE, ASPHALTIC CONCRETE (ASPHALT-RUBBER), AND ASPHALTIC CONCRETE FRICTION COURSE (ASPHALT-RUBBER)

7. The following modifications apply for Asphaltic Concrete Friction Course, Asphaltic Concrete (Asphalt-Rubber), and Asphaltic Concrete Friction Course (Asphalt-Rubber).

(a) In paragraph 3(d), the last sentence is changed to read: If the original calibration specimens are retested, they shall be heated in an oven at 104 ± 11 °C (220 ± 20 °F) for Asphaltic Concrete Friction Course, or at 149 ± 14 °C (300 ± 25 °F) for Asphaltic Concrete (Asphalt-Rubber) or Asphaltic Concrete Friction Course (Asphalt-Rubber), for at least one hour to drive out any condensation or absorbed moisture and to bring them to testing temperature.

(b) In the calbration procedure given in Section 5, the following changes are made:

1) In paragraph 5(g), a "butter mix" shall be used for preparing calibration samples.

2) In paragraph 5(h):

a) At the time of beginning the fabrication of calibration samples for Asphaltic Concrete Friction Course, the temperature of the mixture shall be 104 ± 11 °C (220 ± 20 °F).

b) At the time of beginning the fabrication of calibration samples for Asphaltic Concrete (Asphalt-Rubber) or Asphaltic Concrete Friction Course (Asphalt-Rubber), the temperature of the mixture shall be 149 ± 14 °C (300 ± 25 °F).

c) For Asphaltic Concrete Friction Course or Asphaltic Concrete Friction Course (Asphalt-Rubber), instead of adding mix until the top of the material is mounded approximately 25 to 38 mm (1 to 1-1/2 inches) above the top of the pan, mix shall be added so that the top of the mix is mounded slightly above the top of the pan.

3) In paragraph 5(j), the last sentence is changed to read: If samples are not able to be tested immediately after fabrication, they shall be placed in an oven at 104 ± 11 °C (220 ± 20 °F) for Asphaltic Concrete Friction Course, or at 149 ± 14 °C (300 ± 25 °F), for Asphaltic Concrete (Asphalt-Rubber) or Asphaltic Concrete Friction Course (Asphalt-Rubber).

(c) Paragraph 4 (b) is changed to read: When sampling Asphaltic Concrete Friction Course or Asphaltic Concrete Friction Course (Asphalt-Rubber), an adequate amount of material shall be taken from the truck at the mixing plant and placed into a 19 liter (5 gallon) bucket, or other suitable container, which has been conditioned with a "butter mix". The sample shall be taken at random locations, approximately 300 mm (12 inches) below the surface, within 5 minutes from the time the loading of the truck is completed. Representative samples of Asphaltic Concrete (Asphalt-Rubber) may be obtained either from the truck at the mixing plant, the windrow, or in accordance with Arizona Test Method 104.

(d) In the procedure for testing field samples (Section 6), the following changes are made:

1) At the beginning of the fabrication of field samples of Asphaltic Concrete Friction Course, the temperature of the mixture shall be 104 ± 11 °C (220 ± 20 °F).

2) At the beginning of the fabrication of field samples of Asphaltic Concrete (Asphalt-Rubber) or Asphaltic Concrete Friction Course (Asphalt-Rubber), the temperature of the mixture shall be 149 ± 14 °C (300 ± 25 °F).

CALIBRATION REPORT

8. The calibration report shall contain, as a minimum, the following information:

(a) Make, model, and serial number of the asphalt content gauge.

(b) ADOT project number.

(c) Type of mix.

(d) Calibration number.

(e) Name of test operator.

(f) Identification (type and source) of bituminous material and aggregate materials, (and mineral admixture when used).

(g) Calibration date and background count.

(h) Correlation coefficient of the calibration curve (Fit Coeff.).

(i) Weight and measured count of the dry aggregate blank sample prepared and tested during the calibration prodedure.

(j) Mix design percent bituminous material content.

(k) Percent of mineral admixture if used.

(l) Percentages of bituminous material in calibration samples.

(m) Weight of asphaltic concrete calibration samples.

(n) Measured count of each asphaltic concrete calibration sample.

FIELD SAMPLE TEST REPORT

9. The field sample test report shall, as a minimum, contain the following information:

(a) ADOT project number.

(b) Type of mix.

(c) Calibration number.

(d) Mix design percent bituminous material content.

(e) Date and location of field test sample.

(f) Test number and lot number, if applicable.

(g) Field sample test date and name of test operator.

(h) Background count.

(i) Weight of test sample.

(j) Count time (minutes) of test.

(k) Measured count of test sample.

(l) Gauge measured percent bituminous material.

(m) Percent moisture.

(n) Corrected (actual) percent bituminous material.

(o) When a dry aggregate blank sample is prepared and tested, the following information is also reported:

1) Weight and measured count of the dry aggregate blank sample prepared and tested during the calibration prodedure.

2) Weight and measured count of dry aggregate blank sample prepared and tested during production.

3) Percentage change in counts from the calibration dry aggregate blank sample count.