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METHOD OF TEST FOR DETERMINATION OF BINDER CONTENT OF HOT MIX ASPHALT BY THE IGNITION METHOD

A. SCOPE

This test method describes the procedure to determine the binder content of hot mix asphalt (HMA) by removing the binder via pyrolysis. HMA samples are placed in a furnace and heated past the autoignition point of the binder. The binder is ignited and burned off leaving the aggregate intact.

The type of aggregate in the HMA may affect the results of this test method. Different aggregates may lose weight, to varying degrees due to the pyrolytic action. Accordingly, a correction factor is determined for each type of HMA. A correction factor must be developed for each furnace. This test method employs equipment requiring specific manufacturer operational procedures.

B. REFERENCES

California Test 125 — Sampling Highway Materials and Products Used in the Roadway Structural Sections
California Test 201 — Soil and Aggregate Sample Preparation
California Test 226 — Determination of Moisture Content of Soils and Aggregates by Oven Drying
California Test 304 — Preparation of HMA for Test Specimens
California Test 370 — Determining Moisture Content of Bituminous Mixtures or Graded Mineral Aggregates Using Microwave Ovens

C. APPARATUS

Ignition Furnace, A forced air ignition furnace that heats the sample by either convection method or direct irradiation method. The convection-type furnace must have a minimum temperature capability of 878°F (470°C) and a temperature control capability of ± 9°F (± 5°C). The convection-type furnace must have a blower/fan to move air within the ignition chamber in order to expedite the materials testing and to minimize any smoke or emissions that may be discharged into the laboratory during binder removal. The furnace must also incorporate a means to minimize or eliminate emissions during binder removal.

The furnace must accommodate sample sizes of at least 3000 g.

The ignition furnace must be Type 1.

The Type 1 ignition furnace must have an internal scale (accurate to 0.1 g) capable of measuring the combined weights of the sample and basket assembly. The furnace must have a data collection/processing system that permits the input of factory calibration data and a correction factor. It must also be able to determine and display the weight loss during the test, detect the end point (i.e., when the weight loss during a 3 min ignition interval does not exceed

0.01 % of the initial sample weight), and employ an audible alarm and/or visual display system and/or indicator lights. The furnace must also provide a printout (or ticket) with the elapsed time, temperature, initial sample weight, sample weight loss, correction factor, corrected binder content, and factory temperature compensation. Sample weight after ignition may be verified by weighing on an external scale.

Scale, with a minimum capacity of 6000 g and capable of measuring the combined weighs of the sample and basket assembly accurate to 0.1 g.

Temperature Probe, suitable for measuring sample temperatures within the sample basket(s) or within mixing bowls.

Sample Basket(s), of a size that allows the sample to be spread in relatively thin layers, and that allows air to flow through and around the sample's particles. The sample basket(s) must be stackable and completely enclose the sample.

NOTE: Screen mesh or other suitable material, with maximum and minimum opening of #8 and #30 respectively, has been found to perform well. *Catch Pan*, of sufficient size to hold the sample basket(s) and retain aggregate particles that fall through the sample basket(s) openings during the material testing.

Basket assembly, consisting of assembled catch pan and sample basket(s).

Handling Apparatus, suitable for inserting and removing the basket assembly.

Oven, Capable of maintaining a temperature of 230 F± 9°F (110 ± 5°C).

Mixing Apparatus, in accordance with California Test 304

Sample splitters for aggregates, riffle type in accordance with California Test 201.

Sample splitter for HMA, in accordance with California Test 304.

Safety Equipment, safety glasses or face shield, high temperature gloves, long sleeve jacket, a heat resistant surface capable of withstanding 1202°F (650°C) and a protective cage capable of surrounding the sample baskets during the cooling period.

Miscellaneous Equipment, a pan larger than the sample basket(s) for transferring sample after ignition, spatulas, bowls, and wire brushes.

D. SAMPLING

For developing the correction factor, obtain samples of the aggregate, additives (if any), and the binder that will be used on the project in accordance with California Test 125. For testing, the size of the HMA sample must be governed by the nominal maximum aggregate size of the mixture and must conform to the weight requirement shown in Table 1.

TABLE 1

Nominal Maximum Aggregate Size, inch	Minimum Weight of Mixture Sample, g
1 ½"	4000
1"	3000
3/4"	2000
1/2"	1500
3/8"	1200
# 4	1200

1. Sample sizes must not be more than 400 g greater than the minimum recommended values in Table 1.

2. Prepare the aggregate in accordance with California Test 201 prior to batching for this test method.

E. CORRECTION FACTOR

1. Record the Job Mix Formula (JMF) (including additives and type of binder and/or binder grade to be used for the project). All specimens prepared for the determination of a correction factor must be tested using the JMF gradation that includes the same dry aggregates, additives and binder that will be used on the project. Prepare all loose mixture specimens in accordance with California Test 304,

NOTE: For field laboratories that are not equipped with mixing apparatus but are equipped with an ignition furnace, the mixture samples will have to be prepared elsewhere as specified herein, and shipped to the field for determination of the correction factor for the field laboratory furnace. These samples must be oven dried as outlined in Section F, Step 2, before they are placed into the ignition furnace.

2. Split and batch, in accordance with California Test 304, enough dry aggregate for a minimum of 4 HMA specimens (up to 10 specimens may be required per the JMF).

3. Set the ignition furnace temperature to 1000°F (538°C). Preheat ignition furnace to that specified by the manufacturer. If it is adjustable, the afterburner temperature should be set according to the furnace manufacturer's recommendations for the type of HMA being tested. For the direct-irradiation-type furnace, set the burn profile to the DEFAULT mode.

NOTE: If experience with particular materials indicates correction factors > 0.5 % are likely to result from using the 1000°F (538°C) set point, then the alternate 900°F (482°C) set point may be used to develop the correction factor.

4. Prepare two HMA specimens at the binder target value. Measure and record the weight of dry aggregate used in each specimen as M_{agg}. Measure and record the weight of the binder used in each specimen as M_{ab}. Record the known binder content of each specimen, in percent of total HMA, as $AC_{known\ (1)}$ and $AC_{known\ (2)}$ respectively.

5. Place the second HMA specimen in a 230 F± 9°F (110 ± 5°C) oven until ready to test. Do not preheat the basket(s) assembly.

   Each pair of specimens must be tested within 4 hr.

6. Measure the weight of the basket(s) assembly only and record this weight as M_e.

7. Evenly distribute the first HMA specimen in the sample basket, in as relatively thin a layer(s) as possible. Keep the material away from the solid edges of the basket(s). Whenever multiple stacked baskets are used, also take care to distribute the specimen as evenly as possible amongst the sample baskets.

8. Measure the total weight of the specimen and basket(s) assembly and record it as $M_{initial}$.

9. Place the specimen and basket(s) assembly in the ignition furnace within 15 min after mixing or removal from the 230 F ± 9°F (110 ± 5°C) oven.

10. Calculate the initial specimen weight by subtracting the weight of the basket(s) assembly ($M_{e)}$ from the total weight of the specimen and basket(s) assembly ($M_{initial}$). Record the initial weight of the specimen as (M_s).

NOTE: Verify that the furnace chamber temperature is stabilized at the set point prior to proceeding and between all subsequent tests.

11. After placing the specimen and basket(s) assembly, in the furnace, close the door and verify that the weight indicated by the internal scale is within ± 5 g of the total weight ($M_{initial}$) recorded. Differences of more than 5 g or failure of the scale to stabilize may indicate that the basket(s) assembly is in contact with the furnace wall. If necessary, reposition the basket(s) assembly.

12. Input the initial specimen weight (M_s) based on an external measurement, and a correction factor of zero into the ignition furnace computer.

13. Perform the specimen "burn" in the ignition furnace. When the change in weight of the specimen over a 3 min interval does not exceed 0.01 % of the initial specimen weight (M_s), remove the specimen and basket(s) assembly from the furnace. Place them in the protective cage, and allow them to cool.

14. Record the weight of the specimen and basket(s) assembly after the ignition process as M_{final}. Also, record the total ignition time to obtain M_{final}. This weight and time will be obtained upon completion of the ignition process from the printout or display, along with a printout showing the elapsed time, temperature, initial specimen weight, specimen weight loss, binder content with zero correction factor ($A_{(1)}$), and factory temperature compensation (if applicable).

15. Repeat steps 6 through 13 for the second HMA specimen that was previously placed in the oven as indicated in Step 5.

16. If the difference between the measured binder contents of the two mixture specimens, $A_{(1)}$ and $A_{(2)}$, exceeds 0.15 %, repeat the Steps 2 through 14.

17. Calculate the correction factor as follows:

$$CF = \frac{[A_{(1)} - AC_{known(1)}] + [A_{(2)} - AC_{known(2)}]}{2}$$

Where:

CF = the correction factor, is the average of the differences between the measured and actual binder contents for each specimen expressed in percent by total weight of HMA specimen.

$A_{(1)}$ = binder content of first mixture specimen expressed in percent by total weight of HMA specimen.

$A_{(2)}$ = binder content of second mixture specimen,

AC_{known} = known percent binder added to each Laboratory prepared specimen respectively (by total weight of HMA specimen).

18. If a correction factor exceeds 0.5 %, lower the test temperature to 900°F (482°C) and repeat the test procedure. Use the correction factor obtained at the 900°F (482°C) set point even if it exceeds 0.5 %.

NOTE: The temperature set point used to test field samples of HMA per Section E, "Test Procedure," T_1 must be the same temperature set point used to establish the correction factor in Section D, Steps 3 and 17.

19. The correction factor determined by this test method must be used only with the same furnace and JMF gradation that includes the same aggregates, additives and binder used to establish the factor. If the JMF, component materials and/or the equipment are changed, or if the ignition furnace is moved to a different location, then a new correction factor must be determined.

F. TEST PROCEDURE

1. In accordance with California Test 125, obtain a sample of HMA from the project. The sample size must be sufficient to meet the minimum requirements of Table 1, Section C.

2. Oven dry the sample to a constant weight at 230 F± 9°F (110 ± 5°C) until there is less than a 0.1 % difference in the weight measured for a 60 min interval of drying time, or determine and record the moisture content (M_c) in accordance with California Test 370 and California Test 226.

3. Program the ignition furnace at the same temperature set point used in the determination of the correction factor. If it is adjustable, the afterburner temperature should be set according to the furnace manufacturer's recommendations for the type of HMA being tested. For the direct-irradiation-type furnace, set the burn profile to the DEFAULT mode.

4. Determine the weight of the basket(s) assembly. Record this weight as M_e.

5. Evenly distribute the field sample in the sample basket(s), and in as relatively thin a layer(s) as possible. Keep the material away from the solid edges of the sample basket(s). Whenever multiple stacked sample baskets are used, also take care to distribute the sample as evenly as possible amongst them.

6. Measure the initial weight of the sample and basket(s) assembly. Record this initial weight as $M_{initial}$.

7. Calculate the initial sample weight by subtracting the weight of the basket(s) assembly ($M_{e)}$ from the total weight of the sample and basket(s) assembly ($M_{initial}$). Record the initial weight of the sample as (M_s).

NOTE: Verify that the furnace chamber temperature is stabilized at the set point prior to proceeding and between all subsequent tests.

8. After placing the sample and basket(s) assembly in the furnace, close the door and verify that the weight indicated by the internal scale is within ±5 g of $M_{initial}$. Differences of more than 5 g, or failure of the scale to stabilize, may indicate that the basket(s) assembly is in contact with the furnace wall. If necessary, reposition the basket(s) assembly in the furnace.

   Input the initial sample weight (M_s) based on an external measurement, and enter the established correction factor CF into the ignition furnace computer.

9. Perform the sample "burn" in the ignition furnace until the change in weight of the sample over a 3 min interval does not exceed 0.01 % of the initial sample weight (M_s) prior to ignition.

10. Record the weight of the sample and basket(s) assembly after ignition as $M_{final.}$ Also, record the total ignition time to obtain M_{final}. This weight and time is obtained upon completion of the ignition process either from an external scale and hand recordation or from the device printout or the display. Minimum data recorded must be: elapsed time, temperature, initial specimen weight, specimen weight loss, corrected binder content, the correction factor input, and factory temperature compensation (if applicable).

G. REPORT

Correction Factor Worksheet

1. The report (see example worksheets) must include the following (use of example worksheets is not mandatory):

   a. Date,

   b. Identification of HMA type grading and aggregate, additives (if any), binder, and mix type,

   c. Sample location, contract numbers,

d. Test number, date, time (YYMMDD), tonnage,

e. Furnace make, model, and unique identifier,

f. Correction Factor (CF),

g. Ignition time and incremental time,

h. Weight of sample before and after ignition,

i. Moisture content of HMA (when required), and

j. Corrected percent binder content calculated as follows:

$$AC = \left[\frac{M_{initial} - M_{final}}{M_s} \right] * 100 - CF - M_c$$

Where:

$M_{initial}$ = total weight of the sample and basket(s) assembly prior to ignition, as determined in Section E, Step 8.

M_{final} = weight of the sample and basket(s) assembly after ignition process.

M_s = initial sample weight prior to ignition.

CF = the correction factor, as determined in Section E.

M_c = moisture content of the sample as determined in Section F, Step 2 (zero if sample was initially dried to a constant weight).

H. PRECISION AND BIAS

Data to establish the following precision statements was obtained from a round robin study on carefully batched samples of dense graded asphalt concrete (DGAC). This was reported by the National Center for Asphalt Technology in 1995.

Analyses of the data within laboratory and between laboratory precision provided the following:

Binder Content, %		
	Standard Deviation (S)	**Acceptable Range of Two Results (D2S)**
Within laboratory	0.04	0.11
Between laboratories	0.06	0.17

The data consisted of measurements using a Type 1 furnace and individual batched samples prepared for the round robin testing. There were 4 aggregate types, 4 replicates and 12 laboratories involved.

Developing a calibration factor for each aggregate addresses a known bias. Criteria for other biases such as sampling have not been established.

I. HEALTH AND SAFETY

It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Prior to handling, testing or disposing of any materials, testers must be knowledgeable about safe laboratory practices, hazards and exposure, chemical procurement and storage, and personal protective apparel and equipment.

Caltrans Laboratory Safety Manual is available at:

http://www.dot.ca.gov/hq/esc/ctms/pdf/lab_safety_manual.pdf

End of Text
(California Test 382 contains 10 pages)

CORRECTION FACTOR WORKSHEET (SECTION E)

Sample I.D. _____ Project I.D. _____ Resident Engineer _____

Location of Sample _____ JMF # _____

Furnace Make and Model _____ Serial No. _____

Furnace Temperature Set Point (circle one) 1000°F (538°C) 900°F (482°C)

Testing Laboratory _____ Mobile Lab Unit I.D. _____

_____ Technician Name _____

_____ Date of Test _____

Aggregate Source _____ Type of Binder _____

Supplier/Plant _____

Additive(s) Type(s) and Source(s)_____

_____

Type of HMA and Design Specifications (use the table below):

Type _____ Grading, _____ (Target Binder Content = _____%)

Correction Factor Determination Data

(1)_____ (2)_____ = M_{agg}, weight of the dry aggregate. (Section E, Step 4)

(1)_____ (2)_____ = M_{ab}, weight of the binder. (Section E, Step 4)

(1)_____ (2)_____ = AC_{known}, known binder content in percent of HMA. (Section E, Step 4)

(1)_____ (2)_____ = M_e, weight of basket(s) assembly. (Section E, Step 6)

(1)_____ (2)_____ = $M_{initial}$, initial weight of specimen and basket(s) assembly (Section E, Step 8)

(1)_____ (2)_____ = M_s, initial weight of specimen (Section E, Step 9).

(1)_____ (2)_____ = M_{final}, final weight of specimen and basket(s) assembly after ignition process. (Section E, Step 13).

(1)_____ (2)_____ = Total ignition time (Section E, Step 13).

(1)_____ (2)_____ = Incremental time, if applicable; use "N/A" if not applicable.

(1)_____ (2)_____ = A, measured percent binder content.

_____ = Difference between $A_{(1)}$ and $A_{(2)}$ (Section E, Step 15).

_____ = CF, the Correction Factor (Section E, Step 16).

$$CF = \frac{\left[A_{(1)} - AC_{known(1)}\right] + \left[A_{(2)} - AC_{known(2)}\right]}{2}$$

TEST PROCEDURE WORKSHEET (SECTION F)

Sample I.D. _____ Project I.D. _____ Resident Engineer _____

Location of Sample _____ JMF # _____

Furnace Make and Model _____ Serial No. _____

Furnace Temperature Set Point (circle one) 1000°F (538°C) 900°F (482°C)

Testing Laboratory _____ Mobile Lab Unit I.D. _____

_____ Technician Name _____

_____ Date of Test _____

Aggregate Source _____ Type of Binder _____

Supplier/Plant _____

Additive(s) Type(s) and Source(s)_____

_____

Type of HMA and Design Specifications (use the table below):

Type _____ Grading, _____ (Target Binder Content = _____%)

_____ = M_e, weight of basket(s) assembly. (Section F, Step 4)

_____ = $M_{initial}$, initial weight of sample and basket(s) assembly (Section F, Step 6)

_____ = M_s, initial weight of sample (Section F, Step 7).

_____ = M_{final}, final weight of sample and basket(s) assembly after the ignition process. (Section F, Step 10)

_____ = Total ignition time (Section F, Step 10).

_____ = CF, the Correction Factor (Section E, Step 16).

_____ = M_c, moisture content (Section F, Step 2).

_____ = AC, corrected measured percent binder content.

$$MoistureContent,\% = \left[\frac{Weight_{ORIGINAL} - Weight_{FINAL}}{Weight_{FINAL}}\right] \times 100$$