STATE OF CALIFORNIA—BUSINESS, TRANSPORTATION AND HOUSING AGENCY

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METHOD OF TEST FOR DETERMINATION OF ASPHALT AND MOISTURE CONTENTS OF BITUMINOUS MIXTURES BY HOT SOLVENT EXTRACTION

A. SCOPE

This test method describes the procedure for determining the bitumen and moisture contents of a bituminous mix in one operation using hot solvent.

In the instructions that follow, an earlier model of extractor, which can be identified by the three-way valve, will be referred to as Model A and the later model as Model B. The same general principles apply to both, the difference being that they are equipped with different valve systems.

B. APPARATUS (see Figures 1 to 6, inclusive)

1. Extraction assembly consisting of:

   a. Metal thimble with a finely perforated inner basket for holding the sample.

   b. Top and bottom plates with clamping device.

   c. Condenser and moisture trap.

2. Balance - 4.5 kg capacity, 0.1 g sensitivity.

3. Heat source - Bunsen burner for Model A and Calrod for Model B.

4. Air source consisting of either an air compressor capable of developing 345 kPa or a pneumatic tire pump (foot type preferred).

5. Water supply (for cooling condenser). If running water is not available, the water may be supplied to the condenser by means of a 19-L can fitted with a connection for 6 mm rubber tubing.

6. Drying ovens - use either:

   a. Drying unit (Figures 4 and 5) and blowtorch.

   b. Oven capable of maintaining a temperature of 150 $\pm$ 5°C.*

   c. Microwave oven.*

7. Curing pans - approximately 280 by 180 by 25 mm.

* To be interchangeable with California Test 362, ovens must be capable of drying a 3000-g sample.

8. Small pointed trowels.

9. Beakers - 1000 mL.

10. Mechanical mixing machine - optional.

11. Gloves – heat resistant.

C. MATERIALS

1. Stoddard solvent, Petroleum Naphtha or Naphthol Spirits.

2. Filter paper (Eaton Dikeman No. 652, 140 mm or equivalent).

3. White gasoline (for use when blowtorch is used for heat source) and safety container.

4. 6 mm rubber tubing.

5. Container for microwave oven: must be paper, glass or ceramic.

D. PREPARATION OF SAMPLE

1. Field Samples.

   a. For samples with aggregates larger than 25 mm:

      (1) Prepare a 2000-g sample (including each sample of a set to develop the calibration curve).

      (2) Separate each 2000-g sample into three approximately equal parts.

   b. For samples with aggregates whose maximum size is 25 mm or less:

      (1) Prepare an approximately 750-g sample for mixtures containing paving or liquid asphalt.

      (2) For mixtures containing asphaltic emulsion or cutback, prepare an approximately 850-g sample. (Dry all mixes to a constant mass using either a conventional or microwave oven. Cool to room temperature prior to extracting.)

E. EXTRACTION CALIBRATION CURVE

(Complete only if job aggregate is available. Do not use for emulsion mixes.)

1. Prepare triplicate test specimens with asphalt contents that are at least 1.0 % above and 1.0 % below the Optimum Bitumen Content (OBC) as follows:

   a. Prepare six 750-g aggregate samples using the aggregate and gradation for the project.

   b. Bring aggregate and asphalt to 150 $\pm$ 5°C.

   c. Mix, in triplicate, for 2 min $\pm$ 10 s (hand or machine). Extend mixing time only if aggregate is not well coated. If mixing time is extended, note time and mix all companion samples for the same period.

   d. After mixing, place in a curing pan and cure at room temperature for 15 to 20 h.

2. Extract as per Section F.

F. TEST PROCEDURE FOR EXTRACTION

1. Place filter paper in the metal thimble as shown in Figure 6.

2. Tare the thimble and inner basket to nearest 0.1 g.

3. Place a representative sample of about 750 g in the inner basket and weigh the assembly to the nearest 0.1 g.

4. Clamp the thimble assembly in place in the extraction unit.

5. Turn on the cooling water valve. If using Model A (Figure 1) proceed with steps 6 through 12.

   If using Model B (Figures 2 and 3) skip steps 6 through 12 and proceed with step 13.

MODEL "A" EXTRACTOR ONLY

6. Close the bottom drain valve (B), open the 3-way valve (A) to "Solvent" position, and pour about 350 mL of solvent through the funnel.

7. Turn the 3-way valve (A) to "Heat" position.

8. Apply heat to the extractor to remove moisture from the sample and dissolve the asphalt.

9. After the collected water in the trap reaches a constant amount, shut off heat, then read and record the volume of water (mL) in the trap.

10. Open the 3-way valve (A) to "Solvent" position and pour in an additional 150 mL of solvent.

11. Open the drain valve on the bottom plate.

12. Turn the 3-way valve (A) to "Blow" position and apply air from air pump or compressor (approximately 200 kPa). Skip steps 13 through 19 and proceed with step 20.

MODEL "B" EXTRACTOR ONLY

13. Close the bottom drain valve "B." Turn valves "A" and "D" to open position. Push down on funnel valve "E" to open, and pour about 350 mL of solvent into funnel.

14. Release valve "E" to close, and turn valve "D" off.

15. Apply heat to the extractor to remove moisture from the sample and dissolve the asphalt.

16. After the collected water in the trap reaches a constant amount, shut off heat, then read and record the volume of water (mL) in the trap.

17. Open valves "E" and "D" and pour in an additional 150 mL of solvent.

18. Open drain valve "B" on the bottom plate.

19. Place valves "A", "E," and "D" in the close position and apply air to valve "C" from an air pump or compressor (approximately 200 kPa).

20. If the flow of asphalt-laden solvent slows more than a reasonable amount, release the air pressure, and rotate the stirring crank several turns to aid filtration.

21. After all the dirty solvent has been expelled, release the air pressure (may be released through valve "D" in Model B) and introduce approximately 500 mL of solvent.

22. Expell the solvent and repeat with subsequent charges until the solvent is clear.

23. After the final charge of solvent has been expelled, release the air pressure and remove the sample.

24. Place sample, basket, and thimble in an oven or, if an oven is not available, place the basket containing the sample on the special pan on the lower deck of the dryer. Then place the thimble on top of the dryer as shown in Figure 4.

25. Dry to a constant mass (0.2 g loss or less in 20 min) or for 48 h*, if necessary. Remove from the dryer or oven, allow to cool and weigh. Weigh within 1-h after removing from the dryer or oven.

26. A sieve analysis can then be performed on the aggregate; see California Test 202, Appendix A.

G. CALCULATIONS

1. Calculate the percentage of bitumen using the following formula:

   **Percent bitumen =
   [(B - C - D)/(C - A)] X 100

   Where:

   A = Tare of thimble and basket, g.

   B = Mass of sample + thimble + basket before extraction, g.

   C = Mass of sample + thimble + basket after extraction, g.

   D = mL of water in trap.

2. Establish a calibration curve to adjust the extracted data.

   a. For the calibration curve, average the triplicate specimen results for each percent bitumen. Plot the average extracted asphalt content versus the actual asphalt content on Form No. TL 309 (Figure 7), and connect the points with a straight line.

   b. Use this curve to correct the field sample test results as follows: Locate the extracted asphalt content on the ordinate, proceed horizontally to intersect the calibration curve. At the intersection drop vertically to obtain the actual asphalt content on abscissa.

3. Calculate the percentage of moisture in the sample by the following formula:

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* In the case of absorptive aggregates (Km values of 1.8 or higher), a 48-h drying time in a 150°C oven may be required.

** When 2000 g samples are required, three tests are used, calculate the asphalt content based upon the combined mass of the three samples.

Percent moisture = [D/(B - A - D)] X 100

H. PRECAUTIONS

1. Be sure extraction is complete. Three charges of solvent will normally be sufficient for this purpose.

2. Make sure all connections are tight.

3. Do not use air pressure in excess of 345 kPa.

4. Regulate the water flow through the condenser(s) so that no water vapor escapes during the heating process.

5. Drops of water may collect in the condenser during the heating process. Wash these into the trap by pouring some solvent through the condenser before reading.

6. If a 19 L water can is used to supply the condenser, be sure it does not empty during the test.

7. Heat may cause equipment damage or induce erroneous data if hot materials are weighed on sensitive scales.

I. REPORTING OF RESULTS

1. Report the corrected bitumen content of field-control samples and correlation samples after using the calibration curve established in G.2. Use the letter "C" and the calibration curve number (encircled) with each corrected test result (example - 4.8 % 2 C). Also report actual correction applied.

2. Report bitumen content on cores, recycled AC or research samples on old pavement as calculated in G.1. Add 0.2 % as a correction constant in lieu of using a calibration curve. Use letter "C" with each test result to indicate correction has been applied (example - 4.5 %C).

3. Report the sieve analysis in percent passing each sieve based on the dry mass of the extracted aggregate.

4. Report the percent bitumen and the grading on Form TL 302. Note the test method number (California Test 310) for identification on the report.

J. HEALTH AND SAFETY

Personnel must use heat resistant gloves when working with hot materials. Use proper lifting techniques when handling bags of aggregate. Reasonable care should be exercised to avoid being burned by hot asphalt, aggregate or equipment. Eye protection to safeguard against contact, irritation or injury caused by the solvent is mandatory. Hot extractors must be operated under hoods or in properly ventilated areas.

Observe the following procedures when using solvent:

a. Store in cool, dry, well-ventilated areas away from ignition source, direct sunlight, and oxidizing materials.

b. Use in a well-ventilated area.

c. The wearing of protective gloves and eyeglasses or chemical safety goggles is mandatory.

d. If spilled on skin, immediately rinse with copious amounts of water and consult a physician for reddened or blistered skin. Wash affected area with soap and water.

e. For eye contamination, gently lift eyelids, flush immediately and continuously with large amounts of water, and seek immediate medical attention.

If a blowtorch is used, the gasoline should be stored in a safety container and kept a safe distance from all open flames.

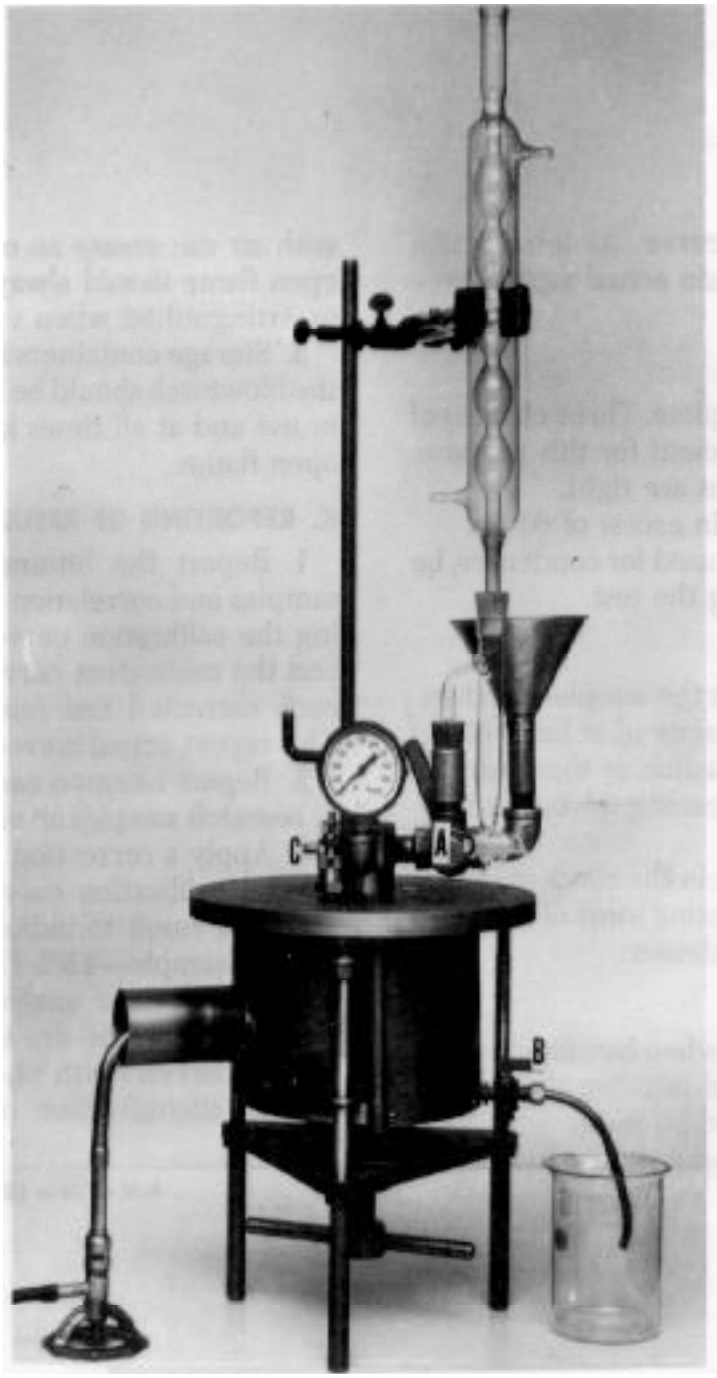
Prior to handling, testing or disposing of any waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0 and 10.0) and Part C (Section 1.0) of Caltrans Laboratory Safety Manual. Users of this method do so at their own risk.

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.

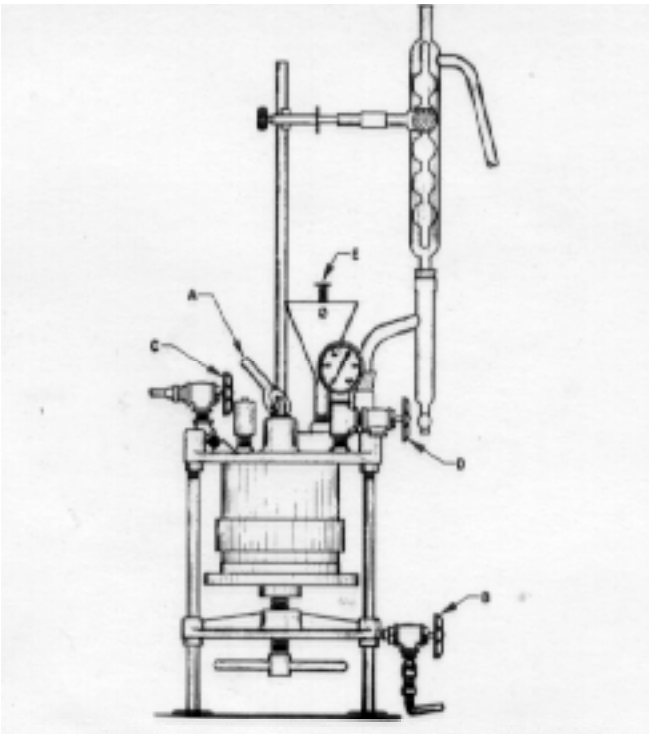
Caltrans Laboratory Safety Manual is available at:

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

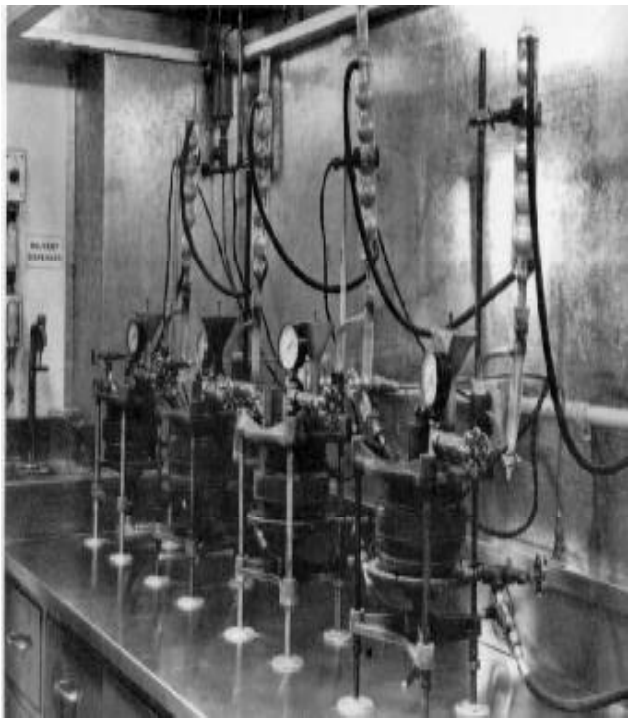
End of Text
(California Test 310 contains 11 pages)



EXTRACTOR – MODEL A
FIGURE 1

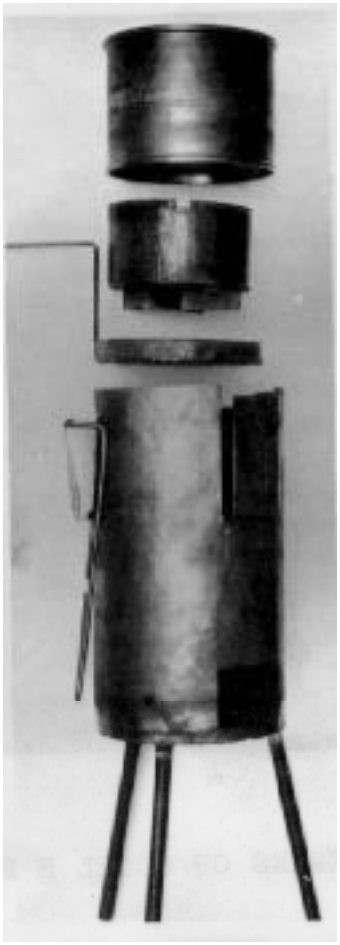


EXTRACTOR – MODEL B
FIGURE 2



SERIES OF MODEL B EXTRACTORS
FIGURE 3

SAMPLE DRYER
FIGURE 4



DRYER ASSEMBLY EXPANDED
FIGURE 5

THIMBLE ASSEMBLY EXPANDED
FIGURE 6

EXTRACTION CALIBRATION CURVE NO 2

TEST METHOD CA 310

DISTRICT 10 CONTRACT NUMBER 123456

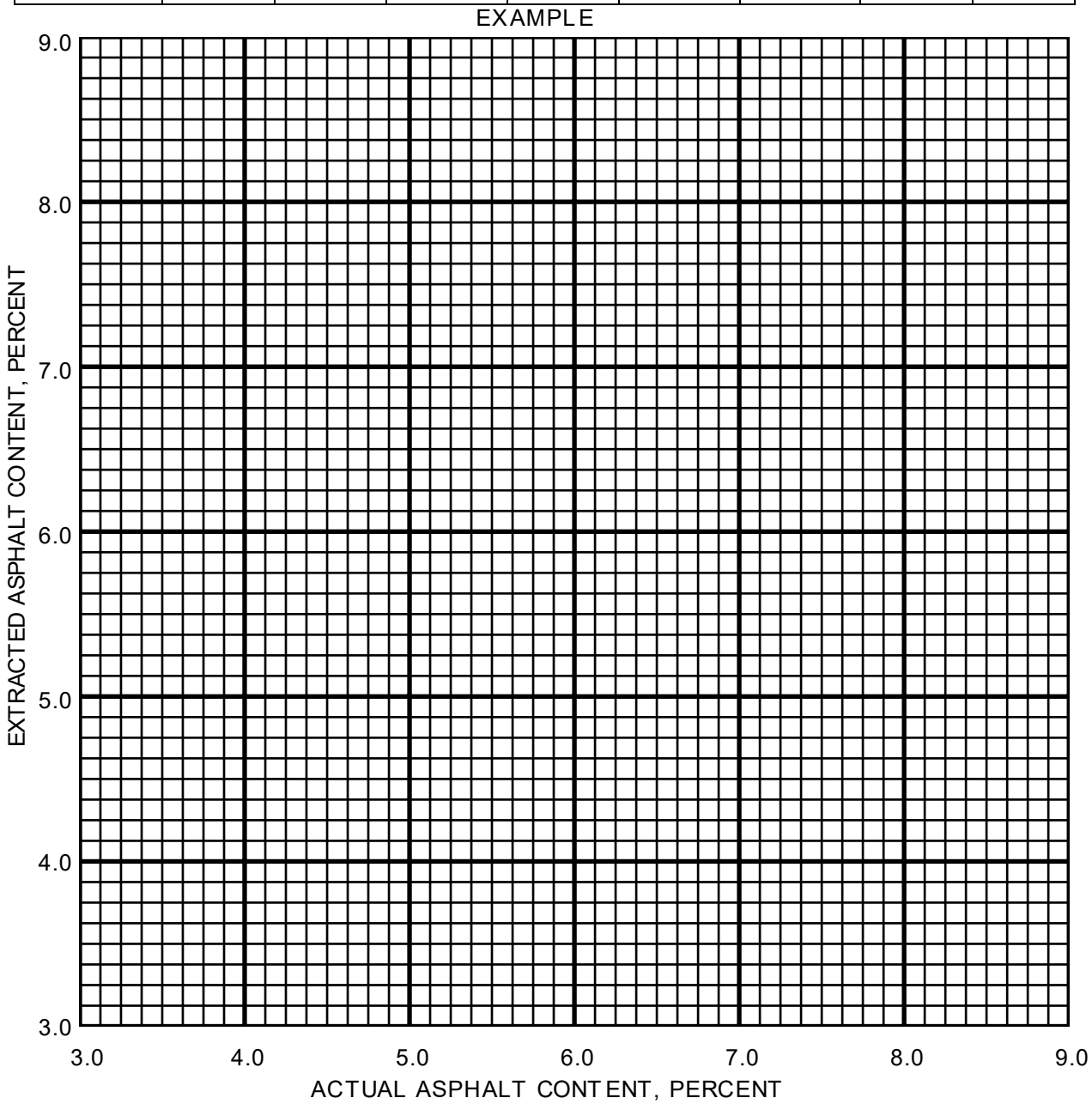
AGGREGATE SOURCE Browns Pt. AGGREGATE Km VALUE 1.4

ASPHALT GRADE AR-4000 ASPHALT SOURCE Chevron-Oakland

DATE July 11, 1984

GRADATION OF AGGREGATE USED

SIEVE SIZE	25.0 mm	19.0 mm	12.5 mm	9.5 mm	4.75 mm	2.36 mm	600 µm	75 µm
% PASSING	100	98	85	72	52	35	18	4



TL-309 (Rev. 5/96)

ACTUAL ASPHALT CONTENT
FIGURE 7