

Method of Test for  
**ASH CONTENT OF ASPHALTIC CONCRETE MIXTURE SOLVENT  
OBTAINED BY REFLUX OR CENTRIFUGE EXTRACTION**  
DOTD Designation: TR 314M-94

**I. Scope**

- A. This method of test is designed to determine residual fines (mineral matter) present in extraction solvent after asphaltic material has been extracted from the asphaltic concrete mixture.
- B. Reference Documents
  - 1. DOTD TR 307 - Bitumen Content of Paving Mixtures by Reflux Extractor.
  - 2. DOTD TR 308 - Bitumen Content of Paving Mixtures by Centrifuge.

**II. Apparatus**

- A. Graduated cylinder - 2000 mL (minimum).
- B. Pipette - 100 mL capacity.
- C. Balance - sensitive to 0.01 g.
- D. Evaporating dish - 100 mL (minimum capacity).
- E. Muffle furnace.
- F. Electric hot plate - thermostatically controlled.
- G. Oven - capable of maintaining a temperature of  $110 \pm 5^\circ\text{C}$ .
- H. Desiccator.
- I. Rubber gloves, thermal gloves, eye protection, apron, tongs - for handling hot materials.
- J. Gas burner.
- K. Ring stand.
- L. Well-ventilated hood.
- M. Worksheet - Total Ash Content, DOTD Form No. 03-22-4177. (Figure 1)

**III. Reagents**

- A. Solvent - 1,1,1-Trichloroethane, technical grade.
- B. Saturated Solution of Ammonium Bicarbonate - ( $\text{NH}_4\text{HCO}_3$ ).

**NOTE 1:** A saturated solution of ammonium bicarbonate is created when 22.5 grams of powdered ammonium bicarbonate is added to and mixed in 100 mL of distilled water at  $21^\circ\text{C}$ .

**IV. Health Precautions**

The solvent listed above is to be used only under a hood or with an effective surface exhaust system. Material Safety Data Sheets (MSDS) shall be maintained at locations where employees handle hazardous materials.

Extreme caution is to be exercised when testing this material due to the high temperature involved.

**V. Sample**

- A. The extracted solvent sample size shall conform to DOTD TR 307 or TR 308.
- B. Care shall be taken to ensure that all the solvent extracted from a particular extraction sample is obtained, and that this extracted solvent does not contain any material from previous extractions.

**VI. Procedure**

- A. Place entire extracted solvent sample in the graduated cylinder, washing any remaining asphalt and fines into the cylinder with small increments of fresh solvent. If the level of solvent falls between 2 graduations, add additional solvent to bring the level in the graduated cylinder up to the next graduation. Record on worksheet as C to the nearest whole number.
- B. Prepare an evaporating dish by heating to approximately  $110^\circ\text{C}$ , cool in desiccator and weigh to the nearest 0.01 g. Record on worksheet as B.
- C. Thoroughly stir the extract in the graduated cylinder and immediately use a pipette to obtain a 100 mL test specimen from the approximate center of the mixture while continuing to stir the mixture with the pipette. Slightly overfill pipette past the 100 mL mark and drain off excess solvent. Drain the 100 mL test specimen into the tared evaporating dish.
- D. Place evaporating dish on hot plate and evaporate solvent carefully under a well-ventilated hood at a temperature of approx-

imately 149°C or the low-to-medium range until all solvent has evaporated. The temperature may need to be regulated to avoid overflow. Discard test specimen if overflow occurs and repeat step VI.C.

- E. Using gas burner and ring stand under a well-ventilated hood, ignite residue remaining in evaporating dish until residue no longer burns.
- F. Place evaporating dish containing residue in a cool muffle furnace. Set at approximately 600°C. Heat until residue becomes gray and powdery in appearance (approximately one hour after reaching set temperature).

**NOTE 2:** *Evaporating dishes are prone to cracking when placed into a preheated furnace.*

- G. Remove evaporating dish from furnace and cool in desiccator to room temperature. Determine weight of evaporating dish containing initial residue. Record on worksheet as E.
- H. Determine weight of initial residue by subtracting the tare weight of the evaporating dish (B) from the weight of the evaporating dish and initial residue (E). Record on worksheet as F.
- I. Determine amount of ammonium bicarbonate solution to be added to the evaporating dish containing the initial residue. Multiply the grams of initial residue (F) by 5 mL/g. Record on worksheet as G.
- J. Add the calculated amount of ammonium bicarbonate solution (G) to the evaporating dish containing the initial residue. Stir and allow to stand in desiccator at room temperature for one hour.
- K. Place evaporating dish containing the residue mixture in the oven set at 110°C and dry to a constant weight. Remove from the oven, place in desiccator and allow to cool to room temperature. Remove from desiccator and weigh evaporating dish containing final residue to the nearest 0.01 g. Record on worksheet as A.

**NOTE 3:** *Constant weight for drying purposes is defined as less than 0.1% weight loss between successive weighings no less than 5 minutes apart.*

## VII. Calculations

Calculate the total ash content (D) to the nearest 0.1 g using the following formula:

$$D = \frac{(A - B) \times C}{100}$$

where:

A = weight of evaporating dish and final residue (ash), g

B = tare weight of evaporating dish, g

C = total volume of extracted solvent, mL

100 = volume of test specimen, mL

example:

A = 54.44 g

B = 54.08 g

C = 1260 mL

$$\begin{aligned} D &= \frac{(54.44 - 54.08) \times 1260}{100} \\ &= \frac{0.36 \times 1260}{100} \\ &= \frac{453.6000}{100} \\ &= 4.536 \\ D &= 4.5 \end{aligned}$$

## VIII. Report

Report the total ash content (ash correction) to the nearest 0.1 g.

## IX. Normal Test Reporting Time

Normal test reporting time is 24 hours.

MATT MENU SELECTION - 09

DOTD 03-22-4177  
 5/94

LOUISIANA DEPARTMENT OF TRANSPORTATION AND DEVELOPMENT  
 MATERIALS AND TESTING SECTION

TOTAL ASH CONTENT  
 (DOTD TR 314)

Project No. 026-06-0018

Material Code 1, 7, 2

Lab No. 58-109298

PURPOSE CODES

Date Sampled 07-23-94

Submitted By 0593

- 1. Quality Control
- 2. Verification
- 3. Acceptance
- 4. Check
- 5. Resample
- 6. Source Approval
- 7. Design
- 8. Independent Assurance
- 9. Preliminary Source Test

Quantity \_\_\_\_\_ Units \_\_\_\_\_ Purpose Code 1

Plant Code H901

Spec. Code 3

PO No. \_\_\_\_\_ Date Tested 07-24-94

Ident. ASH-01

Remarks 1 \_\_\_\_\_

Remarks 2 \_\_\_\_\_

Item No. 501.1.1.1.1

Sampled By: C.B. Date: 07-23-94

Mix Code ..... 06

01-1WC, 02-1BC, 05-3WC, 06-3BC, 09-5A Base, 10-5B Base,  
 15-Shld WC, 16-Shld BC, 17-Drain Blnkt, 18-7WC, 19-7BC,  
 20-8WC, 21-8BC, 22-8FWC, 23-9WC, 24-Perm AB

A. Weight of evaporating dish + final residue, g	<u>54.44</u>
B. Tare weight of evaporating dish, g	<u>54.08</u>
C. Volume of extracted solvent, mL	<u>1260</u>
D. TOTAL ASH CONTENT, g (ASH CORRECTION) [(A-B)x(C/100)]	<u>4.5</u>
E. Weight of evaporating dish + initial residue, g	<u>54.43</u>
F. Weight of initial residue, g [E-B]	<u>0.35</u>
G. Required volume of ammonium bicarbonate solution, mL [5xF]	<u>1.8</u>

Tested By: A.C. Date: 7-24-94 Checked By: R.N. Date: 7-24-94

Person Notified: John Paul Phone Letter By: R.N. Date: 7-24-94

Approved By: Dist. Lab. Insp. Date: 7-24-94

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Total Ash Content Worksheet

Figure 1