Method of Test for
ASH CONTENT OF ASPHALTIC CONCRETE MIXTURE SOLVENT
OBTAINED BY REFUX 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 ± 5°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 - (NH₄HCO₃).

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°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°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

\[
D = \frac{(54.44 - 54.08) \times 1260}{100} = 0.36 \times 1260 = 453.600\ = 4.536\ D = 4.5
\]

**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.
### Total Ash Content Worksheet

**DOTD TR 314M**

**LOUISIANA DEPARTMENT OF TRANSPORTATION AND DEVELOPMENT**
**MATERIALS AND TESTING SECTION**

**TOTAL ASH CONTENT**
**(DOTD TR 314)**

- **Project No.:** 03.64 - 016 - 0018
- **Material Code:** 1, 7, 2
- **Lab No.:** 58 - 109.298

**Date Sampled:** 07-23-94

- **Quantity:** __________
- **Units:** __________
- **Purpose Code:** __________
- **Plant Code:** H, 9, 0, 1
- **Spec. Code:** __________

**PO No.:** __________

**Ident.:** A.S.H - 01

**Date Tested:** 07-24-94

**Remarks 1:**

**Remarks 2:**

**Item No.:** 501, ( ), ( )

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

**Mix Code:** __________

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

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<table>
<thead>
<tr>
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<tbody>
<tr>
<td><strong>A.</strong> Weight of evaporating dish + final residue, g</td>
<td>54.44</td>
</tr>
<tr>
<td><strong>B.</strong> Tare weight of evaporating dish, g</td>
<td>54.08</td>
</tr>
<tr>
<td><strong>C.</strong> Volume of extracted solvent, mL</td>
<td>124.0</td>
</tr>
<tr>
<td><strong>D.</strong> TOTAL ASH CONTENT, g (ASH CORRECTION) [(A-B)(C/100)]</td>
<td>4.05</td>
</tr>
<tr>
<td><strong>E.</strong> Weight of evaporating dish + initial residue, g</td>
<td>54.43</td>
</tr>
<tr>
<td><strong>F.</strong> Weight of initial residue, g [E-B]</td>
<td>0.35</td>
</tr>
<tr>
<td><strong>G.</strong> Required volume of ammonium bicarbonate solution, mL [5xF]</td>
<td>1.8</td>
</tr>
</tbody>
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**Tested By:** J.G. Date: 07.24.94 **Checked By:** R.N. Date: 07.24.94

**Person Notified:** John Poir **Phone:** Letter By: R.N. Date: 07-24-94

**Approved By:** Dist. Lab. Eng. Date: 07-24-94

**Figure 1**