

**METHODS OF SAMPLING AND TESTING**  
**MT 307-04**  
**METHOD OF TEST FOR CENTRIFUGE**  
**EXTRACTION OF BITUMINOUS MIXES**  
**(Modified AASHTO T164 Method A)**

**1 Scope:**

- 1.1 This method of test is used for quantitative determinations of bitumen in hot-mixed paving mixtures and pavement samples for specification acceptance, service evaluation, control and research.

**2 Apparatus:**

- 2.1 *Extraction Apparatus* - consisting of a bowl approximating that show in Fig. 1 and an apparatus in which the bowl may be revolved at controlled variable speeds up to 3600 rpm. The speed may be controlled manually or with a preset speed control. The apparatus should be provided with a container for catching the solvent thrown from the bowl and a drain for removing the solvent. The apparatus preferably shall be provided with explosion-proof features and installed in a hood or an effective surface exhaust system to provide ventilation.

*NOTE 1 - Similar apparatus of larger size may be used.*

- 2.2 *Filter Rings* - felt or paper, to fit the rim of the bowl.
- 2.3 *Oven* - capable of maintaining the temperature at  $230 \pm 9^{\circ}\text{F}$  ( $110 \pm 5^{\circ}\text{C}$ ).
- 2.4 *Pan* - flat, of appropriate size to warm test specimens.
- 2.5 *Balance or scale* - with weights shall have a sensitivity of 0.1 grams.
- 2.6 *Hot Plate* - electric, with adjustable heating rate.
- 2.7 *Cylinders* - Graduated, 1000 or 2000 ml capacity. Optional cylinder, 100 ml capacity.

**3 Reagent:**

- 3.1 *Methylene Chloride* - technical grade.
- 3.2 *1,1,1-Trichloroethane* - conforming to Federal Specification O-T-620a (Int. Amd.3).
- 3.3 *Trichloroethylene* - reagent grade (Note 3) or technical grade Type 1, Federal Specification O-T-634, latest revision.

*NOTE 2 - Reagent grade Trichloroethylene may be required when asphalt is recovered from the solution.*

**4 Precautions:**

- 4.1 The solvent should be used only under a hood or with an effective surface exhaust system in a well-ventilated area.
- 4.2 Trichloroethylene in the presence of heat and moisture may form acids that are extremely corrosive to certain metals, particularly when subject to contact over lengthy periods of time. Proper precautions should be taken to not allow this solvent to remain in small quantities in the effluent tanks of aluminum vacuum extractors.

**4 Precautions: (continued)**

**4.3** Trichloroethylene when stored in a steel container and in continuous contact with moisture may decompose by dehydrohalogenation to form unsaturated hydrocarbon liquids and hydrogen chloride. Steel drums containing trichloroethylene should be stored in a cool dry location, kept tightly sealed and opened as infrequently as possible. Trichloroethylene should be transferred from the drums to clean, dry brown glass bottles for laboratory use. The hydrogen chloride in decomposed trichloroethylene may harden an asphalt during the extraction test.

**5 Sampling:**

**5.1** If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large, flat pan and warm to  $230 \pm 9^\circ\text{F}$  ( $110 \pm 5^\circ\text{C}$ ) only until it can be handled or mixed. Split or quarter the material until the mass of material required for test is obtained.

**5.3** The size of the test shall be governed by the nominal maximum aggregate size in the mixture and conform to the mass requirement shown in Table 1 (Note 3):

**Table 1 Size of Sample**

| Nominal Maximum<br>Aggregate Size<br>Standard Alternate<br>Mm |         | Minimum Mass<br>Of<br>Sample<br>Kg |
|---|---------|------------------------------------|
| 4.75  | (No.4)  | 0.5                                |
| 9.5   | 3/8 in. | 1                                  |
| 12.5  | 1/2 in. | 1.5                                |
| 19.0  | 3/4 in. | 2                                  |
| 25.0  | 1 in.   | 3                                  |
| 37.5  | 1½ in.  | 4                                  |

*NOTE 3 - When the mass of the test specimen exceeds the capacity of the equipment used (for a particular method), the test specimen may be divided into suitable increments, tested, and the results appropriately combined for calculation of bitumen content.*

**6 Procedure:**

**6.1** Determine the moisture content of the material in accordance with Section 5.

**6.2** Place the test portion into a bowl.

**6.3** Cover the test portion in the bowl with trichloroethylene, and allow sufficient time for the solvent to disintegrate the test portion (not over ½ hr.). Place the bowl containing the test portion and the solvent in the extraction apparatus. Dry and determine the mass of the filter ring and fit it around the edge of bowl. Clamp the cover on the bowl tightly and place a beaker under the drain to collect the extract.

**6.4** Start the centrifuge revolving slowly and gradually increase the speed to a maximum of 3600 rpm until solvent ceases to flow from the drain. Allow the machine to stop, add 200 ml (or more as appropriate for mass of sample) trichloroethylene, and repeat the procedure. Use the sufficient solvent additions (not less than three) so that the extract is not darker than a light straw color. Collect the extract and the washings in an appropriate container for mineral matter determination.

**6.5** Remove the filter ring from the bowl and dry in air. If felt filter rings are used, brush off mineral matter adhering to the surface of the ring and add to the extracted aggregate. Dry ring to constant mass in an oven at  $230 \pm 9^\circ\text{F}$  ( $110 \pm 5^\circ\text{C}$ ). Carefully remove all the contents of

6 Procedure: (continued)

the bowl into a metal pan and dry on a steam bath and then to constant mass in an oven or on a hot plate at  $230 \pm 9^{\circ}\text{F}$  ( $110 \pm 5^{\circ}\text{C}$ ). If trichloroethylene or trichloroethane is used as the extraction solvent, the preliminary drying on a steam bath may be omitted. The mass of the extracted aggregate ( $W_3$ ) is equal to the mass of the aggregate in the pan plus the increase in mass of the filter rings.

- 6.6 Determine the amount of material matter in the extract – For this method, any suitable (700 g or higher) centrifuge may be used.
- 6.7 After extracting, place solvent-bitumen mixture into centrifuge starting centrifuge slowly. Gradually increase speed to 700 g or higher for thirty (30) minutes. Remove solvent from centrifuge and discard.
- 6.8 Air-dry the bottles, remove fine material. Weigh material and add to weight of extracted aggregate.

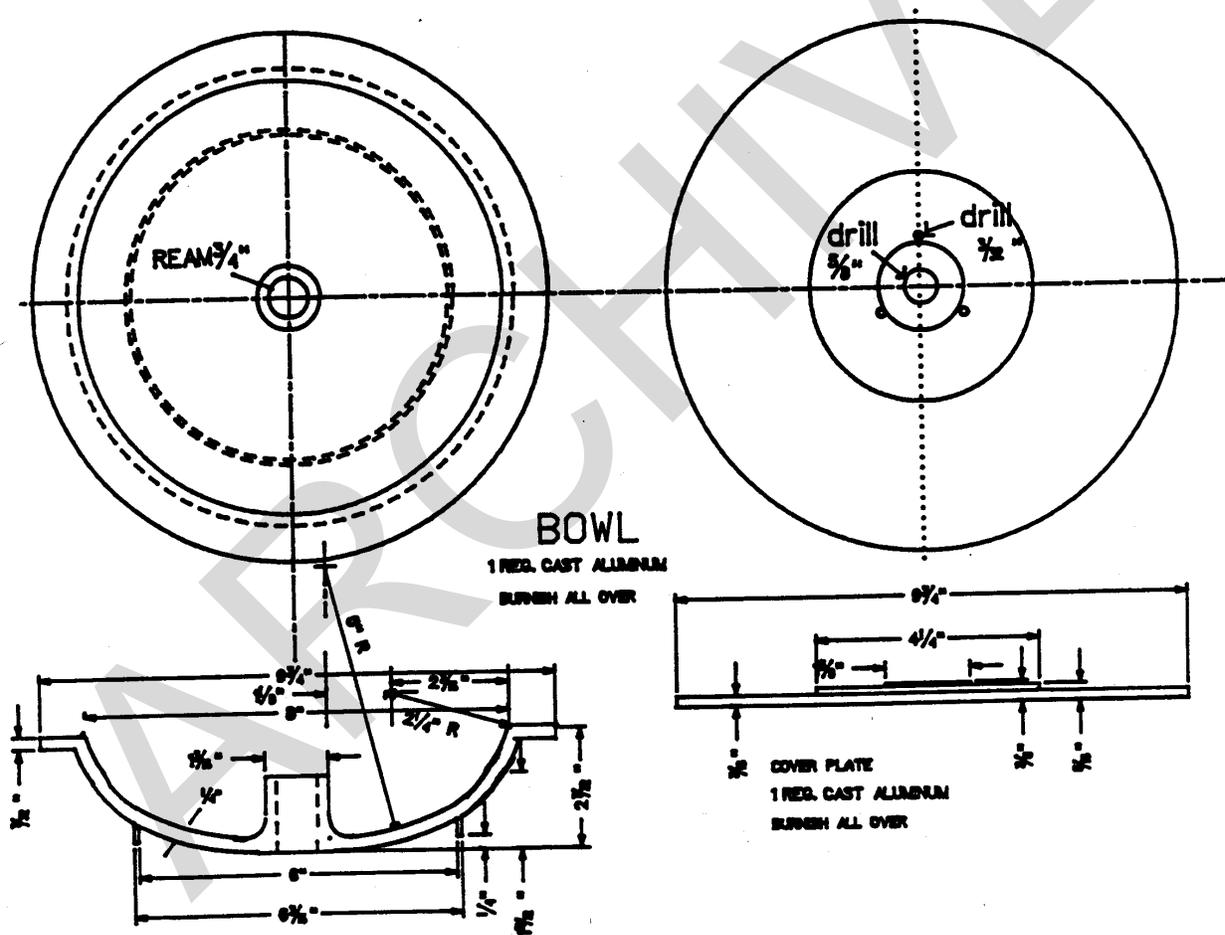


Fig. 1.-Extraction Unit Bowl

## Centrifuge Extraction Worksheet

Lab No. \_\_\_\_\_ Sample No. \_\_\_\_\_ Project No. \_\_\_\_\_

Project Designation \_\_\_\_\_

### Before Extraction

A \_\_\_\_\_ Weight of Sample

B \_\_\_\_\_ Weight of Pan

C \_\_\_\_\_ Weight of Filter

D \_\_\_\_\_ Weight of Filter and Pan

### After Extraction

E \_\_\_\_\_ Weight of Sample, Pan and Filter

E \_\_\_\_\_ - D \_\_\_\_\_ = F \_\_\_\_\_ Wt. after Extraction

A \_\_\_\_\_ - F \_\_\_\_\_ = G \_\_\_\_\_ Wt. of Asphalt

G/A \_\_\_\_\_ = H \_\_\_\_\_ Percent of Asphalt by Total Mix

Weight of Filter after Extraction \_\_\_\_\_

Filter Gain \_\_\_\_\_

Before Wash \_\_\_\_\_ After Wash \_\_\_\_\_ LBW \_\_\_\_\_

| Wt. Ret. | Wt. Pass   | Pct.  |
|----------|------------|-------|
| _____    | 1" _____   | _____ |
| _____    | 3/4" _____ | _____ |
| _____    | 1/2" _____ | _____ |
| _____    | 3/8" _____ | _____ |
| _____    | 4M _____   | _____ |
| _____    | 10M _____  | _____ |
| _____    | 40M _____  | _____ |
| _____    | 200M _____ | _____ |

Fracture \_\_\_\_\_