



**REFLUX
EXTRACTOR**

Introduction

This Reflux Extractor is designed using hot solvent extraction, determines percentage of bitumen in paving mixture. For H-1495 each cone has 500g capacity and for replacement filter paper use H-1497 series. For H-1499 each cone has 1,000g capacity and for replacement filter paper use H-1482 or H-1498 series. Components are available as replacement parts.

Sample Preparation

1. If the mixture is not sufficiently soft to separate with a spatula or trowel, place approximately 4,000g in a large flat pan and warm in the oven at 240°F (115.6°C) only until it can be so handled. Separate the mixture using care not to fracture the mineral particles. Mix and quarter the warm material to obtain a portion for extraction weighing approximately 1,000g. For routine testing, smaller samples maybe used when the maximum size aggregate therein is less than ¼" (6.4mm).
2. At the same time weigh a 500g portion into a metal still, Humboldt 1871, conforming to section 2 (A) of the standard method of test for moisture or volatile distillates in bituminous mixtures (AASHTO) Designation T-110 & ASTM D1461.
3. Dry, if necessary and weigh two sheets of filter paper. Fold each sheet separately on its diameter and fold twice again, one fold being made over the other to make three segments. Open to form a hollow 3-ply cone with a single 1-ply seam and place in wire cones of extractor frames.
4. Weigh each frame with filter paper in cone to nearest 0.1g. Record these tare weights identifying each frame by number (see Note below).
5. Place approximately 500g of the prepared sample for the H-1495 and 1,000g for the H-1499 reflux extractors in each of the filter paper lined cones and weigh the loaded frames to the nearest 0.1g (see Note below). Compact each of the portions by tamping lightly with finger or spatula blade.
6. Assemble the load frames and place in jar. Carefully pour about 500ml of solvent over sample in top frame. At no time should the solvent level contact the tip of the cone in the lower frame. Four or five glass beads placed in jar will facilitate easy boiling of solvent.
7. Place loaded jar on electric hot plate with ceramic pad between glass and the heating element cover with condenser and circulate cold water by just cracking water valve open and waiting for flow.
Caution: Opening the water valve fully will subject the condenser to excessive pressure beyond the 4 psi limit at which units are 100% factory tested.
8. Adjust heater so that solvent boils gently and a steady flow of condensed solvent drips into the top cone. Care must be taken to adjust heat so that each portion of sample is covered with solvent and at the same time the filter cones do not overflow.
9. Continue extraction until solvent running from tip of lower cone appears colorless when viewed against a white background. Then shut off heat but not water through condenser and allow standing until cool enough to handle.
10. Remove frames from jar, dry first in air and then to constant weight in an oven at 240°F (see Note below). If drying at 240°F causes the filter papers to become so brittle they cannot be handled and brushed, they may be dried separately from the aggregate and at a lower temperature.

11. Transfer solvent extract from jar to a 1,000ml graduated cylinder. Wash any remaining asphalt and fines in jar into the graduate with small increments of fresh solvent and record the total volume.
12. Thoroughly stir the extract in the 1,000ml graduate and immediately pour 100ml into a 100ml graduated cylinder. Pour this aliquot into a tare 200ml silica evaporating dish. Wash any remaining asphalt and fines from 100ml cylinder into dish with small increments of fresh solvent.
13. Evaporate solvent carefully in a well ventilate hood and finally ash at a dull red heat (500-600°C) cool and add 5 grams of powdered ammonium bicarbonate and 5ml of water per gram of ash. Stir and allow digesting at room temperature for one hour and then drying to constant weight in an oven at 240°F. Calculate the weight of ash in the entire volume of extract.
14. Determine the water content of the sample in the metal still (section 4B) in accordance with standard method of test for moisture or volatile distillates in bituminous mixtures (AASHTO Designation T-110) and calculate the weight of water in the extraction sample.

Note: *Some agencies weigh filter papers and the sample separately from the frames. After extraction is completed, the filter papers containing the aggregate are transferred to a pan and then placed in the oven. This exposes more surfaces for drying and weighing of the frame is eliminated.*

Calculation

Calculate the percent bitumen in the sample as follows:

$$\text{Bitumen content of dry sample, percent} = \frac{(W1-W2) - (W3 + W4)}{W1-W2} \times 100$$

Where: W1 = Total weight of extraction sample in two cones

W2 = Weight of water in extraction sample

W3 = Total weight of extracted mineral matter in two cones and

W4 = Weight of ash in total extract

Sieve Analysis

If a sieve analysis of the extracted aggregate is desired, it maybe obtained by the standard method of test for mechanical analysis of extracted aggregate (AASHTO Designation T-30). The total weight of extracted mineral matter in the two cones plus the total weight of ash in the extract shall be taken as the total aggregate sample weight. The ash in the extract and the fine mineral matter retained by the filter papers after brushing shall be included in the total weight passing the No. 200 sieve.

Warranty

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