RECOVERY OF ASPHALT FROM EXTRACTION SOLUTION

(An Arizona Method)

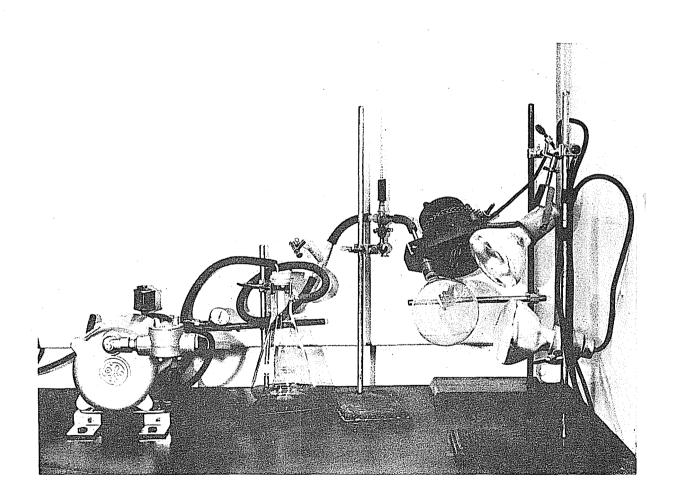
Scope

1. This method describes a procedure to recover an asphalt residue from an extraction solution. The asphalt is recovered with substantially the same properties that it possessed in the asphaltic mix.

Apparatus

2. The apparatus shall consist of the following:

- (a) Stirring hot plate with stirring bar.
- (b) 4000 ml. beaker
- (c)* 1000 ml. beaker
- (d) Funnels



- (e)* Centrifuge-capable of maintaining 1800 rpm and equipped with a minimum of four centrifuge bottles.
- (f) Rotary Vacuum Evaporator apparatus as listed below and as shown assembled in Figure 1:
 - 1) Rotary Vacuum Evaporator
 - 2) 1000 ml. round bottom flask
 - Heat Source two 250 watt heat lamps placed approximately 2 inches from the 1000 ml. round bottom flask.
 - 4) 1000 ml. filtration flask
 - 5) Vacuum Pinch Release Clamp
 - 6) Vacuum Pump capable of maintaining a vacuum of 15 to 20 inches mercury. The pump exhaust must be vented directly into the hood exhaust system.
 - Vacuum gauge with adjustable valve for regulating the vacuum.
 - 8) Thermometer with range of 78° F. to 130° F. in 0.2° F. increments placed in the vacuum line within 4 inches of the Rotary Vacuum Evaporator.

*Required only when utilizing the alternate procedure (Section 4).

Procedure

- 3. NOTE: This procedure may be used if the extraction solution does not contain fine aggregate material.
- (a) Pour the extraction solution into the 4000 ml. beaker which contains a stirring bar.
- (b) Heat at medium setting (#4) while stirring on the Hot Plate, until 400 to 450 ml. of the solution remains.
- (c) Remove Stirring Bar and using Funnel pour solution into the $1000\ \mathrm{ml.}$ round bottom flask.
- (d) Wash inside mouth and top 1/2 of flask with a small amount of extracting solvent.

CAUTION: It is important that a maximum of 500 ml. of solution be in flask to prevent solution from being drawn into the vacuum line.

(e) Place flask on Rotary Vacuum Evaporator, turn on vacuum (which has been adjusted to maintain 15-20 in, mercury) and make sure flask is properly seated in position on the shaft, then turn on the rotor and the heat lamps.

CAUTION: If the vacuum is released during the procedure it is possible that the flask will slide off of the shaft. If it becomes necessary to release the vacuum, provision must be made to hold flask in place with gloved hand.

- (f) Allow to run while a rise in temperature, followed by a decline in temperature is noted. When the temperature drops to a stable condition and either holds at that temperature for 5 minutes or shows a rise of 0.2° F. the removal of all solvent is indicated.
- (g) While holding the neck of the flask with glove suitable for high temperatures turn off the rotor and then the vacuum.

NOTE: If vacuum is maintained at 15-20 in. mercury, there should be no occurence of the asphalt being drawn up into the vacuum line, however, if this does occur, the vacuum should be released immediately by use of the pinch release clamp, adhering to CAUTION following paragraph 3 (e). Using gloves remove flask from shaft, then clean vacuum system. Check volume of solution in flask and reduce to a maximum of 500 ml. if necessary. If reduction is not required, vacuum should be reduced to a minimum of 15 inches mercury. This should prevent further occurences and after adhering to precaution in paragraph 5 (b), the flask can be returned to rotor and the procedure continued.

(h) Remove the flask from the rotor shaft and pour asphalt into a 6 $\,$ oz. tin.

NOTE: When the flask is removed from the rotor shaft it is possible that the small "0" ring on the shaft will be removed with the flask. If this happens a glass rod may be used to remove the "0" ring from the mouth of the flask before the asphalt is poured into the tin.

- (i) Turn off the heat lamps.
- (j) Asphalt is now ready for further testing.

Alternate Procedure

- 4. NOTE: This procedure is required when the use of a centrifuge is necessary for extraction solutions containing fine aggregate material.
- (a) Pour the extraction solution into the 4000 ml. beaker.
- (b) Heat at medium setting (#4) while stirring on the Hot Plate, until 400 to 500 ml. of the solution remains.
- (c) Remove Stirring Bar and pour solution into a $1000\ \mathrm{ml.}$ beaker.
- (d) Pour approximately equal amounts of the solution into centrifuge bottles as necessary. The filled centrifuge bottles must weigh within 1 gram of each other to properly balance the centrifuge. To bring the bottles to the correct weight add clean extracting solvent to the centrifuge bottles as needed.

- (e) Place bottles in centrifuge so that they are properly balanced and run at 1800 rpm for approximately 45 minutes.
- (f) Remove bottles and using funnel pour approximately 450 ml. of solution into the 1000 ml. round bottom flask. The solution must be carefully poured so that the fine aggregate material on the bottom of the centrifuge bottle is not disturbed.
- (g) Wash inside mouth and top 1/2 of flask as in paragraph 3 (d) and follow original procedure to completion.

Precautions

5. (a) All work with extraction solution shall be done under a functioning fume hood.

- (b) Flask should not be removed from rotor until the recovery of asphalt is complete. If it becomes necessary to remove the flask, the flask and the sample shall be allowed to cool, any asphalt inside mouth or top 1/2 of the flask washed down with a small amount of extracting solvent, and the volume checked to not exceed 500 ml. before being replaced on the rotor shaft. (This will prevent the material from being drawn into the vacuum line.)
- (c) Temperatures of material in flask during recovery exceed 300° F. Recovery of asphalt which contains hazardous materials at this temperature, such as sulphur, shall not be attempted with this procedure.