

## VACUUM RECOVERY OF ASPHALT EMULSION RESIDUE (An Arizona Method)

### Scope

1. This method describes a low temperature vacuum procedure for recovery of the asphalt residue from asphalt emulsions. It is not suitable for quantitative recovery of solvents from emulsions containing low boiling range distillates.

### Apparatus

2. The apparatus shall consist of the following:

- (a) Brass stirring rod.
- (b) 8 oz. ointment can.
- (c) 100 ml. stainless steel beaker.

(d) No. 20 sieve conforming to AASHTO designation M 92.

(e) Vacuum recovery apparatus as shown assembled in Fig. 1.

- 1) Vacuum source capable of producing an absolute vacuum within the system of approximately 710 mm (28 in.) mercury.
- 2) Thermometer — shall have a range of  $-5^{\circ}$  to  $+200^{\circ}\text{C}$  ( $23^{\circ}\text{F}$  to  $392^{\circ}\text{F}$ ). The overall length shall be 600 mm (24 in.) and the distance from the bottom of the bulb to the zero point shall be 300 mm (12 in.)
- 3) Stirrer hot plate.

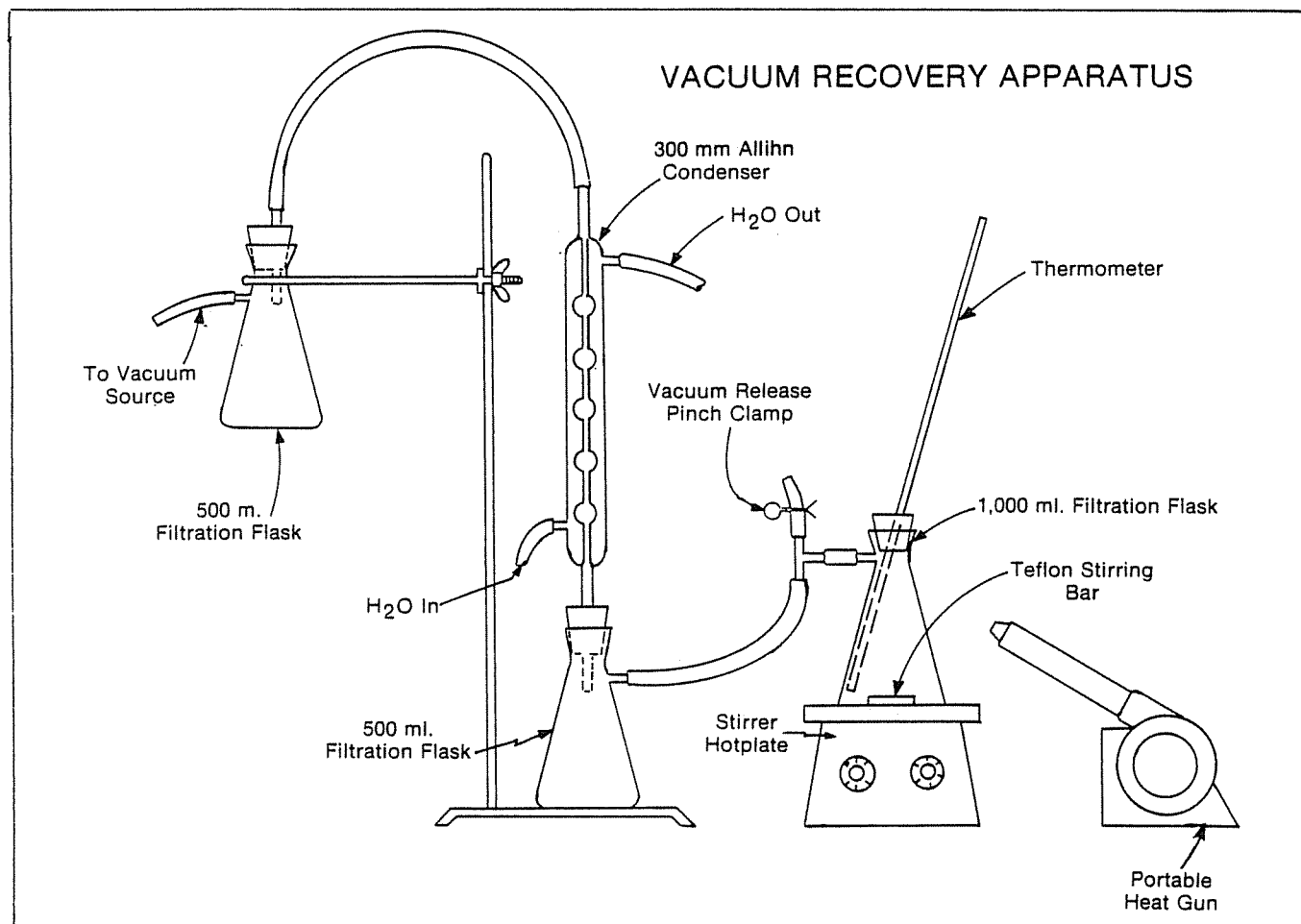


FIGURE 1

- 4) Teflon covered stirring bar.
- 5) 1000 ml. and two 500 ml. filtering flasks with tubulation.
- 6) 300 mm Allihn condenser.
- 7) Portable heat gun.
- 8) Miscellaneous rubber stoppers and connecting hose.
- 9) Vacuum release clamp.

(f) Additional vacuum recovery apparatus for alternate test procedure.

- 1) 8" waterbath — hemispherical.
- 2) 500 ml. Separatory Funnel — pear shape.

### Material

3. Isopropyl alcohol, technical grade.

### Sample Preparation

4. Emulsion shall be thoroughly stirred and strained through a No. 20 sieve just prior to use.

### Test Procedure

5. (a) Pour  $750 \pm 25$  ml. of the strained emulsion into a 1000 ml. stainless steel beaker.

(b) Cool the emulsion to approximately  $25^{\circ}\text{C}$  ( $77^{\circ}\text{F}$ ).

(c) Add isopropyl alcohol (approximately 25 to 100 ml.) in sufficient quantity to break the emulsion.

(d) Knead lumps with brass stirring rod against the sides of the beaker to expel the water from the broken asphalt.

(e) Apply a film of soap around neck of the 1000 ml. vacuum flask to prevent asphalt from sticking to the flask.

(f) Wet hands thoroughly with distilled water. Remove the stirring rod on which the broken asphalt emulsion (now asphalt) is clinging, and with the wet hands gently squeeze out all entrained water while washing asphalt under a constant flow of distilled water. Form asphalt into a rope and drop into the 1000 ml. flask containing the Teflon stirring bar. (Amount of asphalt to be added should be enough to cover bottom of flask approx. 1" in depth). Wash soap off of flask and rinse soap from sample and flask with distilled water making sure water is drained from flask before it is placed on hot plate.

(g) Insert the stoppered thermometer (positioned in the stopper at an angle to prevent contact with stirring bar) into the flask and set on hot plate at a medium high heat setting (#4). The bulb of the thermometer should be 6 mm. ( $\frac{1}{4}$  inch) from the bottom of the flask.

(h) Turn on the water to the condenser, start the stirring bar and open the vacuum line to establish a vacuum of approximately 710 mm (28 in.) mercury in the apparatus.

(i) Turn on the portable heat gun to medium high heat (#6) and use wherever needed to keep condensation from forming inside the vacuum flask.

(j) Allow temperature to rise until it reaches approximately  $90^{\circ}\text{C}$ ., turn hot plate down to setting #2 and then allow the temperature of the residue to rise until it reaches  $115^{\circ}\text{C} \pm 3^{\circ}\text{C}$ . ( $239^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ). Hold at this temperature for at least 1 minute (if possible).

*NOTE: The hot plate temperature will need to be regulated during heating of residue to allow for complete removal of water. (This removal is complete when there is no longer foaming in the residue.) If the  $115^{\circ}\text{C}$  temperature is reached and there is still water present, the flask should be removed from the hot plate, allowed to cool a short time, and then heating resumed at a reduced temperature. As the residue heats up, soap bubbles will form up into the vacuum line and condense in the filtering flask. Some emulsions may react violently between  $60^{\circ}\text{C}$  ( $140^{\circ}\text{F}$ ) and  $105^{\circ}\text{C}$  ( $221^{\circ}\text{F}$ ) and rise up in the flask. Use the vacuum release clamp as often as needed to control the asphalt. DO NOT let asphalt escape into the vacuum line.*

(k) Turn off vacuum and slowly release vacuum through apparatus using release clamp. Stop stirrer, remove thermometer, and pour residue into a 8 oz. ointment can. Residue is now ready for specified tests.

### Alternate Test Procedure

6. This procedure is recommended for use with emulsions that will not break using isopropyl alcohol.

(a) Pour 350 ml. to 400 ml. of the strained emulsion into the separatory funnel.

(b) Place the stoppered funnel into the 1000 ml. flask and carefully set the unit into the preheated water bath (Setting #6).

(c) With the water circulating through condenser, open the vacuum line to establish a vacuum of approximately 710 mm (28 in.) mercury in the apparatus.

(d) Start the stirring bar at slow speed and slowly add the emulsion at a rate which allows the emulsion to break as it leaves the funnel. (This is indicated by the presence of foaming at the end of the funnel.

*CAUTION: If the emulsion is added to the flask too rapidly, it may react violently and rise up in the flask. If this happens quickly return addition of emulsion to proper rate and use the vacuum release clamp to prevent asphalt from entering into the vacuum line.*

(e) Continue adding emulsion to flask until all of it is removed from the funnel.

(f) Close the vacuum line and slowly release vacuum from unit. Stop the stirring bar.

(g) Remove the separatory funnel and the water bath. Wipe bottom of flask dry, insert the thermometer as in 5 (g) and follow original method to completion.