

## METHOD OF TEST FOR RECOVERY OF ASPHALT FROM SOLUTION BY ABSON OR ROTAVAPOR

### 1. SCOPE

- 1.1 This method covers the recovery, by the Abson or Rotavapor Method, of bitumen from asphaltic mixtures with certified A.C.S. trichloroethylene.
- 1.2 This recovered bitumen has properties substantially the same as those it possessed in the asphaltic mixture, and is recovered in sufficient quantities for further testing.

### 2. RELEVANT DOCUMENTS

- 2.1 ASTM D 1856
- 2.2 ASTM D 2172
- 2.3 ASTM Rotavapor (Draft 5)

### 3. EXTRACTION APPARATUS

- 3.1 ROTAREX EXTRACTOR: Either hand or electrically operated, complete with a 1000 g capacity bowl.
- 3.2 TUBING: 10 mm inside diameter neoprene tubing to drain extractor.
- 3.3 TORQUE WRENCH: 9.5 mm drive, calibrated in N·m with a capacity of approximately 22 N·m.
- 3.4 SOCKET: 9.5 mm drive, 19.1 mm size with two slots milled out of the sides, to fit over the steel rod in the extractor unit funnel.
  - i) A 19.1 mm socket with two slots milled out of the sides to fit over the steel rod in the extractor unit funnel, or
  - ii) A fitted socket and socket head each of 1 cm (3/8 in) size.
- 3.5 OVEN: A gravity convection oven of suitable capacity and capable of maintaining the temperature at 93 to 105°C.
- 3.6 CENTRIFUGE
  - 3.6.1 Batch Unit: capable of exerting a minimum centrifugal force of 770 times gravity. This unit must be capable of handling a minimum of two 250 ml capacity glass bottles.
  - 3.6.2 Continuous Unit: high speed centrifuge as described in ASTM D-2172 Section 11.6.2.1.

### 4. ABSON RECOVERY APPARATUS

- 4.1 CYLINDER: Carbon dioxide gas with pressure reducing valve.
- 4.2 VARIABLE ELECTRICAL TRANSFORMERS

- 4.3 ELECTRIC HEATING MANTLE: 2000 ml capacity.
- 4.4 ELECTRICAL HEATING MANTLE: 500 ml capacity.
- 4.5 BOILING FLASK: 2000 ml capacity, round bottom with standard taper joints 24/40.
- 4.6 DISTILLING FLASK: 500 ml capacity, 2 neck with standard taper joints (24/40 and 34/45 sizes).
- 4.7 SUPPORT STAND: Large.
- 4.8 RING SUPPORT: For 2000 ml capacity heating mantle with clamp.
- 4.9 RING SUPPORT: For 500 ml capacity heating mantle with clamp.
- 4.10 GAS FLOWMETER: 10-1900 ml/min air flow capacity.
- 4.11 GAS FLOWMETER SHIELD: For above.
- 4.12 FIBREGLASS MUFFLERS: To fit over distilling flasks, (i) 500 ml size, (ii) 2000 ml size. Made from 50 mm thick fibreglass.
- 4.13 TRICHLOROETHYLENE: Certified A.C.S. grade.
- 4.14 GAS MANIFOLD: With regulating valves for CO<sub>2</sub> gas control, for multiple secondary distillations or use individual valves as required.
- 4.15 CONDENSER: Graham, jacket length 200 mm minimum.
- 4.16 GRADUATED CYLINDERS: Capacity of 500 ml used to measure the rate of flow of the extract during the removal of mineral fines. Use 2000 ml cylinders if they are being used as receivers for the total volume of extract.
- 4.17 FLASKS: Flat bottom, long neck, 2000 ml capacity for (i) recovery of bitumen solution from extraction, (ii) recovery of trichloroethylene from the primary distillation, (iii) 250 ml capacity for recovery of trichloroethylene from the secondary distillation.
- 4.18 BOILING CHIPS
- 4.19 GLASS CONNECTING TUBES: 6.4 mm inside diameter formed as shown in Figures 1 and 2.
- 4.20 RUBBER TUBING: 6 mm inside diameter for condensers.
- 4.21 CENTRIFUGE BOTTLES: 250 ml capacity.
- 4.22 GLASS OR PLASTIC FUNNEL: 100 mm in diameter.
- 4.23 AERATION TUBE: Copper - 4 mm inside diameter and 200 mm in length with one end cut at a 45° angle.
- 4.24 PLASTIC TUBING: 6.4 mm inside diameter for CO<sub>2</sub> gas distribution.
- 4.25 CORK STOPPERS: For distilling flasks - 500 ml and 2000 ml capacity flasks.
- 4.26 THERMOMETER: An ASTM thermometer 7C with a range of -2 to 300°C conforming to the requirements of ASTM Designation E 1.

## 5. ROTAVAPOR RECOVERY APPARATUS

5.1 ROTAVAPOR: Equipped with distillation flask, variable speed motor capable of rotating the flask at the rate of at least 50 rpm, condenser, solvent recovery flask and a heated oil bath. The angle of the distillation flask from the horizontal to the bath is set at approximately 15°. The distillation flask, when fully immersed, should be at a depth of approximately 38 mm (1.5 in.). See Figure 3, 4, and 5 for basic and optional equipment installations. See Note 1.

5.2 WATER BATH: Ice in water bath for cooling of the solvent recovery flask.

5.3 COLD TRAP: A vacuum flask in ice water is satisfactory.

5.4 MANOMETER, GAUGE OR PRESSURE CONTROLLER: Suitable for measuring the specified pressure (vacuum), (See Note 2).

5.5 WOULFE BOTTLE: Used only if pressure controller is included in the system (see Figure 5).

5.6 GAS FLOWMETER: Capable of indicating a gas flow of up to 1000 ml/min.

5.7 VACUUM SYSTEM: Capable of maintaining a vacuum to within  $\pm 0.5$  mm ( $\pm 0.02$  in.) of the desired level up to and including 600 mm (23.6 in.) Hg.

5.8 NITROGEN GAS: Cylinder of nitrogen gas fitted with a pressure reducing valve.

5.9 TRICHLOROETHYLENE: Certified A.C.S. grade.

*Note 1: The Buchi Rotavapor RE-121A has been found to be satisfactory for this purpose.*

*Note 2: The Buchi B-161 digital vacuum controller has been found to be satisfactory for this purpose.*

## 6. RECOVERED ASPHALT CEMENT APPARATUS

3.4.1 BEAKER: Glass, 30 ml capacity, to pour recovered bitumen for kinematic viscosity testing.

3.4.2 PENETRATION CANS

3.4.3 SCREEN: 1.18 mm, 120 mm square.

3.4.4 SCREEN: 150  $\mu$ m, 120 mm square.

## 7. EXTRACTION PROCEDURE

7.1 Place a 2000 g sample into a large flat pan, and heat in an oven maintained at a temperature of 93 to 105°C for a period of 15 minutes.

7.2 Remove the sample from the oven. Using a large spike or a stiff spatula, break down the mix as much as possible.

7.3 Return the sample to the oven for an additional 30 minutes of heating.

7.4 After 30 minutes, remove the sample from the oven, and break down the mix as much as possible.

- 7.5 Transfer about 1000 g of the sample to a balance pan and measure the mass of the mix to 0.1 g
- 7.6 Clean and flush the extractor bowl, the inside of the extractor and the drain tube with fresh trichloroethylene.
- 7.7 Place the measured mass of mix in the bowl and distribute evenly. Add 350 ml of certified trichloroethylene and break up the mix further until all the particles are submerged.
- 7.8 Place the dried filter paper and the lid on the extractor bowl and tighten the lid to 11.3 N·m of torque. Allow the sample to soak for 15 minutes either in the stand or in the extractor.
- 7.9 At the end of the 15 minute period, with the bowl in the extractor and the lid in place, and a 2000 ml flat bottom flask to collect the asphalt solution, start the extractor slowly.
- 7.9.1 With the hand-operated extractor, turn the handle slowly, gradually increasing the handle speed to 50 rpm. Maintain this rate until the solvent ceases to flow from the drain tube.
- 7.9.2 With the electrically-powered extractor, calibrate and set the speed control for a speed of 1800 rpm. Start the machine, slowly letting it increase speed to a maximum of 1800 rpm. until the solvent ceases to flow.
- 7.10 Add 200 ml of certified trichloroethylene to the mix, allowing it to stand for 10 minutes, and repeat steps 4.9.1 or 4.9.2.
- 7.11 Add 200 ml of certified trichloroethylene to the mix. Allow to stand for 5 minutes, and repeat steps 4.9.1 or 4.9.2.
- 7.12 Repeat sections 4.5 through 4.11 with the second batch of 1000 g of mix.
- 7.13 Removal of Mineral Fines - Alternative 1
- 7.13.1 Transfer the asphalt solution to the 250 ml glass bottles, adjusting the amount of solution so that each bottle of solution weighs the same. Place the bottles in the centrifuge, and centrifuge at the maximum rate for 10 minutes.
- 7.14 Removal of Mineral Fines - Alternative 2
- 7.14.1 The following procedure is appropriate when using the SMM high speed centrifuge. Place a clean, previously tared, centrifuge cup in the high speed centrifuge.
- 7.14.2 Start the centrifuge and allow it to reach constant operational speed (at least 9000 r.p.m.). Close the feed funnel control valve and place a suitable receiver at the effluent drain to collect the solvent. Fill the feed funnel with extract. Open the control valve at the bottom of the funnel to allow a flow of 100 to 150 ml/min using a graduated cylinder to measure the centrifuge effluent flow rate. Continue to top up the feed funnel until all the extract has been transferred. Wash the container with several small volumes of fresh solvent and transfer each to the feed funnel. Wash the funnel with fresh solvent. Allow each increment of solvent from these washings to run through the centrifuge until the effluent is essentially colourless. Allow the centrifuge to run several more minutes to evaporate the remaining solvent.

## 8. ABSON RECOVERY PROCEDURE

### 8.1 PRIMARY DISTILLATION, (See Figure 1)

8.1.1 After centrifuging, decant the solutions from both extracted portions into a 2000 ml round bottom flask, add a few boiling chips, and assemble the apparatus. Place the fibreglass cover on the flask, and commence the primary distillation.

8.1.2 Adjust the variable electric transformer so that the distillate comes off in a slow, steady stream into a flask. When the asphalt solution is reduced to a volume of approximately 200 ml, transfer the solution and boiling chips into the secondary distillation flask.

### 8.2 SECONDARY DISTILLATION, (See Figure 2)

8.2.1 Preheat the secondary flask. Assemble the apparatus. Insert the copper tube and thermometer through a cork in the centre neck of the flask. The thermometer is set 6.4 mm from the bottom of the flask. The CO<sub>2</sub> tube is set above the surface of the solution temporarily. Place the fibreglass cover over the flask.

8.2.2 Adjust the variable electric transformer so that the distillate barely comes off in a steady stream. Collect the distillate in a suitable container. When the temperature of the solution reaches 115°C, lower the copper tube until the tapered end touches the bottom of the flask with the open end facing away from the thermometer. Introduce the CO<sub>2</sub> gas at a rate of 100 ml per minute, and maintain a distillation rate of 50 to 70 drops per minute. Increase the CO<sub>2</sub> gas flow rate to maintain the 50 to 70 drops per minute of distillation until the maximum gas flow rate of 900 ml/minute is attained. Increase the temperature of the solution to a maximum of 150°C at a rate to maintain the desired distillate flow of 50 to 70 drops per minute. Maintain a temperature of 150°C and the CO<sub>2</sub> gas flow rate of 900 ml/minute for a period of 20 minutes.

8.2.3 After the distillation is stopped, disassemble the apparatus and pour the residue through a 1.18 mm mesh sieve into a 236 ml container placed on its lid.

*Note 3: This sample may be transferred immediately to the proper size can for the penetration determination. If kinematic viscosity is to be determined pour approximately 20 ml through a 150 µm mesh sieve into a 30 ml beaker and transfer to the appropriate viscosity tube. If reheating is required, heat the sample to the lowest temperature which will result in a consistency which will just allow the asphalt to be poured without the inclusion of air bubbles. Stir carefully in a horizontal fashion. In no case should the temperature be raised to more than 90°C above the softening point of the specimen. Approximately 20 ml is poured through a 150 µm mesh sieve into a 30 ml beaker for kinematic viscosity testing.*

## 9. ROTAVAPOR RECOVERY PROCEDURE

9.1 Heat the oil bath to a temperature of 138°C ± 2.8°C

9.2 Circulate cold water through the condenser

9.3 Apply a vacuum of  $40 \pm 0.5$  mm Hg below atmospheric pressure and draw approximately 600 ml of asphalt solution from the sample container into the distillation flask via the sample line. (for configurations in Figures 4 and 5 transfer all of the extract, normally about 1500 ml, to the 2000 ml distillation flask and fit the flask to the rotator assembly prior the applying the vacuum).

9.4 Begin a nitrogen flow of approximately 500 ml/min through the system. (Note 3). Begin rotating the distillation flask at approximately 40 rpm and lower flask into the oil bath. Initially the immersion depth of the flask will be determined by the need to achieve a controlled solvent evaporation rate. The correct evaporation rate can be observed as a steady controlled stream of condensed solvent being collected in the recovery flask.

*Note 4: Vacuum and nitrogen flow valves may require adjusting depending on location. Also, low flow values are recommended at the beginning of the rotary process when sample volume is large because of the possibility of back flow into the vacuum system. The possibility of back flow can be minimized by regulating the depth of immersion of the flask in the oil bath.*

9.5 When the amount of asphalt solution within the distillation flask appears low enough so that more solution may be added, (Figure 1) discontinue the nitrogen flow. Draw the remaining asphalt solution from the sample container into the distillation flask and readjust the nitrogen flow (Note 4).

*Note 5: The equipment may be modified to allow a continuous flow of solution from the sample container into the distillation flask such that the volume in the distillation flask is maintained at approximately 600 ml. The nitrogen flow is not started until all the solution has entered the distillation flask.*

9.6 When the bulk of the solvent has been distilled from the asphalt and no obvious condensation is occurring on the condenser, immerse the distillation flask to the recommended maximum immersion depth of 38 mm (1.5 in). A one to two minute delay before applying the vacuum is recommended. Slowly apply a vacuum of  $600 \pm 0.5$  mm Hg below atmospheric pressure. Increase the nitrogen flow to approximately 600 ml/min and the spin rate to about 45 rpm (Note 5). Hold or reduce the vacuum if foaming or a bubbly formation occurs. When foaming subsides apply maximum vacuum. Maintain this condition for  $15 \pm 1$  minute.

*Note 5: A faster spin rate exposes more surface area of the asphalt and hence dislodges more traces of solvent from the asphalt. The rotation speed of the flask may also be varied of the flask rotation may be stopped to enhance the removal of solvent. Due to the cooling effect of the nitrogen flow, an increase in the temperature of the oil bath is generally needed to maintain a constant sample temperature. Experience has shown that a typical bath temperature range of  $149 - 157^{\circ}\text{C}$  ( $300 - 315^{\circ}\text{F}$ ) is satisfactory for this purpose.*

9.7 At the end of the 15 minute period, remove the distillation flask from the apparatus and cover the neck loosely with aluminium foil.

*CAUTION: Nitrogen flow should always be discontinued and system opened to atmosphere before vacuum pump is turned off to ensure that a positive pressure is not built up in the system.*

*Place the flask in an oven at 163°C (325°F) for 30 ±1 min (Note 6). After 30 minutes in the oven remove the flask, wipe the neck clean of any oily residue and pour into the containers prepared for subsequent testing of the asphalt as indicated in section 5.2.3.*

*Note 6: Experience has shown that for samples having penetration in excess of about 80 penetration a heating period in the oven at 163°C of fifteen minutes is sufficient to facilitate pouring of the asphalt.*

## **10. SAFETY**

10.1 The use of trichloroethylene requires that testing be carried out under vapour evacuation conditions (i.e. fume hood), because the solvent is toxic to some degree.

10.2 Neoprene gloves and safety goggles must be worn when handling trichloroethylene.

10.3 In case of spills of trichloroethylene appropriate respirators must be worn. (Occupational Health and Safety Act: R.R.O. 1980, Reg. 692, S. 134-C). See also the manufacturer MSDS.

10.4 Eating, drinking or smoking is not allowed in the working area of the laboratory.

## **11. GENERAL NOTES**

11.1 The test must be completed within 8 h.

11.2 It may be necessary to calibrate the gas flow meter to determine the correction for the gas flow rate of 900 ml/minute for CO<sub>2</sub> gas.

11.3 Perform all operations under a fume hood, and avoid inhaling trichloroethylene fumes when transferring solution from the primary to the secondary distillation.

11.4 Wear heat resistant gloves when handling hot equipment.

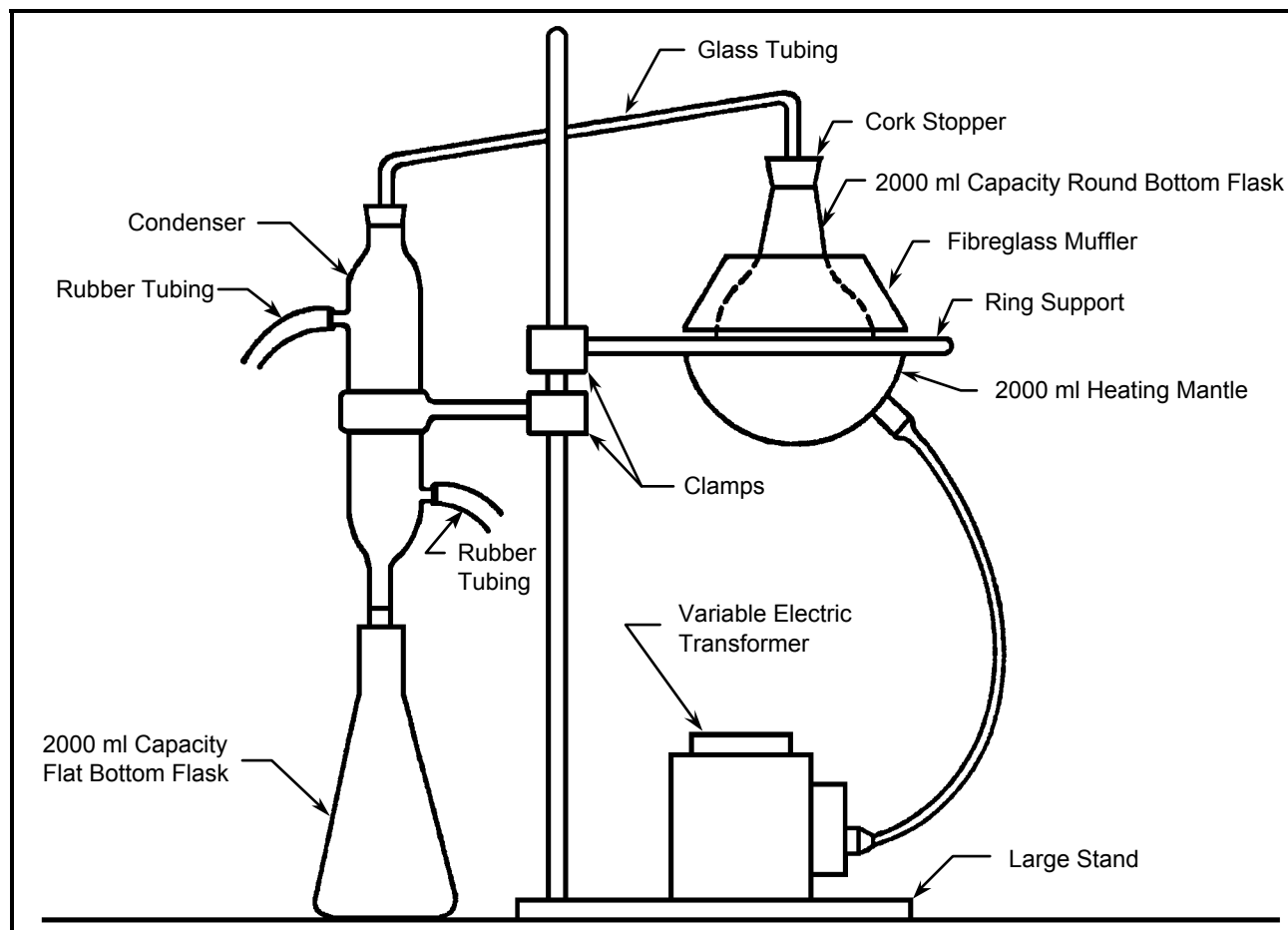


Figure 1  
Abson Primary Distillation System



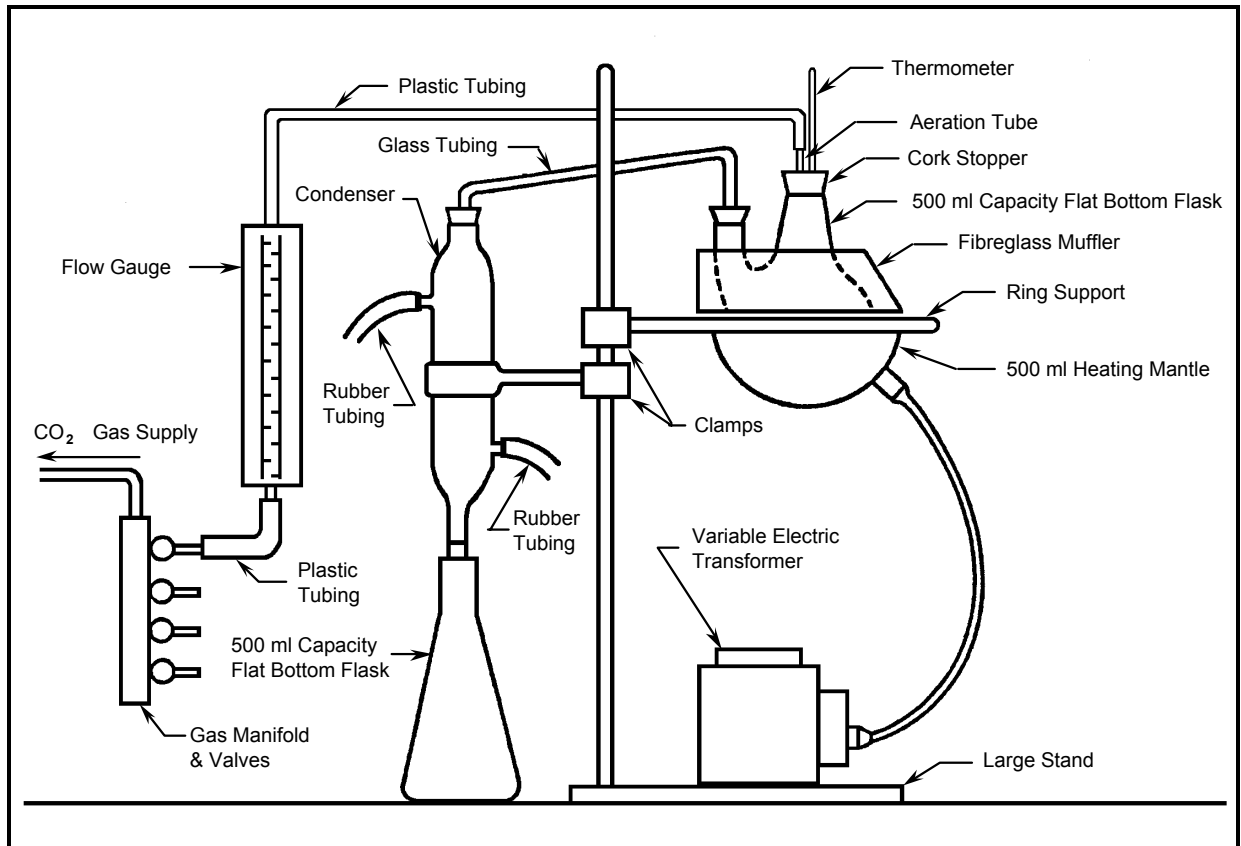


Figure 2  
Abson Secondary Distillation System

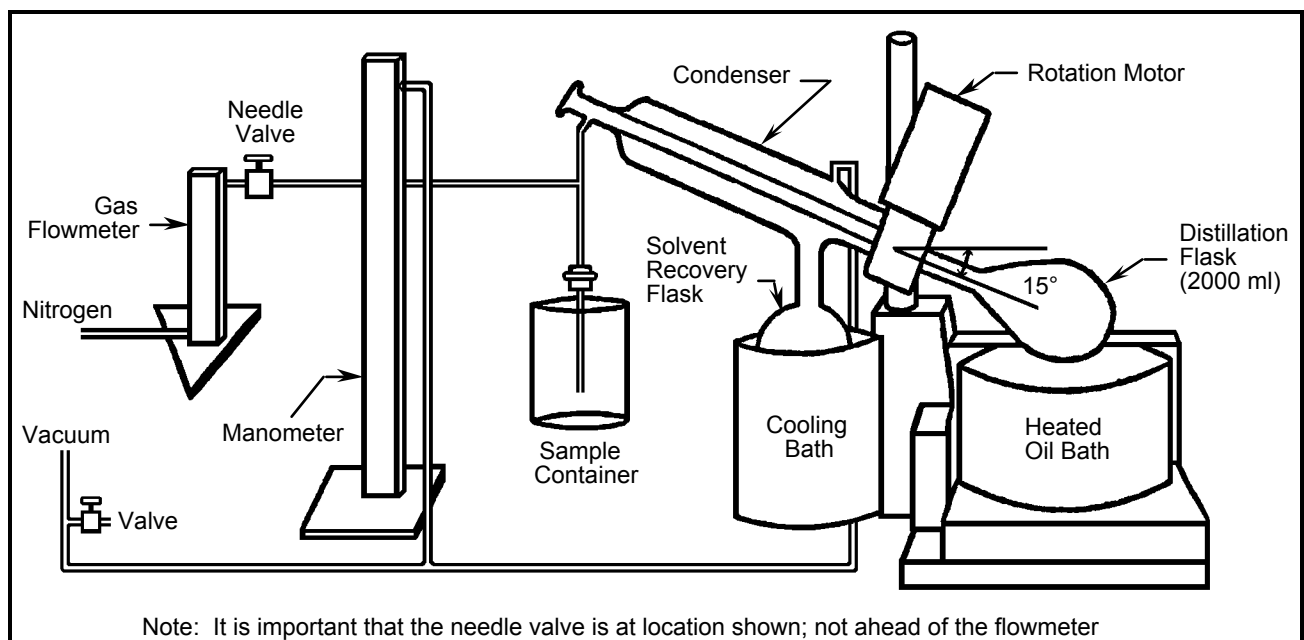


Figure 3  
Rotavapor Apparatus And Recovery System  
(Option A)

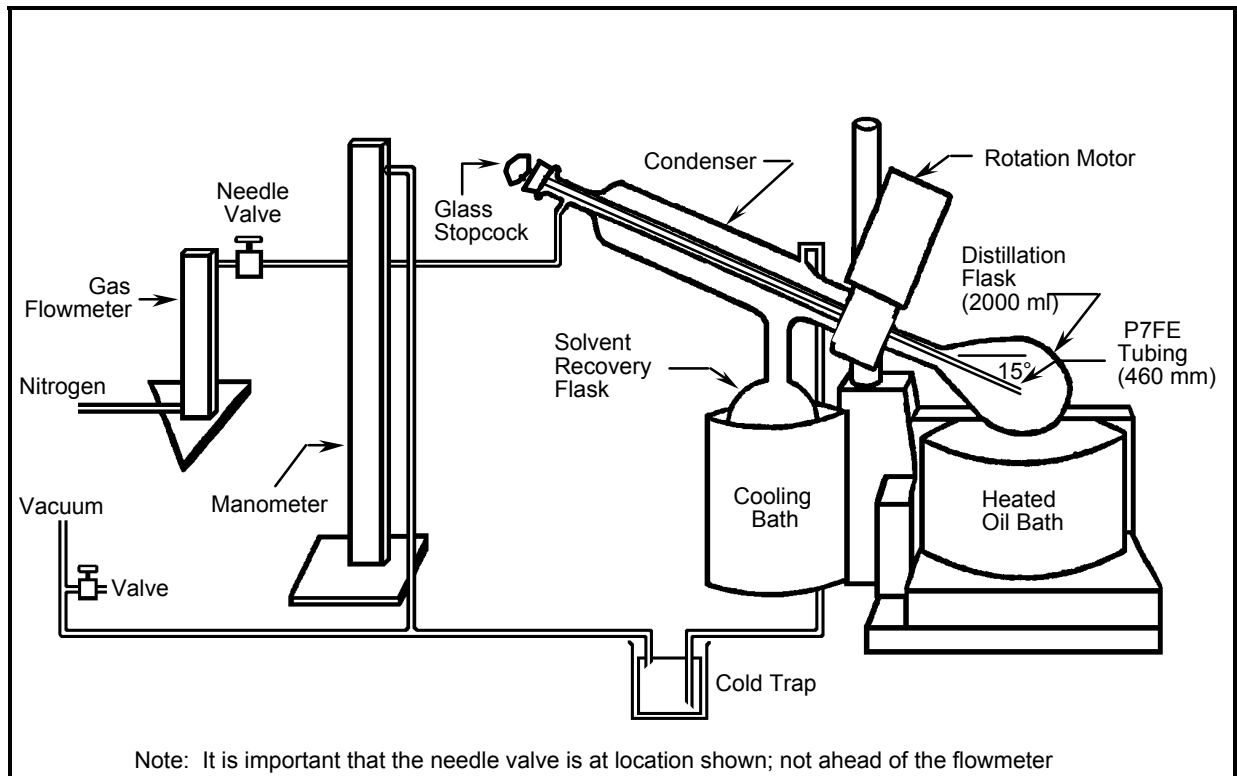


Figure 4  
Rotavapor Apparatus And Recovery System  
(Option B)

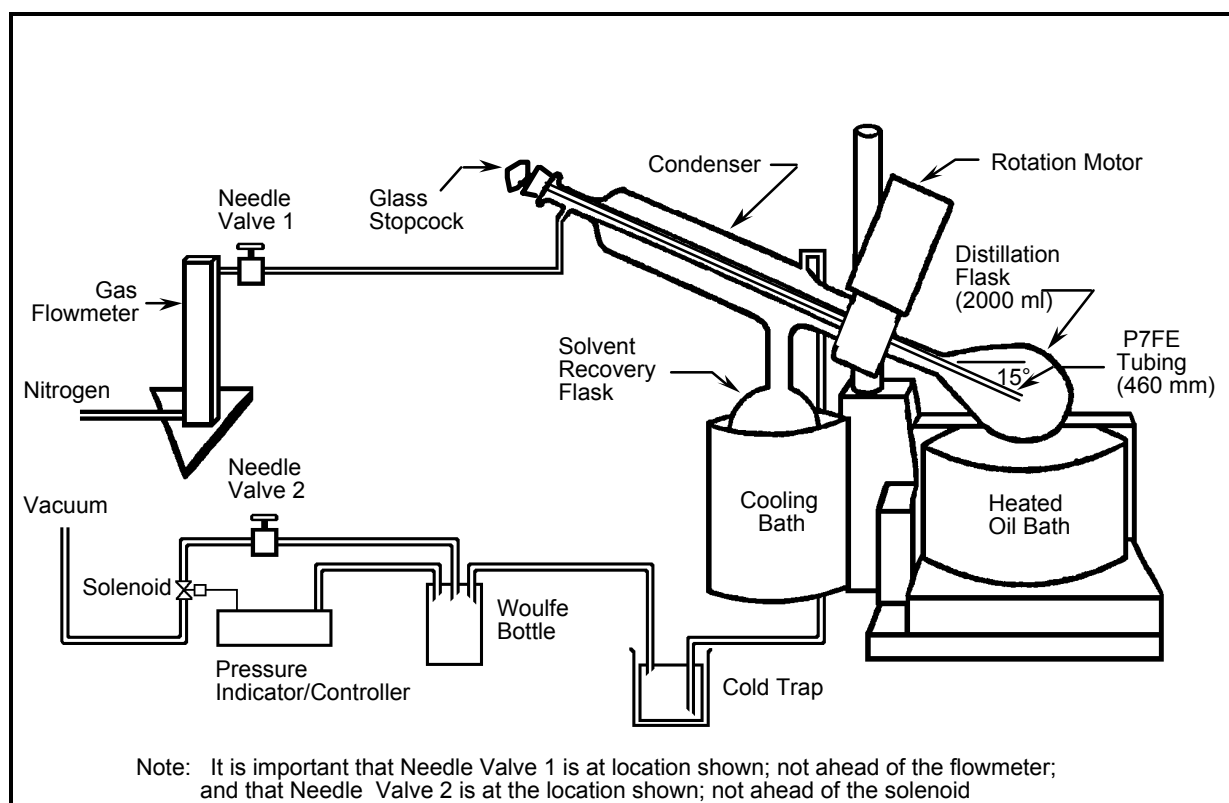


Figure 5  
Rotavapor Apparatus And Recovery System  
(Option C)