

INTERLABORATORY TESTING OF THE SHRP
EXTRACTION AND RECOVERY PROCEDURE
FOR ASPHALT

by
Vidyasagar Jagadam
and
Joe W. Button

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Texas Transportation Institute
Texas A&M University
College Station, Texas

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INTRODUCTION

This report describes the interlaboratory evaluation (round-robin tests) of the extraction and recovery procedure developed in SHRP project A-002a, Task 2.1. This procedure was developed to provide a method to obtain asphalts from new or old paving mixtures with minimum change in their physical and chemical properties.

Background

Separation of asphalt from the aggregates is required in order to study the changes that take place in the physical and chemical properties of the asphalt during plant mixing operations and pavement aging. Extraction is also used when the binder content of a paving mixture is desired. The only acceptable method for this process has been and remains to be solvent extraction. The commonly used procedures are frequently found to be unsatisfactory for the following reasons:

- 1) the asphalt is not completely removed from the aggregate,
- 2) the solvent is not completely removed from the recovered asphalt, and
- 3) the recovered asphalt is hardened by contact with the solvent.

Such errors pose a serious problem for research into properties of hot-mix and road-aged asphalts. Research was carried out by the Texas Transportation Institute (TTI) Chemical Engineering group to eliminate or minimize the above effects. They developed a new extraction and recovery method in an attempt to minimize the effects of temperature, solvent exposure, time of contact with solvent, and thus asphalt hardening. The method developed by these researchers was used in the interlaboratory test program to evaluate the test procedure.

Objective

The objective of this work was to conduct an interlaboratory evaluation of the SHRP extraction and recovery procedure and to provide data that can be used to estimate the repeatability and reproducibility of the procedure

as developed. The data obtained were used to prepare a precision and bias statement based on response variables (penetration and viscosity) of the extracted asphalt. Limited tests were performed to compare the new method to the existing standard methods of extraction and recovery. This was accomplished by applying these methods of extraction in the laboratory on the same paving mixture and comparing the properties of the recovered asphalts.

Scope

The interlaboratory test program was conducted at ten different laboratories with two replicates each of two different asphalt paving mixtures. The two replicates of each test were conducted by the same technician, making it possible to determine the within-laboratory variability (repeatability). The data from different laboratories were used to compute the between-laboratory variability (reproducibility).

DESCRIPTION OF EXPERIMENT

Summary of the SHRP Extraction/Recovery Method

The paving mixture is repeatedly washed and filtered using toluene and toluene with 15 percent ethanol in the extraction/filtration apparatus. Each successive filtrate is distilled under vacuum in a rotary evaporator in a stepwise process with the asphalt being concentrated in a boiling flask. After recovery of the final filtrate, the solution is concentrated to about 300 ml and centrifuged to remove aggregate fines. The decanted solution is distilled under vacuum to remove the extraction solvents. Nitrogen gas is introduced during the final phase of the distillation to aid in driving off any remaining traces of the solvents. The recovered asphalt may then be subjected to further testing as required. The method can be used both for obtaining asphalt for further analyses and for calculation of asphalt content in pavement samples. A detailed description of the new SHRP extraction and recovery procedure is given in Appendix A.

Materials Tested

Two different asphalt concrete plant mixtures were included in the test program. All samples of each of the two mixtures were obtained and packaged at the same time to minimize variability between replicate pairs. Properties of the original asphalts used in the mixtures are given in the Table 1.

Table 1. Properties of Original Asphalts used in Mixtures Subjected to Round Robin testing.

Mix I.D.	Sample I.D.	Pen @ 25°C, dmm	Vis. @ 60°C, Pa.s
Mixture A	Neat A.C.	58	302
	Neat A.C.> TFOT	40	721
Mixture B	Neat A.C.	79	226
	Neat A.C.> TFOT	47	382

Experimental Design

Controlled Variables: The following *control variables* were used for this experiment:

- a) Asphalt concrete mix type, 2 levels (plant mixes)
- b) Solvent type, 1 level
- c) Replicates, 2 levels
- d) Laboratories, 10 levels
- e) Technicians, 1 level

Response Variables: The *response variables* measured for each recovered asphalt specimen included the following:

- a) Viscosity of asphalt at 60°C (140°F), and
- b) Penetration of asphalt at 25°C (77°F).

Statistical Design

A full factorial design was used for this experiment. The asphalt paving mixture specimens were coded to blind the identity of the specimens from the technicians. The full factorial design resulted in a data set by which all the interactions among the controlled variables could be evaluated for each response variable. The two replicates in the design provided the data needed for a reasonable estimate of the experimental error or variance so that the statistical significance of the effects and interactions could be determined.

Although ASTM E 691-87 prefers a minimum of three materials and three replicates each for the interlaboratory study, this study was conducted using only two materials with two replicates each. The primary reasons for this were time requirements and financial constraints of the participants. These tests required the testing apparatus to remain at each laboratory for approximately two weeks and demanded almost two man weeks of technician time to conduct them. Since all of the laboratories that participated in this round-robin testing program were volunteers, it was felt that requesting an additional amount of their resources for this program was excessive.

According to the ASTM, a reproducibility or between-laboratory

variability study requires acceptable data from a minimum of six laboratories. The study described herein was undertaken with 11 laboratories.

FINDINGS

The major emphasis of this study was on determining the repeatability and reproducibility of the SHRP extraction and recovery test procedure and to prepare a precision and bias statement for the test protocol. Two asphalt paving mixtures were chosen for the interlaboratory test program and two replicates were tested for each response variable. The materials were so labelled as to mask their true identity and the technicians operated under the assumption that they were dealing with four different materials.

Round-Robin Participants

The following agencies participated in the round-robin test program:

- 1) Arizona Department of Transportation
- 2) California Department of Transportation
- 3) Colorado Department of Transportation
- 4) CTL Thompson Consulting Engineers, Denver, Colorado
- 5) Florida Department of Transportation
- 6) Georgia Department of Transportation
- 7) Nevada Department of Transportation
- 8) North Dakota Department of Transportation
- 9) Southwestern Laboratories, Houston, Texas
- 10) Utah Department of Transportation
- 11) Texas Transportation Institute Laboratory

Description of the Statistical Analysis

Properties of the neat asphalt used in the two paving mixtures that were subjected to the round-robin testing are given in Table 1. The data from the interlaboratory test program consist of penetration at 25°C and absolute viscosity at 60°C of asphalts extracted and recovered using the new SHRP method on two paving mixtures. A sample data sheet sent to each of the laboratories for their use to tabulate the data is given in Appendix A. All penetration and viscosity data obtained from the interlaboratory program appears in Table 2.

Data from the round-robin testing was analyzed for within-laboratory

Table 2. Data from Round-Robin Testing of SHRP Extraction & Recovery Procedure/Device.

Laboratory	Mixture	Replicate 1		Replicate 2	
		Pen @ 25°C, dmm	Vis @ 60°C, Pa·s	Pen @ 25°C, dmm	Vis @ 60°C, Pa·s
North Dakota DOT	A	26	-	27	1,456
	B	27	779	28	760
California DOT	A	31	1,280	36	848
	B	49	350	37	525
Colorado DOT ¹	A	-	630	-	663
	B	-	401	-	361
Florida DOT	A	35	874	34	916
	B	36	546	32	567
Georgia DOT	A	35	1,063	36	933
	B	34	613	35	562
Nevada DOT	A	38	759	37	699
	B	37	586	37	683
Utah DOT	A	41	807	33	1,054
	B	32	641	32	641
CTL Thompson	A	46	450	38	646
	B	36	422	40	383
Arizona DOT	A	41	602	30	990
	B	41	500	37	661
TTI Materials ²	A	36	814	36	966
	B	38	438	39	543

¹ Ran only viscosity of extracted/recovered asphalt then discarded samples.

² The TTI laboratory included in the round-robin test was different from the one that developed the SHRP procedure.

and between-laboratory variability in accordance with ASTM E691-87 specifications. The "Interlaboratory Data Analysis Software", developed by the ASTM for this specific purpose, was used to perform statistical calculations. Although the interlaboratory testing program began with 11 laboratories, data were not received from the Southwestern Laboratories for either viscosity or penetration. North Dakota DOT, had only one data point for viscosity instead of two, and that value was the highest among all others, being more than twice the lowest viscosity value. Hence it, was treated as an outlier and excluded from the analysis. Colorado DOT discarded the sample after testing for viscosity only and so could not furnish data on penetration. Thus each of the response variables was effectively measured by 9 laboratories.

The statistical analysis of the data consisted of a one-way analysis of variance (within- and between-laboratories) carried out separately for each material. Results obtained from these analyses include: mean values of the response variables, standard deviation, precision statistics (repeatability standard deviation, S_r , and reproducibility standard deviation, S_R), and consistency statistics (k , the within-laboratory and h , the between-laboratory statistics).

Summary of Round-Robin Results

The average penetration value (for all laboratories) for Mixture A was found to be 35.39 dmm, with a standard deviation of 4.11 and a coefficient of variation, C_v , of 11.6 percent. Similarly, the average penetration value for the asphalt used in Mixture B was found to be 35.94 dmm with a standard deviation of 4.52 and a coefficient of variation of 12.59 percent. The asphalt viscosity data from the interlaboratory test program yielded mean values of 833 and 523 Pa·s for Mixtures A and B, respectively. The standard deviation and coefficient of variation were found to be 167.58 and 20.15 percent for Mixture A, and 98.67 and 18.85 percent for Mixture B, respectively. Variability was determined by the consistency statistics (k and h), while the precision statistics (repeatability (S_r) and reproducibility (S_R) standard deviations) were used to prepare the precision and bias statement. The precision and bias statement comprises the last section of the protocol in Appendix A. All results from the statistical analysis are given in Appendix B and their main implications are discussed

below.

For the penetration data, the critical value of the k-statistic, which corresponds to the within-laboratory variability, was 2.41. This means that if the k value for a laboratory and a material is less than 2.41, the within-laboratory variability is not statistically significant for that particular laboratory and material. From Table B2 in Appendix B, the k value for California DOT for Mixture B is 2.58 which is greater than the critical value of 2.41. Thus the penetration of Asphalt B as measured by California DOT has statistically significant within-laboratory variability.

For the viscosity data, the critical value of the k-statistic is 2.41 and all the laboratories exhibit a lower k value for both Mixtures A and B. This indicates that there is no statistically significant within-laboratory variability in viscosity measurement.

Similarly, for penetration data, the critical value of the h-statistic, which corresponds to the between-laboratory variability, was 2.23. None of the h values exceeded this value as can be seen in Tables B1 and B2 of Appendix B. Although the h values for North Dakota DOT vary considerably from the others, they are within the critical limits, thus indicating that the between-laboratory variability for measuring viscosity was not statistically significant. The critical value of the h-statistic for the viscosity data was 2.23 and all the h values for different laboratories and materials were less than this, indicating that there is no statistically significant between-laboratory variance in viscosity measurement.

Based on these findings, a precision and bias statement was developed and added to the test protocol in Appendix A.

Design of Apparatus

The extraction and recovery apparatus was designed to accommodate commercial production as well as appropriate functioning in a research laboratory. The intent was to furnish a design that is functional yet convenient, economical, and safe. Machine drawings for the final design are provided in Appendix C.

Comparison of New Method with Standard Methods

The asphalts were extracted from the two paving mixtures using ASTM

D2172, Method A (cold) and Method B (hot). The binders were recovered using the Abson (ASTM D1856) procedure with extraction Method A and the proposed ASTM rota-vapor procedure with extraction Method B. After the asphalts were extracted and recovered, penetration and viscosity were measured. The resulting data appear in Table 3. A graphical comparison of asphalt properties from the new and existing methods is provided in Figures 1 and 2.

Table 3. Properties of Asphalt Extracted from Mixtures A and B Using Existing Standard Extraction and Recovery Methods.

Mix I.D.	Extraction Method	Recovery Method	Pen @25°C, dmm	Vis @60°C, Pa·s
Mixture A	ASTM D2172, Method A	Abson	42	614
	ASTM D2172, Method B	Rota-Vapor*	44	638
	SHRP	SHRP	36/36	814/966
Mixture B	ASTM D2172, Method A	Abson	53	351
	ASTM D2172, Method B	Rota-Vapor*	52	322
	SHRP	SHRP	38/39	438/543

* Rota-Vapor method is proposed as a standard by ASTM.

The new SHRP method consistently yielded asphalts which were harder than those obtained using the standard extraction/recovery methods. Since the new method was designed specifically to minimize asphalt hardening, it was postulated that more solvent was left in the asphalts recovered by the standard methods. The present study was not designed address this issue, however, work done under SHRP contract A-005 provided limited data in this regard.

Asphalt samples were extracted using ASTM D2172 Method B and recovered using the Rota-Vapor method. Identical samples were extracted and recovered using new SHRP method. Their viscosities and the amount of solvent remaining in the asphalt were measured. One sample, extracted using the SHRP method, yielded a viscosity of 13,664 Pa·s and, when extracted using ASTM D2172, it had a viscosity value of 3,099 Pa·s. The quantities of residual solvent measured 0.15 percent TCE for the hot method, and less than 0.1 percent toluene for the SHRP method. Another sample yielded viscosity

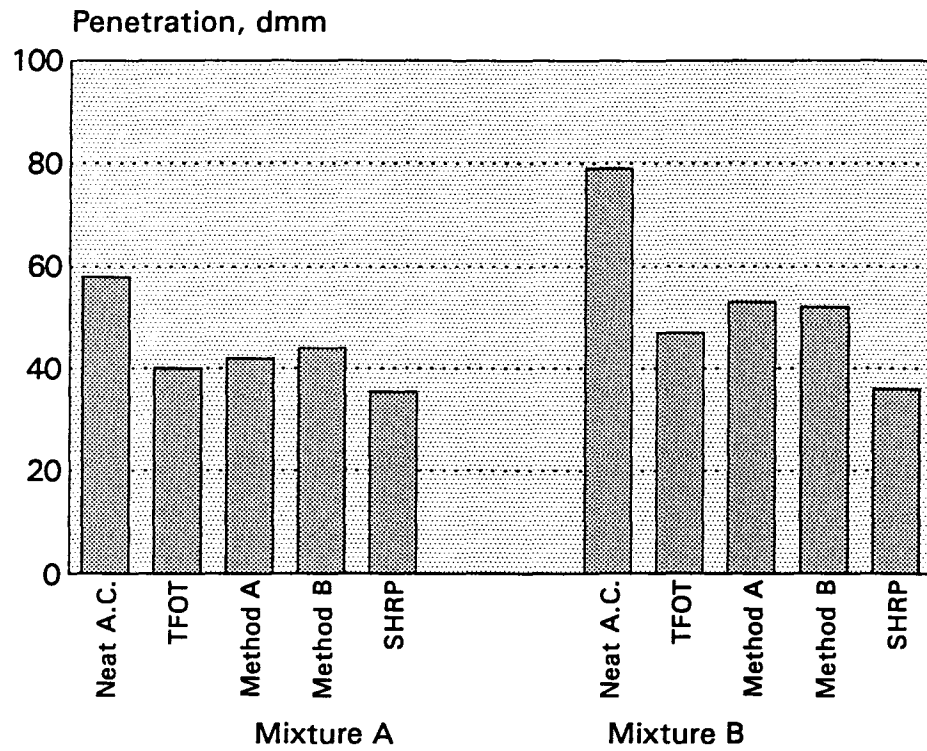


Figure 1. Comparison of Penetration of Asphalts Extracted and Recovered Using new and ASTM Standard Methods.

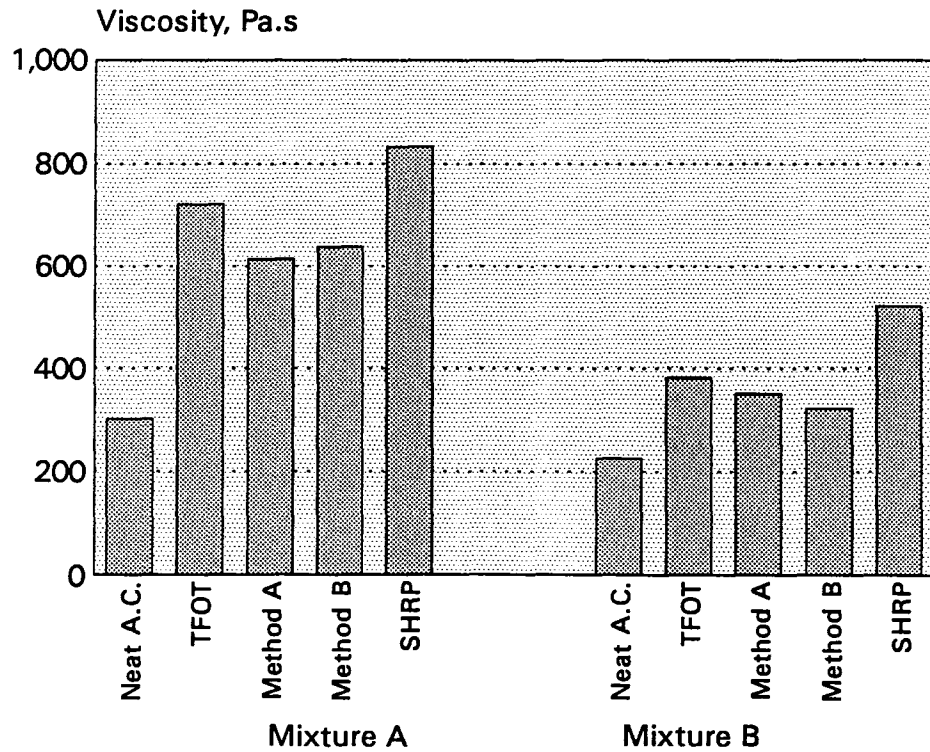


Figure 2. Comparison of Viscosity of Asphalts Extracted and Recovered Using New and ASTM Standard Methods.

values of 9,920 Pa.s for new method and 2,866 Pa.s for the ASTM method, while the residual solvent quantities measured about 0.1 percent for both the methods. These very limited data indicate that the consistently harder asphalts recovered by the new method as compared to the standard methods are not fully explained by the differences in residual solvent. More research is required in this area to make conclusive remarks.

CONCLUSIONS AND RECOMMENDATIONS

1. The new SHRP extraction and recovery method was subjected to an interlaboratory evaluation to provide data that were used to estimate the repeatability and reproducibility of the method. The penetration data indicates that although within-laboratory variability does exist, only one out of nine values for one of two mixtures was significantly different. Viscosity data indicates considerable within-laboratory variability for both mixtures, however, from a statistical standpoint, the values were not significantly different from one another. Similarly, between-laboratory variability was quantified for penetration and viscosity and none of the values were significantly different from one another.
2. Reliability of the test results may be questionable since the number of materials tested was less than that recommended by ASTM E691. The reason for fewer tests was an attempt to minimize technician time requirements of the volunteer participants.
3. The new extraction and recovery procedure can accommodate a maximum of about 1400 grams of asphalt mix which will typically yield about 60 to 70 grams of asphalt cement. It requires about seven hours to complete for one sample and requires continuous attention by the operator.
4. Limited work was performed to compare the new SHRP method to existing ASTM methods. The new method appears to yield harder recovered asphalts than ASTM D2172, Method A and B used with Abson or rota-vapor recovery methods. More research is needed to study quantities and effects of residual solvent in the recovered asphalts.
5. Although not addressed in this study, it is expected that the new procedure cannot be used for determination of aggregate gradation after asphalt extraction as the rotating action in the extraction vessel may cause degradation of some aggregates.

APPENDIX A

Extraction and Recovery Protocol

SHRP EXTRACTION AND RECOVERY PROCEDURE

1. Scope

- 1.1 This test method covers the extraction and recovery of asphalt from bituminous concrete samples. It is to be used when physical and/or chemical properties of the asphalt binder and the binder content are desired; however, it is not recommended for use in determining aggregate gradations.
- 1.2 This procedure may involve hazardous materials, operations and equipment. This procedure does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Applicable Documents

2.1 ASTM Standards:

D979 Sampling Bituminous Paving Mixtures

D2172 Test Methods for Quantitative Extraction from Bituminous Paving Mixtures

D1856 Recovery of Asphalt from Solution by Abson Method

2.2 Other Documents:

TRB papers 890445, 910350, 910352 (January, 1991)

3. Summary of Method

- 3.1 The paving mixture is repeatedly washed and filtered with toluene and toluene with 15% ethanol in the extraction/ filtration apparatus shown (Figures 1 through 11). Each filtrate is distilled under vacuum in a rotary evaporator with the asphalt remaining in the flask. After recovery of the final filtrate the solution is concentrated only to about 300 mL and centrifuged to remove aggregate fines. The decanted solution is distilled under vacuum to remove the extraction solvents. Nitrogen gas is introduced during the final phase of distillation to drive off any remaining traces of solvents. The recovered asphalt (distillation residue)

may then be subjected to further testing as required.

4. Significance and Use

- 4.1 The method can be used both for obtaining asphalt for further analyses and for calculation of asphalt content in pavement samples. The aggregate should not be used for sieve analysis because it undergoes prolonged grinding in the extraction device.

5. Apparatus and Materials

- 5.1 Oven, capable of maintaining the temperature at $230^{\circ}\text{F} \pm 9^{\circ}\text{F}$ ($110^{\circ}\text{C} \pm 5^{\circ}\text{C}$).
- 5.2 Utilities - Vacuum source, nitrogen gas source and cooling water source.
- 5.3 Balance, having an accuracy of at least 0.01% of the sample mass.
- 5.4 Extraction Vessel, a 5" long piece of 6" I.D. Sch 80 aluminum pipe with removable top and bottom $\frac{1}{2}$ " aluminum plates. Top (upstream) plate has a mixing motor mount and $\frac{3}{4}$ " addition port. Bottom (downstream) plate is equipped with a $\frac{1}{4}$ " NDT quick-connect fitting. Four 4" x 1" baffles are mounted in extraction vessel followed by a 10 mesh screen, glass wool plug, 8 micron filter, and 10 mesh backup screen.
- 5.5 Flask, Suction, 500 mL
- 5.6 Round Bottom Flasks, 1000 mL and cork stands.
- 5.7 Gas Flowmeter, capable of indicating a gas flow up to 1000 mL/min.
- 5.8 Buchi Rotavapor RE-111A with transfer and purge tubes.
- 5.8.1 Transfer Tube, $\frac{1}{4}$ " polypropylene tubing 17" long.
- 5.8.2 Purge Tube, $\frac{1}{4}$ " polypropylene tubing 23" long.
- 5.9 Hot oil bath, capable of heating oil to 350°F .
- 5.10 Copper tubing (Note 1), amount adequate to connect apparatus as shown on figure.

NOTE 1 - The quantity of copper tubing needed will be dependent upon the space utilized in setting up the apparatus.

- 5.11 Single Speed Mixing Motor, 1/5 hp, 30 rpm.
- 5.12 Polypropylene Tubing (Note 2), ¼" for transferring solution throughout the procedure.

NOTE 2 - To avoid contamination of the sample due to solvent degradation of the tubing, do not substitute Nalgene or rubber tubing for the polypropylene tubing specified.

- 5.13 Centrifuge, batch unit capable of exerting a minimum centrifugal force of 770 times gravity.
- 5.14 Wide-Mouth Centrifuge Bottles, 250 mL.
- 5.15 Thermometer, having a range of 30 to 580°F (-2 to 300°C).
- 5.16 Glass Wool, borosilicate.

6. Reagents

- 6.1 Toluene, reagent grade.
- 6.2 Ethanol, absolute.
- 6.3 Nitrogen gas, at least 99.95% pure, in a pressurized tank, with pressure-reducing valve.

7. Precautions

- 7.1 Solvents should be used only under a hood or with an effective surface exhaust system in a well-ventilated area.

8. Sampling and Sample Preparation

- 8.1 Obtain samples in accordance with ASTM Method D 979.
- 8.2 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 ± 9°F (110 ± 5°C) only until it can be handled or mixed. Split or quarter the material until about 1000 g or the mass of material (Note 3) required for the test is obtained.

NOTE 3 - This procedure works best for quantities of asphalt less than 60 g. Therefore, if the asphalt content of the mix is known, then the mass of sample required is that which yields about 50 to 60 g of asphalt.

9. Procedure

9.1 Equipment Set Up.

9.1.1 Prepare the extraction vessel by first installing the baffles piece and then place the metal screen on the bottom (downstream) of the baffle. Cut several pieces of glass wool that will sufficiently cover the entire screen about one inch thick. Place second screen, then the first gasket, filter then second gasket and aluminum end piece on extraction vessel, as shown in Fig. 4. Tightly and evenly fasten the end piece with wing nuts. Use a crisscross pattern where opposite nuts are tightened sequentially.

9.1.2 Weigh an amount of pavement sample that will yield approximately 50 to 60 g of extracted asphalt. Place sample in extraction vessel. Put gasket and top end piece on the vessel and tightly and evenly fasten the wing nuts, creating a secure seal.

9.1.3 Prepare the rotary evaporator. Turn on the cooling water. Turn on the oil bath and set the temperature to $212 \pm 4.5^{\circ}\text{F}$ ($100 \pm 2.5^{\circ}\text{C}$). Place six 3 mm glass boiling beads in a 1000 mL round bottom flask. Attach the recovery flask to the rotary evaporator and apply a vacuum of $700 \pm 5\text{ mm Hg}$ (60 mm Hg absolute pressure) to rotary evaporator. Immerse flask 3/4 to 1" into oil bath. The angle of the recovery flask from the horizontal to the bath is set at 15°F . Set the flask rotation at 40 rpm. Clamp the empty condensate flask onto the condenser. Attach transfer tube inside neck of rotary evaporator. Attach filtrate transfer line to external fitting on neck of rotary evaporator.

9.2 Extraction and Filtration

9.2.1 Harge 600 mL of toluene through the 3/4" port on the top of the extractor. Blanket the interior of the extraction vessel by injecting nitrogen through the top port at 1000 mL/min for 1 minute. Close the port with a threaded plug. Attach the extractor to the motor. Start the motor and mix for 5 ± 1 minutes at 30 rpm. Shut off the motor.

9.2.2 Remove the extractor, place it on a stand and attach the quick connect fitting to the filtrate receiving flask. Make sure the

filtrate transfer line is closed. Remove the top port and blanket the extractor with 400 mL/min of nitrogen while filtering. Apply 700 ± 5 mm Hg vacuum to the filtrate receiving flask. Filter until the filtrate flow rate decreases rather abruptly to a rate below about 10 mL/min. (This flow rate can be estimated from the 50 mL increments marked on the Erlenmeyer flask). Shut off the vacuum.

- 9.2.2.1 (Optional) If using the fine filter (Note 4), switch vacuum to a second filtrate receiving flask and apply 700 ± 5 mm Hg vacuum. Filter until filtrate flow rate is below 10 mL/min. Shut off vacuum.

NOTE 4 - The fines filtration step remains in the developmental stages until a durable filter capable of retaining all aggregate fines is discovered.

- 9.2.3 Disconnect the extractor from the quick connect fitting. Repeat the extraction procedure. For the second and third washes use 400 ± 10 mL of toluene. For subsequent washes (Note 5), use 400 ± 10 mL of toluene with 15 volume % ethanol. In addition, mix the second wash for ten minutes and all subsequent washes for 30 minutes or more.

NOTE 5 - It is suggested that after the third wash, the condensate from the primary distillation step be used for extraction solvent. Recycling solvent in this manner allows the entire procedure to use approximately 1500 mL toluene.

9.3 Primary Distillation

- 9.3.1 After filtration, open the filtrate transfer valve and allow solution to flow from the filtrate receiving flask to the recovery flask. Continue the transfer until the filtrate receiving flask is empty or the recovery flask is about 2/3 full.
- 9.3.2 Close the filtrate transfer valve line and distill solvent at

212 ± 4.5°F (oil bath temperature) and 700 ± 5 mm Hg vacuum until the rate of recovery is approximately 1 drop per 10 sec or the next cycle is ready to be transferred.

- 9.3.3 If after the primary distillation step the condensate flask is over half full, empty the flask. Save this solvent for use in subsequent washes (Note 5). After primary distillation of each filtrate, maintain vacuum, temperature, flask rotation, and cooling water flow. Repeat the primary distillation after each filtration (Note 6).

NOTE 6 - It is important to concentrate the asphalt in the recovery flask after each wash and at a low temperature. This minimizes the time and temperature during which the asphalt is in a dilute solution and therefore minimizes asphalt hardening.

9.4 Two-flask method

- 9.4.1 After primary distillation of the first three filtrates, remove the recovery flask (which should contain only small amounts of solvent) and set it aside. Replace it with another 1000 ml roundbottom flask containing six 3 mm glass boiling beads.
- 9.4.2 Carry out the remaining primary distillations using the new recovery flask.

9.5 Final Extraction and Recovery

- 9.5.1 If after a 30 minute wash the filtrate flowing through the transfer tube is light brown color, proceed and perform the final recovery.
- 9.5.2 Pour contents of the current recovery flask into the original recovery flask. Attach original recovery flask to the Roto-Vap.
- 9.5.3 Distill the contents of the recovery flask until it is about 1/3 full.
- 9.5.4 Disconnect the recovery flask and pour the contents into the centrifuge bottles. Fill the bottles so that their weights are equal. Wash any residue from the recovery flask into the centrifuge bottles and centrifuge the bottles at 3600 rpm for

25 minutes. Raise the oil bath temperature to $345 \pm 5^{\circ}\text{F}$.

- 9.5.5 Decant the asphalt-solvent solution into the recovery flask and add six 3 mm glass boiling beads. Attach the flask to the rotary evaporator. Disconnect the transfer tube from the rotary evaporator and replace it with the gas purge tube. Disconnect the filtrate transfer line from the external rotary evaporator neck fitting and replace it with the nitrogen gas line. Apply 700 mm Hg vacuum. Lower the flask 3/4 to 1" into the oil bath.
- 9.5.6 Distill the solvent.
- 9.5.7 When the condensation rate falls below 1 drop every 30 seconds, introduce nitrogen gas at 1000 mL/min. Maintain the gas flow, vacuum and bath temperature for 30 ± 1 minutes.
- 9.5.8 Shut down the oil bath, flask rotation, vacuum, gas flow, and cooling water flow. Remove the evaporating flask and pour the asphalt into a sample tin.

10. Test on Asphalt Cement

Perform tests on recovered asphalt as desired.

11. Precision and Bias Statement

- 11.1. Criteria for judging acceptability of penetration and viscosity of an asphalt sample after the extraction and recovery procedure are provided in the following two subsections.
- 11.2. Penetration

Material	Single Operator		Multi laboratory	
	1 S %	D2 S %	1 S %	D2 S %
Mix A	11.16	31.56	14.04	39.72
Mix B	9.15	25.89	14.15	40.03
Average	10.15	28.72	14.09	39.86

The single-operator coefficient of variation has been found to be 10.15% (1S %). Thus, results of two properly conducted tests by the same operator on similar material should not differ by more than 28.72% (D2 S %).

The multi-laboratory coefficient of variation has been found to be

14.09% (1S %). Thus the results of two properly conducted tests from two different laboratories on similar material should not differ by more than 39.86% (D2 S %).

11.3. Viscosity

Material	Single Operator		Multi laboratory	
	1 S %	D2 S %	1 S %	D2 S %
Mix A	19.66	55.62	24.48	69.25
Mix B	12.97	36.68	20.96	59.29
Average	16.31	46.15	22.72	64.26

The single-operator coefficient of variation has been found to be 16.31% (1 S %). Thus, results of two properly conducted tests by the same operator on similar material should not differ by more than 46.15% (D2 S %).

The multi-laboratory coefficient of variation has been found to be 22.72% (1S %). Thus, results of two properly conducted tests from two different laboratories on similar material should not differ by more than 64.26% (D2 S %).

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[illegible]

* Same technician should perform all penetration tests.

** Same technician should perform all viscosity tests. May be different from one performing penetration tests.

APPENDIX B

Statistical Analysis of Data From Round-Robin Testing

Table B1. Statistical Results from Penetration Data on Mixture A.
(from ASTM Interlaboratory Data Analysis Software)

<i>LAB</i>	<i>AVERAGE</i>	<i>STD DEV</i>	<i>DEV</i>	<i>h</i>	<i>k</i>
1	33.50000	3.53553	-1.88889	-0.46	0.89
2	34.50000	0.70711	-0.88889	-0.22	0.18
3	36.00000	1.41421	0.61111	0.15	0.36
4	37.50000	0.70711	2.11111	0.51	0.18
5	37.00000	5.65685	1.61111	0.39	1.43
6	42.00000	5.65685	6.61111	1.61	1.43
7	26.50000	0.70711	-8.88889	-2.16	0.18
8	35.50000	7.77817	0.11111	0.03	1.97
9	36.00000	0.00000	0.61111	0.15	0.00
<i>AVERAGE OF CELL AVERAGES</i>			35.38889		
<i>STD. DEV. BETWEEN CELL AVERAGES</i>			4.10623		
<i>REPEATABILITY STD. DEVIATION</i>			3.95109		
<i>REPRODUCIBILITY STD. DEVIATION</i>			4.96655		

Table B2. Statistical Results from Penetration Data on Mixture B.
(from ASTM Interlaboratory Data Analysis Software)

<i>LAB</i>	<i>AVERAGE</i>	<i>STD DEV</i>	<i>DEV</i>	<i>h</i>	<i>k</i>
1	43.00000 <i>k</i>	8.48528 <i>k</i>	7.05556	1.56 <i>k</i>	2.58 <i>k</i>
2	34.00000	2.82843	-1.94444	-0.43	0.86
3	34.50000	0.70711	-1.44444	-0.32	0.21
4	37.00000	0.00000	1.05556	0.23	0.00
5	32.00000	0.00000	-3.94444	-0.87	0.00
6	38.00000	2.82843	2.05556	0.45	0.86
7	27.50000	0.70711	-8.44444	-1.87	0.21
8	39.00000	2.82843	3.05556	0.68	0.86
9	38.50000	0.70711	2.55556	0.56	0.21
<i>AVERAGE OF CELL AVERAGES</i>			35.94444		
<i>STD. DEV. BETWEEN CELL AVERAGES</i>			4.52386		
<i>REPEATABILITY STD. DEVIATION</i>			3.29140		
<i>REPRODUCIBILITY STD. DEVIATION</i>			5.08743		

Table B3. Statistical Results from Viscosity Data on Mixture A.
(from ASTM Interlaboratory Data Analysis Software)

<i>LAB</i>	<i>AVERAGE</i>	<i>STD DEV</i>	<i>DEV</i>	<i>h</i>	<i>k</i>
1	1064.000	305.470	231.000	1.38	1.86
2	646.500	23.335	-186.500	-1.11	0.14
3	895.000	29.698	62.000	0.37	0.18
4	998.000	91.924	165.000	0.98	0.56
5	729.000	42.426	-104.000	-0.62	0.26
6	930.500	174.655	97.500	0.58	1.07
7	548.000	138.593	-285.000	-1.70	0.85
8	796.000	274.357	-37.000	-0.22	1.67
9	890.000	107.480	57.000	0.34	0.66
<i>AVERAGE OF CELL AVERAGES</i>			833.000		
<i>STD. DEV. BETWEEN CELL AVERAGES</i>			167.846		
<i>REPEATABILITY STD. DEVIATION</i>			163.817		
<i>REPRODUCIBILITY STD. DEVIATION</i>			203.937		

Table B4. Statistical Results from Viscosity Data on Mixture B.
(from ASTM Interlaboratory Data Analysis Software)

<i>LAB</i>	<i>AVERAGE</i>	<i>STD DEV</i>	<i>DEV</i>	<i>h</i>	<i>k</i>
1	437.500	123.744	-85.944	-0.87	1.82
2	381.000	28.284	-142.444	-1.44	0.42
3	556.500	14.849	33.056	0.34	0.22
4	587.500	36.062	64.056	0.65	0.53
5	634.000	67.882	110.556	1.12	1.00
6	641.000	0.000	117.556	1.19	0.00
7	402.500	27.577	-120.944	-1.23	0.41
8	580.500	113.844	57.056	0.58	1.68
9	490.500	74.246	-32.944	-0.33	1.09
<i>AVERAGE OF CELL AVERAGES</i>			523.444		
<i>STD. DEV. BETWEEN CELL AVERAGES</i>			98.668		
<i>REPEATABILITY STD. DEVIATION</i>			67.885		
<i>REPRODUCIBILITY STD. DEVIATION</i>			109.725		

APPENDIX C

Machine Drawings of the Equipment

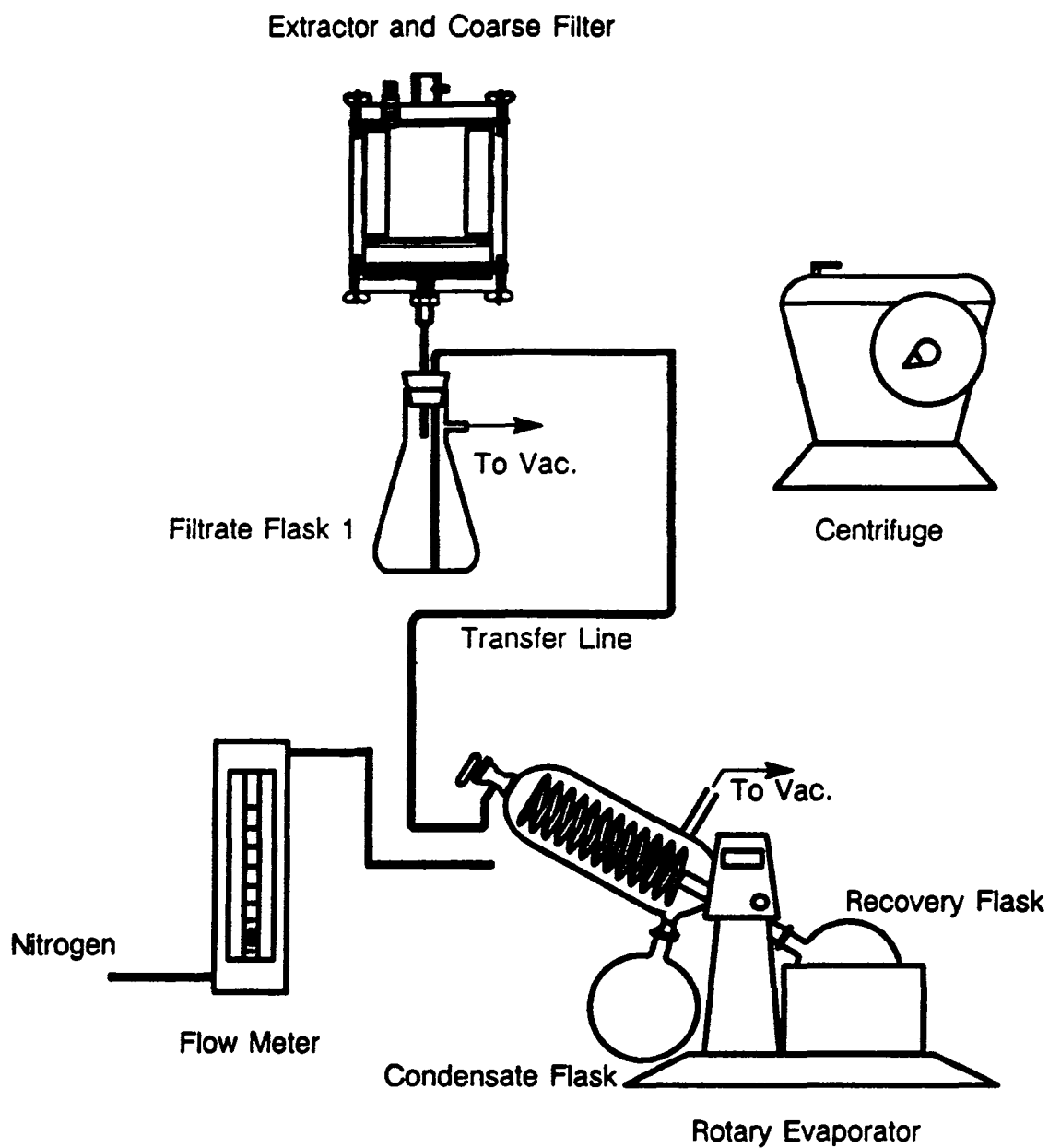


Figure C1. SHRP Asphalt Extraction and Recovery Apparatus.

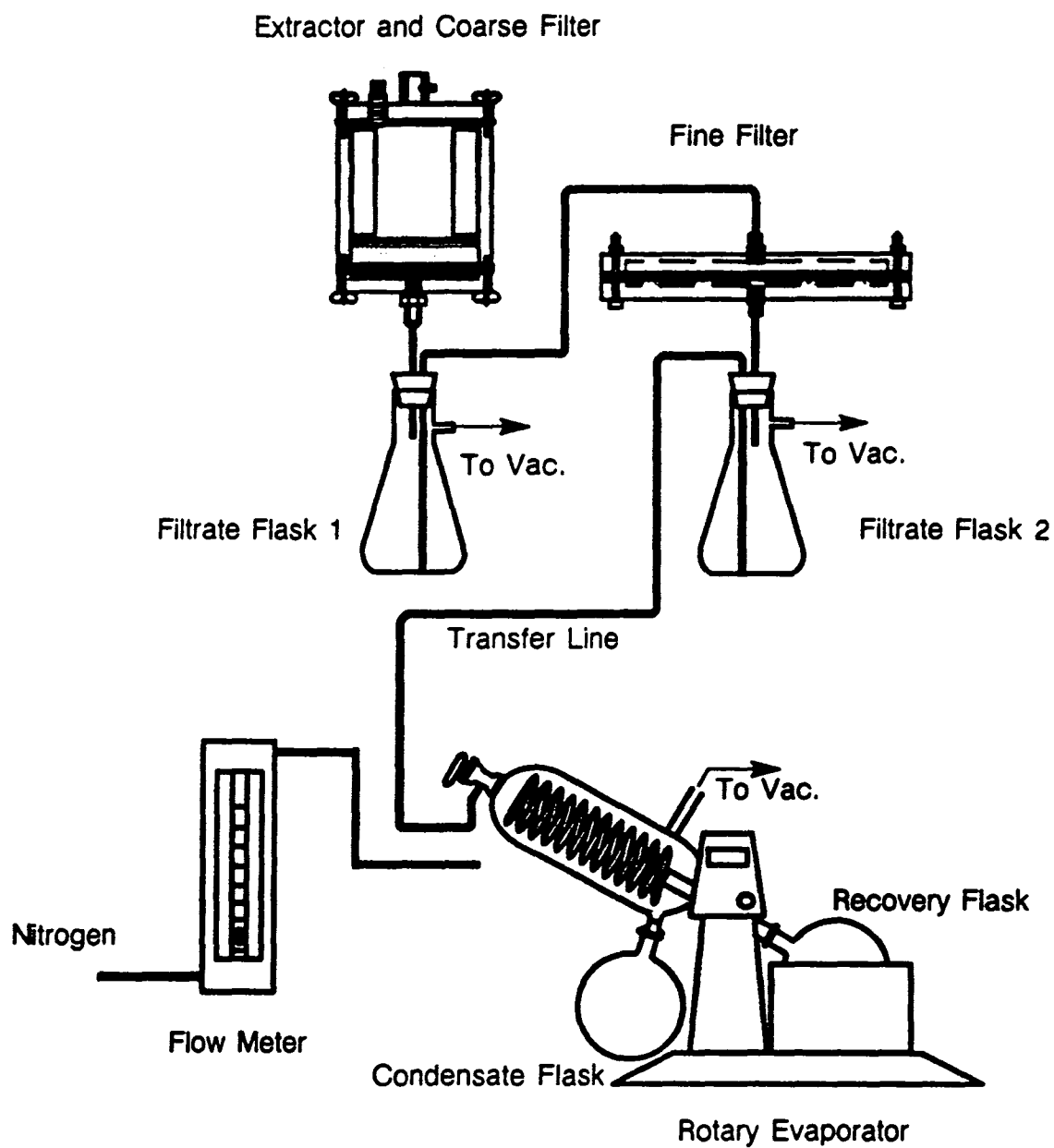


Figure C2. Asphalt Extraction and Recovery Apparatus (with Optional Fine Filter).

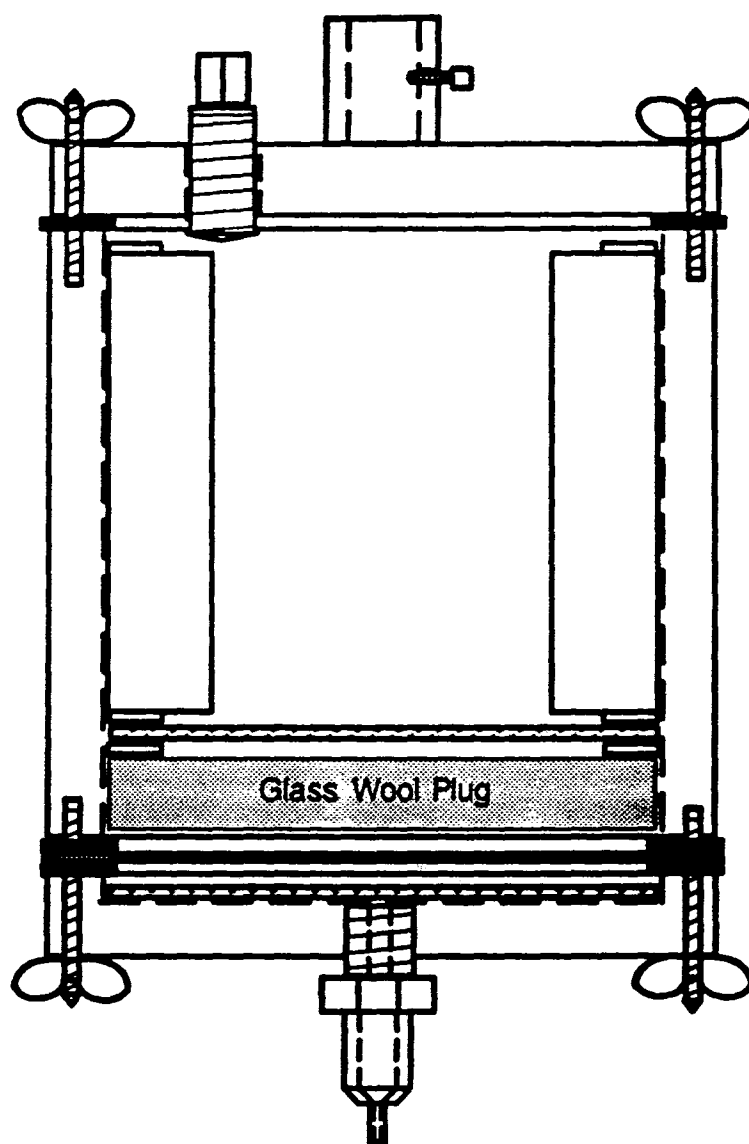


Figure C3. Extraction/Filtration Vessel.

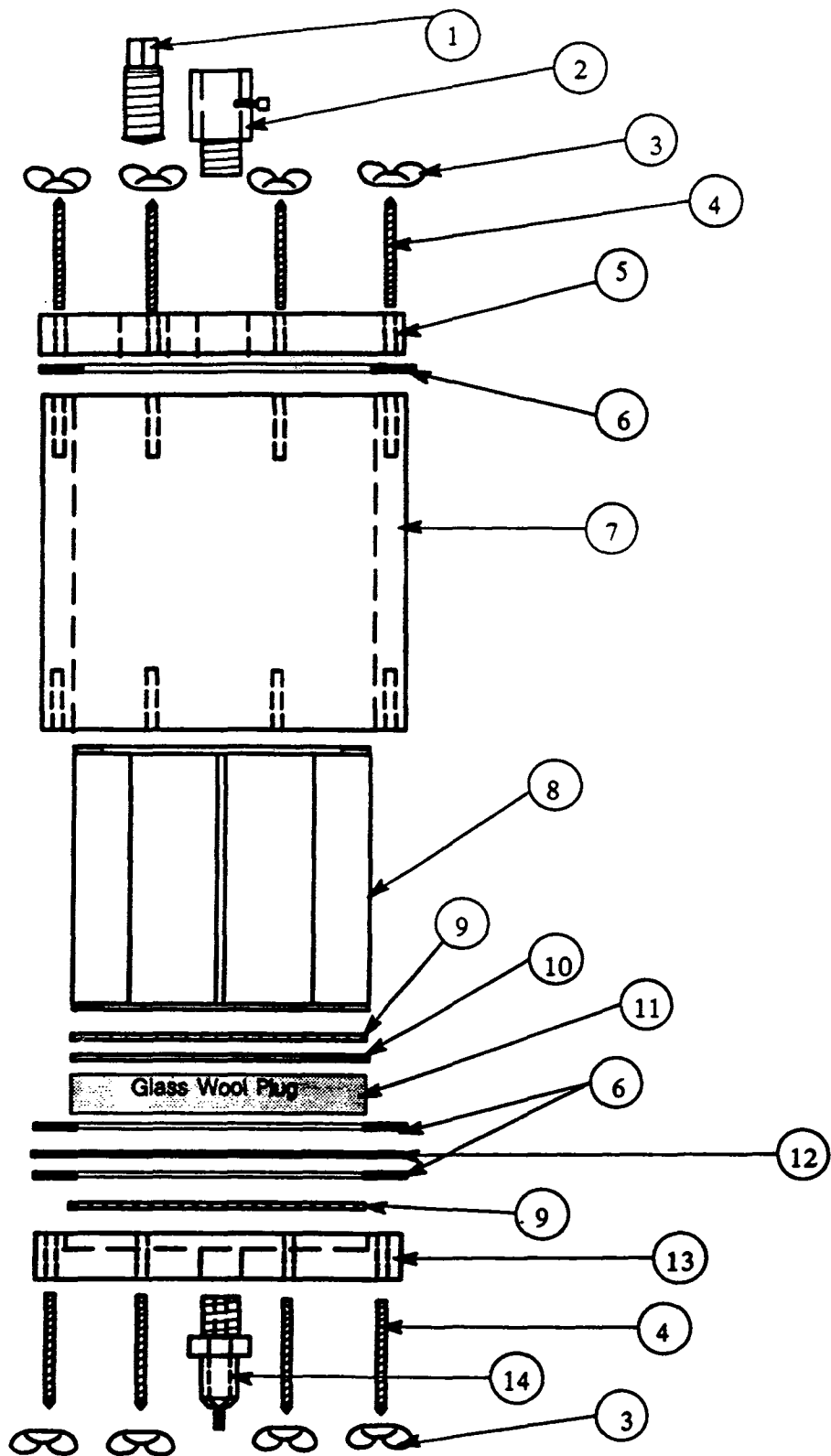
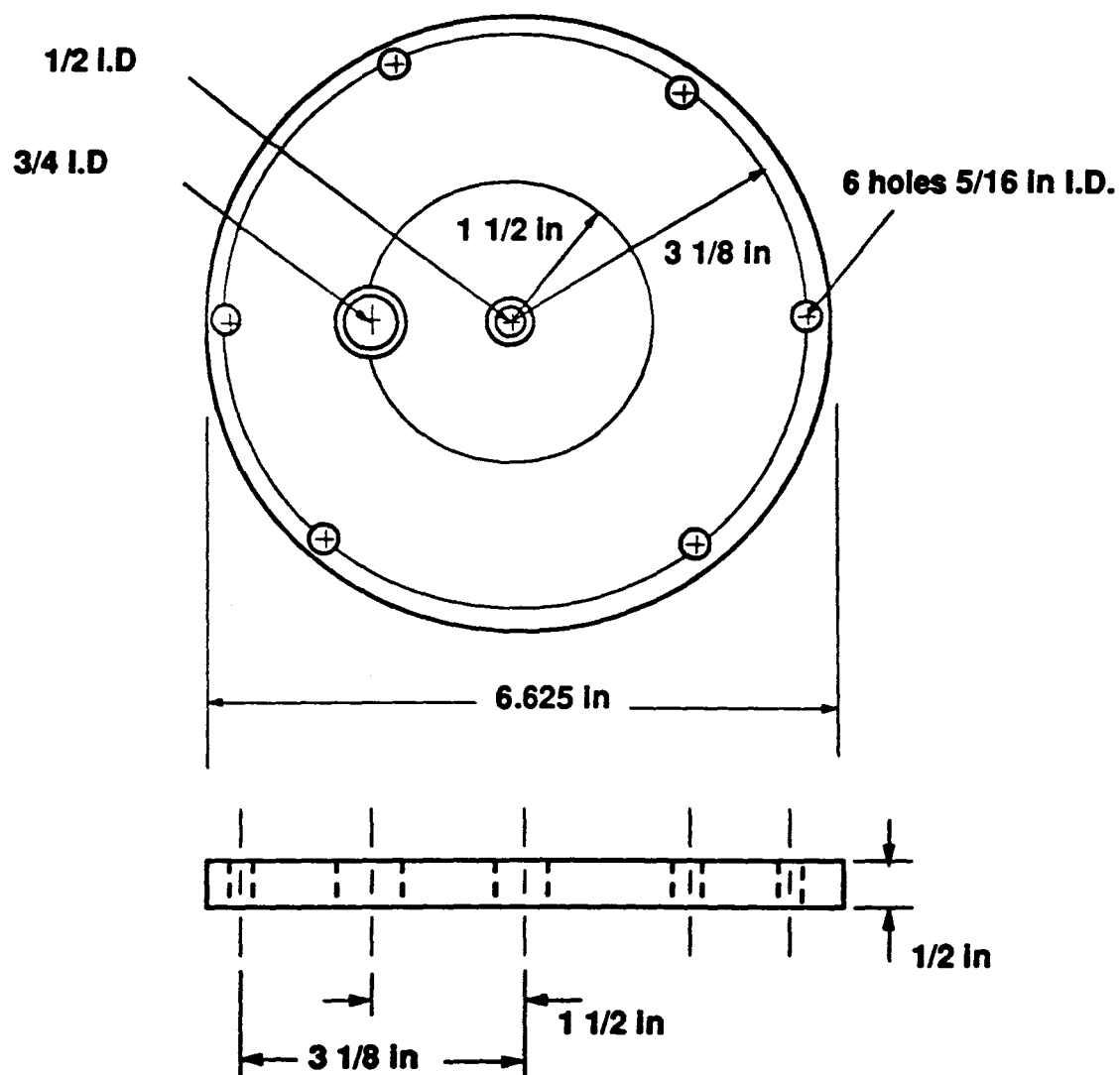
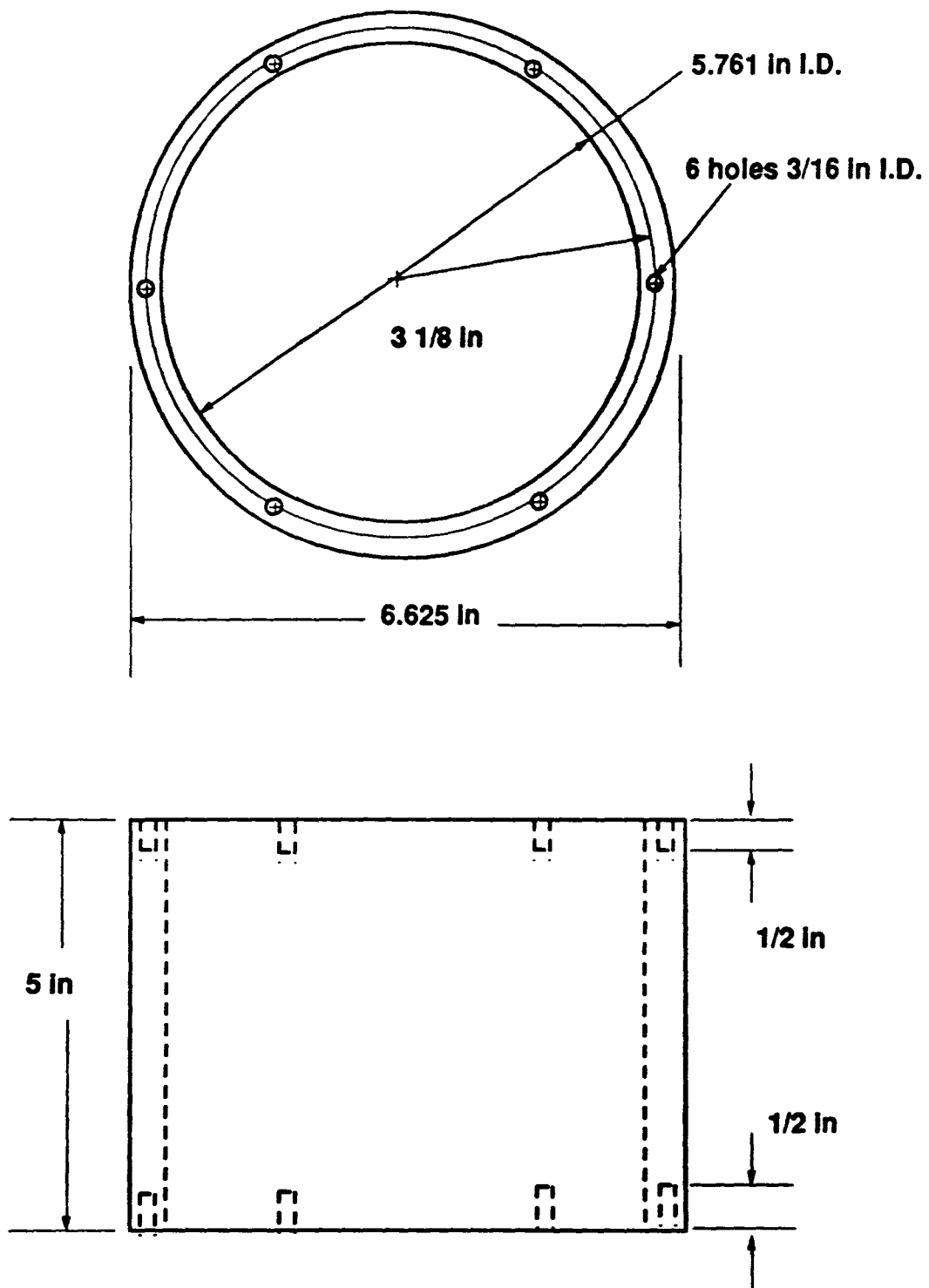


Figure C4. Disassembled View of Extraction/Filtration Vessel.



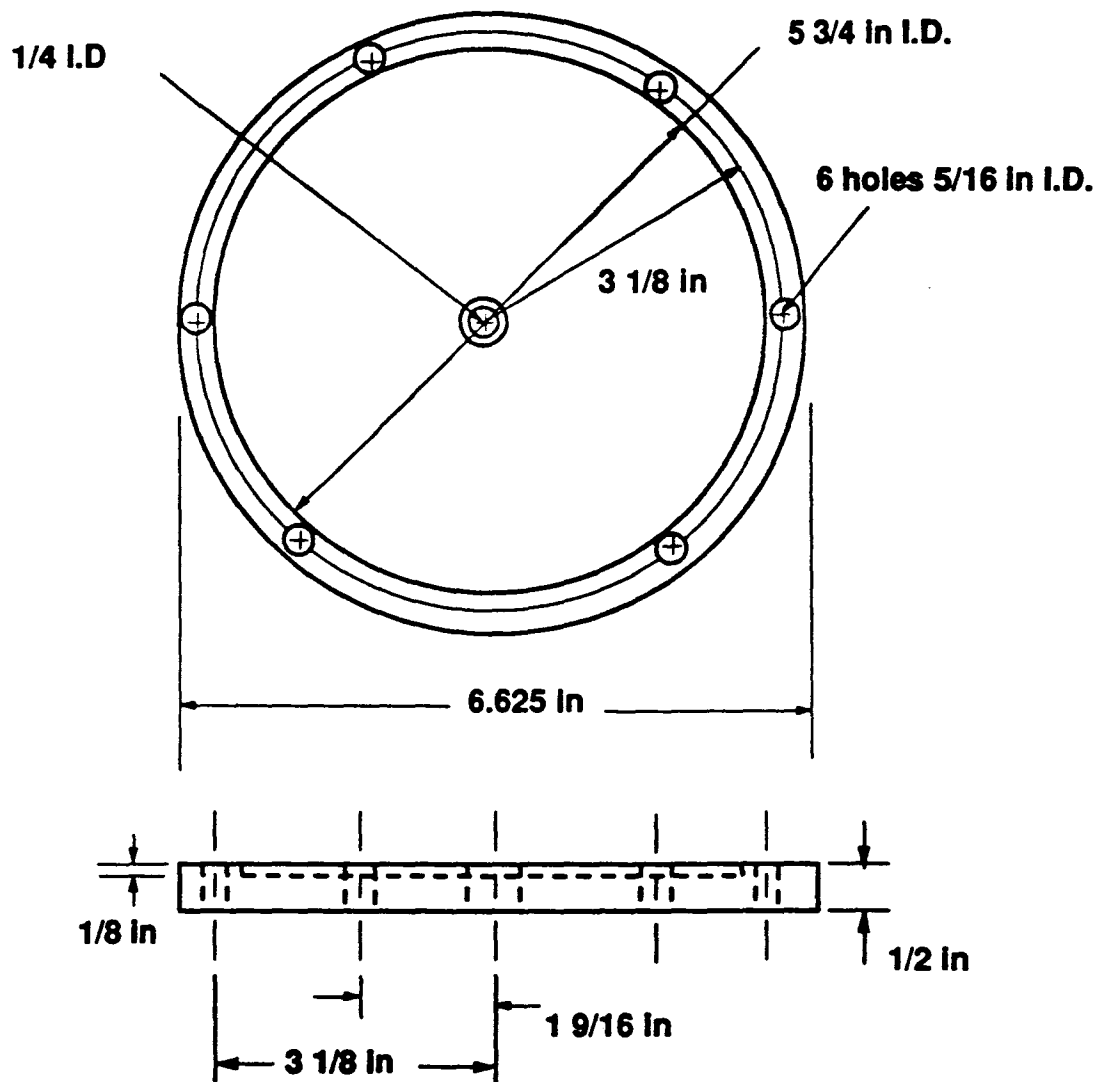
1 Stainless steel plate 1/2 in thick

Figure C5. Details of Extraction Vessel Lid.



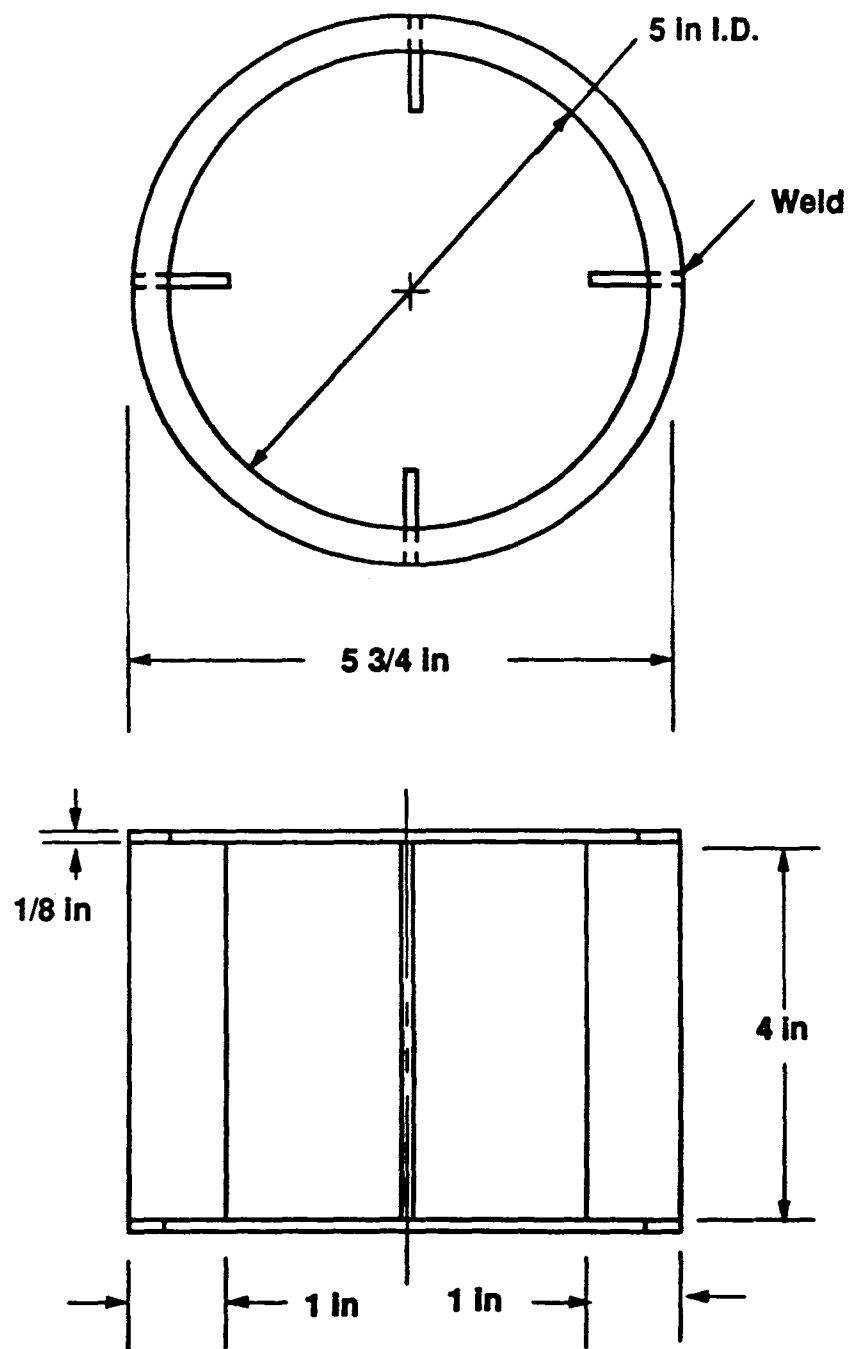
1 Six in Nominal Schedule 80 stainless steel pipe

Figure C6. Details of Extraction Vessel Body.



1 Stainless steel plate 1/2 in thick

Figure C7. Details of Extraction Vessel Bottom.



4 Aluminum plates $\frac{1}{8}$ in thick

2 Aluminum rings $5 \frac{3}{4}$ in O. D., 5 in I.D.

Figure C8. Baffle Housing.

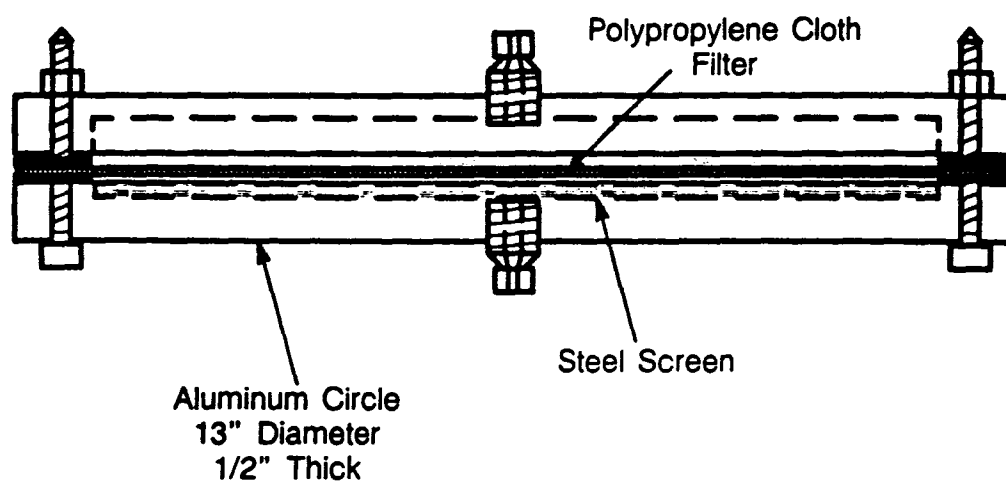
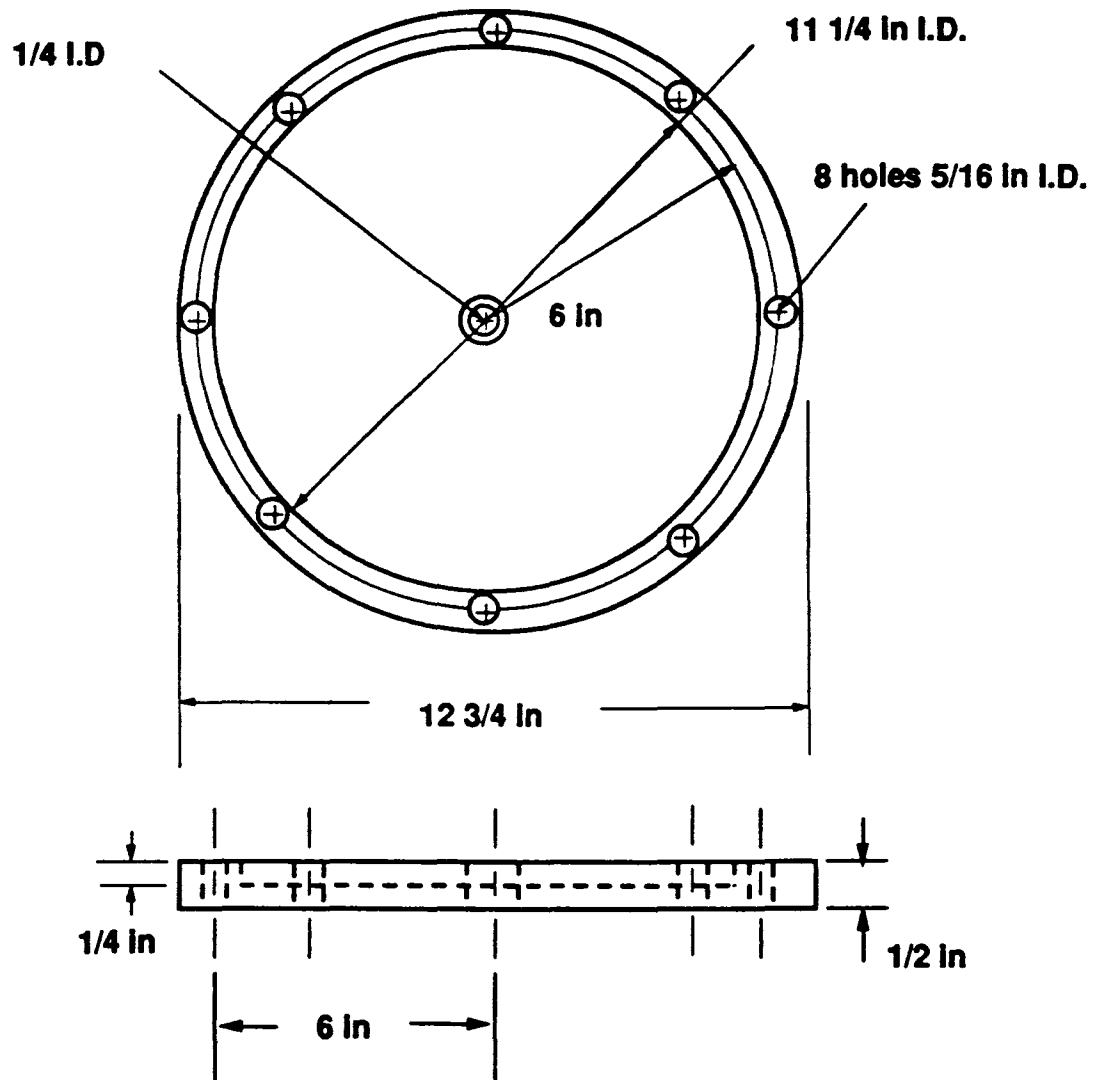
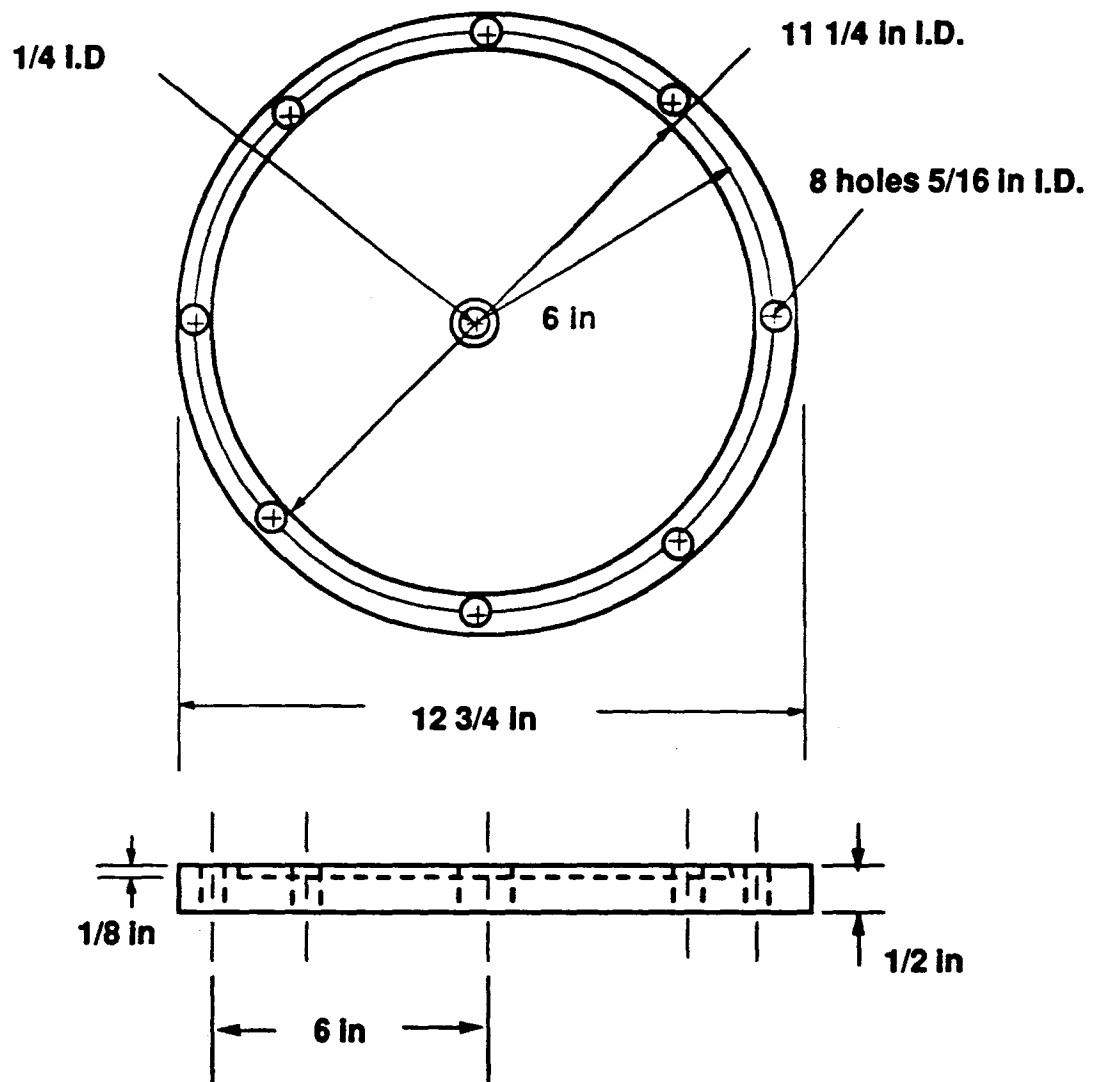


Figure C9. Fine Filter (1-2 Microns) for Second Stage Removal of Fine Aggregate.



1 Stainless steel plate $\frac{1}{2}$ in thick

Figure C10. Housing Top for Fine Filter.



1 Stainless steel plate $\frac{1}{2}$ in thick

Figure C11. Bottom for Fine Filter Housing.

Table C1. Parts List for Extraction/Filtration Vessel.

- 1) 3/4 inch stainless steel NPT plug
- 2) Motor mount 5/8 inch I.D. 1/2 inch NPT fitting
- 3) 12 1/8 inch wing nuts
- 4) 12 2 inch long 1/8 inch screws
- 5) Extraction Vessel Top
- 6) 3 Viton Gaskets 1/8 inch thick, 5/8 inch width with holes to fit over studs
- 7) Extraction Vessel
- 8) Aluminum Baffle Housing
- 9) 2 stainless steel screens 10 mesh
- 10) Aluminum ring 1/8 inch thick 5.75 inch O.D, 3/8 inch width
- 11) Glass Wool
- 12) SW 230 8 micron Polypropylene Twill Weave Filter with holes to fit over studs
- 13) Extraction Vessel Bottom
- 14) Stainless Steel Quick Connect 1/4 inch NPT fitting