

EXTRACTION OF ASPHALT FROM BITUMINOUS MIXTURES BY SOXHLET EXTRACTION

(An Arizona Method)

Scope

1. This procedure is designed to determine the bitumen content of bituminous mixtures by extraction of the asphalt. It may also be used in conjunction with ARIZ 511 - Recovery of Asphalt from Extraction Solution, when small amounts of asphalt are required for further testing.

Apparatus

2. The apparatus shall consist of the following:

(a) Soxhlet Extraction Apparatus, 100 mm inside diameter, assembled as shown in Figure 1. (Cal-Glass for Research, Inc. #LG-103/60 Soxhlet Extraction apparatus has been found suitable for

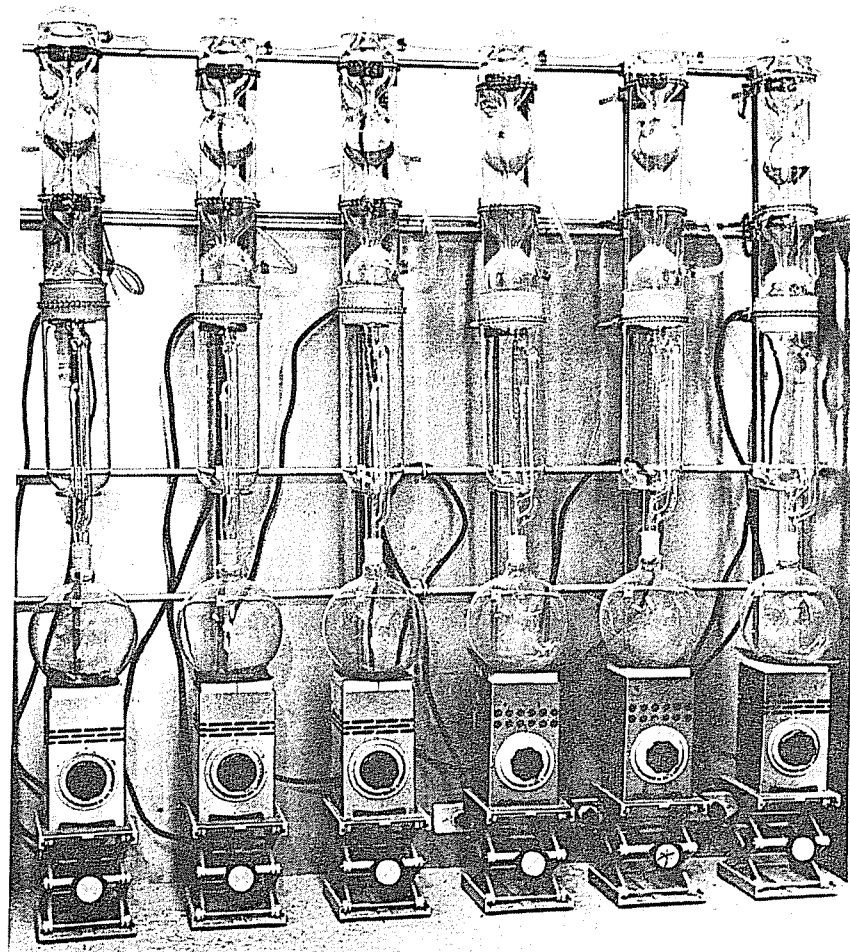


FIGURE 1

this procedure.) The condenser and extractor must be held individually in a fashion which allows the condenser to be moved vertically 4 to 5 inches, and the extractor to be removed and replaced with ease. The Soxhlet Extraction Apparatus shall be used under a functioning fume hood.

(b) Heating device with adjustment for temperature, capable of maintaining a constant flow rate of distillation, and able to hold the 3000 ml. round bottom extraction flask. (Precision Scientific, "Ful-Kontrol-750 watts", or similar is required.)

(c) Adjustable stand for heating device.

(d) Water source with miscellaneous tubing and clamps.

(e) Filter paper (Fluted, 50 cm., Grade 515)

(f) Methylene Chloride, Tech. Grade (Reclaimed Methylene is NOT recommended when solvent is to be subjected to ARIZ 511).

(g) Oven or hot plate capable of maintaining a temperature of $230^{\circ} \pm 9^{\circ}$ F.

(h) Balance, 5 kg. capacity, sensitive to 1 gram.

(i) Spoons, spatulas, pans, and miscellaneous tools.

(j) 1000 ml. beaker.

Sample Preparation

3. (a) A representative 1250 to 1750 gram sample of the bituminous mixture shall be obtained. A representative 500 to 1000 gram sample shall also be obtained for moisture determination (ARIZ 406b - Moisture in Bituminous Mixtures)

(b) When cores are being tested, they shall be heated and broken into small pieces.

Procedure

4. (a) Place a double layer of filter paper in a tared 1000 ml. beaker.

(b) Record weight of filter paper to the nearest gram as "b".

(c) Tare beaker with filter paper.

(d) Firmly place sample into filter paper while in beaker so that it is filled to a maximum of 1 inch below the top of filter paper. Record the weight of the sample to the nearest 1 gram as "i".

(e) Remove filter paper and sample from beaker. Place in Soxhlet Extractor.

(f) Place extractor in holder, slide condenser in place into top of extractor.

(g) Place 8 to 12 boiling chips into the 3000 ml extractor flask.

(h) Add methylene chloride to extraction flask until approximately 2/3 full.

(i) Place the flask on the heating device and raise adjustable stand so that the top of flask fits snugly into bottom of extractor.

(j) Turn on heat and control temperature so that the solvent boils rapidly, and a constant rate of distillation is maintained.

NOTE: *Once the heat has been turned on, the apparatus should not be left unattended for long periods of time until extraction is complete. During the extraction process, the amount of methylene chloride in the extraction flask should not be allowed to fall below the 1/4 full level.*

(k) The solvent normally will not syphon automatically when the liquid level reaches the syphon tube level. The addition of a few ml. of solvent through the condenser should initiate the action.

NOTE: *Caution must be exercised to not exceed 3/4 of the capacity of the extraction flask due to continued additions.*

(l) Continue the recycling until the extraction liquor is clear, usually 8 or 10 cycles will be sufficient.

(m) When extraction is complete, turn off heat and allow apparatus and sample to cool.

(n) Lower extraction flask and remove from heating device. If recovery of asphalt from methylene chloride is desired, subject contents of flask to ARIZ 511 - Recovery of Asphalt from Extraction Solution. If bitumen content and sieve analysis of aggregate is all that is desired, discard contents of flask.

(o) Raise condenser so that the extractor which holds the sample may be removed.

(p) Transfer sample to clean tared pan for drying. Place filter paper in pan also. Thoroughly clean extractor, making certain all material is transferred into drying pan.

(q) Allow excess methylene chloride to evaporate under functioning fume hood, if desired cover sample and filter paper with a piece of aluminum foil which has been punctured 2-3 times, and then dry in oven or on hot plate at $230^{\circ} \pm 9^{\circ}$ F., to constant weight.

NOTE: *A micro-wave oven may be used to dry materials, provided proper attention is given to the use of apparatus; the intensity of heat generated so that sample does not splatter, and the ventilation of the exhaust fan. (If micro-wave oven is used the aluminum foil covering sample is not used.)*

(r) Record weight of dried aggregate (being sure to remove clinging aggregate from filter paper into aggregate pan before weighing) to the nearest 1 gram, as "d".

(s) Weigh filter paper which will contain minus No. 200 material and record to the nearest gram as "a". Discard filter paper.

NOTE: Materials shall be weighed within 1/2 hour after removal from heat to assure moisture has not been absorbed.

Sieve Analysis of Extracted Aggregate

5. The extracted aggregate shall be subjected to ARIZ 201 - "Dry Preparation and Sieving of Coarse and Fine Graded Soils and Aggregates".

Calculation and Example

6. Calculations to determine bitumen content and other data necessary are shown on laboratory card in Figure 2, along with an example.

Report

7. Report all values as required on the Asphaltic Concrete Tabulation laboratory card as shown in Figure 2.

MATERIALS SERVICES
 ASPHALTIC CONCRETE TABULATION

RECEIVED DATE

LAB NUMBER: G 1 3 7
 PROJ CODE: 8 11
 MATL: 12 13
 TYPE: 14 15
 PUR-POSE: 16
 LAB: 17
 SPEC #: 18
 SIZE: 19
 SIZE %: 20

TEST NO.: 22 25
 LOT OR SUFFIX: 26 27
 SAMPLED BY: 28 55 35
 MO.: 36
 DAY: 41
 YEAR: 42
 TIME: 45

LIFT NO.: 46 47
 SAMPLED FROM: 48 63
 RDWY: 64 65
 STATION: 66 69
 PLUS: 71

P = PIT
 E = EXC.
 ORIGINAL SOURCE: 72 74
 STATION OR PIT NO.: 75 78 80
 PROJECT NUMBER: []
 IF MILEPOST, INPUT DECIMAL IN COL. 69

I = IN PLACE IF MILEPOST, INPUT DECIMAL IN COL. 78

REMARKS: G 2 EXAMPLE SOXHLET EXTRACTION
 34 48 61
 62 80

KEYPUNCH INSTRUCTIONS: Duplicate col. 3 thru 7 on all cards

COARSE FACTOR: G 3 071174
 FINE FACTOR: 135468

ARIZ 413
 EXTRACTION TEST

G 6 ARIZ 817
 VOIDS ANALYSIS

	WEIGHTS RETAINED	% RET.	% PASS	SPECS.
3"	12			
2 1/2"				
2"				
1 1/2"	27			
1"	32	0	100	
3/4"	37	93	7	93
1/2"	42	287	13	80
3/8"	47	362	6	74
1/4"	52	541	13	61
#4	57	637	6	
-#4	62	768		
Total	67	1405		

Total Dry Wt. of Pass #4 Split: 406
 $t = \frac{q \times c}{r} + q$

	WEIGHTS RETAINED	% RET.	% PASS	SPECS.
#8	11	87	12	43
#10	13	7	36	
#16	22	11	25	
#30	27	8	17	
#40	30	3	14	
#50	32	3	11	
#100	35	4	7	
#200	37	3		
-#200	38		3.8	
Total	41			

u = DRY WEIGHT
 $u = t - u$
 Asphalt Content Target Value

a. Wt. of filter and - #200: 36 g
 b. Predetermined Wt. of Filter: 29 g
 c. Dry Wt. of - #200 (a - b): 7 g
 d. Dry Wt. of Extracted Agg.: 1398 g
 e. Dry Wt. of Ext. Agg. and - #200 (c + d) (enter in l below and course sieve total): 1405 g
 f. Trap Reading: 25 2.5 ml
 g. Wt. of Moisture Sample: 28 843 g
 h. Moisture Content $\frac{100 \times f}{g - f}$: 0.30 %
 i. Initial Wt. of the AC Sample: 1489 g
 j. Wt. of Water (h x i ÷ 100): 39 4 g
 k. Wt. of AC Less Water (i - j): 1485 g
 l. Dry Wt. of Ext. Agg. and - #200 (e): 1405 g
 m. Wt. of Ext. Asphalt (k - l): 49 80 g
 n. Ext. Asphalt Content (m ÷ k x 100): 52 5.39 %
 o. Asphalt Retention Factor: 56 58
 p. Total Asphalt Content (n + o): 59 5.39 %
 q. O.D. Split Wt. of Pass #4: 402 g
 r. Dry Wt. of Pass #4: 66 767 g

C = Core
 M = Marshall
 H = Hveem

A. Asphalt Absorp. (O.D. Basis): 9 11 %
 B. Asphalt Absorp. (100% of Mix) (100 - p) x A: 12 14 %
 C. Effective Asphalt Content (p - B): 15 18 %
 D. AC Mix Dry Bulk Sp. Gr. (AASHTO T-166): 19 22
 E. Agg. Relative Dry Sp. Gr. $D \times (100 - p)$: 23 26
 F. Combined Agg. Bulk O.D. Sp. Gr.: 27 30
 G. V.M.A. $(1.000 - \frac{E}{F}) \times 100$: 15.5 - 18.0
 H. E.V. (Air Voids) $G - (\frac{D \times C}{1.015})$: 34 36 %
 I. Voids Filled $(\frac{G - H}{G}) \times 100$: 37 39 %
 J. Samp. Max. Sp. Gr. $\frac{100 \times D}{100 - H}$: 40 43
 K. Samp. Max. Dens $J \times 62.3$: 44 47
 L. AC Mix Bulk Density (D x 62.3): 48 51
 Stability: 52 56
 Cohesion or Flow: 57 60
 Asphalt Sp. Gr. = 1.015
 Water Density = 62.3 PCF

WHITE
 YELLOW
 BLUE

Abs. Visc. @ 140°F: 70 121380
 Joe Tester
 TEST OPERATOR

LABMAN, PROJECT SUPERVISOR,
 OR RESIDENT ENGINEER

FIGURE 2