Yardstick for Guidance in Evaluating Quality of Asphalt Cement

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•THE PENETRATION test, performed at 77 F, 100 g, 5 seconds, has been used for over 40 years on original asphalt cements, on residues from oven loss tests, or on recovered asphalts from hot-mixed pugmill operations to measure the hardening effect of aging on asphalt cement. However, this test cannot be depended on now in many areas due to crossblending of crudes from which the asphalt is produced and to the use of additives and inhibitors.

Observations made by the Standard Oil Company of Ohio Laboratory on a special test road built in 1953 on the State highway system near Millersburg, Ohio, showed the inadequacy of the penetration test as a quality guide when the penetration test was run on recovered asphalts. Asphalt cements of the 85-100 penetration grade from different refineries and different crudes were used following the same mixing and same laying procedure with significant differences as given for two of the asphalts in Table 1. The recovered penetrations were approximately the same yet one pavement cracked extensively, while the other remained unaffected. The recovered asphalt when tested for ductile characteristic at 55 F, 1 cm per min clearly identified the difference in the two asphalts.

Other observations pointed out that ductilities on original asphalts run at 77 F, 5 cm per min did not give a definite indication of the sensitivity to hardening of an asphalt cement in the hot-mixing operation. It was observed that there were asphalts which would have ductilities of 100+ cm on the original asphalt at 77 F, 5 cm per min, but which would still manifest early cracking when incorporated in a properly prepared and properly placed and compacted pavement. These asphalts showed a noticeable difference when tested for ductility as supplied at a lower temperature as given in Table 2.

For years various publications (Appendix C) have pointed towards the use of low temperature ductility as a criterion for spotting early cracking asphalt cements not particularly on the original asphalt but more importantly the tests run on the asphalt recovered from cores from the pavement. Paving engineers have observed that cracking occurs as asphalt pavements gradually chill down to lower temperatures in passing from the summer months into the winter period.

Asphalt cements were laboratory tested for expansion and contraction at different temperatures using the linear thermal expansion of penetration grade asphalt test (see Appendix A). Curves (Fig. 1) plotted from these tests reveal definite breaking off or transition points at which the asphalt changes from a plastic to a solid form. These points for asphalt cements were between temperatures of 30 F and 50 F. This same method of testing on asphaltic concrete indicated that transition points were close to 45 F for typical mixtures.

Because it was found by the laboratory that 77 F was too warm a temperature to run ductilities having any significance on original or recovered asphalts, a series of tests were run on both original asphalts and recovered asphalts at 55 F, 1 cm per min. The results are shown in Table 3. All five samples showed a 100+ cm ductility at 77 F, 5 cm per min on the original asphalt, whereas there was quite a difference in the ductility of the samples when run at 55 F, 1 cm per min. The ductilities on the recovered asphalts pointed up even more noticeable differences.

The results of these tests at 55 F, 1 cm per min and others were still not capable of equipping one to separate the good from the bad accurately. Testing ductility at 45 F, 1 cm per min was inaugurated and finally adopted as being a more desirable temperature and speed to evaluate ductile characteristics on both original and recovered asphalt cements.

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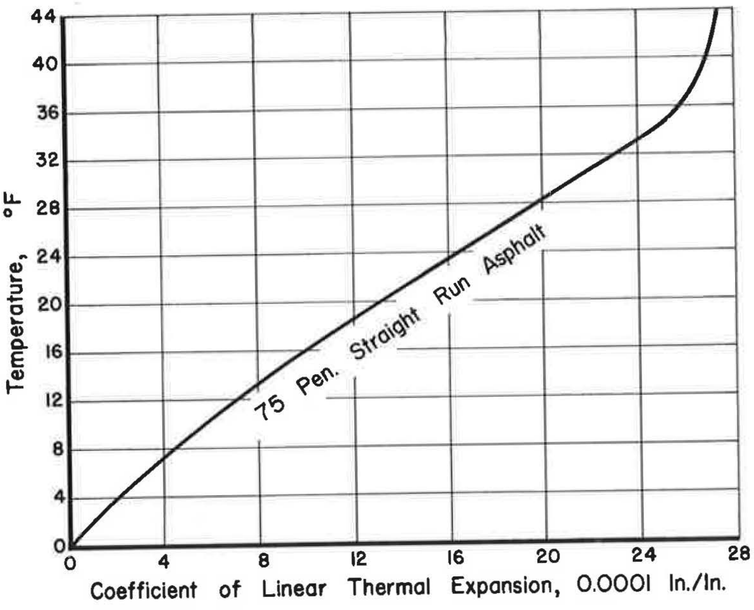


Figure 1.

TABLE 1

ASPHALTS USED IN TEST ROAD 2, 1953

Penetration and Ductility	Asphalt A Cracked After 2 Yr	Asphalt E Not Cracked After 3 Years
Orig. asphalt:		
Pen. at 77 F, 100 g, 5 sec	94	88
Hot-mix plant simulating test recovered asphalt:		
Pen. at 77 F, 100 g, 5 sec	49	48
Duct. at 55 F, 1 cm per min	7	16
Asphalt recovered from cores taken after 3 years:		
Duct. at 55 F, 1 cm per min	6	12

TABLE 2

ORIGINAL ASPHALTS

Test	Asphalt A	Asphalt E
Penetration at 77 F, 100 g, 5 sec	94	88
Ductility at 77 F, 5 cm per min	100+	100+
Ductility at 45 F, 1 cm per min	9	28

TABLE 3

ASPHALT CEMENTS PRODUCED FROM VARIOUS CRUDES, 1958

Asphalt	Original Asphalt			Hot-Mix Plant Simulating Test Run 325 F[1]	
	Pen. at 77 F, 100 g, 5 sec	Duct. at 77 F, 5 cm	Duct. at 55 F, 5 cm	Pen. at 77 F, 100 g, 5 sec	Duct. at 55 F, 1 cm
Crude 1	50	100+	100+	36	16
Crude 2	53	100+	82	43	20
Crude 3	53	100+	28	35	7
Crude 4	54	100+	54	39	10
Crude 5	55	100+	100+	39	19

[1]Recovered asphalt.

TABLE 4

ASPHALT CEMENTS FROM VARIOUS CRUDES, 1958

Crude	Original Asphalt		Hot-Mix Plant Simulating Test[1]	
	Pen. at 77 F, 100 g, 5 sec	Duct. at 55 F, 1 cm	Duct. at 55 F, 1 cm	Pen. at 77 F, 100 g, 5 sec
A	86	100+	30	37
B	89	100+	26	36
C	88	100+	30	40
D	88	100+	55	48
E	91	100+	14	61
F	87	100+	46	57

[1]Recovered asphalt.

TABLE 5

ASPHALT RECOVERED FROM TEST ROAD[1] BUILT IN 1953

Core	Duct. at 55 F, 1 cm	Pen. at 77 F, 100 g, 5 sec
1	5	33
2	5	31
3	8	48
4	8	39
5	6	41

[1] Cracked extensively after three years of service; all cores from same road.

TABLE 6

LOSS OF DUCTILE[1] CHARACTERISTICS

Asphalt	Pen. Range	Pen. Avg.	Oven Weath. Temp. (°F)	Ductility
Str. asph.[2]	85-100	88	-	150+
	70- 85	78	-	130
	60- 70	63	-	43
	50- 60	56	-	39
Mixed and oven weath.[3]	85-100	-	289	19
	70- 85	-	293	13
	60- 70	-	298	8¼
	50- 60	-	302	7½
Mixed and oven weath.[4]	85-100	-	325	10½
	70- 85	-	325	6½
	60- 70	-	325	5¼
	50- 60	-	325	5

[1] Ductility measured at 45 F, 1 cm per min, June 1962.
[2] As delivered to contractor.
[3] Hot-mix plant simulating test; correct temperature for each grade.
[4] Hot-mix plant simulating test; 325 F for all grades.

TABLE 7

DUCTILITY[1] OF ASPHALT CEMENT RECOVERED FROM CORES FROM HIGHWAYS

Location	Age (yr)	Pen. at 77 F, 100 g, 5 sec	Ductility at 45 F, 1 cm	Condition	Air Voids (%)	Asphalt (%)
Fairfield (W. 14-Scranton) Cleveland	14	17	0	Cracked	5.5	6.8
Memphis (Pearl-W. 45) Cleveland	13	41	4¼	Cracked	4.0	7.0
Baltic (W. Blvd.-W. 95) Cleveland	13	28	4	Cracked	3.7	6.7
Maine Turnpike[2] (cored 1961)	14	21	3⅜	Cracked	0.3	6.5
Fairfield (Professor-W. 14) Cleveland	14	78	15½	No Cracks	3.0	6.8
Memphis (W. 45-Border) Cleveland	12	63	9½	No Cracks	1.6	5.8
Clifton (W. 95-Lake Road) Cleveland	13	29	14	No Cracks	4.6	7.8

[1]At 45 F, 1 cm per min.
[2]From different refinery and different crude.

TABLE 8

AVERAGE OBSERVATIONS HOT-PLANT MIXES, ASPHALTIC CONCRETE MIXED AT CORRECT TEMPERATURE

Penetration	Asphalt	
	Orig. Duct.	Recovered Duct.[1]
85-100	150+	15-25
70- 85	115	10-14
60- 70	35	6- 9
50- 60	15+	5- 8

[1]At 45 F, 1 cm.

The asphalt from Crude 3 was the only cement which field tests indicated showed signs of some early deterioration. If these recovered asphalts had been run at 45 F, 1 cm per min, the deficiency would have been much more pronounced.

A test somewhat similar to the widely-known Shattuck test which attempts to simulate the action taking place in a hot-mix pugmill operation in the production of asphaltic concrete mixtures has been standardized. It is identified as the hot-mix plant simulating test (see Appendix B). Its procedure combines an adaptation of the BPR mixing and weathering test with the Abson recovery test.

Samples of asphalt cement produced from different crudes were tested both on the original asphalt and the recovered asphalt from the hot mix plant simulating test. Results are given in Table 4.

Table 5 includes tests on recovered asphalts from a test road in Indiana. This road cracked extensively and the low ductility recordings would identify the lack of stretchability at relatively low temperatures. Had these recovered ductilities been run at 45 F, 1 cm per min, they would have been more clearly defined. Original asphalt was 60-70 penetration at 77 F, 100 g, 5 sec. Tables 4 and 5 were completed prior to adoption of the 45 F, 1 cm per min test condition.

Low ductility in a pavement can be caused by overheating of the asphalt in the pugmill. Table 6 shows the tremendous drop in ductile characteristic which an asphalt experiences when subjected to hot mixing in a pugmill.

Table 6 gives results from four different grades of asphalt cement when mixed at the correct temperature. (Proper mixing temperature for any given asphalt can be determined by the procedure outlined in Appendix B.) These cements were produced from the same crude. The mixing and weathering test simulates mixing in an actual pugmill operation. Extracted asphalt will show ductility at 45 F, 1 cm per min, close to that retained in the highway. If this test reveals an 18-cm ductility at 45 F, 1 cm per min, it is reasonable to expect asphalt mix from a commercial plant to run 25 cm. This difference is due to a specified asphalt content of 4 percent, whereas actual plant mixes will normally contain a higher asphalt content.

It is easy to see why paving engineers pick the softest asphalt consistent with stability to use in mixes. A great deal of work has been performed in an attempt to inhibit this loss of ductile characteristic in the hot-mixing operation. There are some inhibitors and additives available which will contribute materially toward preventing the tremendous drop in ductility.

These data emphasize the desirability of testing asphalt extracted from paving-type mixtures prior to using the asphalt cement in hot-mix construction. The results on such mixes will give a very good indication of what one can expect of the performance of the pavement in time.

Table 7 gives the results of asphalt cement recovered from cores taken from various highways. Pavements that had above 8-cm ductility at 45 F, 1 cm per min after various periods of service were not cracked and not raveled. The pavements showing less than 8-cm ductility at 45 F, 1 cm per min were in each case cracked or raveled.

Samples of paving mixtures produced at various plants and using many different asphalt cements from different crudes and refineries were tested by the laboratory. The test results were averaged and are given in Table 8, indicating that where the softest asphalt cement (85-100 penetration) was used, the original ductility was 150+ cm but recovered ductilities were between 15 cm and 25 cm. All other grades also lost most of their ductile characteristic in the mixing operation.

SUMMARY

Low temperature ductility provides a method of measuring the future service behavior of asphalts and the pavements in which they are incorporated. An improved laboratory method provides a tool whereby the simulation of the hot-mix pugmill operation on a laboratory basis can be duplicated. This procedure will allow an evaluation of the asphalt before it is placed.

It has been determined that any paving mixture which contains an asphalt which shows a ductility exceeding 8 cm should be free from cracking. A value somewhat higher than 8 should be established to provide a safety factor. It is suggested that a minimum ductility at 45 F, 1 cm per min run on extracted asphalt from the paving mixture should be in excess of 25 cm at the start of its service record. This 25-cm minimum ductility correlates with the 18-cm minimum ductility in the hot-mix plant simulating test (Appendix B). The explanation of the difference between 18 cm and 25 cm lies in this test's requiring a specified amount of asphalt (4%) whereas field plants vary upward in their percent of cement used.

Other work in connection with this ductility study has brought out other factors which may contribute to the hardening of the binder in an asphalt pavement:

1. A correct mixing temperature in the pugmill for the particular asphalt cement being used should be adopted. If the correct temperature was exceeded, additional loss of ductility was noted.

2. An asphalt content as high as possible, consistent with maintaining a desired level of stability, should be used. Thin asphalt films should be avoided.

3. The paving mixture should be compacted at the time of construction to show less than 5½ percent voids. Equipment permitting the reading of density tests made on the surface of the pavement immediately following the roller would be most helpful.

Appendix A

LINEAR THERMAL EXPANSION OF PENETRATION GRADE ASPHALT

The purpose of the test is to determine accurately the linear movement of a briquette of asphalt when subjected to controlled variations in temperature.

General Description

A small briquette of solid asphalt is supported in a methanol-water bath. As the temperature is changed, measurements of sample movement are observed with a cathetometer.

Sample Preparation

The specimen is a casting of solid asphalt molded into a 1½- × 1½- × 12- in. briquette, using a demountable brass mold. The mold is coated with a thin film of Dow Corning 200 fluid before casting to minimize sticking. Small threaded Invar metal plugs are carefully embedded into the vertical face of each end of the specimen during the casting. One is used to anchor the specimen rigidly to an Invar frame during the test; the other, to secure a reference point used for cathetometer observations. The underside contact surface of the sample has a thin rubber membrane affixed to it to eliminate the possibility of sample adhesion to the specimen holder, particularly at higher temperatures.

Instrumentation

Elongation measurements are made by following the linear movement of an index point embedded in the free end of the sample at the time it is cast. This movement is measured with a rigidly-mounted cathetometer.

Temperature measurements are made with thermometers embedded in a second briquette of asphalt, also supported in the bath. This second briquette has the same cross-section dimensions as the test specimen. Temperature ranges from -10 to +80 F have been used in this test.

Bath Coolant

The heat transfer medium consists of a methanol-water solution adjusted so that the gravity of the combination is less than the asphalt specimen to minimize its buoyant effect on the specimen.

Procedure

The asphalt specimen is supported in an Invar metal frame with one end of the specimen free and the other anchored to the frame by means of a small threaded Invar plug. The specimen and frame are immersed in an insulated methanol-water bath. The coolant is circulated in this table top bath from an adjoining bath equipped with a coolant pump and provisions for varying temperatures of the heat transfer media under carefully controlled conditions.

Measurements of linear sample movements are made in ± 10 F units. The temperature of the bath is changed 10 F, the specimen conditioned for one hour and a linear measurement made. The temperature is again changed 10 F, the sample held at this new temperature for one hour and a linear measurement again recorded. This sequence with a series either of increasing or decreasing temperatures is followed until sufficient observations are made to record the linear thermal characteristics of the asphalt specimen adequately.

Transition Point

When a sample of asphalt is subjected to slight increases in temperature a definite linear expansion is observed. Above a certain temperature this movement ceases and it is assumed that at this point the specimen has changed from a rigid material to a plastic, amorphous substance. This characteristic and observation is relative and dependent on the test conditions used, but it forms an interesting comparison of asphalt behavior when numerous samples are checked under similar conditions.

Appendix B

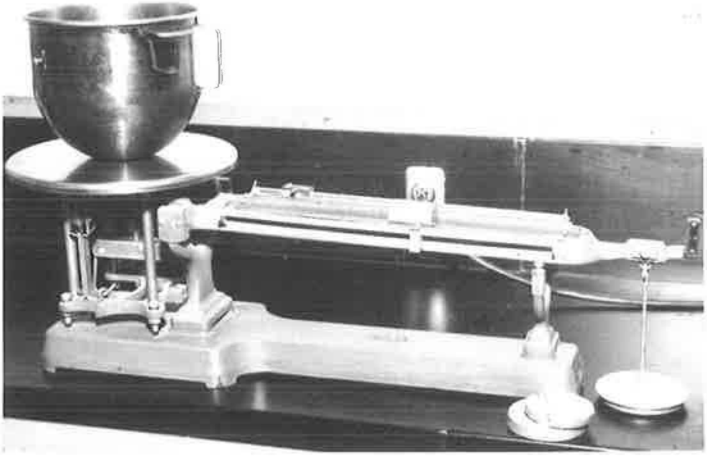
HOT-MIX PLANT SIMULATING TEST

This is a method for determining the sensitivity of an asphalt to mixing and heating at a certain film thickness, measured by ductility at a relatively low temperature and testing speed.

Oven Weathering

The temperature of the asphalt is measured with a Saybolt-Furol viscometer at 120 sec (Fig. 2). The measured temperature becomes the mixing temperature of the asphalt and sand and also the oven temperature in which the mixture is placed for one-half hour after mixing. This temperature, usually falling between 290-310 F for normal 85-100 penetration asphalts, is specified to the nearest degree to insure equal film thickness for all asphalts when mixed with the sand. Plotting of viscosity and temperature on semilog paper aids in finding the 120-sec viscosity temperature.

Figure 2.



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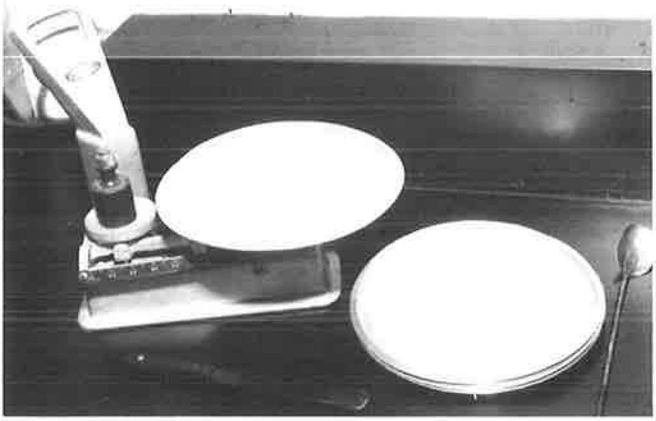

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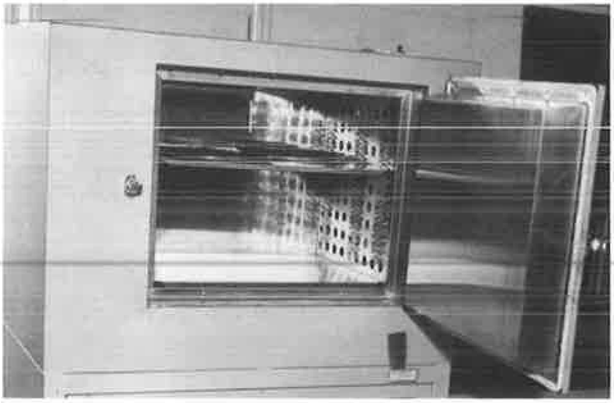
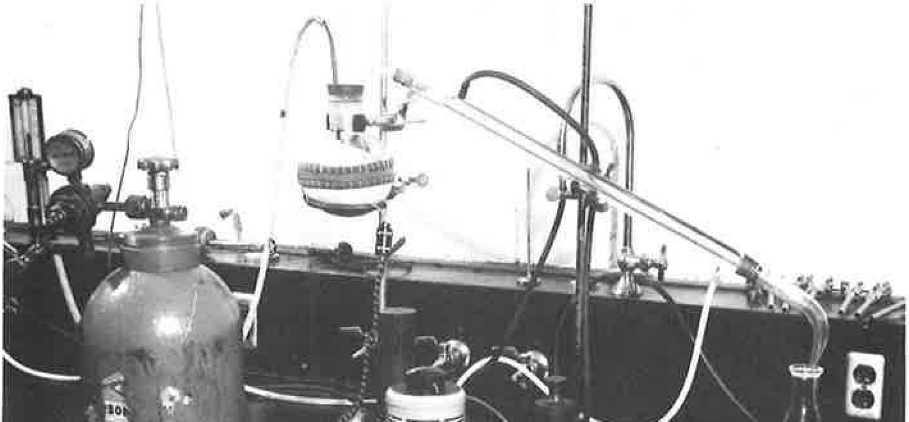


Figure 5.

Figure 6.



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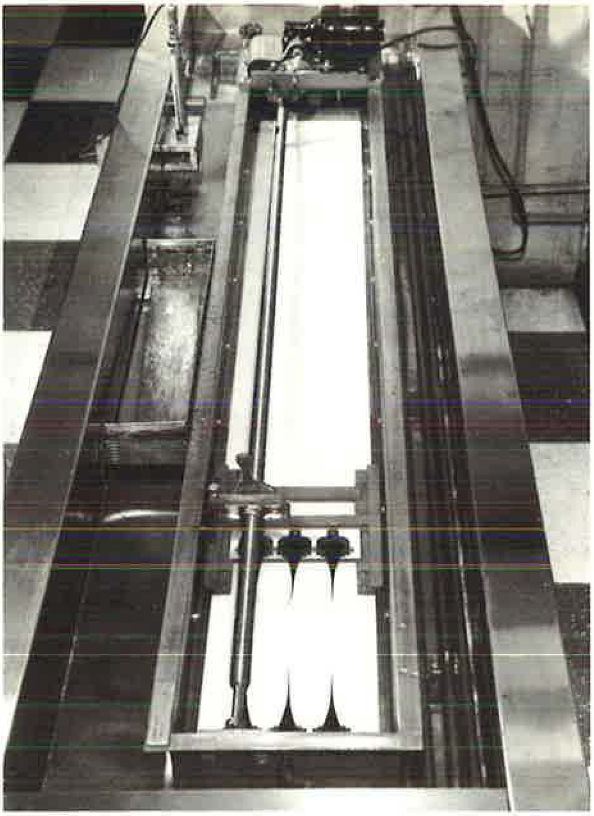

Figure 8.

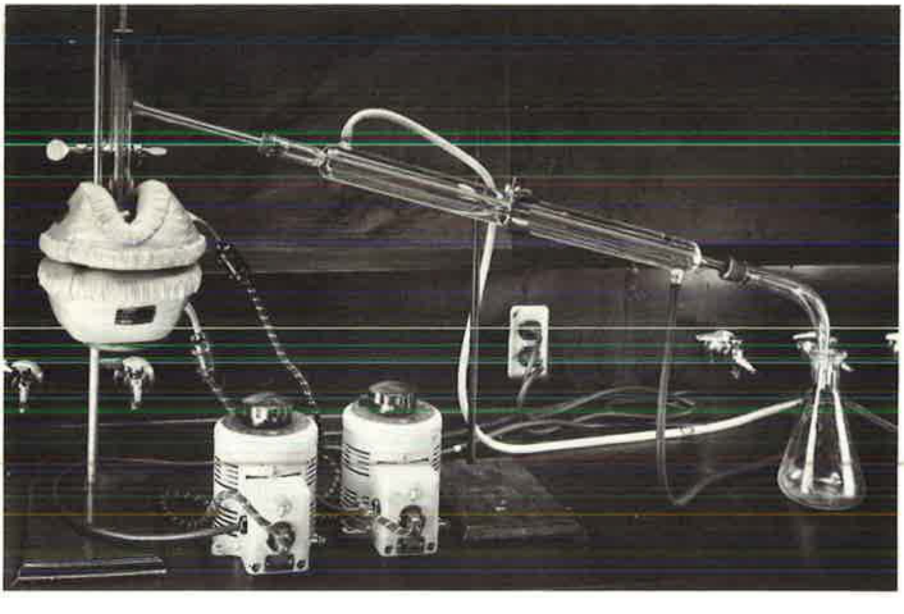

Figure 9.

Exactly 2, 000 g of Ottawa sand (ASTM C-190, 20-30 sieve) is placed in a bowl (Fig. 3). Approximately 160 g of asphalt is placed in a clean container. The sand and asphalt are heated separately in an oven to the 120- sec viscosity temperature. When the asphalt and sand have reached temperature, the previously tared bowl and sand are removed from the oven and placed on the balance. Exactly 80 g of asphalt is placed in the bowl with the sand.

The asphalt and sand are mixed immediately in the Hobart mixer by the preheated, rigid paddle for exactly 2 min. The main shaft speed is set at 60 rpm and causes the paddle speed to be slightly over twice the 60-rpm rate. The heat is maintained within ± 10 F with three 250-w infrared lamps (see Fig. 11). After a 2-min mixing, the bowl with the hot mixture is removed and the hot mixture is then batched into 2 aluminum pizza pans (Fig. 4). Exactly 900 g of mixture is placed into each pan and smoothed out uniformly so that the mixture covers the inside surface evenly in area and in depth. The pans are No. 814 gage, 13-in. O. D., 12-in. I. D. and $^3/_8$-in. deep throughout the I. D. The pans with the mix are placed in a safe place resting on a table and are cooled to room temperature for 1 hour, minimum.

After the mixture has reached room temperature, it is ready for weathering in the oven (Blue-M POM 120, oven accuracy ± $^1/_2$ C, 25- × 20- × 20- in. I. D.; forced convection 50 fpm). The mixture in pans is placed on a shelf in the oven (Fig. 5) for exactly $^1/_2$ hr at the exact temperature which gives a 120-sec viscosity. The $^1/_2$-hr weathering is measured immediately from the time the pans are introduced into the oven. Care should be taken to achieve the 120-sec temperature as quickly as possible. Technique will achieve this temperature within a minute.

After $^1/_2$-hr baking, the pans are removed from the oven and allowed to cool to room temperature for at least 1 hr before recovery. The pans are placed on a flat surface while cooling. (After the oven heating, the sample may be allowed to stand overnight before extracting with benzene.)

The mixture is scraped from the pans into the Dulin Rotarex (Fig. 6) with a putty knife and the benzene added, allowing 250 ml of benzene (C. P.) for each panfull. After soaking about 10 min, the solution is rotated off. The solutions from several identical samples may be combined before distillation.

Asphalt Recovery Procedure

The asphalt recovery procedure is begun with centrifuging the solution. A sample is heated to 250 to 270 F temperature. The mix is broken up with a spoon or spatula and 1, 000 g is introduced into the previously tared Dulin Rotarex bowl. Approximately 250 cc of benzene, C. P. grade, is added and the mixture allowed to soak 20 min. The mixture is then rotated and the benzene-asphalt solution extracted. The remaining aggregate and asphalt may be washed again with benzene (C. P.) and allowed to soak 20 min, then rotated off. The two solutions are then combined.

The solution is placed into two 250-ml centrifuge bottles and centrifuged at 870 g's for 45 min to remove all fines (see Fig. 12). In the hot-mix simulating test this time can be reduced to 15 min because of the small amount of fines present in the Ottawa sand. The solution is decanted from the bottles into a 1, 000-cc spherical distilling flask.

The distilling flask (see Fig. 9) has a side arm 77 mm below the top of the neck and is at a 75^0 angle (see Fig. 13). The flask is heated by two Glas-Col Series O heating mantles controlled by two Variacs. An ASTM 30-180 F thermometer is inserted in the flask and positioned so that the bulb is $^1/_4$ in. from the bottom. The side arm of the flask is connected to a straight innertube condenser.

When approximately 200 ml of benzene has distilled over, heating is suspended and the residual solution is transferred to a 500-ml round bottom, short neck, 50/50 joint flask (Fig. 7), heated by a Glas-Col heating mantle, 500-cc capacity and controlled by one Variac. The flask is stoppered by one size 11 neoprene stopper. Passing through the stopper is a $^3/_{16}$-in. copper tube for the introduction of CO_2 (see Fig. 14). It is important that the distributing holes in the tube foot be directed towards the flask bottom. Also passing through the stopper is an ASTM 85-392 F thermometer, whose

Figure 10.

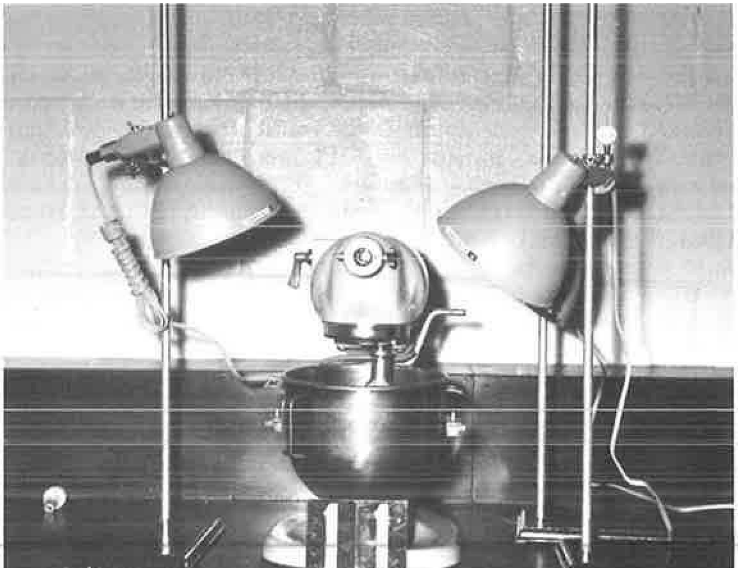

Figure 11.

Figure 12.

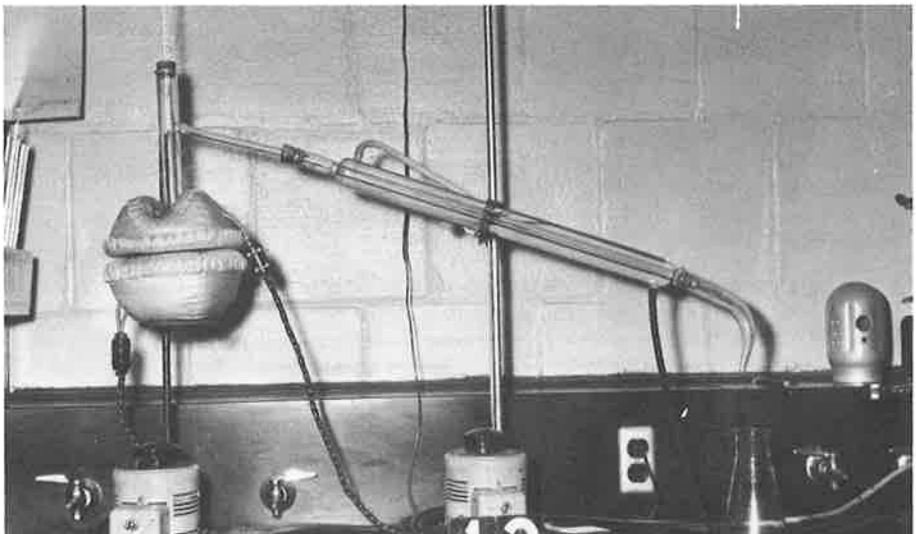

Figure 13.

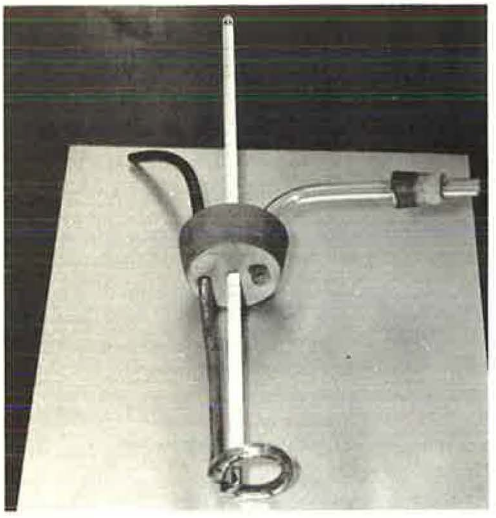

Figure 14.

bulb is centered in the middle of the copper tube foot, 1/4 in. from the flask bottom, and a glass tube for the delivery of benzene vapor to the condenser.

Heat is applied to the flask and the benzene distilled off at the rate of 50-70 drops per minute. When a temperature of 300 F is reached, CO_2 is introduced slowly until 325 F ± 5 F is reached, at which point a rate of 875 cc of CO_2 per min, ± 100 cc, should be obtained. During the CO_2 introduction, it will be necessary to turn the Variac down or possibly off for a short period to avoid overheating. When the drops from the condenser are 30 sec apart the end point of distillation is 15 min additional at 325 F.

The heat is discontinued and the apparatus disassembled. The CO_2 is allowed to run to prevent the copper tubing from clogging. The tube assembly is placed in solvent for cleaning. The asphalt is poured into a 3-oz penetration tin and stirred 1 min to remove the CO_2 bubbles. In no case should the asphalt be permitted to remain in solution more than 10 hr.

Ash

A sample of the asphalt is ashed in accordance with AASHO Method T-111-42. The ash content should be below 0.5 percent. In no case should it be above 1 percent by weight. If a sample yields an ash higher than 1 percent, extract a new sample of asphaltic concrete and increase the centrifuge time accordingly.

Ductility Tests

The recovered asphalt is tested for ductility according to ASTM Designation: D-113-44 at a temperature of 7.2 C ± 0.1 C (45 F ± 0.2 F) and at a rate of pull of 1 cm/min (Fig. 8). In preparing the sample the asphalt is heated in a penetration cup to 300 F with stirring. It is immediately placed in a vacuum bell jar and subjected to a vacuum of 10- 15 mm Hg until bubbling has ceased. It is then removed and poured. In some cases additional heating may be necessary before pouring. The molds are cut off with a sharpened, stiff back putty knife.

Sampling Technique

When obtaining pavement samples as with a coring machine (Fig. 10), cores should be taken in the wheel tracks for maximum sample density. In this area the optimum weather resisting characteristics occur, due to a lowering in the voids content of the mix. Sampling between lanes or at the edge is suspect due to spillage of oil and grease.

Appendix C

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