Just Like



EVALUATION OF THE VACUUM EXTRACTION METHOD FOR DETERMINATION OF ASPHALT CONTENT IN BITUMINOUS MIXTURES



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EVALUATION OF THE VACUUM EXTRACTION METHOD FOR DETERMINATION OF ASPHALT CONTENT IN BITUMINOUS MIXTURES

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State Department of Highways and Public Transportation

5-06-74-179 October 1975

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ABSTRACT

This brief experimental study concerns the comparison of two test methods for determining the asphalt content of bituminous mixtures; the vacuum extraction method as detailed in the Federal Highway Administration's "Vacuum Method of Determining the Composition of Bituminous Aggregate Mixtures" and the centrifuge method as found in the State Department of Highways and Public Transportation Test Method Tex-210-F, "Determination of Asphalt Content of Bituminous Mixtures by Extraction." The data provide the comparison of testing and drying times as well as test precision. The study involves bituminous mixtures consisting of aggregates with varying absorptive qualities. Comments and precautions are also included with regard to experiences with the microwave oven and the vacuum extraction process. Alternate approaches are discussed.

IMPLEMENTATION

The Federal Highway Administration's "Vacuum Method of Determining the Composition of Bituminous Aggregate Mixtures" can be used as a substitute for the Texas State Department of Highways and Public Transportation's Test Method Tex-210-F when test results demonstrate similar accuracy. Alternate methods which are combinations of the two methods show promise and may be used when similar accuracy is supported by tests. Caution should be exercised in the use of the microwave radiation; and the safe use of solvents, other than those mentioned in this report, is questionable.

I. PURPOSE

The purpose of this limited investigation was to provide a brief comparison between the Federal Highway Administration's "Vacuum Method of Determining the Composition of Bituminous Aggregate Mixtures" with the State Department of Highways and Public Transportation's Test Method Tex-210-F, "Determination of Asphalt Content of Bituminous Mixtures by Extraction." See Appendix.

II. CONCLUSION

The test results from this brief study indicate an equality in the capabilities of the two procedures involved. When testing the mixtures included in this investigation, the results from both the vacuum and centrifuge method of extraction were practically identical. Neither method, however, is capable of yielding consistent satisfactory results when testing mixtures containing considerable amounts of highly absorptive aggregates.

The FHWA Vacuum Extraction method as published, was shown to shorten the overall time required for an extraction. This time improvement can be attributed to the use of the microwave oven for drying. The validity of eliminating ash determination should be verified by tests for each particular type of bituminous mixture. A combination of rotary extraction and microwave drying can also be shown to reduce total extraction time. There is also a possibility of eliminating ash determination with the rotary extractor by using a previously evaluated correction for each particular type of mix.

Precautions must be taken when using the microwave oven with materials and/or solvents for which no behavior experience is available. The validity of the vacuum extractor method should also be evaluated for bituminous mixes where no experience is available.

III. TEST METHODS AND EQUIPMENT

The test method and equipment used for the vacuum extraction procedure are those found in the Federal Highway Administration's "Vacuum Method of Determining the Composition of Bituminous Aggregate Mixtures." The equipment used for this procedure was obtained on loan from the Federal Highway Administration. The major pieces of equipment are as follows:

Vacuum Extractor - Soiltest Model AP-520, Serial No. 740806

Vacuum Pump - Gast Model 0522-V3-G18D, Serial No. 0674

Ultrasonic Cleaner - Bransonic 32

Microwave Oven - Litton Model 70-40.05, Serial No. 5957 JP.

The test method and equipment used for the centrifuge method of extraction are found in the State Department of Highways and Public Transportation "Manual of Testing Procedures," Test Method Tex-210-F, "Determination of Asphalt Content of Bituminous Mixtures by Extraction." The field extractor, shown in Figure 1 of this procedure, was the instrument used for all tests of this type.

IV. MATERIALS

The mixtures involved in Table I were made with crushed limestone aggregate.

The coarse aggregate (retained No. 10 sieve) for the mixtures in Tables II, III and IV were crushed lightweight expanded synthetic aggregates from three sources (A, B and C). The fine aggregate (passing No. 10 sieve) was crushed limestone screenings identical to those in the mixtures of Table I. All mixtures in this investigation were made with AC-10 asphalt.

The solvents used for the vacuum extraction test were as specified; denatured ethyl alcohol and methylene chloride. The solvent used for the Test Method Tex-210-F tests was 1,1,1 trichloroethane.

V. PROCEDURE

The aggregates mentioned under "Materials" were obtained and oven dried. The coarse aggregates were sieved to size, 3/8" - No. 4 and No. 4 - No. 10. The crushed limestone screenings were separated into No. 10 - No. 200 and Passing No. 200 sizes. The design gradations and mixtures were made as indicated in the tables. The total weight of each mixture was 1,500 grams and each was kept in the oven for two hours at approximately 250 F prior to extraction. All extraction tests were run in pairs as recorded in the tables. All tests were performed as described under "Methods and Equipment."

VI. DISCUSSION

The Federal Highway Administration has encouraged the use of the vacuum extraction method, accompanied by the ultrasonic cleaner and the microwave oven, for some time. Some personnel of the State Department of

Highways and Public Transportation have expressed interest in the procedure, and indeed, some have had considerable experience with the test method, but no work has been reported which compares this test method with Test Method Tex-210-F.

The advantages of the vacuum extraction method usually cited are speed and the elimination of the "ash" determination after extraction. It was the intent of this study to examine these reported advantages along with the overall efficiency of the two procedures.

The first column of the accompanying Tables contains the design gradation and asphalt content for the mixtures extracted. In order to assure consistency, each batch of mixture for extraction was made by weighing each sized aggregate required by the design and mixing it with the specifiec amount of asphalt. The extraction sample of mixture weighed 1,500 grams. The mixtures containing synthetic aggregate were left in the oven for two hours at approximately 250 F, prior to extraction, to allow for absorption.

The next three columns of the Tables list the two test results and the average of these results for Test Method Tex-210-F.

Immediately following the tabulation of the extraction test results in each Table is listed, Residual Bitumen: No "ash" correction and Correction for "ash." The normal method for determining the residual bitumen after extraction by Test Method Tex-210-F includes correction

for "ash." This value is therefore listed with the extraction test results, as indicated. In order to determine the error introduced by not correcting for the "ash," the <u>No "ash" correction</u> values were calculated and recorded.

The reverse is true for the vacuum method of extraction. No "ash" correction is the norm for this procedure and thus these values are included in the extraction results, as indicated. An "ash" determination was made after each vacuum extraction, the resulting residual bitumen values being recorded in each column as Correction for "ash."

Examination of the test results reveals that with the exception of Table IV the average values of the two extraction test methods are practically identical. One could not expect a better correlation of test data between two test procedures performed by different laboratory personnel. The reason for the higher asphalt content values of the Test Method Tex-210-F in Table IV is not known. Since the two individual Tex-210-F tests agree, it is possible that an error was made in the fabrication of the test samples. Other than this one exception, all data indicate that the precision of the two test methods is equal.

The data support the need for "ash" correction when Test Method Tex-210-F is performed. The figures in Tables II and III may appear to refute this statement but knowledge based upon past experience with synthetic aggregates provides a proper perspective. Many widely used

synthetic aggregates are unusually absorptive. Highly absorptive synthetic and natural aggregates have created extraction problems when used as a major portion of a bituminous mixture. Current methods of extraction do not produce consistent residual bitumen values equal to the actual asphalt contents of bituminous mixtures in which highly absorptive aggregates are used. The No "ash" correction values of Table II are closer to the correct asphalt content than the "ash" corrected values of the Tex-210-F tests. The difference between the values corrected for "ash" and those not corrected for "ash" is caused by dust and not asphalt. The higher value is a result of fines in the extraction effluent, consistent with all the other tests, and therefore is not a "truer" asphalt content value, more closely approximating the original. "Ash" is a real substance in the effluent of Test Method Tex-210-F and must be dealt with and not ignored. When the standard test procedures are followed, Table II shows that the two test methods yield identical asphalt content values, and this is as it should be.

The "ash" from the vacuum extraction method, when testing these four combinations of materials, is insignificant. It is not of sufficient quantity to warrant consideration, however this is not to say that it can be ignored when testing any and all material combinations. Preliminary tests with untested materials are necessary to establish confidence.

It is to be noted at this point that neither procedure provided a satisfactory asphalt content value for the absorptive mixture of Table II. "Testing Time: Extraction of asphalt," following the extraction results in each Table, is just that, the time elapsed from inundation until the extracted aggregate is in the drying pan.

The conventional laboratory soils drying oven was used as standard procedure for drying the aggregate after extraction for Test Method Tex-210-F. The Tables show the time to dry the material to a constant weight and the maximum temperature of the oven during this drying period. The rather low maximum temperature results from the fact that the oven was being used for other laboratory work. The average temperature during these tests is not known. It varied with the use of the oven. Test No. 1 under Tex-210-F in Table II illustrates the price one pays in drying time for opening the oven frequently for other laboratory work. This unrealistic extension of time should be considered when comparing drying times as well as the fact that all drying times could be shortened if oven temperatures had been higher.

For this study the microwave oven was considered as standard equipment for the Federal Highway Administration's vacuum extraction method. The aggregate drying times are recorded in each Table. The maximum sample temperatures are not available for the tests in Tables I and II. These temperatures are listed in Table III.

Problems developed when drying the aggregates in the microwave oven for the mixtures of Table IV. Previous experimentation had established a workable procedure for the drying process. Extracted aggregates were

dried for three minutes and then weighed. They were placed in the oven immediately and dried for three more minutes and weighed. After a third three-minute drying period (total of nine minutes drying time) all extracted aggregate samples tested to date have been dry with respect to constant weight. Sample No. 1 under Vacuum Method in Table IV became excessively hot during the last three-minute drying period. Some particles glowed "red hot." The maximum temperature gradation on the thermometer used was 450 F, thus the recorded value of "450 +." A few seconds after placing Sample No. 2 of this method in the microwave oven for the third three-minute drying time the bowl literally "fell apart." The sample was carefully reclaimed from the oven and the final weight obtained. The actual drying time is unknown. It is something better than six minutes as indicated. Once again, the sample was extremely hot and therefore the temperature unattainable with available test thermometers.

This experience, along with a number of similar incidents prior to this investigation, necessitates a word of caution concerning the use of the microwave oven. It is a piece of equipment of unusual capabilities, but it is also capable of creating hazardous conditions in the laboratory.

Other testing organizations have had similar experiences to ours when drying certain aggregates in the microwave oven. Iron ore and soils and aggregates containing iron are known to react excessively to microwave radiation. They become unbelievably hot very quickly. In this series of tests only one synthetic aggregate extracted sample caused a problem.

The point is that one must take every precaution when submitting an untried aggregate to microwave radiation. The reaction can be quick and extreme.

At this point in the experiment it was noted that a "dead" spot occurred in the middle of the front part of the oven. In this spot no particle of the so-called "reactive" synthetic aggregate could be heated red hot. No cause for this phenomenon could be determined.

Solvents used in the asphalt extraction processes (alcohol-methylene chloride and 1,1,1 Trichloroethane) are generally regarded as non-flashing. However reactive aggregates can produce temperatures as high as 2550 F (melting of stainless steel) which could result in unpredictable solvent decomposition (with toxic gas evolution) or combustion. This would be true especially in an atmosphere higher than normal in oxygen content.

Since 1,1,1 Trichloroethane is the solvent used in all State Department of Highways and Public Transportation field extractions, there was a need to test its reaction to microwave radiation. The aggregate samples were the same as those listed in Table IV and the procedure was identical to that given in Tex-210-F. Results indicated that there were no particular problems in drying aggregates that can be attributed to 1,1,1 Trichloroethane although a slightly longer time was involved compared with the alcoholmethylene chloride solvent.

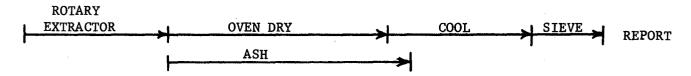
There is one feature of the vacuum extraction method that should be given serious consideration. Proper and adequate evacuation of vapors should be provided in the laboratory. Personnel sensitive to certain solvent and

decomposed solvent vapors can become seriously affected when performing this test. This cautionary statement also applies to the proper evacuation of fumes from the microwave oven when drying extracted aggregate. Adequate ventilation and evacuation of vapors is a prerequisite for all laboratory operations involving solvents or other volatile materials. Good laboratory practice and procedures are essential in providing safe working conditions for all concerned.

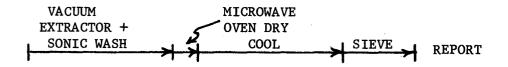
The last item listed in the Tables is "Total Time." This is a summation of the extraction process time and the drying time. None of the times includes sample preparation and weighing and other such activities. The "ash" determination for Test Method Tex-210-F was made during the drying time and therefore added no additional time to the "Total Time." "Ash" determination was extracurricular with regard to the vacuum extraction method and thus not included in any timed sequences.

Some conclusions can be made concerning the "test time" for these two methods. The overall FHWA method was shown to be faster than Test Method Tex-210-F, but contrary to published statements, the actual vacuum extraction procedure takes as much or more time to perform than the centrifuge extraction. Considerable time is consumed removing the extracted aggregate from the vacuum extractor and care must be taken to prevent tearing the filter. The "test time" advantage of the FHWA procedure results entirely from the use of the microwave oven as is illustrated in the following sketch.

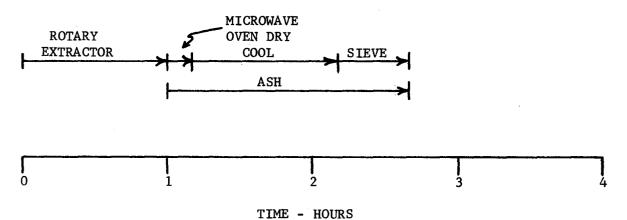
TEST METHOD, TEX-210-F



F.H.W.A. VACUUM EXTRACTOR PROCEDURE



ALTERNATE TEST METHOD ROTARY EXTRACTION + MICROWAVE DRYING



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Elimination of the ash test is not considered to be a factor in reducing "test time" since it can be accomplished concurrently with other test activities. Similar reduction in "test time" is possible with an alternate test method which combines rotary extraction with microwave drying as was shown in the sketch.

Advantages of the vacuum extractor procedure, as a result of eliminating the ash determination, are the savings in man-hours (not test time), the elimination of obnoxious smoke and vapors and the elimination of certain special equipment.

The March 1, 1975 Revision of Tex-210-F will also provide for reduced frequency of ash determinations when using the rotary extraction procedure. This can be done when tests show that an "ash correction" determined on an acceptable frequency gives accurate extraction results. Taking advantage of this provision with rotary extraction procedure should produce man-power savings approaching those provided by the vacuum extractor method. Consideration of a combination method using rotary extraction with microwave drying and reduction in frequency of ash determination may prove advantageous for some laboratories. Proper validation of this approach would be essential however.

This brief investigation indicates that when extracting mixtures consisting of aggregates included in the study, Test Method Tex-210-F and the Federal

Highway Administration's "Vacuum Method of Determining the Composition of Bituminous Aggregate Mixtures" are of equal precision and that under conditions previously discussed the vacuum method <u>can</u> shorten the overall extraction procedure time.

The following recommendations should be considered when use of the FHWA procedure is contemplated:

- 1. For each set of materials and mixes determine by tests similar to those described in this report:
 - a. Accuracy of this test method
 - b. Validity of eliminating ash determination
 - c. Satisfactory drying procedure
 - d. Reaction of aggregates to over-drying
- Establish adequate provision for protecting personnel from exposure to solvent fumes.
- 3. Exercise proper safety precautions in use of microwave oven including check procedures for leakage and posting of warning signs reading "CAUTION MICROWAVE OVEN IN THIS ROOM MAY INTERFERE WITH CARDIAC PACEMAKERS."
- 4. Exercise extreme care in attempts to use solvents for which no previous experience with microwave drying is available.
- 5. For more specific instructions and precautions on the use of the microwave oven, refer to the December 1975 report entitled, "Evaluation of a Microwave Oven in the Laboratory," 5-20-75-242.

TABLE I
Crushed Limestone

			TEX-210-F		VACUUM METHOD		D
o Tan	DESIGN	NO.1 (% BY WT.)	NO.2 (% BY WT.)	AVG (% BY WT.)	NO.1 (% BY WT)	NO.2 (% BY WT.)	AVG. (% BY WT.)
SIZE	(% BY WT.)	(% DI WI.)	(% DI WI.)	(% DI WI.)	(N DI WI)	(A DI WILL)	<u> </u>
Ret. 3/8"	O	0	0	0	0	0	0
3/8"- No.4	38.0	36.4	36.6	36.5	36.1	36.1	36.1
No.4 - No. 10	19.0	20.1	19.8	19.9	20.0	19.9	19.9
Ret. No. 10	57.0	56.5	56.4	56.4	56.1	56.0	56.0
No.10 - No. 200	35.3	34.8	35.2	35.0	34.9	34.9	34.9
Pass.No.200	2.7	3.5	3.2	3.4	3.8	4.0	3.9
Asphalt Content	5.0	5.2*	5.1*	5.2*	5.2**	5.1**	5.2**
Residual Bitumer	n:						
No "ash" cor	rection	5.4	5.3	5.4	**	**	**
Correction fo	or "ash"	*	*	*	5.1	5.1	5.1
Testing Time:	f asphalt	45 min.	45 min.	45 min.	51 min.	46 min.	49 min.
Aggregate Drying	- · · · · · · · · · · · · · · · · · · ·				!		
Conventional	oven	1hr.30min.	1hr.20min.	lhr.25min.	-	<u>.</u> .	-
Max. Temp. (°F)	200	200	200	-	-	-
Microwave Oven		-	<u>.</u>	-	9 min.	9 min.	9 min.
TOTAL TIME:		2hrs.15min.	2hrs.5min.	2hrs.10min	lhr.	55min.	58min.

TABLE II

Crushed Lightweight (Source A) and Limestone Fines

		TEX-210-F		VACUUM METHOD			
	DESIGN	NO.1	NO.2	AVG	NO.1	NO.2	AVG.
SIZE	(% BY WT.)	(% BY WT.)	(% BY WT.)	(% BY WT.)	(% BY WT)	(% BY WT.)	(% BY WT.)
Ret. 3/8"	0	0	0	0	0	0	0
3/8"- No.4	36.4	35.3	35.7	35.5	35.4	35.7	35.6
No.4 - No. 10	18.2	18.8	18.5	18.7	18.8	18.7	18.7
Ret. No. 10	54.6	54.1	54.2	54.2	54.2	54.4	54.3
No.10 - No. 200	33.9	33.8	33.7	33.7	33.5	33.5	33.5
Pass.No.200	2.5	3.5	3.5	3.5	3.8	3.5	3.6
Asphalt Content	9.0	8.6*	8.6*	8.6*	8.5**	8.6**	8.6**
Residual Bitume							
No "ash" cor	rection	8.8	8.9	8.9	**	**	**
Correction f	or "ash"	*	*	*	8.5	8.5	8.5
Testing Time: Extraction o	f asphalt	lhr.3min.	57 min.	lhr.	lhr.8min.	1hr.14min.	lhr.llmin.
Extraction o	Lasphaic	int.Juni.	37 min.				
Aggregate Dryin	g Time:			÷			·
Conventional	oven	2hr.13min.	1hr.20min.	lhr.47min.	_	. –	_
Max. Temp. (°F)	200 (oven	200	200	_	-	_
Microwave Oven		opened frequently)	-	-	12min.	12min.	12min.
TOTAL TIME:		3hrs.16min. (oven opened	2hrs.17min.	2hrs.47min	1hr.20min	lhr.26min.	lhr.23min.

TABLE III

Crushed Lightweight (Source B) and Limestone Fines

			TEX-210-F			VACUUM METHOD			
	DESIGN	NO. 1	NO.2	AVG	NO.1	NO.2	AVG.		
SIZE	(% BY WT.)	(% BY WT.)	(% BY WT.)	(% BY WT.)	(% BY WT)	(% BY WT.)	(% BY WT.)		
Ret. 3/8"	0	0	0	0	0	0	0		
3/8"- No.4	36.4	36.2	36.3	36.3	36.3	36.4	36.4		
No.4 - No. 10	18.2	18.1	18.0	18.0	17.9	17.9	17.9		
Ret. No. 10	54.6	54.3	54.3	54.3	54.2	54.3	54.3		
No.10 - No. 200	33.9	33.6	34.0	33.8	33.5	33.5	33.5		
Pass.No.200	2.5	3.3	3.0	3.1	3.5	3.4	3.4		
Asphalt Content	9.0	8.8*	8.7*	8.8*	8.8**	8.8**	8.8**		
Residual Bitume									
No "ash" cor	rection	8.9	8.7	8.8	**	**	**		
Correction fo	or "ash"	*	*	≯ c	8.8	8.7	8.8		
Testing Time: Extraction o		50min.	45min.	48min.	lhr.	'55min.	58min.		
Aggregate Dryin	-								
Conventional	oven	1hr.15min.	lhr.	lhr.7min.	-	-	-		
Max. Temp. (of)	200	200	200	-	-	- '		
Microwave Oven Max. Temp.(OF	")	<u>-</u>	- -	- -	9min. 425	9min. 450	9min. 438		
TOTAL TIME:		2hrs.5min.	1hr.45min.	1hr.55min.	1hr.9min.	1hr.4min.	1hr.7min.		
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TABLE IV

Crushed Lightweight (Source C) and Limestone Fines

			TEX-210-F			VACUUM METHO	
	DESIGN	NO.1	NO.2	AVG	NO.1	NO.2	AVG.
SIZE	(% BY WT.)	(% BY WT.)	(% BY WT.)	(% BY WT.)	(% BY WT)	(% BY WT.)	(% BY WT.)
Ret. 3/8"	0	o	0	0	0	0	0
3/8"- No.4	36.4	36.2	36.3	36.2	36.3	36.3	36.3
No.4 - No. 10	18.2	18.1	18.1	18.1	18.1	18.1	18.1
Ret. No. 10	54.6	54.3	54.4	54.3	54.4	54.4	54.4
No.10 - No. 200	33.9	33.2	33.0	33.1	32.9	33.1	33.0
Pass.No.200	2.5	3.3	3.3	3.3	3.7	3.5	3.6
Asphalt Content	9.0	9.2*	9.3*	9.3*	9.0**	9.0**	9.0**
Residual Bitume							
No "ash" cor	rection	9.3	9.4	9.4	**	**	**
Correction f	or "ash"	*	*	*	9.0	9.0	9.0
Testing Time:			·			,	
Extraction o	f asphalt	55min.	50min.	53min.	lhr.	45min.	53min.
Aggregate Dryin	g Time:						
Conventional	oven	1hr.35min.	1hr.15min.	1hr.25min.	-	· -	-
Max. Temp. (of)	200	200	200	-	-	-
Microwave Oven Max. Temp.(O	F)	- -	- -	- -	9min. 450+	6min.+? 450+	(See note below)
TOTAL TIME:		2hrs.30min	2hrs.5min.	2hrs.18min.	lhr.9min.	51min.+	(See note below)

NOTE: Bowl broke in oven during third 3 min. drying cycle.

APPENDIX

Texas Highway Department

Materials and Tests Division

DETERMINATION OF ASPHALT CONTENT OF BITUMINOUS MIXTURES

BY EXTRACTION

Scope

This method of test, which is a modification of A. S. T. M. Designation: D 2172, is intended for the determination, by cold solvent extraction, of the percentage of asphalt in the paving mixture. The percentage of asphalt is based on the weight of asphalt and aggregate mixture. The aggregate and fines recovered from this test can be used for the sieve analysis, Test Method Tex-200-F.

Apparatus

1. Extractor or Centrifuge (Figure 1) consisting of a bowl mounted in an assembly such that the bowl may be revolved at a controlled speed. The apparatus is provided with a container or outer shell for catching the solvent thrown from the bowl and a drain for collecting all of the solvent. The extractor should be provided with explosive-proof features and proper ventilation.

Other methods of determining asphalt content of bituminous mixtures which have proven accuracy may be used.

- 2. Filter rings to fit the rim of the bowl.
- 3. Scoop, spatula, and small brush.
- 4. Graduate, 2000 ml. capacity.
- 5. Pipette, 100 ml. capacity.
- 6. Silica evaporating dish, 200 ml. capacity.
- 7. Balance with at least 4500 gram capacity, sensitive to 0.1 gram.
- Analytical balance or a balance sensitive to 0.01 gram.
- 9. Drying oven, capable of attaining a temperature of 200°F . or more.
- Muffle furnace or burner and gas or electric hot plate.
- 11. Large flat pan, No. 2 tin can, beakers, etc.
- 12. Desiccator

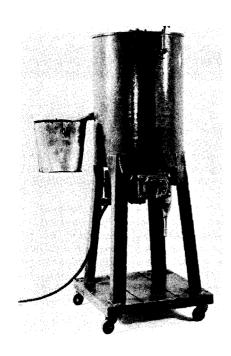


Figure 1

${\tt Materials}$

- 1. Solvent, Benzol, Trichloroethane
- 2. Ammonium Carbonate Solution saturated solution of A.C.S. Grade (NH₄)₂CO₃.

Test Record Forms

Use Work Card, Form No. D-9-F2, for recording results of Extraction Test.

Preparation of Sample

- 1. The bituminous mixtures received for the extraction test may be loose material or a small section of compacted pavement submitted from the roadway. Remove any base material and soil that might be adhering to the compacted asphaltic concrete.
- 2. If the asphaltic mixture is not workable and can not be separated and reduced to laboratory test size, place several thousand grams in a large, flat pan and warm in oven at about 230°F until it becomes workable. Use a trowel to separate the particles of the sample using care not to fracture the aggregate particles.

- 3. Mix, blend, and quarter the mixture continuously until two combined opposite quarters weigh a minimum of 1,000 grams, or, for mixtures not subject to segregation, thoroughly blend the material and take small portions from several places covering the entire area of the pan. Do not use a sample of less than 1000 grams regardless of the maximum size of aggregate, unless sufficient material is not available.
- 4. If the sample consists of hot mix-cold laid material or pre-coated aggregate or pavement which may contain moisture, dry the sample to constant weight at a temperature of min. 200F. If the sample consists of rock asphalt, dry to constant weight at a minimum temperature of 140°F. (See Notes: 3)
- 5. If the extraction is to be run on material before drying, select representative portions of hot mix-cold laid asphaltic concrete, precoated aggregate, or samples of compacted pavements and determine the moisture content according to Test Method Tex-212-F and the hydrocarbon volatile content as outlined in Test Method Tex-213-F.

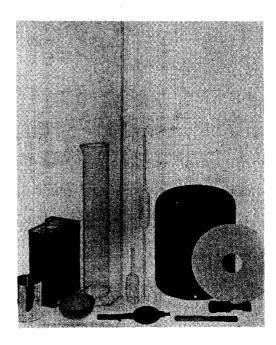


Figure 2.

Procedure

1. Obtain the weight of the laboratory size sample for extraction to the nearest estimated 0.1 gram and record this weight as A.

- 2. Transfer the weighed sample of asphaltic concrete into the extractor bowl, taking care not to lose any of the material, and cover the sample with solvent. If necessary, use a small amount of solvent and wash any material remaining on the balance pan into the bowl.
- 3. Weigh a clean, dry, paper filter ring and place on the extractor bowl. Place the top on the extractor bowl and screw on the funnel-clamp until tight. Place the bowl containing the sample and solvent into the machine, tighten the lock-nut, and secure the extractor lid. Place a container under the drain to catch the liquid extracted from the sample and start the machine revolving.
- 4. When the solvent ceases to flow, stop the motor and collect the discharged liquid in the 2000 ml. graduated cylinder, add approximately 300 ml. of solvent (a No. 2 can about 3/4 full) to the bowl through the funnel, and centrifuge. Collect the discharged liquid, pour back into bowl and centrifuge again. Add the discharged liquid to that already in the graduate. Continue this procedure centrifuging each addition of solvent several times until the extract is reasonably clear and not darker than a light straw color. Stop the motor every time before pouring solvent into funnel. Due to the nature of the extraction process, it is impossible to secure a perfectly clear extract without disintegrating some of the aggregate or using an excessive quantity of solvent.
- 5. Collect and measure the total amount of discharged liquid and record this volume (ml.) as (2).
- 6. Carefully remove the top and filter ring from extractor bowl and brush all clinging aggregate particles back into bowl. Dry the filter paper to constant weight. (See Notes: 3) From this figure, subtract the original weight of filter paper (Step 3) to obtain the weight of fine mineral matter (assumed to be particles passing the 200-mesh sieve) contained in it. Record the weight of the fine mineral aggregate as B.
- 7. Transfer the aggregate from the extraction bowl to a tared pan and dry to constant weight at a min. temperature 140° . If benzol is used as a solvent and the aggregate is to be dried in a gas fired oven, place the pan of material on top of the oven for about 30 minutes to allow fumes to evaporate before placing it in oven to dry completely. Weigh and record the net weight of aggregate as C.
- 8. Obtain the tare weight of a 200 ml. silica evaporating dish. Agitate the extracted liquid collected in the 2000 ml. graduate thoroughly and immediately withdraw a 100 ml. sample from the middle third of the liquid by means of a 100 ml. pipette and suction bulb. Pour the 100 ml. of extract into the tared silica

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dish and evaporate the solvent to dryness on a gas or electric hot plate under a hood or a well ventilated area. (Caution: Depending upon the solvent used, the evaporating fumes may be toxic and flammable.) Ignite the residue over a burner, or, if available, place dishin a muffle furnace for 45 minutes at approximately 1400 F. to ash the bituminous material. Whether burner or muffle furnace is used, continue the process (for an extended period, if necessary) until the ashing is complete. Complete ashing should be obvious from visual observation. Should any doubt arise, the completion of the ashing can be determined by continuing the process to a constant weight condition. Cool and add just enough ammonium carbonate (NH4)2CO3 to wet the ash remaining in the dish. Place the dish on a hot plate and evaporate to dryness at low temperature. Cool the dish and contents to room temperature in a desiccator, if available, and weigh. Record the net weight of the residue as (1). (Use of ammonium carbonate may be omitted, if prior tests do not indicate a need for the re-carbonation of the ash). (An Ash Factor may be used in lieu of ash determination. Frequency of ash determination to verify or change the Ash Factor can be determined by uniformity of plant production or change in materials from those of original design.)

Calculations

The percentage of asphalt in the mixture is calculated by following the indicated steps on Form D-9-F2 or Form D-6-546.

Where:

- A = Weight of total sample in grams
- B = Weight of fine aggregate retained in filter in grams
- C = Weight of oven-dry extracted aggregate in grams
- D = Total number of grams of ash contained in the total volume of solvent
- E = Fine aggregate in the filter plus the extracted aggregate plus the total ash, all in grams
- F = Total loss of sample in grams-residual bitumen plus the moisture and the hydrocarbon volatiles contained in the original sample.
- G = Total loss of sample in percent
- H = Moisture content in percent
- I = Hydrocarbon volatiles content in percent
- J = Residual bitumen or asphalt content in percent

Notes

- 1. If sample is dry when extracted, moisture and hydrocarbon volatiles = 0
- 2. The weight of fine material retained in the filter ring and the weight of ash in the total centrifuged liquid shall be added to the weight of material passing the No. 200 mesh sieve and used in calculating the sieve analysis.
- 3. Drying to a "constant weight" may be accomplished by drying for a specific period of time that has proven by experiment to be adequate or drying to the point that by observation, based on experience, the material is sufficiently dry for testing.

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TEXAS HIGHWAY DEPARTMENT ASPHALTIC CONCRETE EXTRACTION TEST

COUNT Y	H'IGHWAY	PROJECT	CONTROL	
- · -		STATION-		
SPEC. ITEM 350	TYPE "DD"	DESIGN NO. 2-A-72	·	
TOTAL SAMPLE		8_gm.(A) ASH PER 100ML	= e-f =	0.25 gm(1)
		gm.(a) TOTAL SOLVENT.		
ORIGINAL FILTER	=19.	4 gm.(b) TOTAL ASH	= <u> x 2</u> =	gm(D)
FINES IN FILTER	. =a-b=O.	2 gm(B) FINES+AGGR+ASI	H = B + C+D=	$\frac{1477.7}{\text{gm(E)}}$
PANT EXTR. AGGR	<u>=</u> 19 <i>5</i> 9.	4 gm(c) TOTAL LOSS	= A - E =	87.1 gm(F)
PAN	= 487	O gm (d) TOTAL LOSS	= <u>F</u> xI00=	5.6_%(g)
EXTR. AGGR,	=c-d= 1472	.4 gm.(c) MOISTURE CONTE	:NT=	
DISH + ASH PER 100 ml	=91	.77 gm.(e) HYDROCARBON VOL	ATILES CONTENT =	<u>0.1</u> %(1)
		. 52 gm (f) RESIDUAL BITUMEN		

	PER CENT
FINES + AGGR.+ ASH = E X 100=-	94.4
MOISTURE # H =	0.4
HYDROCARBON VOLATILES = =	0.1
RESIDUAL BITUMEN	* 1
TOTAL=	100.0 %

	SIEVE ANALYSIS					
Sieve Size	Grams	Per Cent				
Pase 1/2"	0.0	100.0				
Pass 3/8"	1534.8	98.1				
13/4 - 7/8						
7/8 -3/8						
5/8 — 3/8						
1/2 - 3/8						
$\frac{3}{10} - 4$	536.0	34.3				
1/4-10						
4-10	333.4	21.3				
+10	899.4	57.5				
10-40	175.4	11.2				
40-80	137.4	ප .8				
80-200	208.9	13.3				
Pass 200	56.6	3.6				
Total Loss	87. I	5.6				
Total	1564.8	100.0 %				