Iowa State Highway Commission
Materials Department
Ames Laboratory

Research Project R-238
Final Report

Effects of Aggregates Upon Asphalt Extraction Tests

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by

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TABLE OF CONTENTS

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1.0 INTRODUCTION

The verification of asphalt contents of asphaltic concrete paving mixtures is often done by the extraction test, which consists of using a suitable solvent to extract the asphalt while retaining the mineral aggregate. The validity of the test results is dependent upon several factors. One factor being the effect the aggregate characteristics might have on the extraction process, along with how time and temperature contribute to this effect. Another factor affecting extraction results is the variation of the asphalt content of different samples of the same mixture. If an understanding of the causes of the extraction variation can be gained, then recognition of these variations may allow valid adjustments to be made in the extraction procedure.

2.0 PURPOSE

The purpose of this investigation is to:

1. Study the effects of different aggregates, both limestones and gravels, in their influence on the amount of asphalt that is extractable from a mix.

2. Determine if time and temperature of the asphalt in contact with the aggregate has an influence on the amount of extractable asphalt.

3. Study the effect of surface area variations in relation to asphalt contents.

4. Determine if a correlation exists between extraction results and quality tests of the aggregates.

5. Identify as well as possible the causes for extraction variations.

3.0 MATERIALS

Thirteen sources of crushed aggregates were used in this study. Eleven of these being crushed limestones and two being crushed gravels. One sand was used with these aggregates to build the appropriate gradation.

Five sources of pit run gravels were used.

85-100 pen. asphalt (AB8-185) was used for all mixes.

Identification of mixes is shown in Table I.

4.0 LABORATORY PROCEDURE

1. Description of the extraction procedure. The apparatus is designed to accommodate a 1500 gr. sample.

The extraction procedure used is the reflux method using 1-1-1 trichloroethane as the solvent. The apparatus consists of a 4000 ml. glass beaker which is fitted with a condenser on the top. A stainless steel cylindrical basket, which holds the sample, is attached to the condenser. The bottom of the basket is a #100 mesh sieve held in place by a perforated metal plate. Attached to the bottom of the basket is a metal sump to catch most of the - #100 aggregate. After the refluxing has finished the solution of solvent and asphalt which contains part of the - #100 aggregate is allowed to stand and most of the - #100 aggregate settles to the bottom of the beaker. The solution which still contains a small amount of fines is decanted and an aliquot portion is filtered to determine the amount of fines in suspension. The fines retained in the sump and in the beaker are combined and washed until clean in a Buchner funnel, and then recombined with the coarse portion of the sample and dried. The fines that remain in suspension, which are usually less than 2 grams, are considered in the asphalt content calculation and are considered as - #200 aggregate in the sieve analysis which is then run on the extracted sample.

2. Description of the mixing process. The aggregates are dried and batched in the appropriate proportions. After heating of the aggregates prior to mixing they are placed in the mixing bowl of the Lancaster mixer and the appropriate amount of asphalt added. The mixes are made in 6000 gram batches. The Lancaster mixer consists of a rotating mixing bowl and a counter-rotating mixing paddle.

3. Preparation of aggregates for mixes. The coarse aggregates were separated in the laboratory from top size down to the number 8 sieve and each size was treated as individual portions for the design of composite gradation. All particles of the aggregates passing the number 8 sieve were treated as fine particles for design - the control of the gradation below the #8 sieve was limited to the result that could be obtained by changing proportions of the - #8 material as it naturally occurred in its fraction.

4. Design of gradation of individual mixes. The individual mixes were designed to coincide with the average gradation and composition of previous construction jobs. The mixes consist of the following types:

Two 1"	Type A (Limestone)
One 1"	Type A (Crushed gravel)
One 3/8"	Type A (Crushed gravel)
Two 3/8"	Type A (Limestone)
One 3/8"	Type A (Trap Rock)
Six 3/4"	Type B Class I (Limestone)
Five 3/4"	Type B Class II (Pit run gravel)

The mixes were designed to coincide with the following gradations:

Sieve No.	Percent Passing 1"	3/4"	1/2"	3/8"	4	8	30	50	100	200
Type A 1"	100	92	80	72	53	42	24	14	9.0	6.9
Type A 3/8"				100	75	59	29	17	11	8.4
Type B 3/4" Class I		100	89	82	63	51	28	--	--	8.0
Type B 3/4" Class II	Comply with gradation limits									

5. Design of asphalt contents. The asphalt contents for the various mixtures were determined by our standard method of mix design. (50 blow Marshall Compaction and Hveem Stability.)

6. Testing. Three batches, of 6000 grams each, were made on the 18 different mixtures. The batch designated as "A" was extracted the following day after mixing. The batch designated "B" was placed in an oven at 140°F. for 1 week and then extracted the day following removal from the oven. The batch designated as "C" was aged at room temperature for two weeks and then extracted.

Each batch was divided into four samples of approximately 1500 grams each and extracted and the gradation determined on each of the four samples. The samples were obtained by dumping the 6000 gram batch into a large flat pan, mixing and spreading the material over the bottom of the pan as evenly as possible. The samples were taken by scooping from the pan in the sequence shown in the following diagram so each sample represented two sections across the pan.

Sample 4
Sample 3
Sample 2
Sample 1
Sample 4
Sample 3
Sample 2
Sample 1

5.0 INTERPRETATION OF RESULTS

Evaluation of Test Results of the Different Methods of Treatment of the Coated Aggregates

No appreciable difference in asphalt extraction results could be discovered when comparing the results of the three methods of treatment: A - extraction the day following mixing; B - oven treatment @ 140°F. for 1 week; C - aging @ room temperature for 2 weeks. The average results for each of the separate methods of treatment for the thirteen higher type mixes was the same. The results of the five pit run gravel mixes varied slightly with the average extraction of the A series being 6.11%, the B series 6.07% and the C series 6.09%.

Statistical Analysis of the 18 Mixes

The extraction values of the three different treatment series were combined for this analysis as shown in Table II.

Asphalt Content of Extraction Samples Compared to Surface Area

With the use of the method of determining surface area of aggregates as outlined in the Asphalt Institute's handbook, "Mix Design Methods for Asphaltic Concrete", a good correlation was found between surface areas and asphalt contents. A statistical analysis could not be attempted because only four sample extractions could be compared in each analysis, the reason being, an additional variable in gradation comparisons was introduced when the batch size was limited to 6000 grams. The comparison, on the individual batches, of asphalt content and surface area showed that in 67% of the cases the sample with the high asphalt content also had the high surface area. This high correlation becomes more meaningful if it is noted that comparisons of four extractions were used in which the asphalt content varied from the high value to the low value by only 0.03% and the surface area by 0.6 of a square foot/lb. The average variation of the 54 comparisons, from the high value to the low value, for the asphalt content was .19% and for surface area 1.3 square feet/lb.

Mix #2 was remade and two samples taken; these two samples were individually separated into a coarse and fine fraction on the No. 4 sieve as well as could be done. Extractions and gradations were then performed separately on these resulting fractions. The data is shown in Table III and Fig. 1. It is suggested from this limited information that the film thicknesses coating the coarse and fine aggregate are approximately the same.

Comparison of Results of Higher Type Mixes with the Pit Run Gravel Mixes

The thirteen higher type mixes, upon extraction, all showed a higher asphalt content than the intended amount. If all the asphalt had been extracted, the possibility of a slightly higher result might be the normal expectation, after removal of the asphalt, because fines could be lost as dust and considered as asphalt in the weighings. The results averaged 0.05% high in asphalt content when compared with the intended amount. Mix No. 1 (Table II) which averaged .17% high in asphalt content was repeated with the same result and cannot be explained.

The pit run gravel mixes upon extraction all showed a lower asphalt content than the intended amount. These results averaged .16% lower than the intended amount. Fig. 2 shows the relation between the amount of shale in the + #16 material as plotted against the difference between the average extraction results and the intended asphalt content. The diagram indicates a high degree of correlation between shale content and extractable asphalt. For clarification we define extractable asphalt as that asphalt that goes into solution in the normal procedure of the extraction process.

Correlation of Quality of Aggregates with Extraction Results

There is no indication of any correlation existing between the results of the quality tests of the aggregates and the extraction results obtained. The quality results of the principle components, along with the type of aggregates and their locations are shown in Table IV.

6.0 CONCLUSIONS

This study showed quite clearly that time and temperature of asphalt in contact with aggregate had no influence on extraction results. It also showed that a correlation does not exist between extraction results and the quality of the aggregate as determined by the freeze and thaw test and the Los Angeles abrasion test. The extraction test results on limestone or higher type mixes indicated good accuracy, but with slightly high results. The extraction test results on the pit run gravel mixes showed less accuracy with low results, with the accuracy directly related to the amount of shale in the mixes. A high correlation was found to exist between extraction results and surface area, which indicates a considerable amount of the variation in asphalt contents of the same mixture must be attributed to sampling error.

It is recommended that a procedure be initiated by a laboratory control method, to determine the variation in extraction results due to the characteristics of the aggregate. With this information, accuracy of the test method would be improved. It would also be informative to consider surface area variations when evaluating asphalt content variations.

7.0 TABLES

TABLE I

Identification of Mixes

Mix No.	Size	Type	Principle Component
1	3/4"	B Class I	Limestone
2	3/4"	B Class I	Limestone
3	3/4"	B Class I	Limestone
4	1"	A	Limestone
5	3/4"	B Class I	Limestone
6	3/4"	B Class I	Limestone
7	3/4"	B Class I	Limestone
8	1"	A	Limestone
9	3/8"	A	Limestone
10	3/8"	A	Limestone
11	3/8"	A	Trap Rock
12	3/8"	A	Cr. Gravel
13	1"	A	Cr. Gravel
14	3/4"	B Class II	Pit Run Gravel
15	3/4"	B Class II	Pit Run Gravel
16	3/4"	B Class II	Pit Run Gravel
17	3/4"	B Class II	Pit Run Gravel
18	3/4"	B Class II	Pit Run Gravel

TABLE II

Statistical Analysis

Mix No.	No. of Samples	Intended Asphalt	Avg. of Extractions	Upper Value	Lower Value	Standard Deviation	Coefficient of Variation
1	12	4.00	4.17	4.33	4.01	.0993	2.3
2	12	5.00	5.04	5.29	4.83	.1172	2.3
3	12	5.50	5.52	5.64	5.43	.0585	1.0
4	12	4.25	4.26	4.34	4.22	.0396	0.9
5	12	4.50	4.58	4.76	4.41	.1248	2.7
6	12	4.00	4.01	4.12	3.79	.1116	2.7
7	12	4.00	4.00	4.31	3.85	.1188	2.9
8	12	4.25	4.26	4.53	4.11	.1184	2.7
9	12	5.00	5.08	5.21	4.98	.0625	1.2
10	12	5.00	5.05	5.22	4.95	.0748	1.4
11	11	5.00	5.05	5.14	4.98	.0524	1.0
12	12	4.75	4.84	4.95	4.77	.0553	1.1
13	11	4.00	4.03	4.16	3.96	.0715	1.7
14	12	6.50	6.34	6.50	6.15	.0971	1.5
15	12	6.00	5.75	5.92	5.63	.0901	1.5
16	12	6.00	5.86	5.99	5.72	.0835	1.4
17	12	6.00	5.86	6.05	5.74	.0900	1.5
18	12	6.75	6.63	6.84	6.34	.1390	2.0

TABLE III

Mix #2

Sieve No.	3/4	1/2	3/8	4	8	16	30	50	100	200
	Sieve Analysis % Passing									
Sample No. 1 (Fine)				100	84	68	50	28	18	12
Sample No. 1 (Coarse)	100	78	62	23	16	16	15	12	8.4	5.7
Sample No. 1 (Coarse and Fine Combined)	100	90	82	63	52	43	34	20	13	9.0
Sample No. 2 (Fine)				100	84	69	49	28	18	12
Sample No. 2 (Coarse)	100	76	63	26	17	16	15	12	8.7	6.1
Sample No. 2 (Coarse and Fine Combined)	100	88	82	64	52	44	33	21	14	9.3

	% Asphalt Content	Surface Area Sq.Ft./Lb.	Film Thickness in Microns
Sample No. 1 (Fine)	6.78	56.3	6.3
Sample No. 1 (Coarse)	2.99	24.0	6.2
Sample No. 1 (Coarse and Fine Combined)	5.03	42.1	
Sample No. 2 (Fine)	6.79	56.9	6.2
Sample No. 2 (Coarse)	3.11	25.4	6.2
Sample No. 2 (Coarse and Fine Combined)	5.05	42.8	

TABLE IV

Mix No.	F & T "A" Percent Loss	Los Angeles Abrasion Percent Loss	Principle Component Type of Aggregate	Location
1	2.0	24	Limestone	Conklin Quarry Johnson County
2	0.5	33	Limestone	Behr Quarry Clinton County
3	1.5	30	Limestone	Cotter Quarry Louisa County
4	1.7	27	Limestone	Early Chapel Quarry Madison County
5	12.0	27	Limestone	Greene, Iowa Butler County
6	16.0	28	Limestone	Floyd, Iowa Floyd County
7	6.3	26	Limestone	Anderson Quarry Jefferson County
8 *	2.3	26	Limestone	Crescent Quarry
8	4.2	25		Pottawattamie County
9	1.4	20	Limestone	Smith Quarry Benton County
10	4.4	--	Limestone	Crescent Quarry Pottawattamie County
11 **	0.4	--	Trap Rock	
11	3.9	32	Limestone	Ferguson Quarry Marshall County
12	- -	--	Cr. Gravel	Milford, Iowa Dickinson County
13 *	0.8	23	Cr. Gravel	Milford, Iowa
13	1.4	--		Dickinson County
14	7.1	22	Pit Run Gravel	Johnson Pit Polk County
15	6.9	28	Pit Run Gravel	Sac County
16	6.7	35	Pit Run Gravel	Hardin County
17	- -	--	Pit Run Gravel	Floyd County
18	4.7	--	Pit Run Gravel	Butler County

* Duplication of mix numbers indicates a coarse and fine sample of the same aggregate was used in the mix.

**Indicates principle component aggregates of different types.

Fig. 1

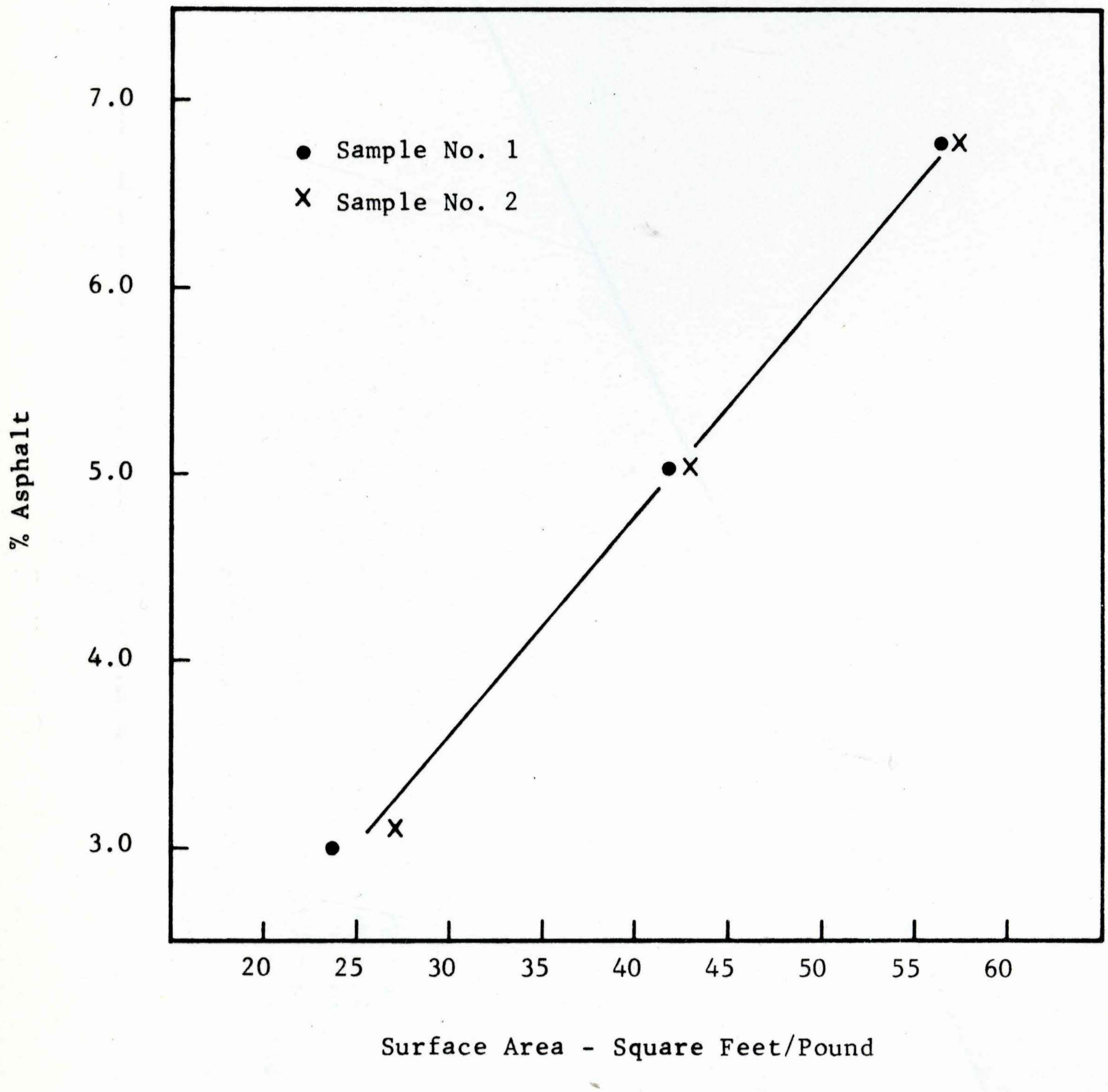
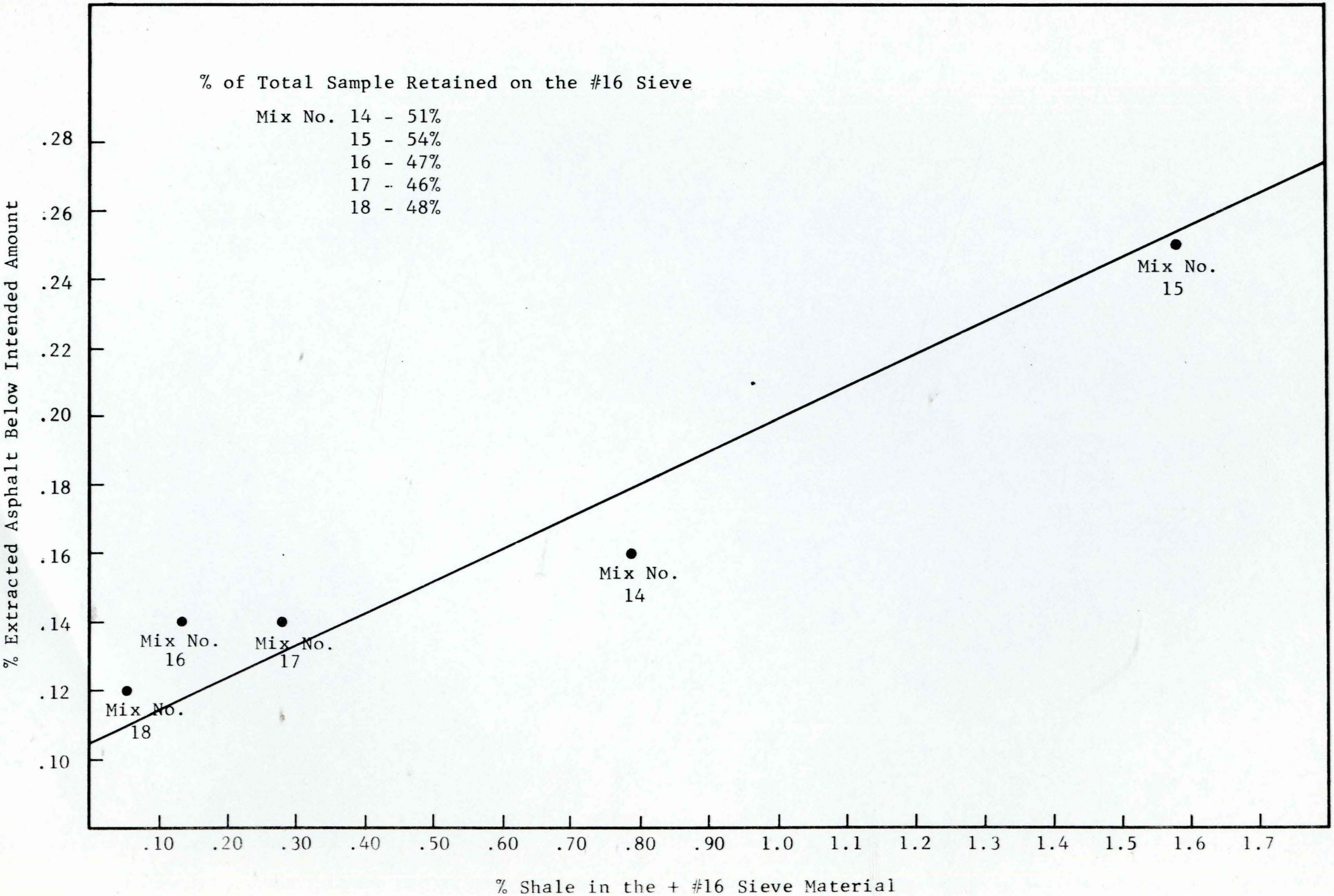

7.0
6.0
5.0
4.0
3.0
% Asphalt
Sample No. 1
Sample No. 2
20
25
30
35
40
45
50
55
60
Surface Area - Square Feet/Pound

Fig. 2



% of Total Sample Retained on the #16 Sieve
Mix No. 14 - 51%
15 - 54%
16 - 47%
17 - 46%
18 - 48%
% Extracted Asphalt Below Intended Amount
.28
.26
.24
.22
.20
.18
.16
.14
.12
.10
Mix No. 15
Mix No. 14
Mix No. 16
Mix No. 17
Mix No. 18
.10
.20
.30
.40
.50
.60
.70
.80
.90
1.0
1.1
1.2
1.3
1.4
1.5
1.6
1.7
% Shale in the + #16 Sieve Material