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16. ABSTRACT

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One of the first test procedures, including a measure of aggregate roughness and absorption, used for determining the asphalt content for bituminous mixes was the C.K.E. test method described by F.N. Hveem (1). This method is still in use in California.

Briefly, this procedure consists of saturating a representative fraction of the aggregate passing the #4 sieve with kerosene, then centrifuging for 2 minutes under a force equal to 400 times gravity. The coarse aggregate represented by the 3/8 inch x #4 is soaked in SAE 10 oil for 5 minutes, then drained for 15 minutes in a 140°F oven. From the amount of kerosene retained by the fines, and the amount of oil (SAE 10) retained by the coarse aggregate sample, the surface constants (Kf and Kc) are determined. Through a series of nomographs the surface constants for both the fine and coarse aggregates are converted into an oil ratio. In this test method no attempt is made to differentiate between the amount of kerosene or oil retained on the surface of the aggregate from that absorbed by the aggregate.

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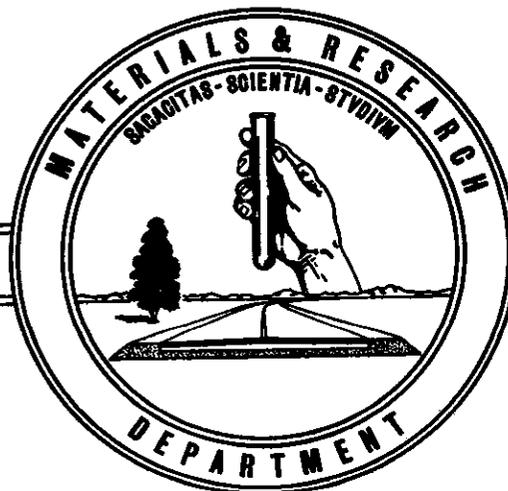


MODIFIED CENTRIFUGE KEROSENE EQUIVALENT TEST

By
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Introduction

In the early years of road construction the Resident Engineer had to be a Materials Engineer as well as a Construction Engineer. Usually he did not have laboratory reports to aid him in selecting the optimum asphalt content to use for a particular aggregate source. Most of the time he relied on his past experience and generally decided upon the percent of asphalt by visual appearance of the mix. This method for selecting an asphalt concrete mix design eventually gave way to a more scientific approach which included test procedures such as the Stain Test, proportioning by voids ratio and a number of formulas usually based upon the sieve analysis and surface area of the aggregate.

One of the first test procedures, including a measure of aggregate roughness and absorption, used for determining the asphalt content for bituminous mixes was the C.K.E. test method described by F. N. Hveem(1). This method is still in use in California.

Briefly, this procedure consists of saturating a representative fraction of the aggregate passing the #4 sieve with kerosene, then centrifuging for 2 minutes under a force equal to 400 times gravity. The coarse aggregate represented by the 3/8 inch x #4 is soaked in SAE 10 oil for 5 minutes, then drained for 15 minutes in a 140°F oven. From the amount of kerosene retained by the fines, and the amount of oil (SAE 10) retained by the coarse aggregate sample, the surface constants (Kf and Kc) are determined. Through a series of nomographs the surface constants for both the fine and coarse aggregates are converted into an oil ratio. In this test method no attempt is made to differentiate between the amount of kerosene or oil retained on the surface of the aggregate from that absorbed by the aggregate.

The C.K.E. test used for determining the oil ratio proved to be very successful, particularly when the aggregates were not crushed or angular. Beginning in the early 1950's, test data was presented which showed that blends of crushed and non-crushed aggregates provided a more stable mix than those containing only rounded aggregate. Since then the increased use of crushed aggregates in asphalt concrete mixes raised some questions about the reliability of the C.K.E. test results, particularly in regard to surface area. Since surface area constants are based on rounded aggregates, it was obvious, as shown in Figure 1, that the estimated surface area would be considerably less than the true surface area for crushed aggregate mixes. The erroneous surface areas, and the absence of a procedure for ascertaining absorption, led to a study to revise the C.K.E. test.

There were three objectives in the study to modify the C.K.E. test. The first was to find a method for measuring the surface

area of aggregate mixes without the use of surface area factors. The second objective was to develop a procedure for determining the absorptive properties of aggregate mixes. The third was to incorporate these new methods for determining surface area and absorption into one workable test procedure for predetermining the asphalt content for any given aggregate mix.

This paper will present the data which led to a new procedure for measuring separately surface area and absorption for aggregate mixes. This new procedure will be referred to as the Modified Centrifuge Kerosene Equivalent Test (Modified C.K.E.).

A statistical study is presented in this report on repeatability and reproducibility for surface area and absorption results as determined by the Modified C.K.E. test.

Surface Area Determination

Background

The surface area as determined by the Surface Area Method⁽²⁾ as well as the C.K.E. test is based upon a computed surface area from constants assigned to the various aggregate sizes. In computing the surface area the following assumptions are made:

- a. Specific gravity of the aggregate is 2.65.
- b. Particles are spherical in shape.
- c. Average diameter of each particle of the fraction is equal to the arithmetical mean of the sieve openings within this fraction.

Using the ASTM sieve sizes the approximate surface areas, or constants, are presented in the following table for the various sieves.

Table 1

Table of Surface Area Equivalents

Table 1 10 Sieves			Table 2 7 Sieves			Table 3 6 Sieves			Table 4 4 Sieves			Table 5 4 Sieves			Table 6 3 Sieves			
Sieve No.	Con- stant		Sieve No.	Con- stant		Sieve No.	Con- stant		Sieve No.	Con- stant		Sieve No.	Con- stant		Sieve No.	Con- stant		
Pass.	Ret.		Pass.	Ret.		Pass.	Ret.		Pass.	Ret.		Pass.	Ret.		Pass.	Ret.		
270		300				260	200		260	200		260	200		260	200		260
200	270	200	200		120													
100	200	120	100	200														
50	100	60				50	200	90	50	200	90							
30	50	30	30	100	46							30	200	72				
16	30	16				16	50	22										
8	16	8	8	30	12				8	50	17				8	200	45	
4	8	4	4	8	4	4	16	6				4	30	9				
3/8	4	2	3/8	4	2 1/2	3/8	4	2 1/2	3/4	8	2	3/4	4	2	3/4	8	2	
3/4	3/8	1	3/4	3/8	1 1/2	3/4	3/8	1 1/2	3/4	8	2	3/4	4	2	3/4	8	2	

As shown in the above table, the smaller the particle size the larger the surface area, or constant, per pound of material. Therefore, it is the smaller size particles which make up the major portion of the surface area for any given aggregate mix.

When using constants for two identical gradations, the computed surface area for both samples will also be the same, even though one sample may be from well rounded aggregates while the other sample may be from a rough textured angular aggregate source. Computed surface areas from assigned constants for the various sieve sizes were not accepted without opposition. For instance, an investigational study by Ziegs and Nuessel⁽³⁾ on the gradation of 36 fine powders showed conclusively that it is quite possible for various fillers to comprise completely different fractions of fines which are beyond reach of the normal sieves. The procedure used by Ziegs and Nuessel to separate these fines into their various fractions was the Gonells⁽⁴⁾ air separation method.

In the final analysis, many researchers had concurred that computed surface areas were at best, only an approximation.

Surface Area of Glass Beads

The first phase in developing a test procedure for directly measuring surface area was to find a material which was not absorptive, and also extremely uniform in shape. Glass beads met these qualifications.

It was found that 100 g of glass beads after being soaked in kerosene for 10 minutes then centrifuged for two minutes in 400 times gravity, retained 0.2 g. The calculated total surface area of the glass beads (100 g) was 3.75 square feet per pound. By varying the amount of beads, the surface area was also altered. After several points had been plotted, a surface area nomograph was made presenting the relationship between the amount of kerosene retained by glass beads and surface area.

From the original surface area data on glass beads, the nomograph as shown in Figure 2 was developed. This nomograph is used in the Modified C.K.E. test for determining surface area for aggregate mixes.

Gradation and Weight of Sample

After the surface area nomograph (Figure 2) was completed showing the amount of kerosene retained by glass beads is proportional to surface area, the question of gradation and weight of the aggregate sample was raised.

Since the interest was in finding the surface area of aggregate mixes, it was logical to use the grading for the sample that would eventually be used in construction.

The size of the regular centrifuge cups limited the weight of the composite aggregate mix sample to 100 g. It was realized that, by using a composite aggregate sample (ranging from 3/4 inch to dust), the possibility of error would increase when using this relatively small sample. It was also apparent that the technicians must use care in combining such a small sample to be representative of the required grading curve. Therefore it was felt that testing larger aggregate mix samples would result in more realistic measurements in reference to surface area. To accommodate a larger test sample, special centrifuge cups (Figure 3) were made. In subsequent tests using 200 and 300 g samples, test results indicated there was no significant difference in the amount of kerosene retained between the larger and regular 100 g samples when the results of the larger samples were reduced to the amount of kerosene retained per 100 g.

As shown in Table 2, the surface area results were more accurate when using the larger centrifuge cups, however, the difference in results were not significant to justify the added expense of replacing our normal centrifuge cups with the larger diameter cups.

Comparison of Computed Surface Area vs Surface Area as Determined by the Modified C.K.E. Test

The inaccuracies of using surface area factors for determining surface area of rough textured aggregate were apparent, however, it was felt that if a non-absorptive, well rounded, polished aggregate mix was used, there should be a rather close correlation between the surface area as determined by surface area factors and by the Modified C.K.E. method. To further control the surface area, the passing 200 material was limited to 3 percent, and no material passing the 270 sieve was used. The gradation and calculated surface area are presented in Table 3. As shown, the calculated surface area for this sample is 37.9 sq. ft. per pound. The surface area as determined by Figure 2 was 35 sq. ft. per lb.

The calculated surface area for a nonabsorptive extremely angular, coarse textured sample with the identical gradation as shown in Table 3 would obviously have the same calculated surface area as the well rounded sample, that being 37.9 sq. ft. per pound. Surface area result as determined by the Modified C.K.E. test was 70 sq. ft. per pound for the nonabsorptive coarse textured sample.

The above examples, as noted, were nonabsorptive specimens. However, when using the Modified C.K.E. test to determine the surface area on absorptive aggregate samples, some of which were rather smooth and rounded, it was found that the surface areas were extremely high, even higher than some all crushed nonabsorptive specimens. The first thought was that there must be some of the smaller particles, which appeared to be round that were actually rough in texture. Microscopic examination of the smaller aggregates did not substantiate this hypothesis. It was then thought that the minus 200 material was extremely fine, therefore increasing the surface area. However, when duplicate samples were prepared with the minus 270 material removed it was found the surface areas of the duplicate samples were slightly lower, but were still higher than some of the nonabsorptive crushed specimens having the same gradation.

The next thought was that the procedure requiring the aggregate samples to be placed in a kerosene bath for 10 minutes prior to centrifuging was excessive.

A survey of available literature revealed that Donaldson, Loomis and Krchma⁽⁵⁾ had presented data showing a rapid increase in absorption occurs during the first few minutes of the kerosene soaking period for alumina aero-gel beads, and a slight increase in absorption after 10 minutes.

Data from other researchers, as well, clearly showed that the soaking period of 10 minutes was excessive for absorptive samples and that in addition to measuring the amount of kerosene retained on the surface the amount of kerosene absorbed was also being included in the test result.

Tests indicated that if absorptive aggregates were placed in the centrifuge cup and 20 ML of kerosene quickly poured over the sample, it was possible to have the electric powered centrifuge in operation within two seconds after introducing the kerosene. From this finding the following procedure was developed.

"Two Point" Method for S.A. Determination

Several 105 g aggregate samples from the same source and identical grading were prepared. These samples were placed in a 230°F oven until reaching a constant weight. They were then taken from the oven and cooled to room temperature. The weight was accurately adjusted to 100 g. One sample was placed in the centrifuge and 20 ML of kerosene was quickly poured over the sample, the centrifuge was in operation within two seconds. The next sample was allowed to soak for a brief period of four seconds then centrifuged. This procedure was continued varying the soaking times. Two typical absorption rate curves are presented in Figure 4,

for absorptive and nonabsorptive samples. Each point is an average of two tests. As shown, the rate of absorption for the sample is quite slow during the first few seconds, however, from 10 seconds to 60 seconds the rate of absorption increases. After two hours a slight increase in absorption was noted. For the nonabsorptive sample no significant increase in the amount of kerosene retained was noted.

From this data, it was obvious that if a line was plotted between the two and four second points and projected until it intersected the ordinate represented by "0" seconds, the result would be an accurate means of determining the surface area. This method for measuring the surface area was successfully used for over a year, for both absorptive, and nonabsorptive aggregate samples. However, the time required for establishing two points was considered excessive particularly when the results from one sample, providing the centrifuge was in operation within 5 seconds, were not significantly different from the surface area results obtained by the "Two-Point" Method.

Absorption

In addition to surface area, absorption is also an important factor that must be considered in determining the proper amount of asphalt to uniformly coat the aggregate in an asphalt concrete mix. Washborn⁽⁶⁾ and Peek and McLean⁽⁷⁾ have shown that the rate a liquid travels through a single capillary is a function of the size of that capillary, the surface tension of the liquid being used, the wetting properties of the liquid, the viscosity and density of the liquid. As Nevitt and Krchma⁽⁸⁾ pointed out, the process of absorption in aggregates is even more complex. It involves not only the pore sizes, their various shapes and lengths, but also the fact that asphalt is an eminently complex substance, which further complicates the mechanism of absorption.

It has also been reported by Nevitt and Krchma,⁽⁸⁾ that absorptive aggregates may reduce the asphaltic film thickness by 25 percent through absorption after mixing and fabricating (Figure 5). The present C.K.E. test does not include any test procedure for measuring the amount of absorption per se. For instance, the minus 4 material, or fines, is placed in a centrifuge cup and then placed in a kerosene bath. The sample is permitted to remain in the bath until the sample is saturated, this generally required from 3 to 10 minutes; after soaking period has been completed, the sample is centrifuged for 2 minutes at 400 times gravity. Upon completing the centrifuging operation, the sample is reweighed and the amount of kerosene retained is used for computing a surface constant (Kf) for the fine material.

For the coarse aggregate, a sample is taken from the material which passes the 3/8 size and retained on the 4 sieve; this 100 g sample is then submerged in an oil (SAE #10) bath for 5 minutes, then placed in a 140°F oven and permitted to drain for 15 minutes. After draining, the sample is reweighed and the amount of oil retained is used in computing the surface constant (Kc) for the coarse aggregates. As shown, both the coarse and the fine material are required to soak for a given period of time permitting the liquids (oil and kerosene) to be absorbed. Consequently, the amount of oil retained by the coarse, and the amount of kerosene retained by the fine material are actually equal to the liquid retained on the surface, as well as the liquid absorbed and not equal to the absorption alone.

As shown by Donaldson, et al,⁽⁴⁾ when aggregate specimens are exposed to kerosene, absorption begins immediately and generally continues for approximately ten minutes. These researchers also show that no significant amount of kerosene absorption occurs after 30 minutes.

Many test procedures have been published for measuring the amount of absorption by aggregates, one of the first was presented by Goshorn and Williams.⁽⁹⁾ Briefly, their method is to determine the volume of asphalt used by each fraction as representative in the preliminary mix design. The volume of the aggregate is also determined. Then by coating a known volume of aggregate with a known volume of asphalt, the volume of the AC mix, if no absorption occurs, would equal the volume of the asphalt plus the volume of the aggregate. However, if the combined volume is less than the separate volumes, then the difference is the amount of the asphalt that is absorbed.

The opponents to this test procedure pointed out the test required too much time, and also the procedure for removing the entrapped air, when determining the volume of the AC mix, was not adequate. The Rice Method⁽¹⁰⁾ for determining the percent absorption is somewhat similar to the Goshorn and Williams procedure. However, the Rice Method does not require as much time to complete, and also provides a vacuum procedure for removing the entrapped air from the AC test specimen.

The main objection to the above-mentioned test methods is that no provisions are made to determine the additional amount of asphalt absorption that will continue after more than a few hours. In other words, these methods determine that amount of asphalt which has been absorbed in approximately 1 or 2 hours. As pointed out by investigators, it is important to know the amount of asphalt that is absorbed shortly after mixing and fabricating; however, it is more significant to know the maximum amount of asphalt

that will be absorbed. As shown by Nevitt and Krchma, asphalt absorption at room temperature continues for 6 weeks; however, absorption may occur up to 6 months.

Brown(11) presented a unique approach to determining the absorptive properties of aggregates. In his study, instead of using asphalt as the test liquid for determining the amount of absorption, he used kerosene, which would be more readily absorbed than asphalt. Brown's procedure for measuring absorption is: Two 1000 g aggregate samples are used, both having identical gradations. One sample is a known non-absorptive specimen, while the other is unknown. Both samples are placed in separate sacks and soaked in kerosene for 30 minutes, followed by wringing and whirling out the excess kerosene. The difference between the soaked and dry weights of the unknown sample, minus the weight of the retained kerosene by the known non-absorptive sample, is equal to the amount of kerosene absorbed which can be calculated as a percentage of the aggregate weight.

Brown's procedure was modified by Donaldson, et al.(4) The size of the sample was reduced from 1000 to 100 g, and a centrifuge replaced the hand wringing and whirling operation. The size of the centrifuge cups, and the force used to remove the excess kerosene from the samples are the same as used in the C.K.E. test.

In both of the above-mentioned tests, the procedures consist of measuring the additional kerosene retained by an unknown as compared to a known nonabsorptive blank of the same gradation. Obtaining an "identical" gradation between two aggregate specimens from different sources is practically impossible, as was shown by Ziegs and Nuessel,(2) consequently, comparing the amount of kerosene retained between unlike specimens could be extremely misleading.

Determining Percent Absorption by the Modified C.K.E. Test

In the surface area determination study it was determined that absorption can be held to a minimum providing the elapsed time from adding kerosene to the sample and starting the centrifuge operation does not exceed 5 seconds.

It was also determined, and substantiated by other researchers, that only a slight increase in kerosene is absorbed by aggregates after soaking in a kerosene bath for 10 minutes. It seemed logical that if the amount of kerosene retained on the surface was subtracted from the total kerosene capacity for any given aggregate mix, the result would be equal to the amount of kerosene absorbed.

The method developed was to take the same aggregate sample used in determining the surface area and soak it for 10 minutes in a kerosene bath. The sample is then centrifuged for 2 minutes at 400 times gravity. The sample is then reweighed. The difference in weight after the first centrifuging (surface area determination) and the second centrifuging (total kerosene capacity) is equal to absorption.

Absorptive Limits for Aggregates

The percent absorption as obtained by the Modified C.K.E. Test has been extremely valuable as a research tool in evaluating many aggregate sources throughout California. In several cases where aggregate sources have been found to have an absorption value greater than 0.6 percent, the pavements constructed from these sources have shown early signs of failure. This was reported in a study by Zube and Cechetini (12).

The problem of setting absorption parameters for aggregate sources is extremely difficult and one of the reasons is the pore size distribution of an aggregate. For example, two aggregate samples from different sources have the same surface area and absorption (0.6 percent). The asphalt content for both sources, as determined by the Modified C.K.E. test is 6.5 percent. After mixing, and during compaction of test specimens one sample flushed asphalt at 6.5 percent, while the other sample showed no evidence of containing an excess of asphalt. The probable reason one aggregate mix could accommodate 6.5 percent asphalt while the other aggregate mix could not is due to the different pore sizes distribution of the two aggregate mixes.

As stated by Lee, (13) the pore size distribution has a direct effect on the degree of absorption. Lee also showed that there was a linear relationship between pore-size greater than 0.5 micron and asphalt absorption, however, this relation was not found in pores less than 0.1 micron.

Generally, absorptive aggregate sources can be used in the construction of asphalt concrete pavements with success providing the asphalt content as determined by the Modified C.K.E. test can be accommodated without flushing. However, on some projects the recommended asphalt content was lowered when flushing occurred during compaction of test specimens at the asphalt content demanded by the Modified C.K.E. test. In these cases, we have had some failures, generally raveling or transverse cracking due to expansion.

The example mentioned above is the type of problem which makes it difficult to set an absorption limit. Of course, a limit could be set at 0.7 percent which would probably reject a few acceptable

aggregate sources. Another possibility would be to accept all absorptive aggregate sources providing they could accommodate the asphalt content as recommended by the Modified C.K.E. test without flushing.

A program has been initiated which tentatively sets a maximum absorption limit of 0.7 percent. Each of the California Division of Highway's 11 district laboratories will evaluate the aggregate sources in their district. From the data received from all the laboratories involved in this program a realistic maximum absorption limit will be obtained.

Modified Centrifuge Kerosene Equivalent Test Procedure

The Modified Centrifuge Kerosene Equivalent Test (Modified C.K.E.) is used for measuring the surface area and absorption of aggregate mixes. This revised method also provides a procedure for predetermining the asphalt content for aggregates proposed for use in bituminous mixes.

The complete procedure is presented in Appendix "A". Also included are the two monographs used to determine the surface area, and the percent of asphalt.

Briefly, the procedure consists of a 100 g of composite material ranging from coarse to fine and representative of the designed gradation, with 3/4 inch aggregate being the maximum size. The 100 g sample is placed in a tared centrifuge cup and 20 ML of kerosene is quickly poured over the sample. Within 5 seconds after the kerosene is introduced, the centrifuge must be in operation at 400 times gravity. The sample is spun for two minutes then weighed. The amount of kerosene retained is proportional to its surface area. The same sample is then placed in a kerosene bath with additional kerosene poured on the top, the sample remains in the kerosene bath for 10 minutes. After the 10 minute soaking period, the sample is again centrifuged for two minutes at 400 times gravity, then again weighed. The weight of the retained kerosene is equivalent to the total liquid capacity. Therefore, the total liquid capacity, minus the kerosene retained by the surface area of the sample is equal to absorption. Knowing the surface area, the type of asphalt that will be used, and the percent absorption, one can easily convert this information into an asphalt content recommended for this particular aggregate mix.

When comparing the asphalt content as determined by the C.K.E. and Modified C.K.E. test, it is generally found that there is a close correlation between the two test results particularly when the aggregates are nonabsorptive or slightly absorptive. However, when the absorption values are above 0.5 percent the

asphalt content results from the two test procedures deviates considerably, with the Modified C.K.E. test results being higher. This is shown in Table 4. In Table 4, it is shown that as the absorption value increases above 0.5 percent, the amount of asphalt as determined by the Modified C.K.E. test is generally higher than the asphalt content as obtained by the C.K.E. test. The reason for this difference can generally be attributed to absorption. For instance, if the percent of absorption is subtracted from the percent of asphalt recommended by the Modified C.K.E. test, the remaining asphalt percentage would coincide with that amount recommended by the C.K.E. test.

The bituminous engineers who are now using the Modified C.K.E. test have stated the absorption value has aided them in making asphalt content recommendations for aggregate mixes particularly when absorptive aggregates are used.

Reproducibility and Repeatability

A statistical analysis was made on surface area and absorption as determined by the Modified C.K.E. test. The three aggregate sources chosen for this analysis varied in surface area as well as in the degree of absorption.

From each of the three sources sufficient material was obtained to complete this phase of the study. All of the test samples conformed to our 3/4 inch maximum (medium) grading requirements.

After the samples had been prepared, the 105 g samples were evenly divided between two laboratory technicians with each technician receiving 27 samples from each of the three aggregate sources for a sum total of 81 samples.

Each technician ran nine samples from each aggregate source, or 27 samples the first day. This was repeated for three days.

The model equation used for analysis of samples from each source was:

$$X_{ijk} = \mu + O_i + R_j(i) + E_k(ij)$$

Where: X_{ijk} = an observation
 O_i = A randomly selected operator
 $R_j(i)$ = A run of 9 tests which were performed at a time selected by the operator
 $E_k(ij)$ = testing error

Since all effects were considered to be random, the following expected mean squares were developed from the model equation:

Source	Freedom (D.F.)	Mean Square (EMS)
O_i	1	$V(E)+9 \cdot V(R)+27 \cdot V(O)$
$R_j(i)$	4	$V(E)+9 \cdot V(R)$
$E_{k(ij)}$	48	$V(E)$

From this, the following estimates were obtained as follows:

VARIANCE

Test reproducibility = $V(E)$

Intraoperator repeatability = $V(E)+V(R)$ (same technician, different days)

Interoperator repeatability = $V(E)+V(R)+V(O)$ (different technicians, and different days)

95% confidence limits were then obtained by multiplying the square root of the variance (σ) by 1.96. The resultant values are given below:

	Grand Mean	95% Confidence Limits & Std. Deviation					
		Reproducibility		Intraoperator Repeatability		Interoperator Repeatability	
		\pm	σ	\pm	σ	\pm	σ
Surface Area							
R-4320	56.778	+5.95	3.04	+9.69	4.95	+9.69	4.95
70-1681	66.259	+5.79	2.95	+6.79	3.45	+7.27	3.71
70-1559	52.333	+3.70	1.89	+3.82	1.95	+4.24	2.16
Absorption							
R-4320	0.850	+ .1255	.064	+ .1348	.069	+ .1544	.079
70-1681	0.333	+ .1067	.054	+ .1145	.058	+ .1157	.059
70-1559	0.163	+ .1016	.052	+ .1016	.052	+ .1044	.053

Summary and Conclusions

Surface Area

1) The factors as used in the C.K.E. test for ascertaining the surface area are reliable for round aggregates. However, when using these factors for computing the surface area of crushed aggregates, the results can be 100 percent lower than the true surface area.

2) Surface area for aggregate mixes was found to be proportional to the amount of kerosene retained after being centrifuged for two minutes at 400 times gravity. This is true providing the centrifuge is in operation within 5 seconds after pouring the kerosene over the aggregate sample.

Absorption

- 1) The Modified C.K.E. test presents a method for measuring absorption by aggregate mixes.
- 2) The Modified C.K.E. test can be used as an aid in making asphalt content recommendations.
- 3) Setting absorption parameters for any aggregate mix is difficult. Two absorptive aggregate mixes (0.6 absorption) from unlike sources may have a different rate for absorbing asphalt. One aggregate mix may absorb the 0.6 percent during the mixing period, while the other sample may require a longer period of time. Usually the recommended asphalt content for slow absorbing mixes is lower than results from the Modified C.K.E. test.
- 4) A tentative maximum absorption limit has been set at 0.7 percent. However, the 11 district laboratories throughout California will evaluate their own aggregate sources. When all the data from the district laboratories are analyzed a maximum absorption limit will be specified.

Modified C.K.E. Test

1. The Modified C.K.E. test measures separately surface area and absorption.
2. The Modified C.K.E. test has been used for several years as a research tool for determining the asphalt content for absorptive aggregate mixes.
3. The asphalt contents as obtained from the C.K.E. and Modified C.K.E. Tests are generally agreement for nonabsorptive mixes.
4. The time required for running the Modified C.K.E. test is much less than required for the C.K.E. test.

Reproducibility and Repeatability

Surface Area

1. The reproducibility for one technician performing 27 tests (9 samples from each group) in one day with a 95 percent confidence

limit ranges from ± 3.7 to approximately ± 6.0 sq. ft. per lb., with the greatest variance being for the sample with the highest absorptive value. This indicates that the technician may have been slightly erratic in getting the centrifuge in operation within 5 seconds.

2. The ± 3.7 sq. ft. per lb. variance for the nonabsorptive sample would appear to be the error that can be expected due to the actual surface area difference between like samples. Most of this error can be contributed to preparation of test samples.

3. When the same technician (intraoperator) tests identical samples on different days surface area results vary from approximately ± 4 to ± 10 sq. ft. per lb. with the greatest variance again being the sample with the highest absorption value approximately the same variation is noted when comparing results from two technicians (interoperator) for identical samples.

4. There are two probable causes for the variation in surface area results between technicians, those being: difference in gradings when samples were prepared and speed in getting the centrifuge in operation after introducing the kerosene, this is extremely critical particularly for absorptive aggregate mixes.

Absorption

1. The 95 percent confidence limit for reproducibility, ranges from approximately ± 0.1 to ± 0.13 percent, with the greatest variance again being for the absorptive samples.

2. The repeatability varied for the same technicians testing identical samples on different days (intraoperator) from ± 0.10 to ± 0.13 percent. The repeatability between two technicians testing identical samples on different days varied from ± 0.10 to ± 0.15 percent with the sample having the highest absorption also having the greatest variance.

3. It is felt that the results from this statistical study for determining the reproducibility and repeatability makes the Modified C.K.E. test an acceptable test method for determining the surface area, absorption qualities of aggregate mixes.

LITERATURE CITED

1. F. N. Hveem, "The Centrifuge Kerosene As Used in Establishing the Oil Content for Dense Graded Bituminous Mixtures", Association of Asphalt Paving Technologists, Vol. 13, p. 9, 1942.
2. T. E. Stanton, "Role of the Laboratory in the Preliminary Investigation and Control of Materials for Low Cost of Bituminous Pavements", HRB, Dec. 1934.
3. Dr. C. Ziegs, and Dr. H. Nuessel, "Grading and Surface Area of Mineral Fillers", Bitumen (Germany), N7, 8, 9, 1932.
4. Gonell, "The Determination of Fineness of Cement", 1928, S/786 FF.
5. J. A. Donaldson, R. J. Loomis, and L. C. Krchma, "The Measurement of Aggregate Absorption", Association of Asphalt Paving Technologists, Vol. 16, p. 358, 1947.
6. Washburn, Phys. Rev. 17, 273, 1921.
7. Peek and McLean, Ind. Eng. Chem. Anal. Ed. 6, 85, 1934.
8. H. G. Nevitt and L. C. Krchma, "Absorption of Liquid Bituminous Cements by Aggregates" Association of Asphalt Paving Technologists, Vol. 13, 1942.
9. J. H. Goshorn, and F. M. Williams, "Absorption of Bituminous Materials by Aggregates", Association of Asphalt Paving Technologists, Vol. 13, 1942.
10. J. M. Rice, "Asphalt Lost by Absorption into Aggregate Particles", Mix Design Methods for Asphalt Concrete (MS-2), 1962.
11. Brown, Socony-Vacuum Oil Company Technical Bulletin No. 21.
12. E. Zube and J. Cechetini, "Expansion Contraction of Asphalt Concrete", HRB #104, 1965.
13. Dah-Yinn Lee, "The Relationship Between Physical and Chemical Properties of Aggregates and Their Asphalt Absorption", Association of Paving Technologists, Vol. 38, 1969.

Table 2

Test	3 in. I.D. Cups 300g Sample		2-1/16 in. I.D. Cups 100g Sample	
	Kero.Ret. (g)	Surface Area (Sq.Ft./Lb.)	Kero.Ret. (g)	Surface Area (Sq.Ft./Lb.)
	Per/100g	Per/100g		
1	1.9	41	2.1*	45
2	2.4	51	2.6	55
3	1.6	35	1.5	33
4	2.2	47	2.3	49
5	1.5	33	1.5	33
6	2.0	43	2.2	47
7	1.6	35	1.6	35
8	1.5	33	1.3	29
9	1.4	31	1.3	29

*All above results are the average of three tests.

Table 3

<u>Screen Size</u>	<u>% Passing</u>		<u>S.A. Factor</u>	
3	100%	x		2.0
4	87	x	2	1.7
8	73	x	4	2.9
16	57	x	8	4.6
30	42	x	14	5.9
50	28	x	30	8.4
100	16	x	60	9.6
200	3	x	160	<u>4.8</u>

37.9 surface area
sq.ft.per lb.

Table 4

Test No.	Grade Asphalt	C.K.E. S.A.*	Mod. C.K.E. S.A.*	C.K.E. AB(%)**	% Asphalt		% Asphalt Rec. Range
					C.K.E.	Mod. C.K.E.	
1	SC-800	17.9	23	0.0	3.2	3.2	3.2 - 3.5
	120-150	"	"	"	4.0	3.8	3.5 - 3.8
	85-100	"	"	"	4.4	4.1	3.6 - 3.9
	60-70	"	"	"	4.8	4.3	3.8 - 4.1
2	SC-800	26.3	54	0.1	4.4	5.0	4.1 - 5.4
	120-150	"	"	"	5.4	5.4	5.3 - 5.6
	85-100	"	"	"	5.7	5.6	5.3 - 5.6
	60-70	"	"	"	5.8	5.7	5.5 - 5.8
3	SC-800	22.0	33	0.2	4.2	4.1	4.1 - 4.4
	120-150	"	"	"	4.7	4.5	4.3 - 4.6
	85-100	"	"	"	5.2	4.8	4.5 - 4.8
	60-70	"	"	"	5.5	5.0	4.6 - 4.9
4	SC-800	30.9	42	0.2	4.5	4.6	4.5 - 4.8
	120-150	"	"	"	5.5	4.9	4.9 - 5.2
	85-100	"	"	"	5.8	5.2	4.9 - 5.2
	60-70	"	"	"	6.0	5.3	5.0 - 5.3
5	SC-800	22.6	33	0.3	3.9	4.2	4.3 - 4.6
	120-150	"	"	"	5.0	4.7	4.5 - 4.8
	85-100	"	"	"	5.2	4.9	4.5 - 4.8
	60-70	"	"	"	5.4	5.1	4.7 - 5.0
6	SC-800	26.1	39	0.3	4.2	4.5	4.5 - 4.8
	120-150	"	"	"	5.1	4.9	4.7 - 5.0
	85-100	"	"	"	5.4	5.2	4.9 - 5.2
	60-70	"	"	"	5.6	5.4	5.2 - 5.5
7	SC-800	27.6	56	0.6	5.1	5.8	5.6 - 5.9
	120-150	"	"	"	5.7	6.2	5.9 - 6.2
	85-100	"	"	"	6.0	6.4	6.2 - 6.5
	60-70	"	"	"	6.2	6.6	6.2 - 6.5
8	SC-800	28.3	68	0.7	5.2	6.6	6.3 - 6.6
	120-150	"	"	"	5.9	6.8	6.3 - 6.6
	85-100	"	"	"	6.1	7.0	6.3 - 6.6
	60-70	"	"	"	6.4	7.1	6.5 - 6.8

*S.A. = Surface Area

**AB = Absorption

PARTICLE SURFACE TEXTURES

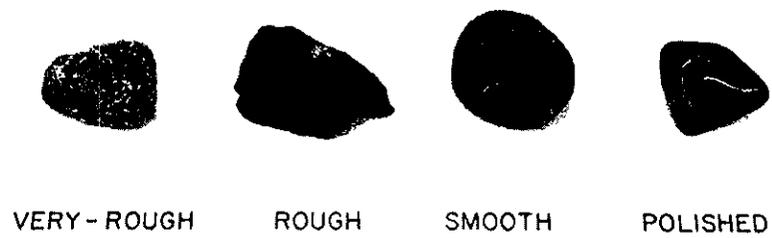


Figure 1

The Surface Area for the Above $3/4 \times 1/2$ in.
Aggregates are Totally Different Due to Their Surface Texture.

SURFACE AREA CHART

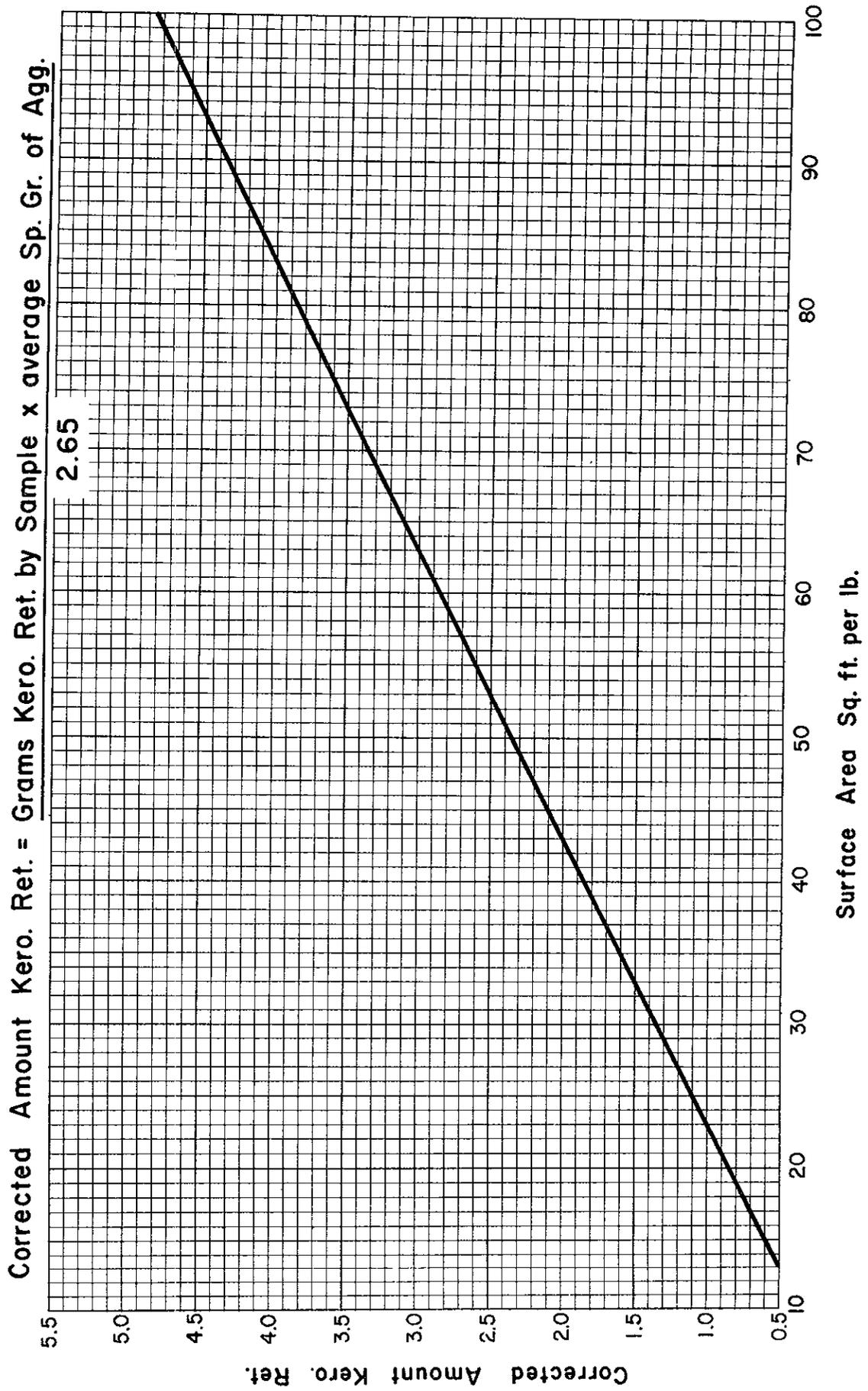


Figure 2

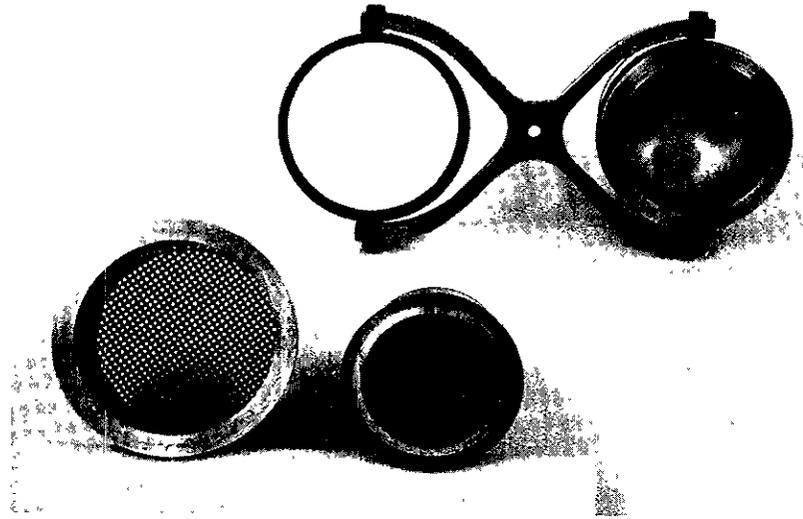
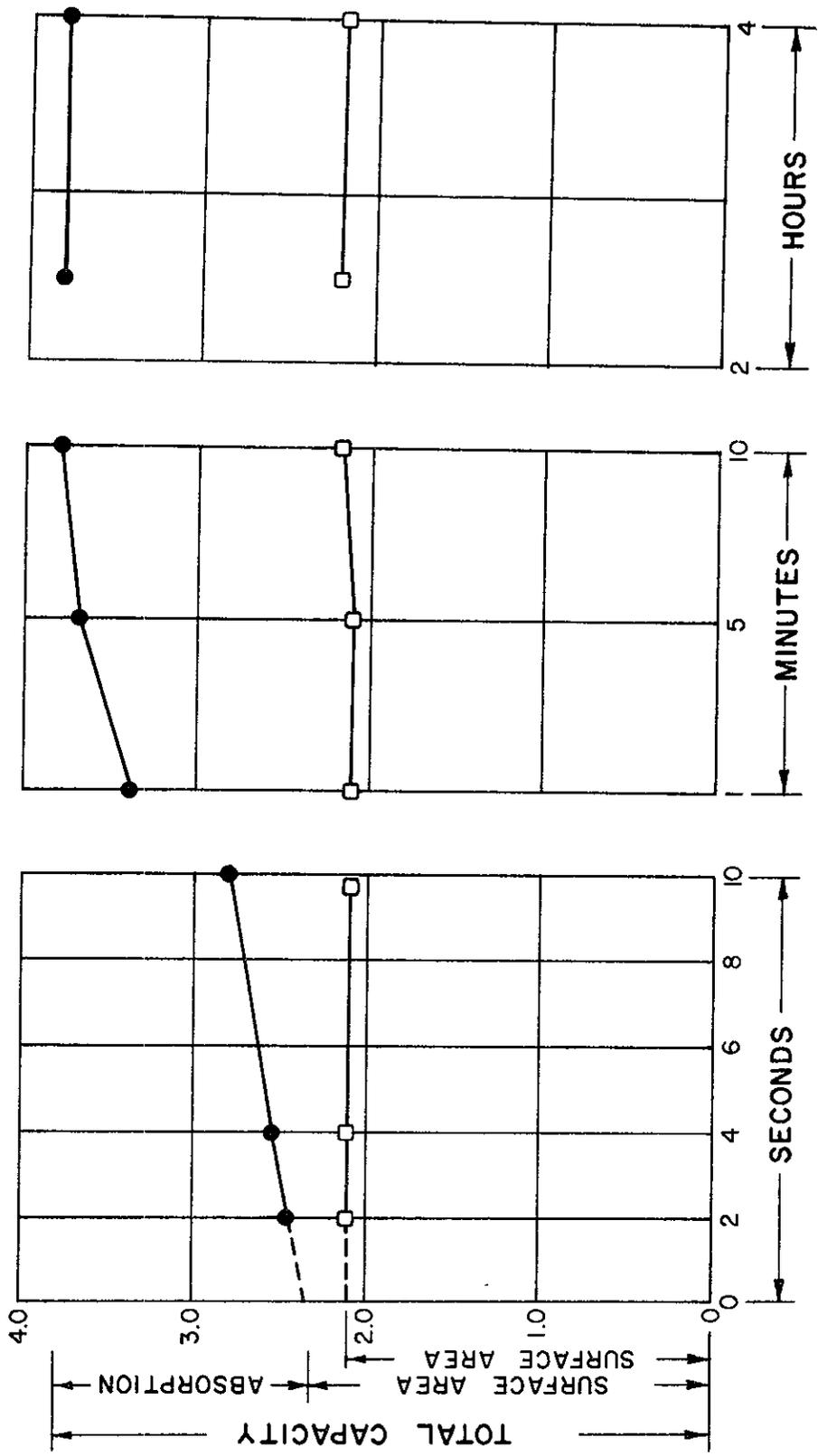


Figure 3

Diameter of Larger Centrifuge Cup is 3.0 Inches
While the Normal Centrifuge Cup on Right Foreground has a
Diameter of 2.75 Inches.



□ - NON - ABSORBITIVE AGGREGATE MIX
 ● - ABSORBITIVE AGGREGATE MIX

Figure 4

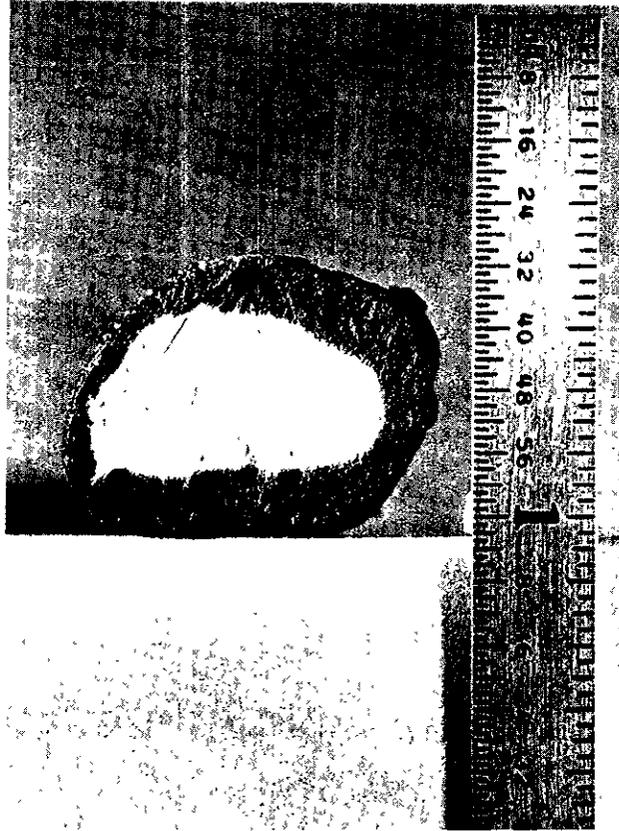


Figure 5

As Reported, Absorptive Aggregates Can Reduce the Asphaltic Film Thickness By 25 Percent.

APPENDIX A

Method of Test for Determination of Surface Area and Absorption of Aggregates Used in Bituminous Mixtures.

SCOPE

The test furnishes a measure of the surface area and absorption of aggregate gradations used in bituminous mixtures.

PROCEDURE

A. Apparatus

1. Centrifuge (power driven) capable of exerting a force of 400 times gravity (400G) on a 100 gm sample (see Figures 1-A and 2-A).

$$\text{Required rpm of centrifuge head} = \frac{\sqrt{14,000,000}}{r}$$

Where r = radius in inches to center of gravity of sample.

2. Centrifuge cups, 2-13/16 in. in height, and 2-1/16 in. inside diameter, complete with perforated brass plate .031 in. thick, with a minimum of 100 holes, 0.62 in. diameter, per square inch (see Figures 3-A and 4-A).
3. Torsion balance, 500 gm capacity \pm 0.1 gm accuracy.
4. Timer with sweep second hand.
5. Hot plate or 230 \pm 9 F oven.
6. Round tin pans, 4-1/2 in. diameter, 1 in. deep.

B. Materials

1. Kerosene
2. Filter paper, Eaton-Dikeman Co. size 5-1/2 cm., No. 613.

C. Test Record Forms

1. Work card, Form T-302, for recording test data.

D. Tests and Calculations

1. Nomenclature

- (a) S.A. = Surface area of aggregate particles.

Surface area of aggregate particles (S.A.) is determined by the weight of kerosene retained on the superficial surface of a 100 gram composite test sample after being wetted with approximately 20 ml. of kerosene for not more than 5 sec., and then centrifuged for 2 min. at 400G (see Figure 5-A).

- (b) Ab = absorption (%). The total amount of kerosene retained minus the amount required to wet the superficial surface.

After the weight of kerosene for surface area determination has been obtained, the centrifuge cup containing the specimen is placed in a pan containing kerosene to the depth of approximately 1/2" and an additional 35 ml. of kerosene is then poured over the sample.

The sample is allowed to stand in the kerosene bath for 10 minutes after which it is again centrifuged for 2 min. at 400G and weighed. The weight after second centrifuging minus weight after first centrifuging equals absorption (Ab).

2. Procedure for determining surface area (S.A.)

- (a) Batch in a small pan exactly 100 gms of the required individual sizes to furnish the desired grading. (Maximum size should not exceed 3/4"). Then add approximately 5 grams of fine material (passing No. 4 material) to compensate for moisture.
- (b) Place in 230 ± 9 F oven and dry the sample to constant weight. Allow to cool to room temperature.
- (c) Place sample in tarred centrifuge cup containing filter paper and adjust weight of material to exactly 100 gms. (Remove only passing No. 4 material when adjusting weight.)
- (d) Place cup in centrifuge and quickly pour 20 ± 2 ml. of kerosene over sample.
- (e) Centrifuge immediately for 2 min. at 400 x gravity. (Centrifuge must be in operation within 5 seconds after adding kerosene.)
- (f) Weigh sample to nearest 0.1 gram and record amount of kerosene retained. Correct for specific gravity.

Corrected kerosene retained =

$$\frac{\text{Weight retained} \times \text{Average Specification Gravity of Aggregate}}{2.65}$$

$$\text{Average specific gravity} = \frac{100}{\frac{\% \text{ Coarse}}{\text{Sp.Gr. Coarse}} + \frac{\% \text{ Fines}}{\text{Sp.Gr. Fines}}}$$

This formula is applicable to any percentage by weight of coarse or fine aggregates, either crushed or uncrushed or a combination of both as long as the summation of the percentages totals 100.

- (g) Use the surface area chart (Figure 5-A) to determine surface area (S.A.).

3. Procedure for Determining Absorption (Ab).

- (a) After step (f) under Surface Area, the centrifuge cup containing the test sample is placed in a small pan containing 1/2" of kerosene, and approximately 35 ml. of kerosene is poured over the sample. Allow sample to remain in bath for 10 minutes.
- (b) Remove sample from bath and centrifuge 2 min. at 400 x gravity.
- (c) Weigh and record amount of kerosene retained and make corrections for specific gravity as shown in paragraph 2f.
- (d) Subtract the value obtained in paragraph 2f from the value obtained in 3c and record the result as absorption (Ab).

4. Establishing Approximate Asphalt Content.

- (a) Using surface area determined from Figure 5-A proceed across chart (Figure 6-A) to grade of asphalt being used. Hold point and proceed vertically downward to diagonal line representing absorption. Hold point and proceed horizontally to bitumen ratio. The bitumen ratio determined by this method has been found to be generally within $\pm 0.5\%$ of the recommended asphalt content as determined by Calif. Test Method 304.

Example:

After the first soaking and centrifuging cycle the amount of kerosene retained after correction for

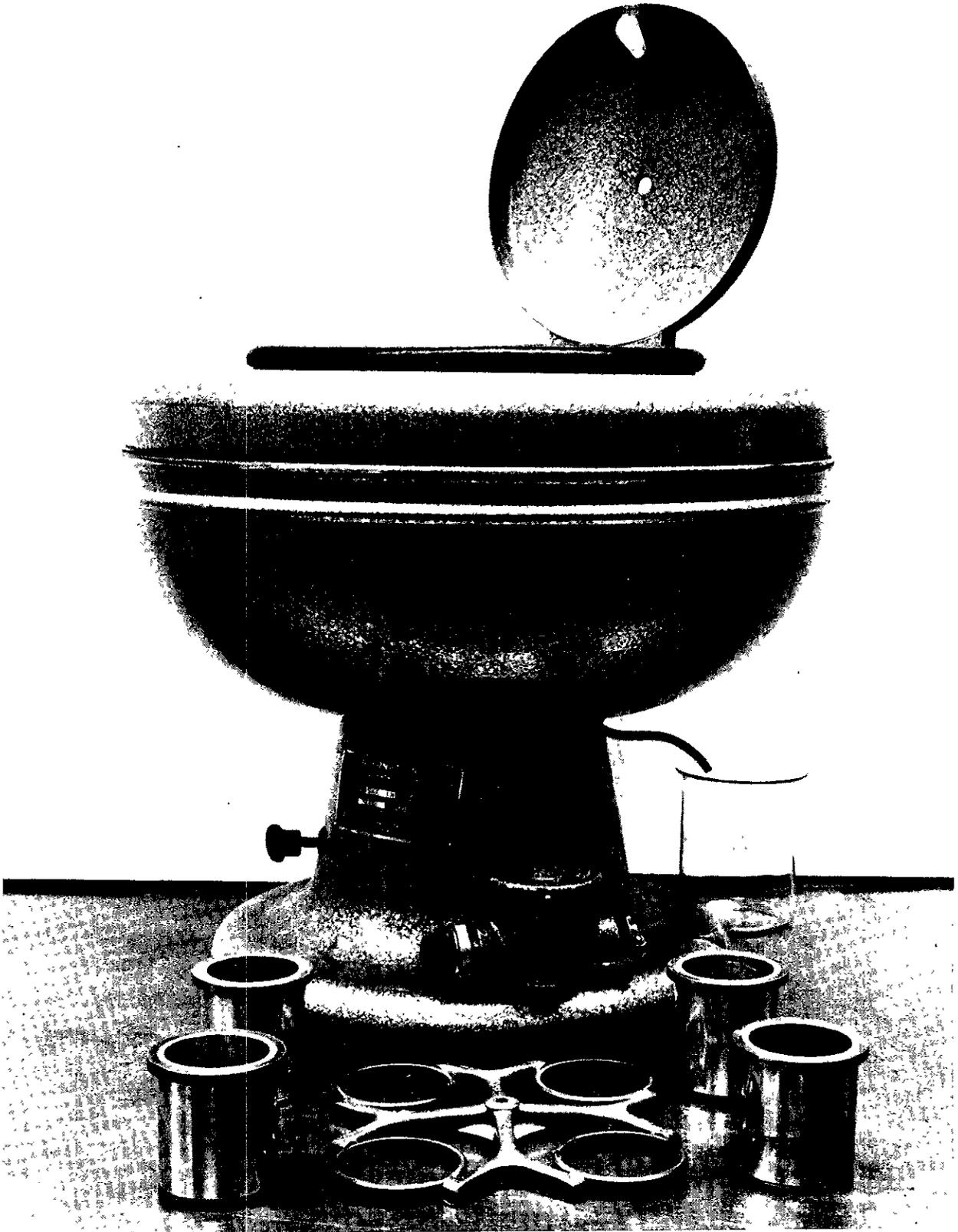


Figure 1-A

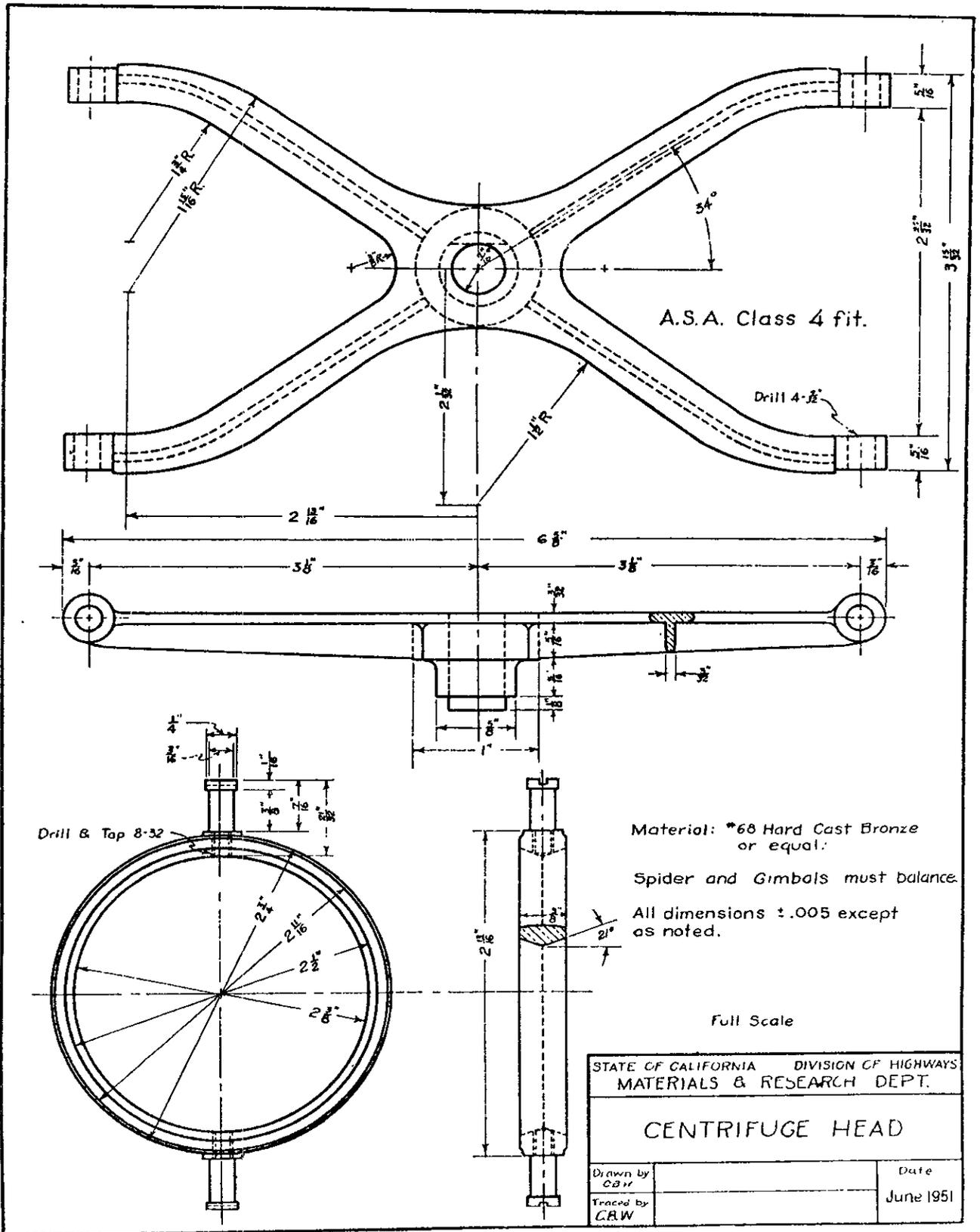


FIGURE 2-A

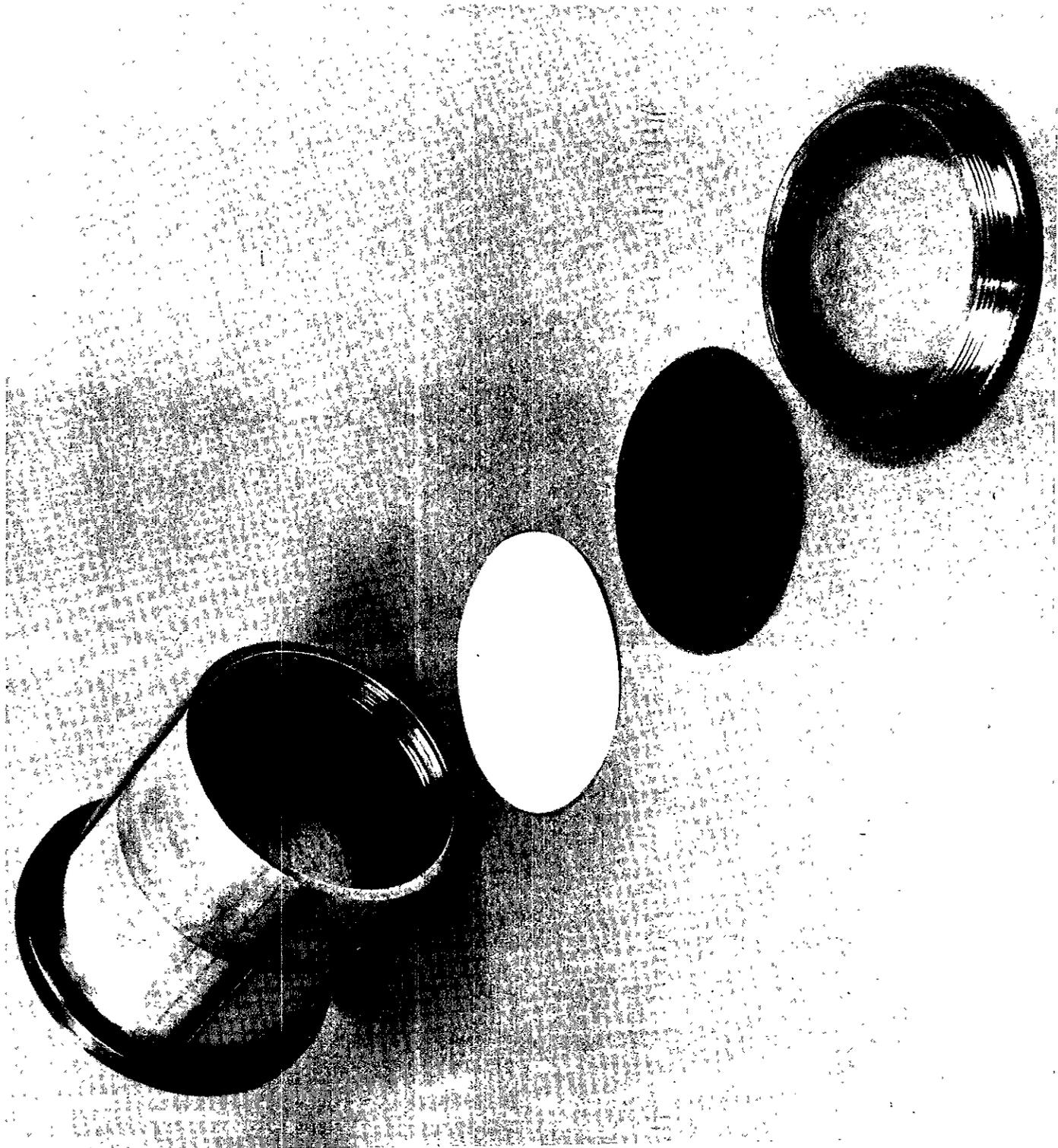


Figure 3-A

SURFACE AREA CHART

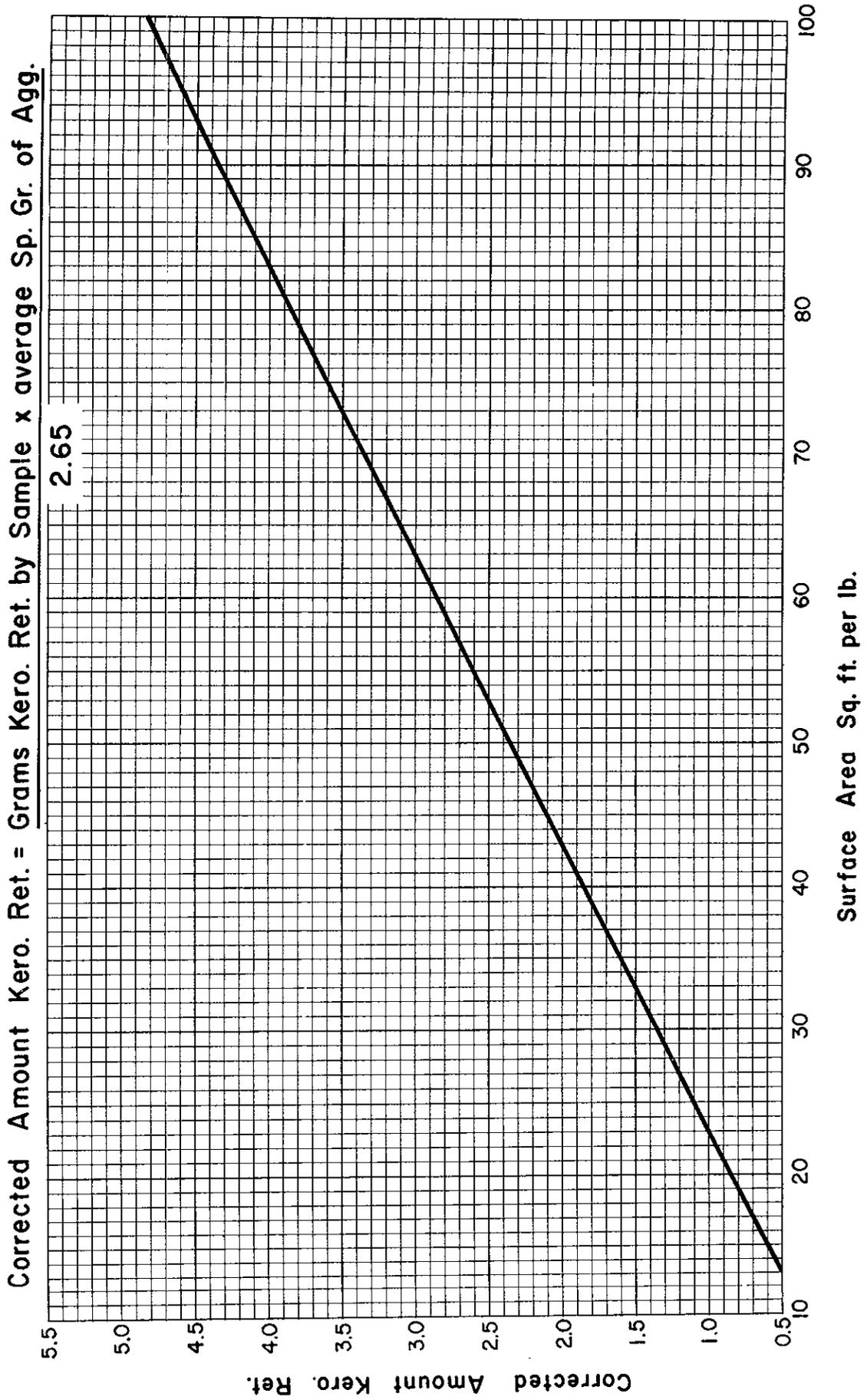
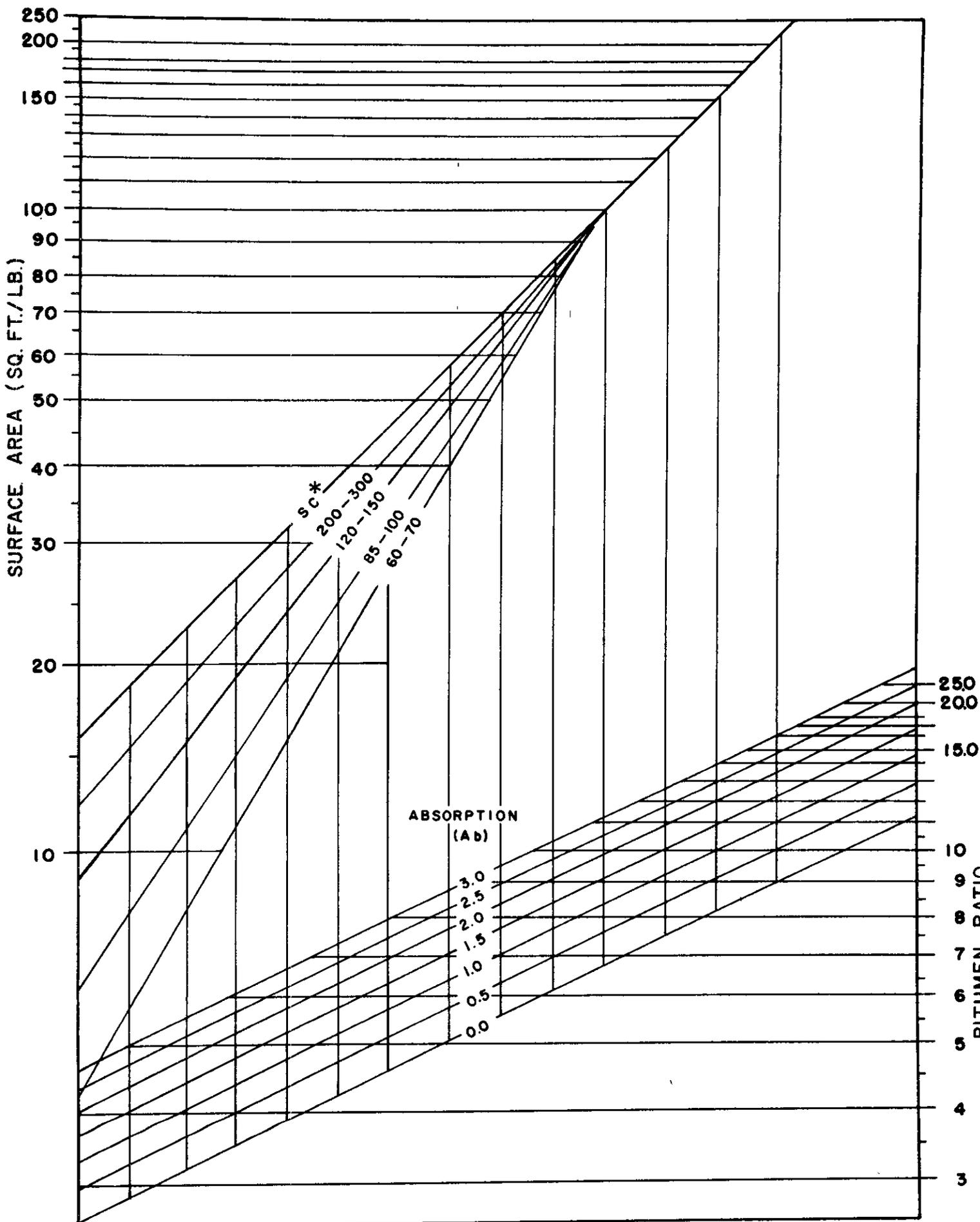


Figure 5-A



*INCLUDES ALL GRADES OF LIQUID ASPHALT

Figure 6-A