Improved Methods of Estimating Stripped Area in Asphalt Stripping Tests

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The work described in this paper was directed toward developing a better method than visual estimation for determining the extent of stripping. No attempt was made to improve on the sample preparation techniques specified in the various state highway procedures. Measurement of the extent of stripping was approach in two ways: (1) indirect and (2) direct measurement of area. In the indirect methods, some factor other than area, that is related to either the coated or uncoated area, is measured. Indirect measurement methods generally have greater possibilities for being conducted as routine tests. The early work on indirect measurement methods, such as dye-adsorption techniques, showed that a direct measurement method would be required as a research tool to evaluate any other methods that might be developed. The ordinary visual estimation method is not sufficiently reliable for this purpose. Consequently, emphasis was shifted to the development of a direct measurement test as the first step toward the ultimate goal of a routine test that might be accepted by the state highway departments.

● THE development of a satisfactory direct measurement method to be used as a research tool for the development of more routine tests has been accomplished. However, some additional work is required to prove its accuracy conclusively and to determine what simplifications can be made to reduce the time required for a measurement. In this direct method, an apparatus is used which permits visual examination of the stripped sample systematically, and allows the operator of the instrument to sum up the total stripped area which he observes. The apparatus consists of a motor-driven stage to move the sample relative to the operator's eye, a low-power microscope with cross-hairs to fix and pinpoint the operator's view, and an electrical counting circuit to aid the operator in recording the relative areas of coated and uncoated aggregate viewed. The components of this experimental apparatus cost approximately $300; the low-power microscope represents $200 of the cost. Recent work showed that a less refined optical system could be used at a much lower cost; the total cost with the simpler system would be about $125.

Results with this method, the mechanical integration method, were compared with unaided visual estimates on samples of limestone and quartz aggregate, coated with an RC-2 cut-back asphalt using various curing times and with different concentrations of additive[1] added. The mechanical integration method gave greater reproducibility between operators than did visual estimation. Measurements on the same sample by three operators, only one of whom was experienced in the use of the apparatus, resulted in values ranging from 4.9 to 5.7 percent stripping. Visual examination of another sample by five operators, two of whom were experienced in visual estimation, resulted in values of from 9 to 35 percent; 9 percent was probably closer to the true value. Mechanical integration did not, however, show any significant improvement in precision over that obtained when visual estimates were made

[1] A heat-stable additive marketed by the Carlisle Chemical Works, Incorporated.

only by a highly experienced operator. The results also showed that the precision of the mechanical integration method is better than the reproducibility of replicate samples. This fact indicates that any really significant improvement in stripping tests will require improvement in the entire stripping test procedure.

Limited study was made of an automatic optical method. In this method the sample is illuminated with polarized light so that a photocell viewing the sample through a Polaroid filter will receive only light that is diffusely reflected from the uncoated aggregate. The Polaroid filter system eliminates nearly all of the light that normally would be reflected from the asphalt surface. Preliminary work has shown that differences in the extent of stripping can be detected. Additional work is required to design an apparatus that will have satisfactory sensitivity and reproducibility.

Two indirect chemical methods were studied: (1) precoating aggregate with a dye or other coating and measuring the amount dissolved from the bare aggregate areas, and (2) direct leaching of the bare aggregate area with a dilute acid. Precoating the aggregate with fluorescein dye was not satisfactory when wet aggregate was used since the asphalt displaced the dye from the surface of the aggregate during the coating operation. The use of a zinc oxide precoating on the aggregate eliminated this displacement by asphalt but analysis for zinc after stripping proved to be expensive and time consuming. When tests were conducted on dry aggregate, the fluorescein dye precoating method showed promise. Although the stripping values calculated from fluorescein tests were much higher than visual estimates or values obtained by mechanical integration on the same sample, it is believed that the data obtained from the fluorescein method may provide useful indications of incipient stripping.

Although tests on the aggregate leaching method showed some promise in preliminary work, the stripping is accelerated by the leaching solution. This additional stripping is a serious problem which was not solved.

EXPERIMENTAL WORK

The over-all stripping test procedure, except for the final step of measuring area stripped, generally was the same as that employed in Bureau of Roads test specifications.[2] Washed aggregate, screened to minus ¼ plus ⅜-inch mesh, is mixed with 6 percent asphalt (based on dry aggregate) in this procedure. The Battelle tests varied from those of the Bureau of Roads in the following respects: larger aggregate samples were used (500 grams instead of 95 grams), and the sample was spread out in a layer in a Pyrex glass tray instead of being placed as a lump in a beaker during the stripping phase of the test. Mixing was generally done by hand; in a few tests mixing was done by rotating sealed cans containing the aggregate and asphalt on revolving rolls. Curing of cut-back asphalts was generally done at room temperature, and stripping was carried out with neutral water at room temperature. Where wet aggregate was used, a specific degree of wetness was achieved by draining excess water from the aggregate sample for a set time interval; uniform wetness was achieved by agitating while draining.

Shell 85/100 asphalt (Sewaren, New Jersey plant) and Shell RC-2 cut-back (midcontinent crude) were used in the studies. Ohio river gravel (quartz) and Marble Cliff limestone were the aggregates used. Concentrations of additive employed ranged from 0.1 to 2.5 percent based on the asphalt.

Direct Measurement of Stripped Area

The early work done on indirect measurement methods (fluorescein dye and aggregate leaching, described later in this paper) showed that the usual visual estimation of stripping is not satisfactory as a standard of comparison for other tests being developed. Accordingly, emphasis was placed on development of a direct measurement method which could still utilize the human eye if properly aided or which could utilize photocell measurements. The major portion of the work on direct measurement methods was on devising an apparatus that would aid and systematize the operator's viewing of the sample.

Although the sensitivity of the human eye is good in differentiating between areas of uncoated aggregate and highlights reflected from the asphalt, the ability of the human mind to compare the relative areas of coated and uncoated aggregate is not particularly good. Ac-

[2] Bureau of Public Roads private communication.

cordingly, emphasis was placed on aiding the operator to make this comparison. Basically what is required is some sort of scanning and area-summing system.

A similar problem is faced by metallographers in estimating the relative quantities of the various phases in an alloy when observed under a microscope. A technique has been developed for this case that involves superimposing a grid on photomicrographs and determining alloy composition by counting the number of grid intersections that lie over the different metal phases; the relative counts for each phase then represent the volume percentage of those phases in the alloy. Initial studies of the application of this technique to measurement of the stripped area in aggregate-asphalt mixes were encouraging. Although it was necessary to superimpose the grid on the test sample rather than on a photograph, and the time required for a single determination was excessively long, reproducible results were obtained. On the basis of these results, a mechanical device was constructed to simplify the scanning and counting operations.

Mechanical Integration Methods. This mechanical device consisted basically of a motor-driven stage for moving the sample relative to the operator's eye, a low-power (both 7× and 3× were used) microscope with cross-hairs to fix and pinpoint the operator's vision, and an electrical counting circuit to aid the operator in recording the relative areas of coated and uncoated aggregate. A schematic diagram of the assembled apparatus is shown in Figure 1. The apparatus costs approximately $300, of which $200 is the cost of the microscope. A limited amount of work indicated that the microscope can be replaced with no impairment in performance by a 3/8-inch pipe nipple having cross-hairs attached to the lower end of the nipple. This change brings the total cost of the apparatus down considerably (by about $200). The procedure in measuring stripping with the mechanical integrator is as follows.

The sample of asphalt and aggregate contained in a 6- by 11-inch Pyrex glass tray is placed on the movable stage, and the microscope is focused on the sample surface. The operator then turns on the motor and watches

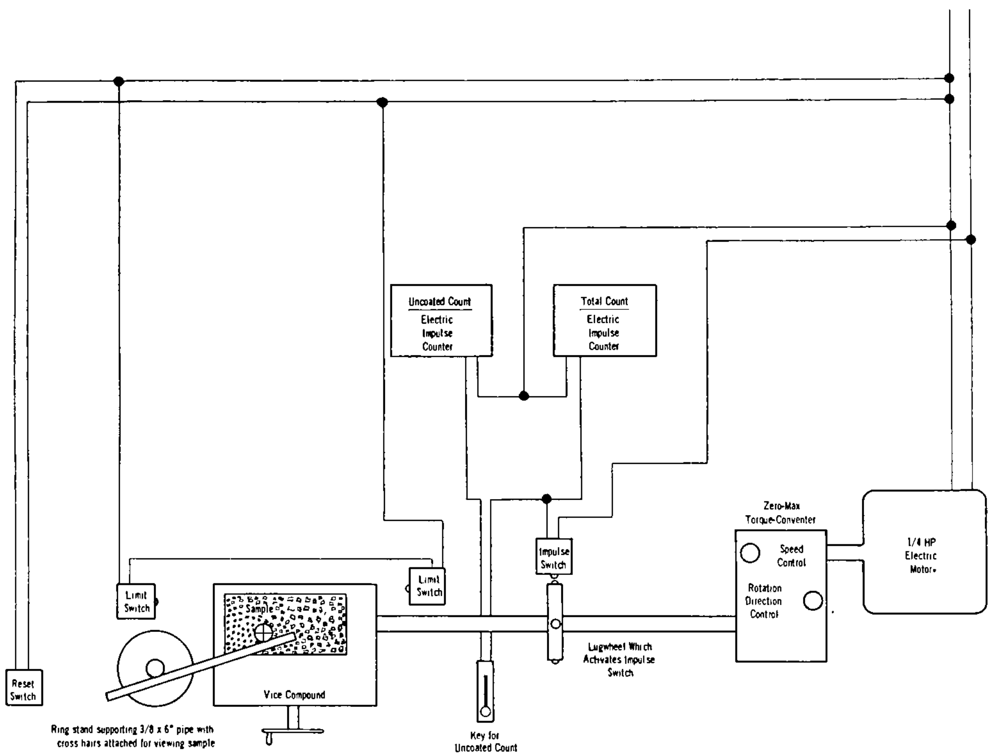

Figure 1. Schematic drawing of mechanical integration.

the sample at the cross-hair intersection. When the edge of a noncoated area appears to touch the cross-hair intersection, he depresses a key (actuating a microswitch); when the noncoated area has passed and asphalt appears to touch the cross-hair intersection, he releases the key. At the completion of a traverse, the limit switch stops the drive motor; the operator then indexes the sample stage to a new traverse position (parallel to the previous one) and proceeds as before.

Electrical impulses originating from actuation of a second microswitch by lugs on a wheel driven by the motor are recorded on a solenoid counter; the number of counts recorded on this counter (the total-count counter) is proportional to the length of traverse. When the key is depressed by the operator, the electrical impulses are also fed to a second counter (the part counter); the number of counts on this counter is proportional to the total length of uncoated area along the traverse line. The ratio of the part count to the total count gives the fraction of the aggregate (along the line traversed) that is not coated. This lineal ratio is directly proportional to the ratio of noncoated to total area, if there are sufficient traverses made from one end of the sample to the other. Thus, the data obtained represent directly the fraction of the total aggregate area that is not coated with asphalt, i. e., percentage stripped.

In the standard procedure used in evaluating the method, 10 traverses were made over a 5- by 5-inch area of the sample as one reading. Ten such readings (100 traverses) were made at random spots over the total sample area. These 10 readings were averaged to give the stripping determination for that sample. Each reading, consisting of 10 traverses, takes about 10 minutes. The complete sample determination, consisting of 10 readings, requires about 1¾ hours.

Comparison of Various Operators: Three operators (one experienced with the apparatus and two with no experience) determined the percentage of stripping on a single sample. Two of these operators (one experienced) ran determinations on two other replicate samples. The results are recorded in Table 1.

Variance analysis of the data on Sample 8D shows that the results obtained for one sample by these operators (experienced and inexperienced) are not significantly different. The same is true of the results obtained by the two operators on Sample 1E. With Sample 9D, however, the difference between the results obtained by the two operators is significant. This result indicates that some samples may vary so much across the surface that more than 500 lineal inches must be viewed on a sample of 58 square inches.

Data on visual estimations are given in Table 2. These data can be compared with those in Table 1. The large variation in results of visual estimates by different operators emphasizes a very serious drawback to the visual estimate method. The mechanical integration method has largely overcome this problem of reproducibility between operators.

Variation in Results on Replicate Samples: An experiment was conducted to obtain information on sample replication. Six supposedly identical samples were mixed on one particular day. After stripping, the degree of

TABLE 1
MECHANICAL INTEGRATION RESULTS OBTAINED BY DIFFERENT OPERATORS*
Observed Percentage Stripping

Reading	Sample 8D			Sample 9D		Sample 1E	
	Operator no. 1†	Operator no. 2	Operator no. 3	Operator no. 1	Operator no. 2	Operator no. 1	Operator no. 2
1	3.52	5.25	8.21	8.68	7.73	3.07	4.51
2	4.73	5.88	2.38	7.49	7.11	1.82	2.33
3	4.97	6.61	2.88	7.77	5.59	1.92	2.31
4	6.37	6.72	4.20	5.54	5.17	1.78	3.80
5	6.24	7.09	4.07	8.30	5.76	2.54	2.38
6	5.59	4.09	4.99	7.40	6.32	3.01	2.49
7	5.16	4.78	6.33	6.76	7.02	3.45	3.59
8	6.06	6.33	5.51	7.44	6.92	2.99	2.38
9	6.14	4.67	5.57	7.61	6.88	2.54	2.65
10	4.48	5.52	—	8.04	6.46	2.42	2.53
Mean, $\bar{x}$......	5.33	5.69	4.90	7.50	6.50	2.55	2.90
Variance, v..	0.85	1.00	3.18	0.76	0.63	0.34	0.61

* All samples were dry quartz aggregate coated with RC-2 cut-back containing 0.4 percent additive and cured for ¾ hour before stripping.
† Operator no. 1 was experienced; Operators nos. 2 and 3 had no experience with the mechanical integrator before making their determinations on Sample 8D.

TABLE 2
RESULTS OF VISUAL ESTIMATION BY FIVE DIFFERENT OPERATORS ON TWO SAMPLES*
Estimated Percentage Stripping

Sample	Operator no. 1	Operator no. 4	Operator no. 5	Operator no. 6	Operator no. 7
Quartz 3A......	8.7	9.0	28.3	30.0	35.0
Limestone 3A...	10.9	19.0	40.0	35.0	50.0

* Operators no. 1 and no. 4 had considerable experience with visual estimation; Operators no. 5, no. 6, and no. 7 had no previous experience.

TABLE 3
MECHANICAL INTEGRATION RESULTS OBTAINED ON REPLICATE SAMPLES*
Observed Percentage Stripping

Reading	Sample					
	1E	2E	3E	4E	5E	6E
1	3.07	2.70	2.37	2.49	2.06	2.13
2	1.82	2.63	1.86	3.02	2.20	3.00
3	1.92	3.53	1.99	2.45	2.24	2.16
4	1.78	2.37	1.41	3.40	2.56	2.14
5	2.54	2.52	2.37	2.29	2.42	2.04
6	2.01	3.61	2.19	3.37	1.85	2.82
7	3.45	2.30	2.13	3.39	2.48	2.33
8	2.99	2.57	1.48	2.82	3.01	2.13
9	2.54	3.02	2.12	2.39	3.39	2.53
10	2.42	2.82	2.58	3.34	3.64	2.19
Mean, $\bar{x}$	2.55	2.81	2.05	2.90	2.59	2.35
Variance, v	0.34	0.21	0.14	0.21	0.34	0.11

* All samples were dry quartz aggregate coated with RC-2 cut-back containing 0.4 percent additive and cured for ¾ hour before stripping.

stripping was measured by the mechanical integration procedure. The results are shown in Table 3. Variance analysis of the results showed that the difference between replicate samples was significant. The supposedly identical samples themselves contributed more to the over-all variance than did the apparatus and operator.

The three samples listed in Table 1 were supposedly identical samples prepared at different times. The data in Table 1 show that the difference in the results with replicate samples is much greater than the difference between results obtained by experienced and inexperienced operators. Thus, any major improvement in the reproducibility of the stripping test will require a reduction in the variation among replicate samples. Although these results indicate that additional work on improving the precision of the mechanical integration itself may not be warranted, work on reducing the time required to conduct a mechanical integration without reducing over-all accuracy or precision would be justified.

Effect of Curing Time and Additive Concentration: As a further comparison of the mechanical integration and the visual estimation methods, measurements were made on samples cured for various times before stripping and on samples coated with asphalt containing various amounts of additive. Variations in curing times and additive concentrations employed were sufficiently large that a significant change in degree of stripping should be produced. For a test method to be useful, it should be possible to detect such differences by the method.

Measurements were made by both the mechanical integration method and by visual estimation on the same samples. At the same time, measurements were made by the fluorescein method that is described later in this paper. Three curing times, ¾ hour, 4 hours, and 22 hours were used, each concentration or cure time being replicated three times; quartz aggregate (precoated with fluorescein) and an RC-2 cut-back containing 0.4 percent additive were used. Each sample was estimated visually once. The standard 10 readings (100 traverses) were taken with the mechanical integrator. The data are given in Table 4.

TABLE 4
EFFECT OF CURE TIME ON AMOUNT OF STRIPPING AS MEASURED BY DIFFERENT METHODS OF DETERMING STRIPPING*

Curing Time	Average Percentage of Stripping of Three Samples as Measured by					
	Mechanical integration		Visual estimation		Fluorescein	
	Mean	Variance	Mean	Variance	Mean	Variance
hours						
¾	6.15	2.24	8.67	1.35	44.13	5.42
4	4.75	0.66	5.33	0.35	34.83	3.89
22	3.29	0.08	3.33	0.30	32.44	5.48

* Dry quartz aggregate coated with RC-2 cut-back containing 0.4 percent additive.

With each of the methods, the change in percentages of stripping with curing time is significant. Thus, the methods are sensitive enough to detect changes produced by varying the curing time. Although there is reasonably good agreement between the values found by the mechanical integration and visual estimation methods, drastically different values were found by the fluorescein method. The probable explanation for this difference is discussed later in this paper.

The variance was lower for the visual estimation method than for the mechanical integration method; low variance is normally associated with high precision. The low variance in this case, however, may be a result of operator bias or lack of sensitivity in detecting small differences between replicates rather than a result of intrinsic high precision in the visual estimation.

In the tests on the effect of additive concen-

TABLE 5

EFFECT OF ADDITIVE CONCENTRATION ON AMOUNT OF STRIPPING AS MEASURED BY DIFFERENT METHODS OF DETERMINING STRIPPING*

Amount of Additive	Average Percentage of Stripping of Three Samples as Measured by					
	Mechanical integration		Visual estimation		Fluorescein	
	Mean	Variance	Mean	Variance	Mean	Variance
percent						
0	30.68	19.09†	41.67	8.34†	67.70	5.26
0.4	5.72	0.36	4.50	0.25	28.16	4.94
1.5	1.19	0.09	0.60‡	0.04	10.40	0.69

* Dry quartz aggregate, RC-2 cut-back, ¾-hour cure.
† High variance where no additive was used may indicate that additive reduces the effect of variations in the aggregate on stripping as well as reducing the amount of stripping.
‡ Value reported was "less than 1 percent." Value in table was arbitrarily selected for use in statistical analysis of data.

tration on stripping, three concentrations of additive were used (0, 0.4, and 1.5 percent). Quartz aggregate, precoated with fluorescein, was coated with RC-2 cut-back containing the additive, and was cured for ¾ hour; each additive concentration was replicated three times. The results are summarized in Table 5.

As was the case when curing time was varied, measurements by each of the methods indicated a significant change in stripping as the additive concentration was varied. The variance of the results with visual estimation again was lower and again may be misleading if taken to mean high precision.

Since there is no way of providing samples having accurately known degrees of stripping, it is not possible to test the accuracy of the mechanical integration procedure directly. Another possible approach to the problem of evaluating the method would be to measure known white areas randomly distributed on a black background. The mean value obtained with the mechanical integrator could then be compared with the known true value of percentage of white area. Use of such a "spatter card" would make it much more convenient to determine best minimum distance between traverse lines, optimum traverse speed, etc.

Optical Methods. Initial tests on determining the degree of stripping by measuring the amount of light diffusely reflected from an illuminated sample were complicated by interfering reflection from the asphalt surface. Recent tests have demonstrated that the interfering reflection can be minimized and possibly eliminated by the use of a polarized light source and a polarizing viewing filter. By using a photo multiplier tube with the polarized light and polarizing filter, it is possible to measure small differences in uncoated areas of aggregate. Additional work would be required to determine the effect of variations in the color or reflectivity of the uncoated aggregate on the accuracy and reproducibility of this optical method.

Indirect Measurement of Stripped Area

Tests based on indirect measurement of areas stripped depend on the measurement of some variable that is a function of area stripped. Several tests of this nature have been suggested:

1. Measurement of compressive strength.
2. Measurement of gas adsorption, where gas would preferentially adsorb on aggregate or on asphalt.
3. Measurement of dye adsorption, where dye would adsorb from solution preferentially on aggregate or asphalt.
4. Leaching of some material from aggregate where either the amount of material leached or the rate of leaching is a function of area of stone exposed.
5. Dissolving of some material placed on the aggregate surface prior to coating with asphalt, where the amount of material dissolved from aggregate is a function of the amount of aggregate area exposed.

Work has been done by other investigators on compressive-strength tests and dye adsorption. The work on dye adsorption evidently has been abandoned. Work on compressive strength is being continued by a number of investigators. Work was done for this study on leaching the aggregate surface and on dissolving a precoating material from the aggregate surface.

Fluorescein Dye as a Precoating Material. The following test method was developed for utilizing fluorescein dye as an aggregate precoating material. The aggregate sample, previously sized and washed, is placed in a boiling aqueous solution of fluorescein for one minute. The aggregate and solution is then dumped into a wire basket and mechanically agitated until dry. The normal coating, curing, and immersion operations are then carried out. As stripping occurs, the exposed dye dissolves in the stripping water. Comparison of the dye concentration from the test sample with the dye concentration from a control sample (aggregate precoated with fluorescein and immersed in water without coating with asphalt) gives the relative amount of stripping that has occurred in the test sample. Analysis for fluorescein concentration requires either a spectrophotometer or a colorimeter; there is the possibility that comparisons of the stripping solution with color standards may be a satisfactory routine procedure.

Tests on Hot-Mixed Samples: Some early work was done with the fluorescein method on hot-mixed samples. This work indicated that a correlation existed between the results with the fluorescein method and visual estimates. This phase of the work was terminated at the suggestion of members of the Highway Research Board and work was concentrated on tests with cut-back asphalts.

Results With Wet Aggregate and Cut-Back Asphalt: Subsequent work with cut-back asphalt (RC-2) and wet aggregate showed that the amount of stripping indicated by the fluorescein determination varied widely from visual estimates, especially where the asphalt that contained additive displaced the water, and consequently fluorescein, from the surface of the aggregate.

Results With Dry Aggregate and Cut-Back Asphalt: Data on the percentage of stripping of dry aggregate as measured by the fluorescein method were given earlier in this paper in Tables 4 and 5. These data were obtained at the same time as data from mechanical integration measurements and visual estimates on the same samples. In these tests the aggregate was precoated with fluorescein before mixing with the asphalt. Samples of the stripping water were taken for fluorescein determination and were compared with control samples not coated with asphalt. The correlation between the results with the fluorescein method and the results with the other two methods is not very good, although the fluorescein results show a reasonable correlation with change in cure time and with change in additive concentration. The fact that the stripping indicated by fluorescein is so much higher than either the visual estimates or that measured by mechanical integration may indicate that the adhesion between the asphalt and aggregate is poor even though most of the aggregate surface is covered with asphalt. If large areas of aggregate are covered by asphalt but not adhesively bonded with the asphalt, the stripping water can work its way from uncoated regions into those poorly bonded regions and leach out the fluorescein. Consequently, results from fluorescein measurements may correlate better with immersion-compression test results, and possibly with road tests, than do visual estimates or mechanical integration (both of which measure only area uncoated and do not measure poorly bonded areas). Thus, the fluorescein method might provide useful data, if its precision can be improved.

Zinc Oxide as a Precoating Material. A brief study was made of zinc oxide as a precoating material that might eliminate the difficulties encountered with fluorescein on wet aggregate. The zinc oxide coating is not soluble in the neutral water used to wet the aggregate. Consequently, the zinc oxide is not displaced from the aggregate surface when the asphalt-additive mixture displaces water from the aggregate during the coating step. The zinc oxide coating which becomes exposed as the asphalt strips from the aggregate can be dissolved in mildly acid solutions. Analysis of the concentration of zinc in the solution will then permit calculation of the percentage of stripping which has occurred. The results obtained in tests with zinc oxide indicate that it might be better as a precoating material than fluorescein for tests on wet aggregate. Work with zinc oxide was discontinued, however, because the analysis for zinc proved to be expensive and time consuming. It is also probable that the zinc oxide coating would affect the adhesion characteristics of the aggregate.

Leaching of the Aggregate Surface. A study was conducted on using the aggregate surface itself as an indicator of the extent of stripping by leaching with a weak acid solution. Measurement of the rate of dissolving (by measuring rate of acid depletion) should be a measure of the area of uncoated aggregate. In this work limestone and hydrochloric acid were used; hydrochloric acid might be used with other carbonate-bearing aggregates such as dolomite; hydrofluoric acid might be used with silica aggregates. The results showed some promise in that there was some correlation between stripped area and rate of acid depletion; however, in the brief study undertaken, the precision was poor. The evolution of carbon dioxide from carbonate-bearing aggregates caused an additional difficulty by promoting accelerated stripping. Additional study would be required to determine whether this leaching method might correlate satisfactorily with area stripped, strength tests, or road tests.

RECOMMENDED FUTURE WORK

Additional work should be done on "mechanical integration" to obtain better proof of its accuracy and to fix exactly the procedure to be used to obtain specific degrees of precision. Measurements should be made of "spatter cards" in which the relative black and white areas were known exactly. These cards could be useful in optimizing the mechanical integration procedure for precision and economy of testing.

It is highly probable that the automatic optical instrument based on illuminating the sample with polarized light can be developed into an accurate method of determining stripping. Such a method is extremely attractive from the viewpoint of simplicity in operation. The method shows so much promise that additional work to determine its ultimate possibilities is definitely warranted. The bulk of the work on the optical method would have to follow the additional work on the mechanical integration method, since the mechanical integration method would be the primary standard for evaluating the optical method.

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