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A study made by the California Division of Highways in cooperation with the U.S. Department of Commerce Bureau of Public Roads

16. ABSTRACT

A modified method has been developed for using the ASTM Tracer Salt method is not as good as the Dye solution procedure.

The percentage of stripping determined by the Dye and Tracer Salt methods was compared with the surface water abrasion test results on a number of different aggregates. The results show that the abrasion loss and percentage of stripping are related.

Water may induce partial to serious raveling and disintegration at the surface of a pavement. The entrance of water into the pavement may cause stripping of the asphalt. Such action may alter the properties of the pavement to the extent that failures will occur under traffic action.

One of the important aspects of the problem is the development of tests that will provide a quantitative measure of the effect of water on pavement performance. These tests should be performed on paving mixtures containing the entire aggregate gradation with the design asphalt binder content.

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STATE OF CALIFORNIA
HIGHWAY TRANSPORTATION AGENCY
DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS

FINAL REPORT ON STUDY OF THE EFFECTS OF WATER ACTION ON BITUMINOUS MIXTURES

A STUDY MADE BY THE
CALIFORNIA DIVISION OF HIGHWAYS
IN COOPERATION WITH THE
U.S. DEPT. OF COMMERCE
BUREAU OF PUBLIC ROADS

August 1965

65-09

State of California
Department of Public Works
Division of Highways
Materials and Research Department

July 15, 1965

M&R Project 230068
HPR 1 (1) D-3-5

Mr. L. R. Gillis
Assistant State Highway Engineer, Operations
1120 N Street
Sacramento 7, California

Dear Sir:

Submitted for your consideration is:

FINAL REPORT

ON

STUDY OF THE EFFECTS OF WATER ACTION ON BITUMINOUS MIXTURES BY MEASURING STRIPPING THROUGH THE USE OF TRACER SALT AND DYE SOLUTION METHODS AND SURFACE WATER ABRASION TEST.

Study made by . . . . .Pavement Section
Under general direction of. . . . . .E. Zube
Work supervised by. . . . . .J. Skog & G. Kemp
Report prepared by. . . . . .J. Skog

Very truly yours,

JOHN L. BEATON
Materials and Research Engineer

Attach.

cc: CGBeer
ACEstep
JFJorgensen
PCSheridan
Districts

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SYNOPSIS

A modified method has been developed for using the ASTM Tracer Salt technique for measuring the percentage of stripping of the total mix gradation. Studies indicate that the repeatability of the modified Tracer Salt method is not as good as the Dye solution procedure.

The percentage of stripping determined by the Dye and Tracer Salt methods was compared with the surface water abrasion test results on a number of different aggregates. The results show that the abrasion loss and percentage of stripping are related.

INTRODUCTION

Water may induce partial to serious raveling and disintegration at the surface of a pavement. The entrance of water into the pavement may cause stripping of the asphalt. Such action may alter the properties of the pavement to the extent that failures will occur under traffic action.

One of the important aspects of the problem is the development of tests that will provide a quantitative measure of the effect of water on pavement performance. These tests should be performed on paving mixtures containing the entire aggregate gradation with the design asphalt binder content.

Recently ASTM Committee D-4 has completed an extensive study of a new method for measuring the stripping of bitumen aggregate mixtures, known as the Tracer Salt Method, (1). In this method, a sample of aggregate passing the 3/8" sieve and retained on the 1/4" sieve is coated with Lithium Chloride salt solution and dried in an oven. Portions of this treated aggregate are coated with bitumen and cured for a designated time. Original Lithium chloride treated control samples (raw aggregate), and bitumen coated materials are placed in 4 oz. screw top jars, covered with distilled water, and allowed to stand for 16-18 hours at room temperature. The Lithium chloride content of the water, from the control and bitumen coated samples, is determined by means of a flame photometer. From these values, the per cent of retained coating or percentage of stripping may be calculated.

The completion of the work on development of the Tracer Salt procedure by Committee D-4 led to a proposal to attempt to modify the method in order to use the entire mix gradation with the design asphalt binder content. Further, a study was proposed for comparing the modified Tracer Salt method with the Dye technique and Surface Water Abrasion test developed in the Pavement Section of the California Division of Highways, Materials and Research Department. The latter two methods are described in a paper published in the 1963 proceedings of the AAPT (2).

This final report will present results on the objectives outlined above.

CONCLUSIONS

Tentative modifications in the original ASTM technique for the Tracer Salt test have provided a method for measuring the percentage of stripping of the total mix gradation.

Studies indicate that the Dye method has better repeatability than the proposed Tracer Salt procedure. The results clearly indicate that every care must be taken in sample preparation since these studies were performed under very strict control of all possible variables. It is apparent that further extensive studies will be required by a number of organizations before adequate control over reproducibility is achieved.

Some aggregate sources may produce anomalous results when the Dye test is used for measuring stripping. This appears to be caused by aggregate sources which have grading fractions with widely different dye uptake-percentage of stripping ratios. Our studies to date indicate that such situations occur only rarely.

Using five aggregates of widely different stripping tendencies, a satisfactory relation was found for the percentage of stripping by the Dye and Tracer Salt methods. For all practical purposes the percentage of stripping is the same by either method.

There is also a definite relation between the percentage of stripping and the surface abrasion loss.

Considering the three methods under study, the surface abrasion test appears to provide the best indication of pavement resistance to water action.

DEVELOPMENT OF A MODIFIED TRACER SALT TEST METHOD

The first objective of this project was to modify the existing Tracer Salt method in order to use the entire mix gradation with the design asphalt binder content. The principal change was in the Lithium chloride coating process of the raw aggregate. In the ASTM method, a 90 gram sample composed of aggregate passing the 3/8" sieve and retained on a 1/4" sieve, is immersed in 45 grams of tracer salt solution for one minute. The sample is stirred with a spatula to ensure complete surface coverage. The sample is transferred onto a

1/4" sieve, and the excess solution is drained off. The coated aggregate is oven dried at 250°F with frequent light stirring by the operator.

When a 250 gram sample containing the entire mix gradation was placed in a container and 125 grams of Lithium chloride added and the excess solution decanted off, some of the fines were lost. Our next approach was to add 50 mls. of Lithium chloride to cover the sample which was spread out in a shallow pan. The sample was stirred, and all liquid was evaporated in an oven at 250°F. The 50 ml. quantity proved to be excessive and "caking" was quite pronounced. After a series of trials with a number of different aggregates 25 ml. of solution was found to adequately coat the aggregate with only a slight excess. The modified test method is presented in Appendix A.

After development of a modified test method, a test series was performed to check repeatability. Two aggregate sources of quite high stripping tendencies were selected. The results are shown in Table A. The standard deviations for Lithium chloride demand indicate about equal repeatability for both uncoated and coated systems. The standard deviations for the percentage of stripping are quite high, and the repeatability is not considered satisfactory.

In order to obtain more information on the repeatability of the Tracer Salt method, a test series was performed in which the Dye Technique and Tracer Salt method were compared using three aggregate sources of varying percentages of stripping. The results are shown in Table B. The results from the Dye test show very good repeatability in both Trials 1 and 2 while the Tracer Salt method shows greater variation. We note, however, that both methods show rather wide differences between Trials 1 and 2 even though the repeatability for each trial is quite good, especially with the Dye procedure. This comparison indicates that factors beyond those involved in the determination of dye and Lithium chloride demands on uncoated and coated samples are important in the final percentage of stripping. These are probably associated with variables concerned with sample preparation, mixing, curing, and stripping. Further studies are required in order to determine the specific reasons for the variations noted in average percentages of stripping in Trials 1 and 2.

COMPARITIVE RESULTS USING TRACER SALT METHOD, DYE SOLUTION METHOD, AND SURFACE WATER ABRASION TEST.

The percentage of stripping by the Tracer Salt and Dye solution methods was compared with the Surface Abrasion test result using five different aggregate sources. The results are shown in Table C and Figs. 1 and 2.

The two methods for measuring stripping check quite closely with two of the aggregate sources, but vary from 10 to 16% for the other three sources.

The Surface Abrasion test results are compared with the percentage of stripping by the two methods as shown in Fig. 2. The percentage of stripping as measured by the two methods appears to be related to the amount of loss attained with the Surface Abrasion test. However, one aggregate source shows very high stripping by both methods with a low loss in the Abrasion test. It is very difficult to explain this finding. There is the possibility that one portion of the fine grading is very hydrophilic and strips badly as compared to other portions, thereby, providing a final high figure. In the compacted specimen, subjected to the surface abrasion test, this portion may be protected by other materials of the matrix which show greater resistance to stripping. The abrasion loss would, therefore, represent the overall resistance of the matrix to water action. On this basis we may conclude that the surface abrasion loss is the best indication of the resistance to water action of the paving mixture.

In the Dye test the amount of dye that is held by the aggregate will depend on the degree of adsorption, absorption, and surface area. As an example, materials having low adsorption and high stripping when combined with those having high adsorption and low stripping may provide misleading final results. The Tracer Salt technique, involving the coating of an inert salt on the surface of the aggregate should provide better results for the percentage of stripping on aggregate combinations of this kind. This is illustrated in Table D and Fig. 3. The results from the Tracer Salt test provide the expected linear relation between percentage of stripping and increasing additions of uncrushed to crushed material from the same source. The dye test does not show this relation and indicates a marked increase in stripping with a small addition of uncrushed material. The surface abrasion results confirm the Tracer Salt results as shown in Fig. 4. An explanation for the anomaly in the dye test results is shown in Fig. 5. We note that the uncoated sample provides a direct relation between dye uptake, and percentage of crushed material in the sample under test. However, this relation is not attained with the stripped sample.

The above noted study was repeated on another aggregate source. The results, shown in Table D and Fig. 6, indicate excellent agreement between dye and tracer salt methods and a direct relation between the percentage of stripping by the two methods and additions of uncrushed material. The dye uptake curve for the stripped sample for this system is compared with that previously found in Fig. 7. We note in this case that the expected linear relation is found.

The anomaly found for the dye test has not been encountered in the bulk of the aggregate sources tested with this method. We, therefore, conclude that a false figure for percentage of stripping by the dye technique will only rarely occur. In any case, these studies indicate the importance of developing tests that will provide a measure of the resistance to water action of a compacted specimen which closely simulates the pavement. In this case, the surface abrasion test appears to consistently measure the overall effect of water on the pavement.

REFERENCES

1. Proposed Method of Test for Stripping of Bitumen Aggregate Mixtures (Tracer Salt Method). Report of ASTM Committee D-4, Sixty-sixth Annual Meeting of ASTM, June 23-28, 1963.

2. "New Test Methods for Studying the Effect of Water Action on Bituminous Mixtures". - J. Skog and E. Zube, A.A.P.T. Vol. 32, p. 380, 1963.

TABLE A

REPEATABILITY OF LITHIUM CHLORIDE

TRACER SALT METHOD

Aggregate Sample No.	Test. No.	P.P.M. Lithium Chloride		% Stripping
		Uncoated	Coated	
64-3187 & 64-3189 Asphalt A Operator #1	1	8	5	63
	2	11	7	64
	3	11	9	82
	4	9	5	56
	5	10	7	70
	6	11	8	73
	7	11	9	82
	8	11	8	73
	Ave $\bar{x}$	10	7	70
	n	7	7	7
	S.D.	± 1.2	± 1.0	± 9.2%
59-1022 & A Asphalt A Operator #1	1	14	8	57
	2	14	8	57
	3	13	8	62
	4	13	7	54
	5	12	5	42
	6	12	5	42
	7	13	6	46
	8	13	6	46
	Ave $\bar{x}$	13	7	51
	n	7	n	7
	S.D.	± 0.8	± 1.4	± 7.7%

TABLE B

REPEATABILITY OF TRACER SALT AND DYE METHODS.

Sample No.	Percentage of Stripping			
	Dye Method		Tracer Salt Method	
	Trial 1	Trial 2	Trial 1	Trial 2
63-5013 Asphalt A Operator #1	14.5	21.0	41.2	27.3
	14.7	21.0	38.9	33.4
	15.0	21.0	35.0	25.0
Average	14.8	21.0	38.4	28.6
S.D.	0.3	--	3.1	3.7
59-1770 & 59-1771 Asphalt A Operator #1	37.9	33.9	33.4	25.0
	38.6	35.7	31.3	31.6
	39.1	33.9	33.4	35.0
Average	38.5	34.5	32.7	30.5
S.D.	0.6	1.0	1.3	5.1
64-3187 & 64-3189 Asphalt A Operator #1	47.2	62.8		
	45.9	61.9		
	42.7	60.0		
Average	45.3	61.6		
S.D.	2.3	1.4		

SUMMARY

Sample No.	Average Percentage of Stripping and Standard Deviation			
	Dye Method		Tracer Salt Method	
	Trial 1	Trial 2	Trial 1	Trial 2
63-5013	14.8	21.0	38.4	?8.6
	S.D. = 0.3	--	3.1	3.7
59-1770	& 71 38.5	34.5	32.7	30.5
	S.D. = 0.6	1.0	1.3	5.1
64-3187	& 89 45.3	61.6		
	S.D. = 2.3	1.4		

TABLE C

COMPARISON OF PERCENTAGE OF STRIPPING
BY TRACER SALT AND DYE SOLUTION METHODS
WITH SURFACE ABRASION LOSS.

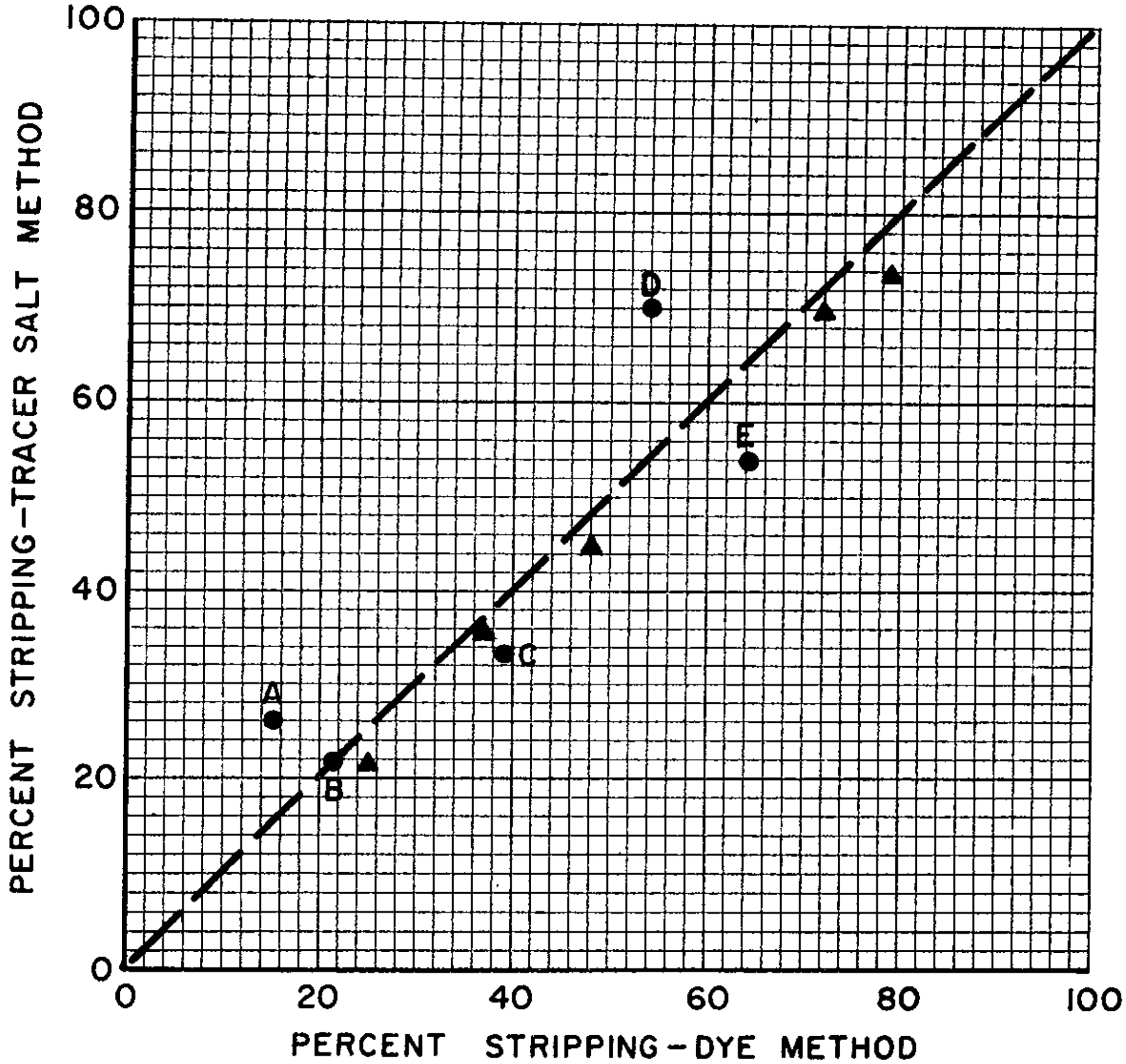
Code No. (see Figs. 1 & 2)	Aggregate Number	Asphalt Source & Grade	Average Percentage of Stripping		Average Abrasion Loss-Gms.
			Dye Method	Tracer Salt Method	
A	63-5013	"A" 85-100	15	26	0
B	64-3190,91, 92,93 & 2194	"A" 85-100	21	22	0.1
C	59-1770 & 1771	"A" 85-100	39	33	19
D	64-3187 & 3189	"A" 85-100	54	70	16
E	59-1022 & A	"A" 85-100	64	54	94

TABLE D

EFFECT OF MINERAL SURFACE ON PERCENTAGE
OF STRIPPING BY DIFFERENT TEST METHODS
USING ASPHALT A, 85-100 PENETRATION GRADE

Aggregate Number and Mix Combination	Percentage of Stripping*		Abrasion Loss* Gms.
	Dye Method	Tracer Salt Method	
59-1021A 100% crushed	25	22	0.1
59-1021 & A 75% crushed 25% as rcvd.	37	36	3.8
59-1021 & A 50% crushed 50% as rcvd.	48	45	14.1
59-1021 & A 25% crushed 75% as rcvd.	72	70	31.7
59-1021 100% as rcvd.	79	74	48.8
59-1022A 100% crushed	29	21	4.3
59-1022 & A 75% crushed 25% as rcvd.	67	27	5.1
59-1022 & A 50% crushed 50% as rcvd.	65	34	9.0
59-1022 & A 25% crushed 75% as recvd.	73	33	32.2
59-1022 100% as rcvd.	66	46	61.3

* Results are average of two tests.



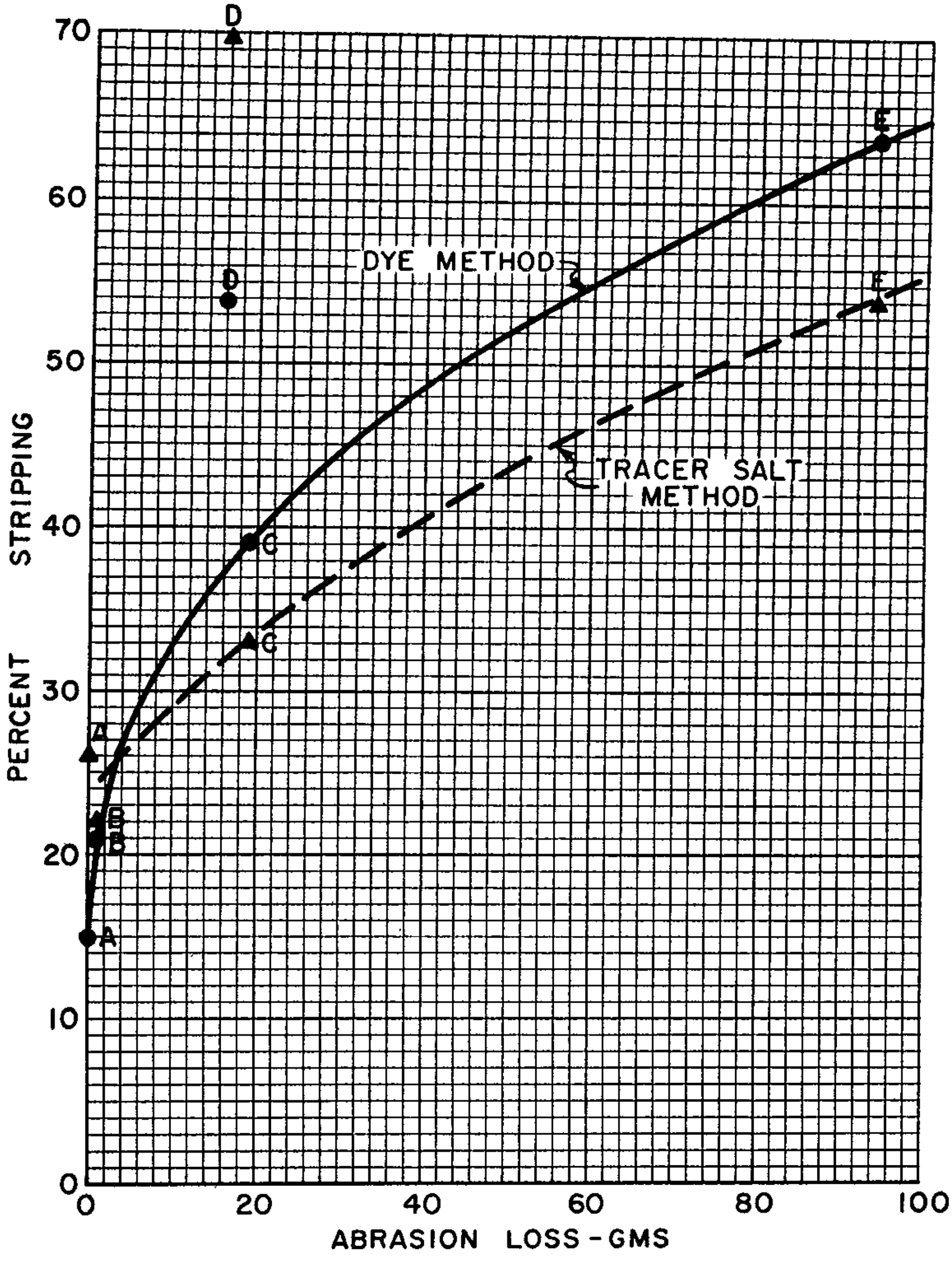
COMPARISON OF PERCENT STRIPPING BY TRACER SALT AND DYE SOLUTION METHODS

KEY

● VARIOUS AGGREGATE SOURCES, TABLE C

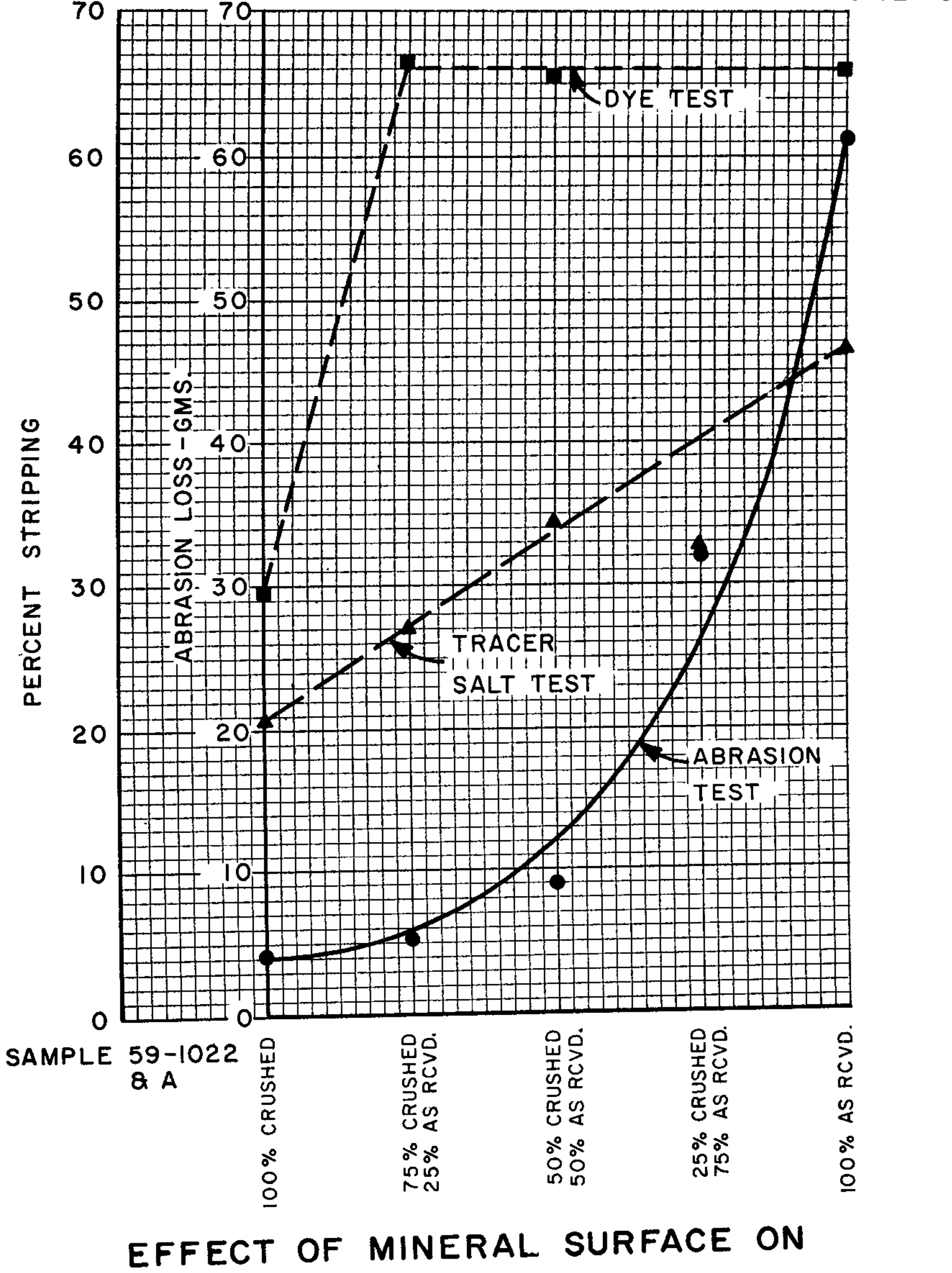
▲ SERIES 2, AGGREGATE 59-1021A, TABLE D

FIGURE 2



PERCENTAGE OF STRIPPING BY TRACER SALT AND DYE SOLUTION METHODS COMPARED WITH SURFACE ABRASION LOSS

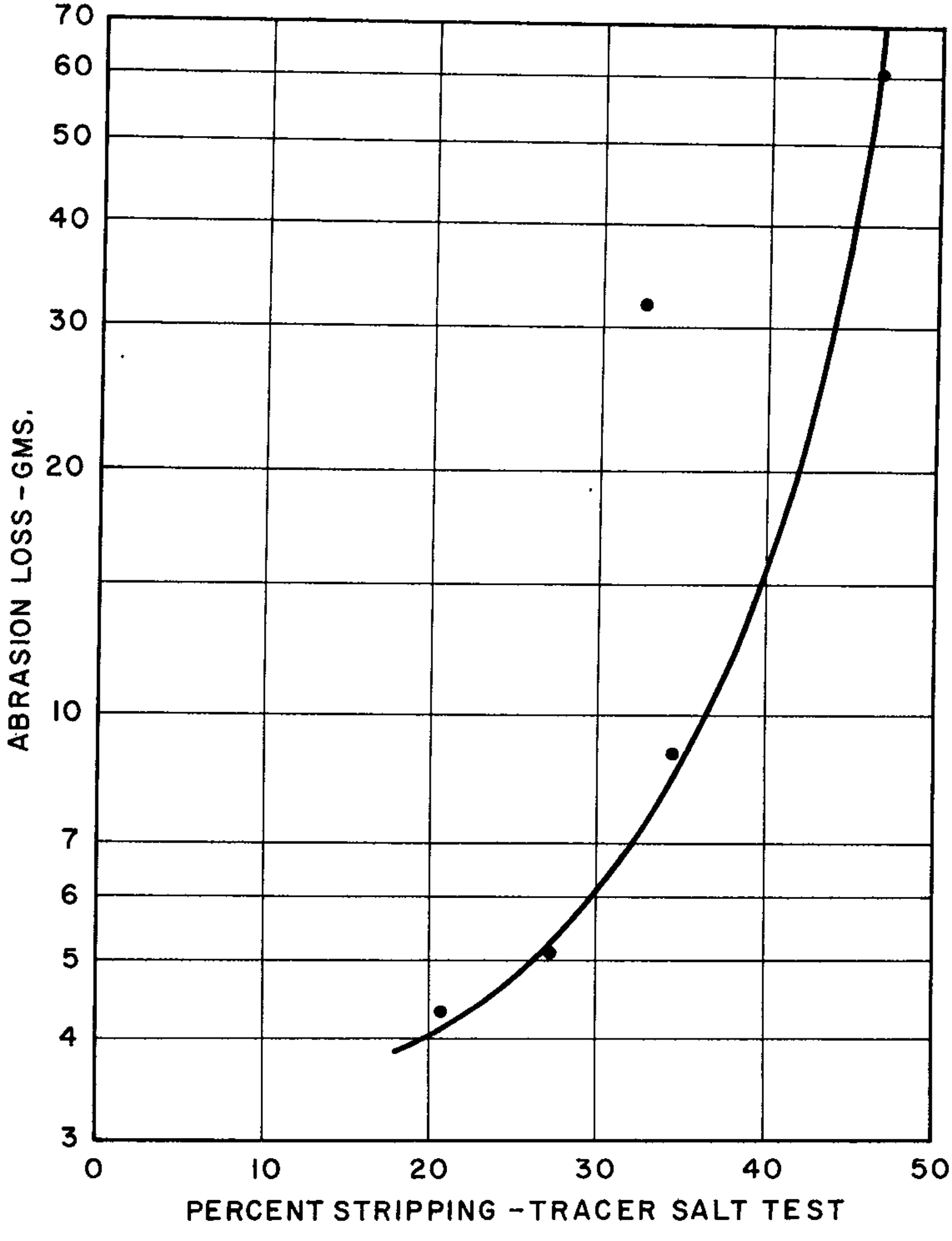
KEY
● DYE METHOD
▲ TRACER SALT METHOD



EFFECT OF MINERAL SURFACE ON PERCENTAGE OF STRIPPING BY DIFFERENT TEST METHODS

SERIES #1

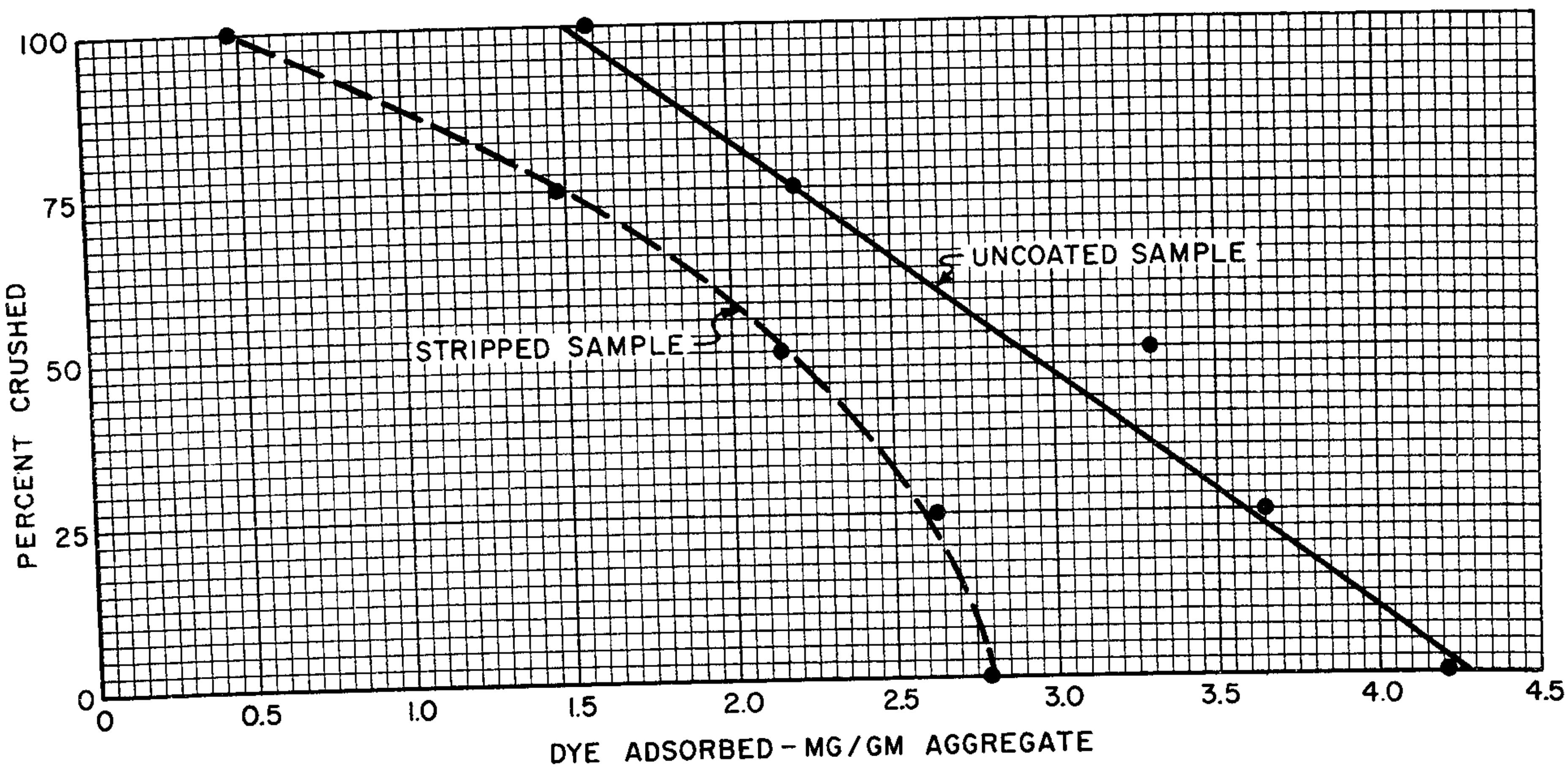
FIGURE 4



RELATION BETWEEN ABRASION LOSS AND PERCENTAGE OF STRIPPING BY TRACER SALT TEST

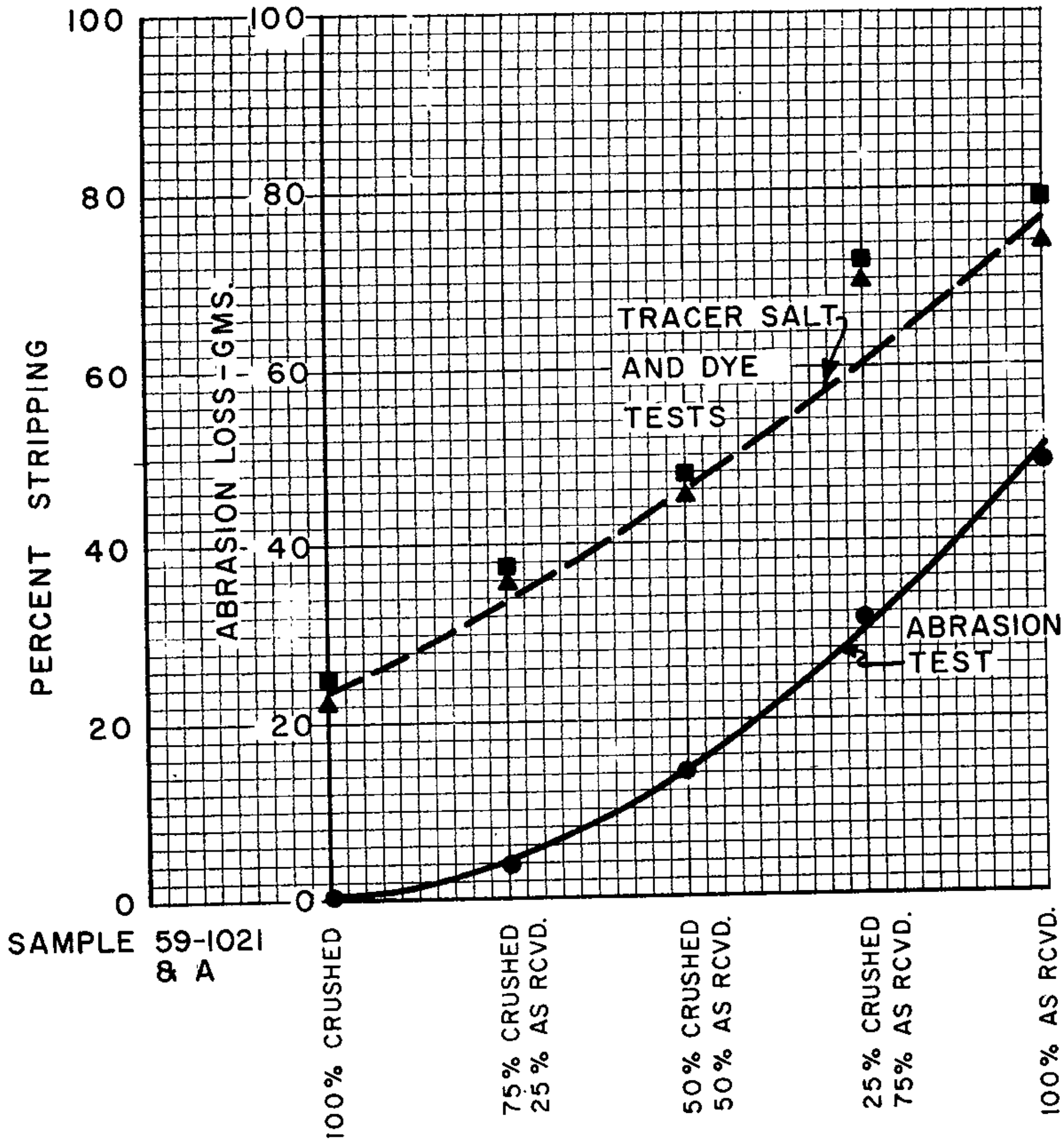
AGGREGATE # 59-1022 & A

FIGURE 5



DYE ADSORPTION PATTERN AS PERCENTAGE OF CRUSHED MATERIAL IS INCREASED

SAMPLE 59-1022 & A, SERIES #1



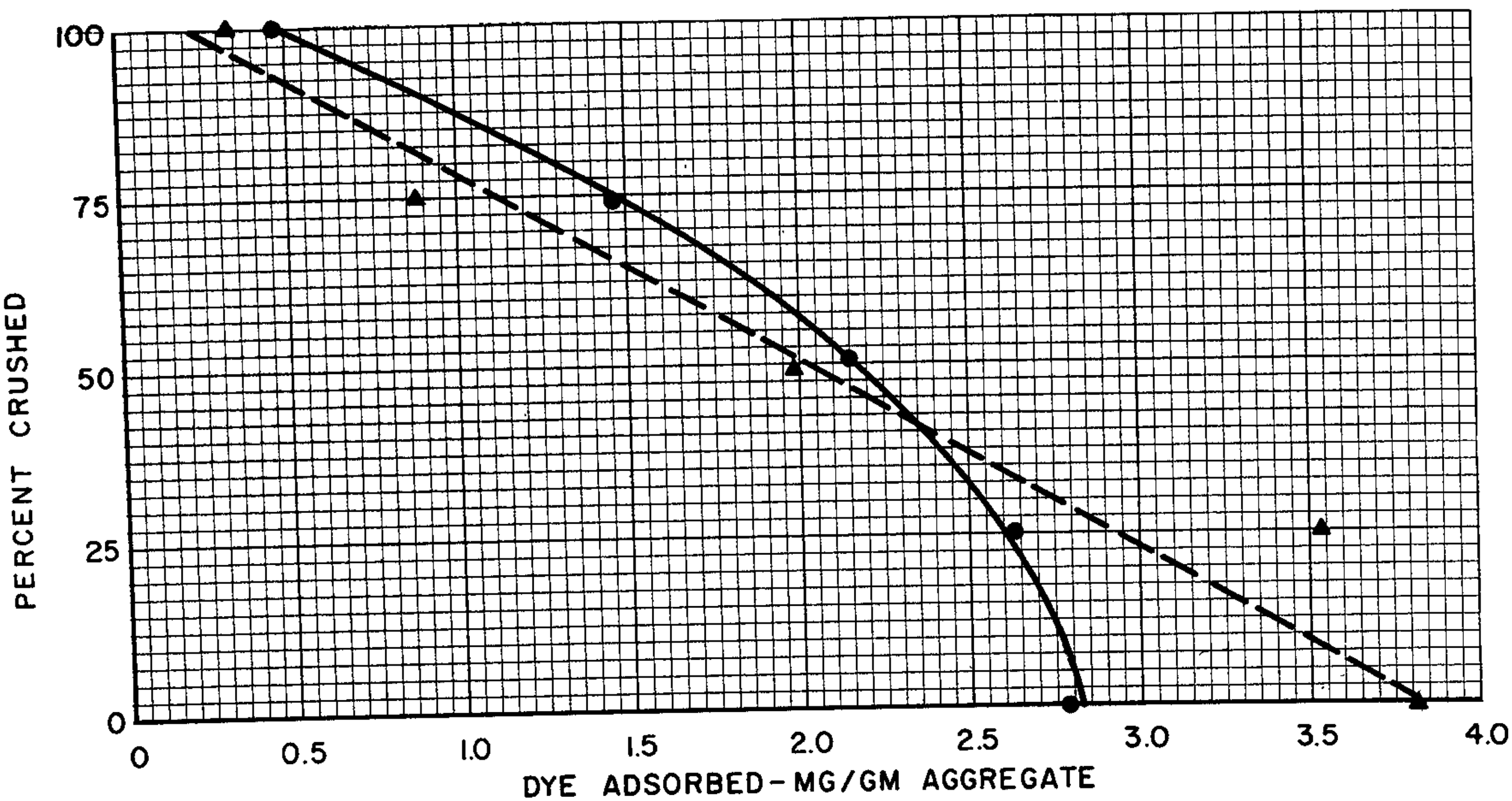
EFFECT OF MINERAL SURFACE ON PERCENTAGE OF STRIPPING BY DIFFERENT TEST METHODS

SERIES #2

KEY

■ DYE TEST

▲ TRACER SALT TEST



DYE ADSORPTION PATTERN FOR STRIPPED SAMPLES

KEY
● 59-1022 & A, SERIES 1
▲ 59-1021 & A, SERIES 2

FIGURE 7

APPENDIX A

Tentative Method of Test For Quantitative Determination of Film Stripping Through Use of Lithium Chloride Tracer Salt Solution

SCOPE:

This method provides a means for the quantitative determination of the resistance of bituminous mixtures to stripping of the asphalt from the aggregate particles, through the use of a lithium chloride tracer salt technique and a flame photometer for indicating the extent of the stripped surface.

PROCEDURE:

A. Apparatus

1. Flame Photometer - Perkin Elmer Model 52a (or equivalent)

2. California Type Mechanical Stripping Machine with special adaptors to hold 16 oz. bottles, Test Method No. Calif. 302-B.

3. Mechanical Mixing Machine (optional) Test Method No. Calif. 304-C.

4. Constant Temperature Oven, capable of maintaining a temperature of 250°F $\pm$ 2°F.

B. Materials

1. Lithium Chloride (LiCl), anhydrous Cp.

C. Preparation of Lithium Chloride Stock Solution

Prepare a solution (250 $\pm$ 10 ppm Li+) by dissolving 1.527 gms. of anhydrous Cp lithium chloride (LiCl) in 1 liter of distilled water.

D. Preparation of Samples

Process the aggregate in a manner comparable to construction processing; i.e., wash if washing is to be employed; otherwise, test as received.

Place four 250 gm. samples of the aggregate in shallow pans (preferably of aluminum or stainless steel). Pour 25 mls. of the stock solution onto the sample and stir with a spatula for 1 min. or until the entire sample has been coated. Place the sample in a 250°F oven for a period of 1-1/2 - 2 hrs. stirring occasionally to prevent caking. Remove the sample from the oven and allow to cool before storing in a clean, dry container for testing. Treat each of the four samples of any aggregate in the same manner.

E. Mixing the Samples

Coat two 250 gram samples of aggregate with the design bitumen content and cure for 15 hours at 140°F.

F. Tests and Calculations

1. Procedure for stripping the coated and uncoated samples.

   a. Place the cured bitumen coated samples in 16 oz. jars and allow to cool. Add 200 ml. of distilled water and place the jars in the stripping machine for 30 minutes.

   b. Follow the same procedure for the uncoated samples.

2. Procedure of preparation of samples for lithium determination.

   a. Allow the sample to remain undisturbed for a period of time to allow the fines to settle.

   b. Filter out about 50 mls. of the solution of each sample until the liquid becomes quite clear.

3. Procedure for determining the concentration of lithium.

   a. Use Perkin Elmer Flame Photometer Model 52A (or equivalent).

   b. Determine Li+ in ppm using standard solutions made from LiCl reagent grade and a predetermined calibration curve.

4. Procedure for calculating the percentage of stripping.

   a. Calculate the percentage of stripped coating as follows:

$$PSC = 100 \frac{(A)}{(B)}$$

Where:
PSC = Percentage of stripped coating.
A = mean of two lithium values in parts per million of the stripping waters of the coated cured, and stripped samples.
B = mean of two lithium values in parts per million of the stripping waters of the uncoated samples.

   b. Report the percentage of stripping to the nearest 0.1%.