REPORT OF COMMITTEE ON STABILIZED ROADS C A HOGENTOGLER, Chairman

REPORT OF SUBCOMMITTEE ON USE OF PORTLAND CEMENT W H Mills, Jr, Chairman

## CHEMICAL DETERMINATION OF CEMENT CONTENT OF SOIL-CEMENT MIXTURES FROM CEMENT HARDENED BASES

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## SYNOPSIS

This investigation was instituted by Subcommittee No 2 of the Project Committee on "Stabilized Roads," of the Highway Research Board in July, 1940, to investigate the "Chemical Determination of the Cement Content of Soil-Cement Mixtures from Cement Hardened Bases"

After preliminary inquiry had shown that two basic methods had been used in determining cement content it was decided to ask a number of laboratories to cooperate in testing standard samples by the two methods Twenty-four laboratories participated in the investigation

The results obtained by the laboratories were remarkably uniform with both methods but it was felt that the test procedure recommended in the Committee report, which is based upon determination of CaO content in samples of the raw soil, cement and soil-cement mixture, offers wider application in that it may be used with soils of higher lime content than are usually met

A preliminary report made in December, 1940 briefly reviewed the results of a questionnaire sent to each State, asking whether determinations of cement content in cement hardened bases had been made, and if so, what method was followed, and with what results. At that time, it was indicated by 43 replies, that only nine states had attempted to make such determinations. The methods submitted by five States indicated that one of two basic procedures was followed.

One procedure required the determination of the CaO content in samples of the raw soil, cement, and soil-cement mixtures, after which these values were used in calculation of the percentage of cement in the soil-cement mixtures, using the proportionality law applied to mixtures This procedure was basically followed by Michigan, Missouri, Texas, Georgia, North Carolina and South Carolina

The other procedure involved the chemical determination of the Ca value

in the soil-cement mixture obtained from the roadway, and a similar determination on a control sample, which was prepared to contain approximately this percentage of cement using the original soil and cement, as components. These values were then used in calculating the cement content of the roadway mix, as described by Vaughan of Mississippi<sup>1</sup> Similar procedures were reported as being used by Arkansas, Mississippi and Oklahoma

This preliminary report outlined the procedure used by Michigan, Missouri, Oklahoma, and Texas, together with a report by Wales of Arkansas, indicating the values obtained on a typical soilcement stabilization project.

On January 23, 1941 the States and the Public Roads Administration were invited to participate in tests of prepared

<sup>1</sup> F W. Vaughn, "Progress in Soil-Cement Construction—Moisture and Compaction Control and Determination of Cement Content," *Proceedings*, Highway Research Board, Vol 19, p 524 (1939). standard samples, following one or more basic procedures. The Division of Tests of the Public Roads Administration and 23 States accepted.

A complete set of standard samples. together with full details, consisting of two basic procedures, uniform data sheets, and supplementary information, were forwarded to each of these cooperating laboratories. The methods of analysis to be used were procedures suggested by Missouri and Oklahoma. with only slight editorial changes, as it was believed these methods would best bring out discrepancies in the two basic procedures, into which all previously suggested methods appeared to fall. The standard samples consisted of soil-cement samples A and B, a raw soil sample, and a cement sample. Participants were requested to criticise the suggested methods in detail, offering alternate procedures, changes in technique, and such other pertinent information as might develop.

After receiving reports from 20 of 24 participants, a report of "Cooperative Tests Results on Standard-Soil-Cement Samples" was forwarded to participants as of August 1, 1941. A special issue of this was later transmitted to the remaining states, which had not taken part in the investigation.

The criticism and suggestions contributed by 15 laboratories, in response to our request, were briefed in the August 1st report.

Examination of this criticism in general, indicated the following points:

- 1. About half of the participants presenting criticism definitely favored Method 1, as being the more accurate The reasons expressed for this viewpoint included mathematical soundness of procedure, and limitation of variables.
- 2 There was some indication that Method 2 was more rapid, under certain conditions.
- 3. There was much more criticism against Method 2 than Method 1 This was principally directed at the balancing of errors, which is an inherent part of the procedure Some laboratories indicated

other objections of a varied nature. There seemed to be no particular preference among operators for Method 2 over Method 1 but rather, the reverse

4 Several laboratories have referred to the question of when and how the necessary samples of raw soil, cement, and soilcement mixture should be taken We believe that the reliability of results is entirely dependent on the degree of accuracy employed in taking samples, however, the question of sampling technique is considered beyond the scope of this project A brief description of the sampling procedure used on a project this season is given in the appendix

Supplementary criticism offered by two cooperating laboratories after the issuance of the August 1 report, will be found in the appendix to this report.

Alternate methods suggested by Laboratories 19, 13, and 21, when reporting results on cooperative tests, were studied to determine their possibilities and limitations They may all be considered as basically similar to Method 1, although there are some distinct differences. Collectively, we believe that all would prove generally satisfactory, but that each had one, or more features, contributing to increased complexity, loss of time, or reduced application.

- 1 Briefly, the use of platinum by Laboratory 19, was considered undesirable, if avoidable, and it was believed that ignitions to remove organic material, ultimately made solution more difficult, and that, except for special cases, it tended to unduly increase the time consumed by the analysis; however, we believe that the method is accurate
- 2 The method suggested by Laboratory 13 has the advantage of extreme simplicity, yet it does not seem that it could be widely used without adjustment of the sample weight by the individual laboratory to take care of variations of lime content in cements throughout the country It appears, also, that the simultaneous removal of the siliceous material, iron and aluminum hydroxides, and residual material would make the filtration very slow, or impossible in some cases, so that the usefulness of the method as a broad general procedure is debatable.

As a rule, most analysts would feel that there would be a tendency for the iron to occlude some lime—a condition offset in Method 1 by a double precipitation of iron

- 3 An alternate method suggested by Laboratory 21, gave very satisfactory results on the standard samples and appeared to be more rapid than Methods 1 or 2 The principal criticism may be directed at the solvent used, and the necessity for making the solution almost neutral, yet preventing the precipitation of iron The addition of ammonium oxalate, followed in turn by boiling, and conversion from a neutral to a faintly ammoniacal solution, would seem to make the residue on the filter consist of insolubles, hydroxides, and calcium oxalate which would be objectionable to analysts who prefer to titrate a solution in which the solute 1s essentially calcium oxalate. In practice, the iron appeared to be entirely oxidized, thus causing no difficulty, other than possibly increasing the color
- 4 We have made some tests using a method previously submitted by Laboratory 4 Although generally similar to Method 1, the alternate provided for removal of silica by dehydration, and provided for only one precipitation of iron, a procedure subject to some criticism, due partly to increased time, but also for technical reasons Although satisfactory accuracy was obtained, it was believed that the procedure recommended herein would be more generally satisfactory among laboratories throughout the various states

A careful study of the cooperative tests indicated that the results obtained by the participants possess a degree of uniformity higher than might ordinarily be expected (see Tables 2 and 5, Appendix). In general, about two-thirds of the participants were within  $\pm 0.1$  per cent of the general average, considering both samples and both methods. It further appeared that, barring a major error, results within  $\pm 0.5$  per cent could ordinarily be expected, and that much closer results would generally be obtained when the analyst became fully experienced with the method. The uniformity obtained was particularly encouraging in view of the rather wide spread of values obtained by CaO determinations on the soil, cement and soil-cement mixtures, as indicated by the difference between the high and low values (Table 3, Appendix). A similar situation was noted when the high and low Ca values, obtained by Method 2, were examined (see Table 4, Appendix) These variations in determinable factors may be attributed principally to lack of familiarity with the particular procedure, inasmuch as these variations were not generally found in the results submitted by laboratories which have previously conducted such tests in their routine activities

It is important to observe that standard sample B was identical with sample A except that it was hydrated and cured, as in practical stabilization work, and that such hydration resulted in lowering the calculated cement content from the • original 80 per cent to 77 per cent

We have considered this method of test from the standpoint that the determination in question should of necessity be as simple as possible in order that results might be rapidly obtained. It is not expected that such results will be as accurate as those based on the orthodox methods, however the value obtained must be relatively accurate to serve the purpose

After full consideration of the merits of each method, we believe that, over a broad field, Method 1 offers wider application, in that it may be used with confidence with soils having higher line conare usually tent than encountered. Method 2 is often faster when the analyst is familiar with the soil involved, particularly so, when a number of consecutive routine analyses must be made over a project, in which the raw soil is fairly uniform, and low in lime On the other hand, it has been pointed out, in criticism, that much more variation in the results reported by Method 2 might have been obtained if the raw soil sample had contained 12 per cent Ca instead of 02 per cent.

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This brief discussion will serve to illustrate the technical factors which were considered together with suggestions made by cooperating laboratories. This information has been embodied in the following revised test procedure, based on original cooperative test Method 1.

## METHOD FOR THE DETERMINATION OF CEMENT CONTENT OF SOIL-CEMENT MIXTURES FROM CEMENT HARDENED BASES

#### 1. Object

The object of this test is to determine the cement content of samples of soilcement mixtures taken from a soil-cement stabilized roadway. This procedure describes the method of analysis, utilizing samples of the raw soil, cement, and soilcement mixture.

## 2. Apparatus

Glassware—beakers, funnels, graduate cylinders, rods, sample bottles, weighing. bottles, policemen, volumetric flasks, pipettes, burettes, etc.

Analytical Balance, with Class S weights.

Filter paper—Whatman No 1, 11 and 15 cm diameter; Whatman No 41, 15 cm. diameter; and Whatman No 2, 11 or 15 cm. diameter.

Supplementary equipment, such as electric ovens, hotplates, small riffle, No 40 sieve with bottom pan and cover, cast iron mortar and pestle together with a ball mill if possible.

## 3. Samples Required

A properly identified sample of the raw soil which should be entirely representative of the soil phase of the soilcement mixture to be analyzed The raw soil sample should be reduced to 40 mesh size, or finer, and sufficiently oven-dried to remove all free moisture, prior to the analysis.

A properly identified sample of the ce-

ment, which should be entirely representative of the cement phase of the soilcement mixture to be analyzed The cement sample should be oven-dried to remove all free moisture. There should be no lumps.

A properly identified sample of the soilcement mixture to be analyzed. This material should be reduced to 40 mesh size, or finer, and should be oven dried to remove all free moisture

- Note 1 Samples of raw soil and cement should be taken at selected sampling points, just prior to mixing operations Soil-cement samples should be removed later at the same points, when the project has been completed It is extremely desirable that all components be accurately sampled at the same locations, so that the various components will be chemically representative
- Note 2 200 g of each component is enough for a gross laboratory sample This sample is obtained by reducing the field sample in bulk, and, if necessary, in particle size, through the use of drying, riffling, and grinding processes A 25 g sample is removed from the gross sample for the use of the analyst

## 4. Method of Analysis

Weigh out on the analytical balance, the following amounts of raw soil, cement, and soil-cement mixture, receiving the samples in 250 ml. beakers; raw soil 5 g., soil-cement mixture 5 g, cement 1 g. To these portions, add 50 ml. of (1-1) hydrochloride acid, cover and boil lightly for 5 min on the hotplate.

Note In the case of the cement sample, it is usually preferable to first add 40 ml of water then stir to obtain a thorough mixture 10 ml of hydrochloric acid should then be added, followed by sufficient light boiling to obtain decomposition of the cement Vigorous or extended boiling of soil or cement samples is seldom necessary, and often results in much slower filtration

Add 25 ml. of hot water to the beakers, stir and allow to settle momentarily, then decant the contents through a Whatman No 1 filter paper, preferably of 15 cm. diameter. The filtrate should be received in a 250 ml. volumetric flask When the liquid has passed through the filter paper, wash the residue once by decantation, using hot water, then transfer it to the filter, using a stream of hot water. The beaker should be rapidly policed—the loosened material being transferred to the filter paper The material on the filter should then be washed an additional four times, each washing consisting of 10-15 ml. of hot water directed in a stream from the wash bottle Very small amounts of residue will occasionally pass through the filter. These ordinarily may be disregarded.

Note In the case of the soil and soil-cement sample, the bulk of the residue sometimes slows filtration appreciably No difficulty is usually encountered from cement samples, and, as a rule, soil samples may be filtered and washed in less than 30 min Some soil-cement mixtures require more time, but, if this period exceeds an hour, subsequent filtration in similar cases, may be more rapid if a No 41 paper is substituted for the No 1 used Slow filtration, in such cases is generally caused by excessive boiling, resulting in gelation of the silica, which materially retards filtration

When washing has been completed, discard the filter, and dilute the filtrate in the volumetric flask to 250 ml. with cold water. The temperature of the solution should be near the calibration point of the flask. Agitate the flask to thoroughly mix the contents, then remove 50 ml and transfer to the original 250 ml. beaker, using a 50 ml. pipette. Dilute to 100 ml Make the aliquot slightly ammoniacal, boil 1-2 min, and allow the hydroxides to settle.

Note If the samples contain ferrous iron, it is desirable to add a few drops of mitric acid, before precipitation of the hydroxides

Filter the hydroxides through a Whatman No 1 (or 41) filter paper, 11 cm, receiving the filtrate in a 600-ml beaker. Wash the original 250-ml beaker into the filter once with a stream of hot 2 per cent ammonium nitrate, and follow by washing the hydroxide precipitate once

or twice with hot 2 per cent ammonium nitrate. Set the filtrate aside, and place the original beaker under the funnel. Perforate the paper with a rod, and wash the hydroxides down into the original beaker, using a stream of hot 2 per cent ammonium nitrate to remove most of the precipitate from the filter paper. The paper is then treated with 20 ml. of hot (1-3) hydrochloric acid; direct the acid over the paper with a glass rod. Wash the paper several times with hot water, after which it is discarded Dilute the hydroxide solution to 75 ml. Make the solution slightly ammoniacal, and boil 1-2 min Allow the precipitate to settle then decant through a No. 1 paper, as before, receiving the filtrate in the 600-ml. beaker previously set aside. Wash and police the beaker, in which precipitation took place, finally washing the precipitate on the filter three or four times with 2 per cent ammonium nitrate solution. Discard the hydroxide precipitate. Add 2 ml of ammonium hydroxide to the filtrate, which will now have a volume of 250-350 ml Heat the solution to boiling and add 10 ml of hot saturated ammonium oxalate solution. Keep the mixtures near boiling until the precipitate becomes granular, then set aside on a warm hot plate for 30 min. or more Before filtering off the calcium oxalate, verify completeness of precipitation, and make sure that a slight excess of ammonia is present. The mixture is filtered through a Whatman No. 2 filter paper, 11 or 15 cm , or, if preferred, a Whatman No 42, making sure that all precipitate is being retained The beaker, in which precipitation took place, should be efficiently cleaned with a rubber policeman, and the contents transferred to the filter by a stream of hot water. The filter should be washed 8-10 times with hot water (75 ml. max) using a stream from the wash bottle

Note Some analysts prefer to wash the filter 4 times each with 2 per cent ammonia, and hot water, in the order stated The filter paper and contents should be removed and transferred to a 400-ml. beaker containing 125 ml water and 6 ml sulphuric acid If preferred, the precipitate may be removed from the paper, by appropriate means The solution is heated to  $85^{\circ}$ C. and titrated, in the usual way, using a N/10 volumetric solution of potassium permanganate The volume of N/10 permanganate used in titrating the soil, cement and soil-cement mixture should be corrected by a "blank," carried along at the same time.

## 5. Calculation

Using the corrected amounts of N/10 potassium permanganate obtained above, calculate the percentage of CaO in the soil, cement and soil-cement mixture. It should be noted that the aliquots titrated are equivalent to 1 g. of soil or soil-cement, and 0.2 g. of cement.

1 ml. N/10 KMnO<sub>4</sub>=0.0028033 g CaO

Calculate the percentage of cement in the soil-cement mixture by the formula

 $X = \frac{C-B}{A-B}$  100, in which X = per-

centage of cement by weight in the mixture

 $A \doteq$  percentage of CaO in cement, B=percentage of CaO in raw soil.

- and
- C=percentage of CaO in soil cement mixture

A more rapid procedure may be employed by calculating the cement content X by the following formula:

$$X = \frac{(ml \ KMnO4 \ for \ S-C \ mixture) - (ml. \ KMnO4 \ for \ Soil)}{5 \ (ml \ KMnO4 \ for \ cement) - (ml. \ KMnO4 \ for \ Soil)} 100$$

When applying this formula, it is necessary to use a standard N/10 permanganate solution. It is necessary that the same solution be used in titrating all components A blank is not required

When hydrated soil-cement mixtures are being analysed, the value X obtained above, is in terms of hydrated cement Such values may be converted to an approximate equivalent of dry cement by multiplying X by the factor 1 04.

In conclusion, we believe that the determination of the cement content of soilcement mixtures through the use of the method just described will be found entirely practicable, and that good results will be obtained when it is properly used. This belief seems entirely justified by the cooperative tests previously described, and, in addition, by our experience through the extensive use of this method in testing a large number of samples obtained from a soil-cement project recently constructed Full details of the latter will be found in the appendix to this report.

We recommend that this method be considered for adoption as a standard method for the "Chemical Determination of Cement Content in Soil-Cement Mixtures from Cement Hardened Bases."

It is further believed that such determinations of the cement value on completed projects will be of material assistance in the development of better construction methods for use in soil-cement stabilization.

#### APPENDIX

#### SAMPLING AND TESTING, PROJECT UNDER CONSTRUCTION

During the progress of the investigation of "The Chemical Determination of the Cement Content of Soil-Cement Mixtures from Cement Hardened Bases," we were fortunate to have under construction approximately 68 miles of soil-cement stabilization constructed by the Mixed-in-Place method.

Soil Groups A-2, A-3 and A4-2 were found in place in the completed roadway. Wet-dry and freeze-thaw durability tests had been made to determine the cement content required in accordance with A S T M 559 and 560-40 T. It was recommended that 8, 9 and 10 per cent cement by volume be placed

During the early stages of construction, 14

sampling locations were selected, which were intended to fully represent the several typical soils Instructions were furnished the field forces, for securing samples of raw soil from the roadway and of the cement when applied, and samples of cores from the final mixture

Near the end of the pulverization process, soil samples were taken at each of the following selected locations

Five samples were taken on a diagonal through the center line from -10 ft on one edge to +10 ft on the opposite edge One sample was taken at the center line, one 18 in from each edge and one at each quarter point A shovel full of soil through the depth of the pulverized material was taken at each of these points and these were combined, quartered and split to obtain about 05 gal of soil sample ~

Cement samples were secured, at the same station, by selecting a portion from each of five bags when dumped on the roadway These individual cement samples were combined, quartered and split to obtain a composite sample of 0.5 lb

These preliminary raw soil and cement samples were properly identified and forwarded to the materials laboratory

About 30 days after final completion of the project, cores were drilled at the same locations to check the thickness of the completed stabilization, to determine the cement content both as to position in the roadway and at various depths and for information on the compressive strength of typical cores Three or more cores were taken at the exact points previously sampled Each was properly identified and numbered and the top properly marked for later identification. In all, 47 cores were drilled and delivered to the laboratory for further study and test

The raw soil samples submitted to the laboratory were air-dried, broken up by hand, then ground in a ball mill until they passed the 40mesh sieve This sample was thoroughly riffled, split and reduced to a laboratory sample of 200 g The cement sample was riffled and split to obtain a 200-g. sample The cores, when ready for determination of the cement content, were placed in the laboratory rock crusher for preliminary reduction, afterwards air-dried to remove most of the moisture This reduced mixture was ground to 40-mesh size in the ball mill and was then riffled and split to a laboratory sample of 200 g. Seven cores were selected and divided into portions representing the top, middle, and bottom of the stabilized base Each of these portions was handled separately. These laboratory samples consisting of 61 soil-cement, 14 cement and 14 raw soil samples were riffled and split to obtain 25-g samples for the chemists'

use, and the latter were then dried over night before chemical analysis was started

The method used in the chemical analysis was that to be recommended to the Highway Research Board in the preceding report The soils were analyzed first, then the cement, and finally the mixtures, after which the cement content of each was calculated The results, as taken from our record of the project, are attached Analyses were conducted in duplicate and were repeated when any variation between duplicates was enough to cause a change in the tenth per cent digit of the cement value This was only necessary in three or four cases

Examination of Table 1 shows considerable variation between the values in column 7, the "per cent cement designed and placed, by weight," and the last column, the "Cement Content, by Weight, in the Completed Road" Appreciable differences in the cement content are indicated in the roadway within short distances Detrimental variation between the top, middle, and bottom of the treatment was found in only two instances as in core 12-E and 13-A

We believe the cement contents shown herein are correct and represent the actual cement in the completed roadway. There is no allowance for hydration in these results as the designed and placed percentage by weight is calculated for the dry weight of the soil on the basis of 4-hr delayed moisture density curves Our reliance on these results is fully justified by the condition of the road after some five months service under traffic. Inspection shows plainly the differences in erosion at these locations, directly comparable to the percentage of cement herein determined.

In this case, the determination of the cement content definitely indicates one of the discrepancies or shortcomings of the finished roadway It is also very evident that, if chemical analyses had been made immediately following the completion of each day's work, fluctuations in cement content would have been discovered at a time when corrective measures could have been most effectively applied

A well planned investigation entirely suitable, to this purpose is not unduly time-consuming, and we believe it is well worth the effort. In the investigation of this project, the processing of samples required the services of two operators for about a week, while the chemical work was accomplished in approximately 10 days, with two and three operators. In the average project activity, the time required would likely be reduced, inasmuch as special care was observed in this case, to fully test the suitability of procedure involved in the investigation in progress for the Highway Research Board.

## SOILS

## TABLE 1

# **Record of Cores and Determination of Cement Content (Method 1)**

		Compress			Percentage	centage of cement			
Core No	Station	Location	Thickness	sive strength	Designed a	nd placed	0		
				sq in	Volume	Weight	analysis by	wt	
1-A	140 + 00	3 Lt	in 60	_	9	- 73	Top 1/3 Mid 1/3 Bot 1/6	78 78 83	
1-B 1-C	140 + 00 140 + 00	7 Lt 3 Rt	57 55	337 327	9 9	73 73		5760	
1.0	$140 \pm 00$	9 Rt			9	73		63	
2-A	$158 \pm 00$	10 Rt	1		. 8	68		37	
2-B	$158 \pm 00$	C L			8	68		86	
2.0	$158 \pm 00$	10 L t	5.6		Ř	68		48	
3.4	$178 \pm 00$				Ř.	70		81	
3-A 2 B	$178 \pm 00$		60		8	70		6.8	
3-0	$178 \pm 00$	6 P+	65		8	70		7 35	
3-0	1/0 + 00	1 D+		200	0	65		7 2	
4-A 4-B	$190 \pm 00$ $190 \pm 00$	8 Lt	59	352	8	65	(Top. 14	52	
4-C	190 + 00	8 Rt	55		8	65	$\begin{cases} 10p & 7s \\ M_1d & \frac{1}{5} \\ Bot & \frac{1}{6} \end{cases}$	55	
5-A	216 + 00	CL	56		8	67		70	
5-B	216 + 00	9 Lt	4 7		8	67	1	58	
5-C	216 + 00	9 R t	60		8	67		51	
6-A	235 + 00	CL	60	531	9	72		70	
6-B	235 + 00	10 Lt.	65		9	72		62	
6-C	235 + 00	5 Lt.	5 2		9	72		57	
6-D	235 + 00	5 Rt.	58	503	9	72		74	
6-E	235 + 00	10 Rt	67	451	9	72		49	
7-A	256 + 00	C. L.	49		9	72		43	
7-B	256 + 00	6 Lt	53	674	9	72		90	
7-C	256 + 00	6 Rt	55	635	9	72		75	
8-A	318 + 00	CL	52	405	10	81	•	68	
8-B	318 + 00	7 Lt.	66	527	10	81		55	
8-C	318 + 00	7 Rt	48	655	10	81		59	
9-A	332 + 00	CL.	50		9	75		89	
9-B	332 + 00	8 Lt	53		9	75	(Top. 14	68	
9-C	332 + 00	8 Rt.	55		9	75	Mid. 1/3	8 1 8 3	
10-A	404 + 00	CL	60		9	73	1,200 /8	31	
10-B	404 + 00	10 Lt	63	360	9	73		71	
10-C	404 + 00	10 Rt	60	569	9	73		63	
11-A	420 + 00		59	266	9	73		74	
11-B	420 + 00	8 Lt	60	382	9	73	(Top 1/4	94 76	
11-C	420 + 00	8 Rt	62		9	73	Mid 1/3 Bot 1/4	7373	
12-A	454 + 00	CL.	30		9	73	/8	4 1	
12-B	454 + 00	3 Lt	4 5	581	9	73	1	61	
12-C	454 + 00	9 Lt	75		9	73		81	
12-D	454 + 00	3 Rt	45	617	9	73	1	70	
	1		1	1	1				

# Completed Soil-Cement Roadway (6 in x 24 ft)

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Core No		Station Location	Thickness	Compres-	Percentage of cement			
	Station			sive	Designed	and placed	Comp road chem	
				sq in	Volume	Weight	analysis by wt	
12-E	454 + 00	9 Rt	<sup>1n</sup> 6 5		9	73	Top 1/5 6 8 Mid 1/5 6 6 Bot 1/5 5 6	
13-A	494 + 00	C. L	.63		10	82	$\begin{cases} Top \frac{1}{3} 56 \\ Mid \frac{1}{3} 55 \\ Bot, \frac{1}{4} 44 \end{cases}$	
13-B	494 + 00	12 Lt.	7 2	422	10	82	64	
13-C	494 + 00	12 Rt.	75	322	10	82	7 0	
14-A	508 + 00	CL.	61		10	83	11 1	
14-B	508 + 00	12 Lt	62		10	83	$\begin{cases} Top \frac{1}{5} 10 9 \\ Mid \frac{1}{5} 10 7 \\ Bot \frac{1}{5} 11 1 \end{cases}$	
14-C	508 + 00	12 Rt.	•		10	83	5 6	

## TABLE 1-CONTINUED

	Percentage of cement found									
Sample*		A		В						
Method	1	1 2		1	2	3				
Laboratory No 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24	NRR 82 7780 8080 80 82 81 81 7280 82 80 7682 80 7680 80 NRR 81 80 80 80 NRR 81 80 81 81 81 81	NRR 80 78 79 80 <sup>b</sup> 82 <sup>b</sup> 78 <sup>a</sup> 79 91 79 79 80 79 80 79 78 <sup>b</sup> NRR 80 NRR 79 80 80 80 80 80 80	79 74 80 77 , 80	NRR 80 77 78 78 79 79 79 79 79 79 79 79 79 79 76 79 NRR 78 78 78 77 86 <sup>bc</sup> 78 78 78	NRR 79 77 77 80 <sup>b</sup> 80 75 <sup>6</sup> 77 78 75 78 75 78 71 77 <sup>b</sup> NRR 77 NRR 78 77 NRR 78 78 77	75 72 76 77 8				
Average <sup>d</sup> Hıgh Low	8 0 8 8 7 2	80 91 78		78 86 72	77 80 71					

TABLE 2 **COOPERATIVE TEST RESULTS ON STANDARD SAMPLES\*** 

Note

<sup>a</sup> Average of three results

<sup>b</sup> A revised value adjusted to conform to rule 4, Supplementary Instructions

<sup>c</sup> It is not clear whether outlined method was employed

(NRR) No report received, as of August 1, 1941 <sup>d</sup> Eliminating high and low results

<sup>o</sup> The original mixture contained 8% cement, by weight Samples A were withdrawn from this dry mixture. The remainder was compacted at optimum moisture and cured, then reprocessed and prepared as Sample B

\* This table taken from "Report on Cooperative Tests" issued to participants on August 1, 1941. Revised to date.

Laboratory No	% CaO soil	% CaO cement	% CaO ''A''	% CaO ''B''	% Cement "A"	% Cement "B"
1					NRR	NRR
2	0 10 <sup>b</sup>	63 65 <sup>b</sup>	5 35b	5 20 <sup>b</sup>	8 2	8.0
3	0 15	64 10	5 10	5 10	77	77
4	0 25	64 30	5 40	5 25	80	78
5	0 30	63 80	5 40	5 20	8.0	77
6	0 30	63 60	5 <u>4</u> 0 <sup>b</sup>	5 35 <sup>b</sup>	8 Ob	8 05
7	0 25 <sup>b</sup>	63 45 <sup>b</sup>	5 45 <sup>b</sup>	5 25 <sup>b</sup>	8 2 <sup>b</sup>	79
8	0 20ª	63 35ª	5 30ª	5 20ª	8 1ª	7 91
9	0 10	64 00	5 30	5 15	81	79
10 ,	1 60 <sup>b</sup>	71 35	6 60 <sup>b</sup>	6 65 <sup>b</sup>	72	72
11	0 30	64 35	5 45	5 25	80	77
12	020	61 95	5 25	5 10	82	79
13	0 35	63 85	5 40	5 25	80	77
<b>14</b>	0 35 <sup>b</sup>	62 95 <sup>b</sup>	5 10 <sup>b</sup>	5 15 <sup>b</sup>	76	76
15	0 25 <sup>b</sup>	64 00 <sup>b</sup>	5 40 <sup>b</sup>	5 30 <sup>b</sup>	8 Op	7 9b
16					NRR	NRR
17	0 35 <sup>b</sup>	62 70 <sup>b</sup>	5 45 <sup>b</sup>	5 25 <sup>b</sup>	8 1 <sup>b</sup>	7 8 <sup>b</sup>
18	0 35	65 10 <sup>b</sup>	5 55 <sup>b</sup>	5 35 <sup>b</sup>	80'	78
19	0 20	64 35	5 30	5 20	80	77
20					NRR	NRR
21	0 30	64 85	5 50	5 30	80	77
22	0 25 <sup>bc</sup>	64 15 <sup>be</sup>			8 8b	8 6 <sup>b</sup>
23	0 20	62 90	5 30	5 10	81	78
24	0 25	63 25	5 35	5 15	81	78
Average <sup>d</sup> .	0 26	63 82	5 38	5 23	8.0	78
ligh	1 60	71 35	6 60	6 65	88	86
low	0 10	61 95	5 10	5 10	72	72

TABLE 3 COOPERATIVE TEST RESULTS ON STANDARD SAMPLES\* Data by Method 1

Note:

.

<sup>a</sup> Average of three

<sup>b</sup> A revised value adjusted to conform to rule 4, Supplementary Instructions.

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\* It is uncertain whether outlined method was employed

(NRR) No report received, as of August 1, 1941. <sup>d</sup> Eliminating high and low results.

\* This table taken from "Report on Cooperative Tests" issued to participants on August 1, 1941. Revised to date.

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		Sampl	e "A"		Sample "B"						
Laboratory No	Cement control, %	Ca found, grams	Ca-cement factor	Cement found, %	Cement control, %	Ca found, grams	Ca-cement factor	Cement found, %			
1				' NRR				NRR			
2	81	0 1831	0 4560	80	79	0 1799	0 4543	79			
3	77	0 1656	0 4240	78	77	0 1641	0 4240	77			
4	79	0 1822	0 4652	78	78	`0 1788	0 4645	77			
5	80	0 1765	0 4464	79	7 8 <sup>b</sup>	0 1706	0 4445	77			
6	8 Ň	0 1797	0 4475	8 0 <sup>b</sup>	80	0 1781	0 4475	8 Ob			
7	80	0 1844	0 4488	8 2 <sup>b</sup>	80	0 1795	0 4488	80			
8	7 58	0 1685ª	0 4300ª	78ª	7 5ª	0 1615ª	0 4291ª	7 5ª			
ŏ	80	0 1790	0 4532	79	80	0 1736	0 4532	77			
10	75	0 1853	0 4060	91	75	0 1796	0 4060	78			
11	80	0 1830	0 4632	79	75	0 1715	0 4560	75			
12	80	0 1780	0 4525	79	80	0 1758	0 4525	78			
12	7 8	0 1855	0 4626	80	78	0 1804	0 4626	78			
14	7 5	0 1798	0 4560	79	75	0 1613	0 4560	7 1			
15	7 010	0 2445	0 6193	7 8b	7 9b	0 2380	0 6193	7 7b			
16		0 2110		NRR				NRR			
17	1	· ·		c				c			
19		]		NRR		1		NRR			
10	90	0 1848	0 4504	8.0	75	0 1776	0 4602	77			
19	00	0 1040	0 1001	NRR				NRR			
20	90	0 1874	0 4764	79	80	0 1851	0 4764	78			
21		0 10/4	0 1/01	c				C			
22	0 0 0	0 1790	0 4473	1 80 1	77	0 1707	0 4379	78			
23		0 1602	0 4216		7 5	0 1657	0 4290	77			
24	00	0 1092	0 4210	00							
Amoread	0.0	0 1803		8.0	78	0 1745		77			
Average"	00	0 2445		91	80	0 2380		80			
rign		0 1656	· ·	78	7 5	0 1613	1	7 1			
LOW	1 13	0 1030		1							

**TABLE 4** COOPERATIVE TEST RESULTS ON STANDARD SAMPLES\* Data by Method 2

Note

<sup>a</sup> Average of three.
<sup>b</sup> A revised value adjusted to conform to rule 4, Supplementary Instructions.

<sup>c</sup> Did not report on this method.

<sup>d</sup> Eliminating high and low results.

(NRR) No report received, as of August 1, 1941.

\* This table taken from "Report on Cooperative Tests" issued to participants on August 1, 1941

#### SUPPLEMENTARY CRITICISM <sup>1</sup>

Laboratory 4 comments that "apparently no laboratory except our own is disturbed by the fact that the co-operative tests were made on a soil with low calcium content We still hold the opinion that tests of such low calcium soil showed the usefulness of Method 2 without revealing its limitations as they should have been revealed had a high calcium soil been used in the tests We respectfully suggest that some of the other laboratories make trial analyses of soil-cement mixtures in which the raw soil is fairly high in calcium, such as we described in our first report, and that their findings be Most of the participating laboratories agree with this contention This method follows fairly closely to A. S. T M C114-40 for the determination of calcium oxide in portland cement The final determination of calcium oxide, however, is done volumetrically The alternate method suggested by Laboratory 19 appears to be satisfactory but requires more time than Method 1 Unless it can be shown that organic matter in the soil interferes with the calcium oxide determination, the expenditure of the additional time required seems unjustified. This method calls for three washings of the calcium oxalate precipitate with 01 per cent ammonium

TABLE 5 ,
COOPERATIVE TEST RESULTS ON STANDARD SAMPLES*
Distribution of Results

	. Sample		A .	В		
_	Method	1	2	1	2	
_		%	% -	%	%	
1	Laboratories varying from average cement value $\pm 1\%$ or less	66 6	66 6	76 2	66 6	
2	Laboratories varying not more than $\pm 2\%$ from average cement value	81 0	94 4	90.5	83 3	
3.	Laboratories varying not more than $\pm$ 5% from average comment value	90.5	04.4	90.5	04.4	
4	Laboratories varying more than $\pm$ 5% from average	, , , , , , , , , , , , , , , , , , ,				
	cement value	93	30	93	30	

\* This table taken from "Report on Cooperative Tests" issued to participants on August 1, 1941 Revised to date

made available to the participants in the cooperative projects Several of the laboratories comment on the importance of sampling Is a "sampling procedure" considered to be a part of this investigation? Since we suspect that sampling procedure is more critical than the difference between analytical procedures, it will be very interesting to see the sampling procedures proposed for use with the Chemical Methods."

Laboratory 2 adds that "an examination of the data indicates that, with the exception of a few erratic results, both methods give fairly accurate and reproducible results. We personally prefer Method  $1 \ldots$  As we feel that it is the more accurate of the two methods.

<sup>1</sup>Submitted after review of the August 1, 1941 report

oxalate followed by three washings with hot water, whereas most procedures call for at least six washings with hot water to remove the soluble oxalate The method proposed by Laboratory 13 is considered shorter than Methods 1 or 2 and appears to possess some merit as a rapid control-procedure Pending additional work in our laboratory we could not definitely comment on the accuracy or reproducibility of the method The alternate method proposed by Labortory 21 is shorter than Methods 1 or 2 but longer than the alternate method of Laboratory 13, and does not appear to have any advantages over the Laboratory 13 method This method presents the difficulties of handling and washing a combined oxalate and R<sub>2</sub>O<sub>2</sub> precipitates and titrating an iron colored solution with permanganate"