HIGHWAY RESEARCH REPORT

DEVELOPMENT OF A METHOD FOR DETERMINING LIME CONTENT OF TREATED SOILS

67-37

STATE OF CALIFORNIA

TRANSPORTATION AGENCY

DEPARTMENT OF PUBLIC WORKS

DIVISION OF HIGHWAYS

MATERIALS AND RESEARCH DEPARTMENT

RESEARCH REPORT

NO. M & R 633295

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

February 1, 1968

Lab. Authorization No. 633295

Mr. J. A. Legarra
State Highway Engineer
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Dear Sir:

Submitted for your consideration is:

A

REPORT

ON

DEVELOPMENT OF A METHOD FOR DETERMINING
LIME CONTENT OF TREATED SOILS

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Very truly yours,

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REFERENCE: E. Zube, C. Gates and M. Hatano, "Development of a Method for Determining Lime Contents of Treated Soils", State of California, Department of Public Works, Division of Highways, Materials and Research Department. Research Report 633295-1, June 1967.

ABSTRACT: Research was directed towards modifying Test Method No. Calif. 338 entitled, "Determination of Cement Content in Cement Treated Aggregates by the Method of Titration". While this method is usable for lime treated soils, some improvement in the method was required to refine the test.

Some of the modifications made were: shortened the time required for test; determined the effect of curing time; used a flocculating agent to more easily detect the titration end point, developed a method for more accurately establishing a calibration curve and eliminated use of a chart for the initial and secondary addition of acid.

A brief statistical study performed with the new method indicated that the test is reliable and precise.

KEY WORDS: testing, testing methods, field tests, mixing efficiency, lime, lime contents, liming of soils.

The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the U. S. Department of Transportation, Federal Highway Administration, Bureau of Public Roads.

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INTRODUCTION

During the past few years, the increased use of lime for treating poor quality soil for use in the highway structural section has necessitated the development of a reliable rapid field test to determine lime content of the mixed material.

The stabilization of soils with lime essentially started in 1948 when California became interested in lime treatment and constructed two small experimental highway projects in the Sierra Nevadas, one near Truckee and the other near Georgetown.* The success of these projects led to the use of lime on other small projects during the next decade. During the period around 1960 to 1965, various organizations in the State started to use lime extensively for treating clay soils.

All of the lime jobs consisted of road mixing the in-place or imported material. It was possible to calculate the amount of lime going into a job but it was very difficult to determine if the lime was being uniformly distributed and mixed. The lack of a reliable rapid field test for determining percent lime in lime treated soils made it extremely difficult to control the different, important variables which contribute to uniform mixing.

Around 1957, a test entitled "Determination of Cement Content in Cement Treated Aggregate by the Method of Titration" (Test Method No. 338)** was developed in California. In 1960, this test was included in the California Standard Specifications. While this method is usable for lime treated soils, the test was designed primarily for determination of percent cement in freshly mixed cement treated aggregates.

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*These projects are described in an article entitled "Experimental Use of Lime for Treatment of Highway Base Courses", by E. Zube, published in ARBA Bulletin No. 181.

**State of California, Department of Public Works, Materials Manual, Testing and Control Procedures, "Determination of Cement Content in Cement Treated Aggregates by the Method of Titration"(Test Method No. 338).

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With the widespread use of lime and increasing use of the test, it became apparent that some improvement in the test was necessary. Problems such as time of titration, quantity of initial and subsequent additions of acid, establishing a calibration curve and use of a flocculant were some items that needed to be modified or changed. It is the purpose of this report to describe the research work performed and present the test method which was developed.

CONCLUSIONS

A suitable rapid field test for determining percent lime in a lime treated soil was developed. Basically, the test consists of adding hydrochloric acid in small controlled amounts to a sample of aqueous solution from a lime treated soil.

The time required to perform this test is 45 minutes not including sampling and sample preparation time. Four specimens can be tested at one time.

A statistical study indicated that the test is able to determine percent lime in a lime treated sample to within $\pm$ 0.38 percent of the actual percentage 95 percent of the time. These tests were performed on laboratory prepared samples.

The test method developed is attached to this report as Appendix A and was placed in the Materials Manual as Part IV of Test Method No. 338 on August 15, 1967.

Background Information

Test Method No. Calif. 338 basically consists of two test methods, namely the Acid-Base and the Constant Neutralization. The method most commonly used for determining percent lime in lime treated soils is by the "Constant Neutralization" method. The test is based upon the continuous neutralization of an aqueous solution from the lime treated soil for a specified time interval. This is accomplished by adding sufficient hydrochloric acid to just neutralize the OH ion which is continuously being liberated during the hydration of the lime. Phenolphthalein is added to the solution to indicate the end point of titration. The amount of acid used is proportional to the lime content of the treated soil sample. Determination of percent lime in a field treated sample is performed by applying the test result to a calibration curve established by testing a sample made up of known amounts of lime, water and soil.

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Since the Constant Neutralization Test is presently being used in California in almost all cases, it was felt that modification of this method would be the most practical from an economic standpoint. It would also mean that training of technicians would be easier since the test is similar to the present Constant Neutralization Test for cement and all of the present equipment could be used. Therefore, research efforts were directed towards modifying the Constant Neutralization Test for cement.

DISCUSSION

Development and modification of the Constant Neutralization Test method for lime involved investigating the various problems encountered through experience. Each problem was treated separately with the overall effect being considered. Therefore, the problems, research performed and recommendations are reported as individual studies but not necessarily presented in the order studied.

I Development of a suitable method for establishing a calibration curve.
Problem: A straight line calibration is used for testing cement treated soils. The calibration curve for lime is curvilinear.
Objective: Develop a simple method for establishing a calibration curve for use with testing of lime treated soils.

II Determine effects of loose curing time.
Problem: Lime treated soils are initially mixed and often allowed to loose cure up to two days before final mixing. This introduces some error into the method if tests on both the calibration curve and field samples are not performed with the same curing period.
Objective: Develop a suitable method to control this variable.

III Development of a suitable method for determining the end point of titration.
Problem: Soils normally of the clay type are lime treated. This causes the aqueous solution of lime and soil to become murky during the test which results in difficulty of determining the end point of titration.
Objective: Experiment with various chemicals and methods to clarify the murky solution.

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Discussion and Analysis:

The test data on Figures 1, 2, 3, and 4 indicates curing time and amount of lime have the most effect on the calibration curve and caused it to be curvilinear.

Tests were performed on 5 additional soils with various curing times and 5% lime (Figure 5). The data shows the same curvilinear relationship between percent lime and milliliters of acid as the previous tests. Again curing time and percent of lime had the most effect on the calibration curve.

In order to attain optimum accuracy from this test, the data show that a number of tests at various percentages of lime must be performed in order to establish an accurate calibration curve. Since this adds to the time and difficulty in performing the test, an alternate procedure was established.

Present California specifications permit lime content variations during construction, not to exceed plus or minus one percent of the planned lime content. Therefore, the calibration points are established at plus and minus one percent of the planned lime content. This gives accurate tests at the specification limits and still makes it suitable as a rapid control test. Any minor errors, due to curvilinearity between the calibration points, does not affect construction compliance with specifications. However, if further refinement is desired, a calibration curve can be established at the planned lime content in addition to plus and minus one percent of the planned lime content.

Deviations in curing time between field and calibration samples can cause errors if corrective adjustments are not made. Methods of construction will usually determine the necessary curing time for field samples. The curing time for the calibration samples should approximate this same time. However, this is not always possible. Reasonable differences in time between the field and calibration curing is permissible when the following correction is applied:

1. Establish a curing time versus grams of hydrochloric acid curve using the planned lime content, aggregate and water from the particular job (Figure 6 showing a planned lime content of 4%).

2. Establish a calibration curve with 3 and 5% lime by preparing laboratory samples having approximately the same curing time as the field control sample. This would be an estimate based on field conditions. The example (Figure 7) shows a calibration curve for a 16 hour curing period.

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3. If the unknown field control sample actually had a 20 hour loose curing time, then the graph (Figure 6) shows a correction factor of +2.0 grams (curve values for 47.5-45.5 grams).

4. If the unknown field sample at 20 hours curing time required 48 grams of acid to titrate, then the corrected grams of HCl would be 50 (48+2.0) giving 4.0 percent lime as shown on Figure 7.

The curing time versus grams of acid correction curve need only be determined once during a contract unless there is a change in the source of material.

Figures 1 through 5 indicate that time is critical during the first hour. It is therefore advisable to wait until at least one hour has elapsed since mixing before testing field samples.

For materials that are completely mixed and tested during the same day, a 4 or 5 hour curing time for the calibration curve is normally adequate for all field sample testing that day.

For materials that are mixed over a period of two days and testing is made during the second day, one curing period for the calibration sample extending into the second day is normally adequate for all field samples tested during the second day.

The closer the time between calibration test and field sample test, the less correction will be needed and the more accurate will be the results. Plan the calibration time so that it will fall towards the middle of or at least near the curing time for the field samples.

Research on Problem III - Development of a suitable method for determining the end point of titration.

Two chemicals were used as flocculating agents to settle the clay particles and clear the solution. The first chemical was sand equivalent stock solution (calcium chloride, glycerine and formaldehyde) and the second was Dow Chemical's Separan NP10. These solutions were used individually in varying amounts and concentrations.

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Discussion and Analysis:

Tests were performed with both chemicals and three operators. The operators were in aggrement that the clarity of the lime, soil, and water solution and determination of the titration end point was better discernible when using the Separan NP10. Therefore, all further experiments were performed with Separan NP10.

Separan NP10 is a commercial item and comes in a powder form. The manufacturer recommends mixing NP10 with water to give a 0.05% solution. This concentration appears adequate for the titration test.

In general, the NP10 appeared to slow down the reaction and less acid was used as compared to a sample tested without NP10.

One disadvantage of NP10 is that vigorous agitation causes some dispersion in the flocculated particles. This again results in a murky solution.

Three methods of adding NP10 were explored in detail.

Method 1: Fifty milliliters of NP10 solution and 200 milliliters of water were added to the lime treated soil at the beginning of the test.
Result: The clay particles were flocculated but became dispersed towards the end of the test. This is undesirable since the red color for detecting the end point of titration is pink towards the end of the test and is difficult to see. This method was abandoned as not being suitable.

Method 2: 50 milliliters of NP10 were added after 30 minutes of testing time had elapsed.
Result: A large initial addition of acid was needed. Very small amounts of acid were required in the latter stages of the test. The solution of lime soil and water was relatively clear at the end of the test.

Method 3: 50 milliliters of NP10 solution and 200 milliliters of water were added to the lime treated soil at the beginning of the test. An additional 50 milliliters of NP10 solution were added after 25 minutes of testing time had elapsed.
Result: Frequent, small additions of acid were required during the first 25 minutes. The solution was clear and only small amounts of acid were required during the next 15 minutes.

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The following Table A show the test data on Methods 2 and 3:

TABLE A

Sample No. & Lime	Mls. Acid Used Method 2	Average	Mls. Acid Used Method 3	Average
66-3723 (16 hr. cure) (4% of 73.2% $Ca(OH)_2$)	51.3 52.7	52.0	49.7 49.0	49.4
66-3723 (1 hr. cure) (4% 91.2% $Ca(OH)_2$)	65.3 64.9	65.1	61.1 61.0	61.1
66-1413 (18 hr. cure) (4% 73.2% $Ca(OH)_2$)	50.1 48.4	49.3	45.7 45.1	45.4
66-1413 (18 hr. cure) (4% 91.2% $Ca(OH)_2$)	64.5 63.0	63.8	64.1 61.3	61.4
64-3136 (1 hr. cure) (4% 88.3% $Ca(OH)_2$)	61.0 62.2	61.6	57.3 58.2	57.8

The total amount of acid required for method 2 and 3 varied. Method 3 consistently used less acid and had a smaller spread between the pairs of tests performed in 4 out of 5 samples. Consequently method 3 was adopted as being the most suitable.

Although the NP10 flocculated the clay particles, it was still difficult to see the titration end point. A strong light was placed above the plastic containers and this seemed to help. However, it was determined that the best solution to this problem is to place a good light source behind the plastic containers (Figure 8). Each field kit would have this light source as part of its standard equipment.

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STATISTICAL STUDY

A brief statistical study was made to indicate the reliability of this test. Figure 9 shows a scatter diagram of tests performed on samples with various known percentages of lime versus percent of lime determined by testing. The data indicate that on laboratory prepared samples, one operator was able to determine lime content within ±0.38% of the planned lime content approximately 95% of the time. The coefficient of correlation was 0.995 (1.0 is direct correlation) for this series of tests.

Figure 10 shows a control chart of averages which gives an indication of the centering process and Figure 11 shows a control chart of ranges which gives an indication of the dispersion of the process. These two charts are used as statistical tests to give an indication of the reliability and precision of the test. The data is shown on Table B.

From the control chart of averages (Figure 10) it is evident that the test is reliable with very narrow control limits. The overall test averages do not deviate more than 0.1 percent from the planned lime content.

The control chart of ranges (Figure 11) shows a maximum dispersion of 0.3% between four specimens tested for each subgroup. The control limits are 0.5% or less.

For use as a rapid field construction control test, the charts indicate that basically, the test is reliable and precise.

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TABLE B

Sample No.	% Lime Planned		
	2	4	6
	By Test		
A1	2.1	4.0	6.1
2	2.2	4.1	6.1
3	2.1	4.1	6.1
4	2.1	4.0	6.2
B1	1.9	3.9	6.2
2	1.9	4.0	6.0
3	2.0	4.0	6.0
4	2.0	4.1	6.0
C1	2.1	4.1	6.1
2	1.9	4.0	6.2
3	2.0	4.1	6.0
4	2.0	4.1	6.2
D1	1.8	3.9	5.9
2	1.8	3.9	5.9
3	1.8	3.9	6.0
4	1.8	3.9	6.2
E1	2.1	3.9	6.1
2	2.1	4.1	6.0
3	2.1	4.0	5.8
4	2.0	4.0	5.8

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COST OF EQUIPMENT

At the present time, Test No. Calif. 338 entitled, "Determination of Cement Content in Cement Treated Aggregate by the Method of Titration", requires about $800 worth of equipment to implement the test in the field.

While research was being performed on this project for lime treated soils, some new, recently manufactured equipment was utilized and substitutes found for other items. This brought the cost of equipment down to about $200. Smaller agencies, such as city and county road departments, with limited funds will now be able to purchase this equipment and perform the test. Those agencies that are presently performing the titration test for cement with the more expensive equipment will be able to continue using the same equipment for lime treatment.

The following is a list of equipment with the approximate cost of each item:

		Approximate Cost
1.	Device for permitting slow, easily controlled addition of acid	$ 10.00
2.	Burette stand and clamp	4.00
3.	Ten 2-quart polyethylene containers	10.00
4.	Two 5-gallon plastic carboys	16.00
	Misc. tubing, stoppers, clamps, etc.	5.00
5.	Glass dropping bottle	.50
6.	Four stainless steel stirring rods	2.50
7.	3/8" sieve 12" diameter	17.50
8.	1½" sieve 12" diameter	20.00
9.	O Haus Dial-O-Gram (1600)	50.00
10.	O Haus 505M (Powder Scale)	22.50
11.	Phenolphthalein solution 1 qt.	3.00
12.	HCl acid 4-6 lb. bottles	10.00

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13. Fluorescent light		$ 25.00
14. 4-50 Ml. graduates		10.00
15. Separan NP10 powder (5 lbs.)		15.00*
	Total	$221.00

*Only about 0.5 pound is needed to start the test since 0.5 gram is used for each 1000 Mls of water. However, 5 pounds is the minimum amount that can be obtained commercially.

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Figure 1

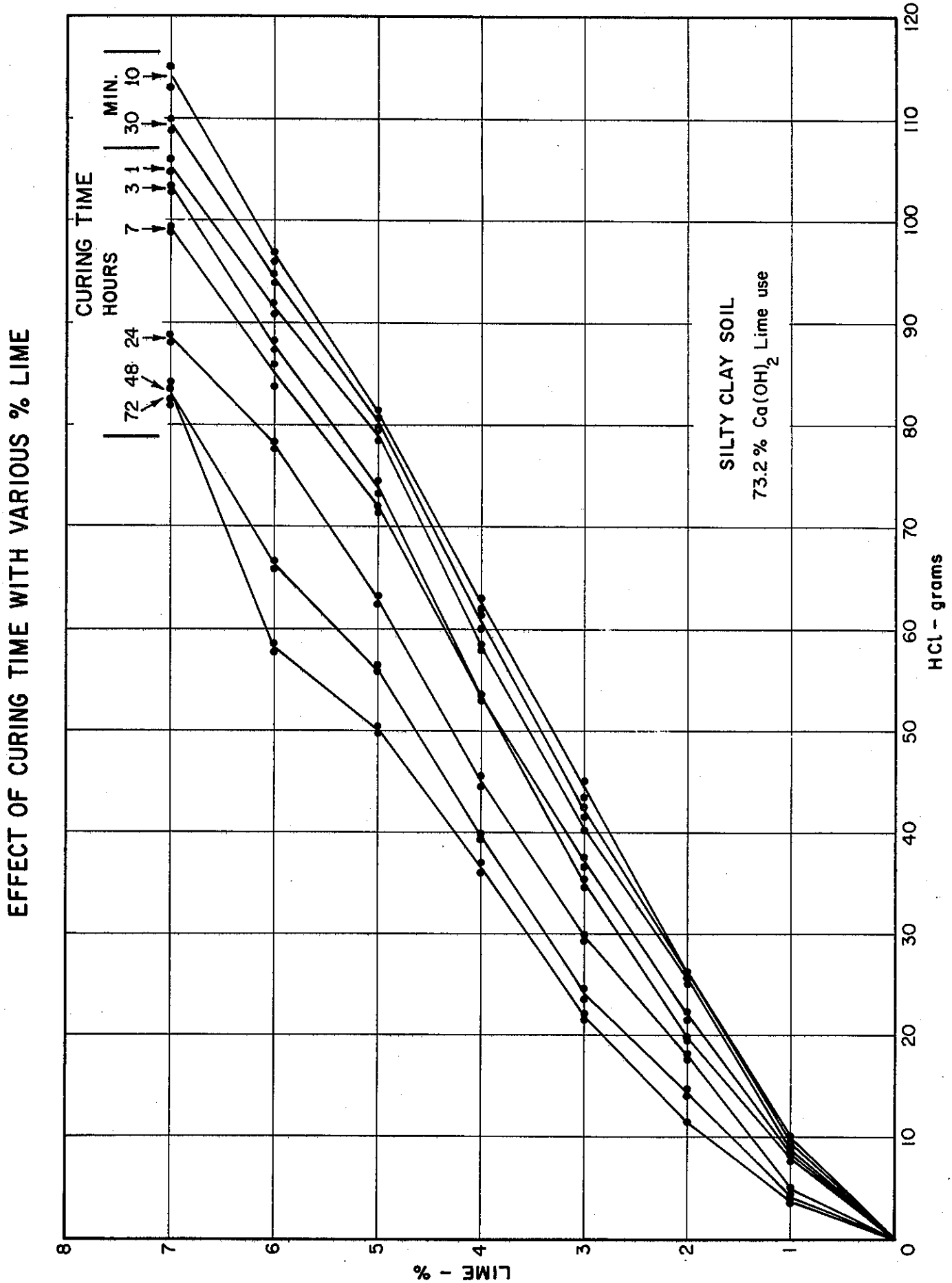

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Figure 2

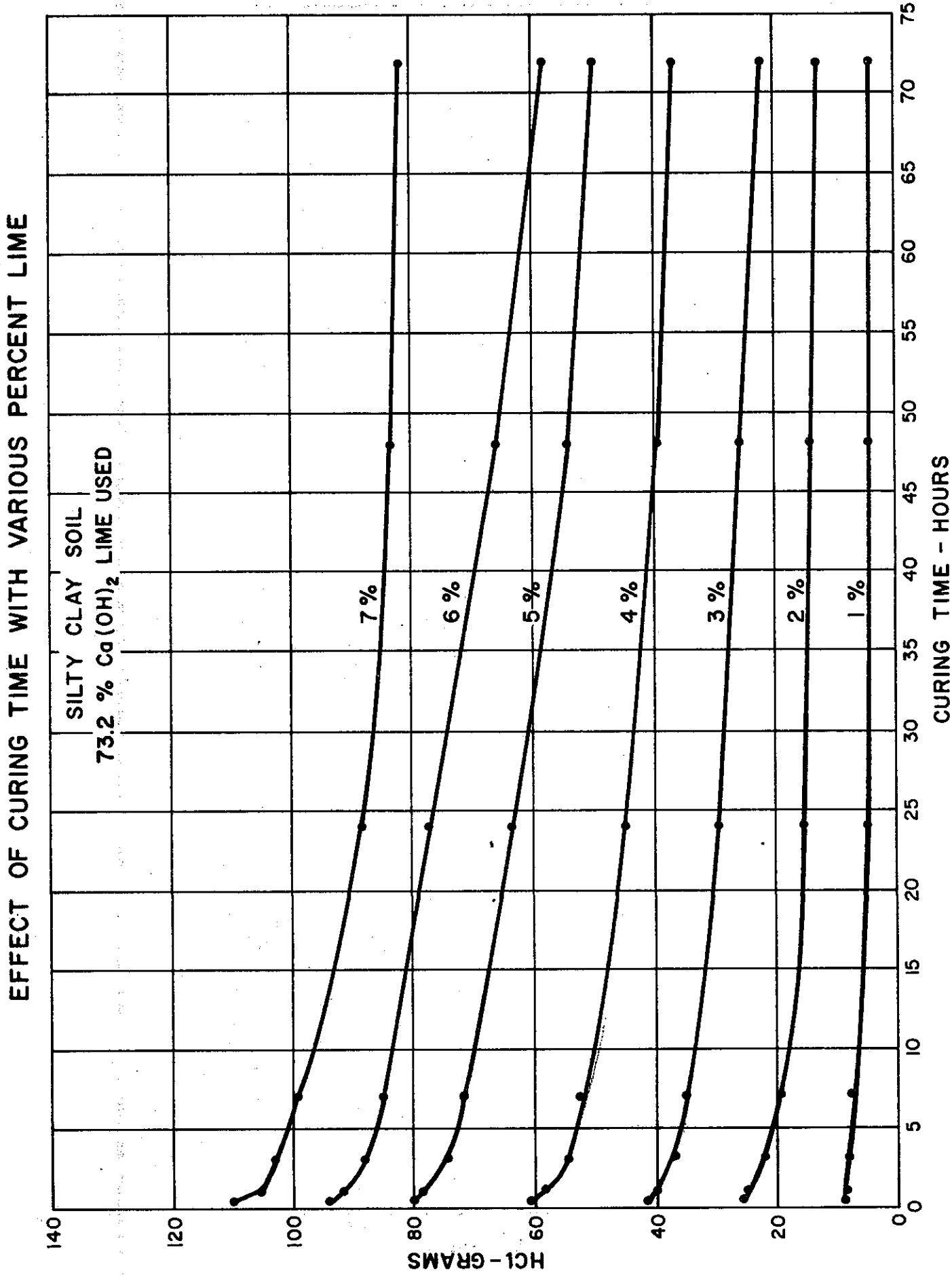

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Figure 3

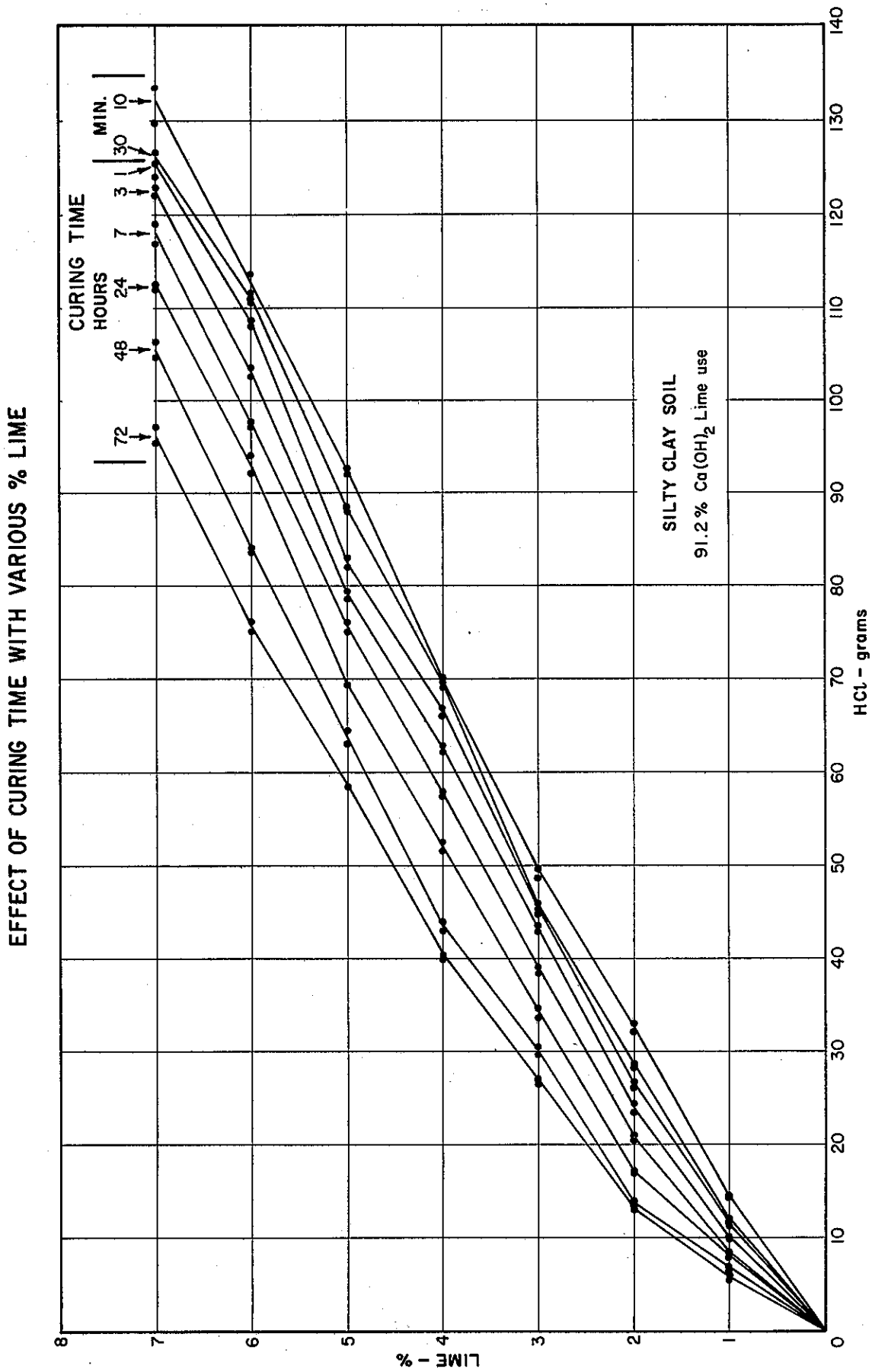

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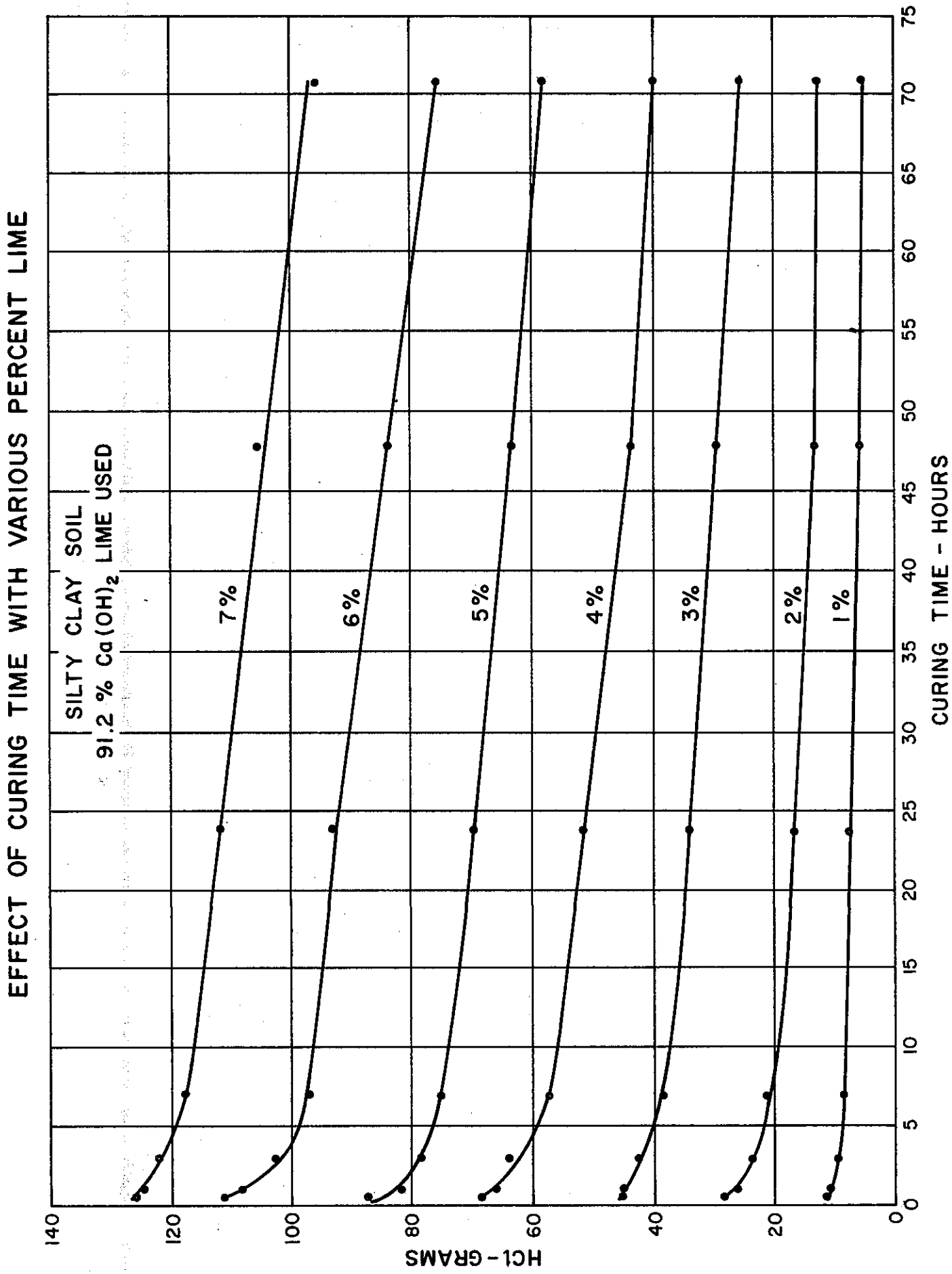
Figure 4



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EFFECT OF CURING TIME WITH VARIOUS TYPES OF SOIL AND 5% LIME

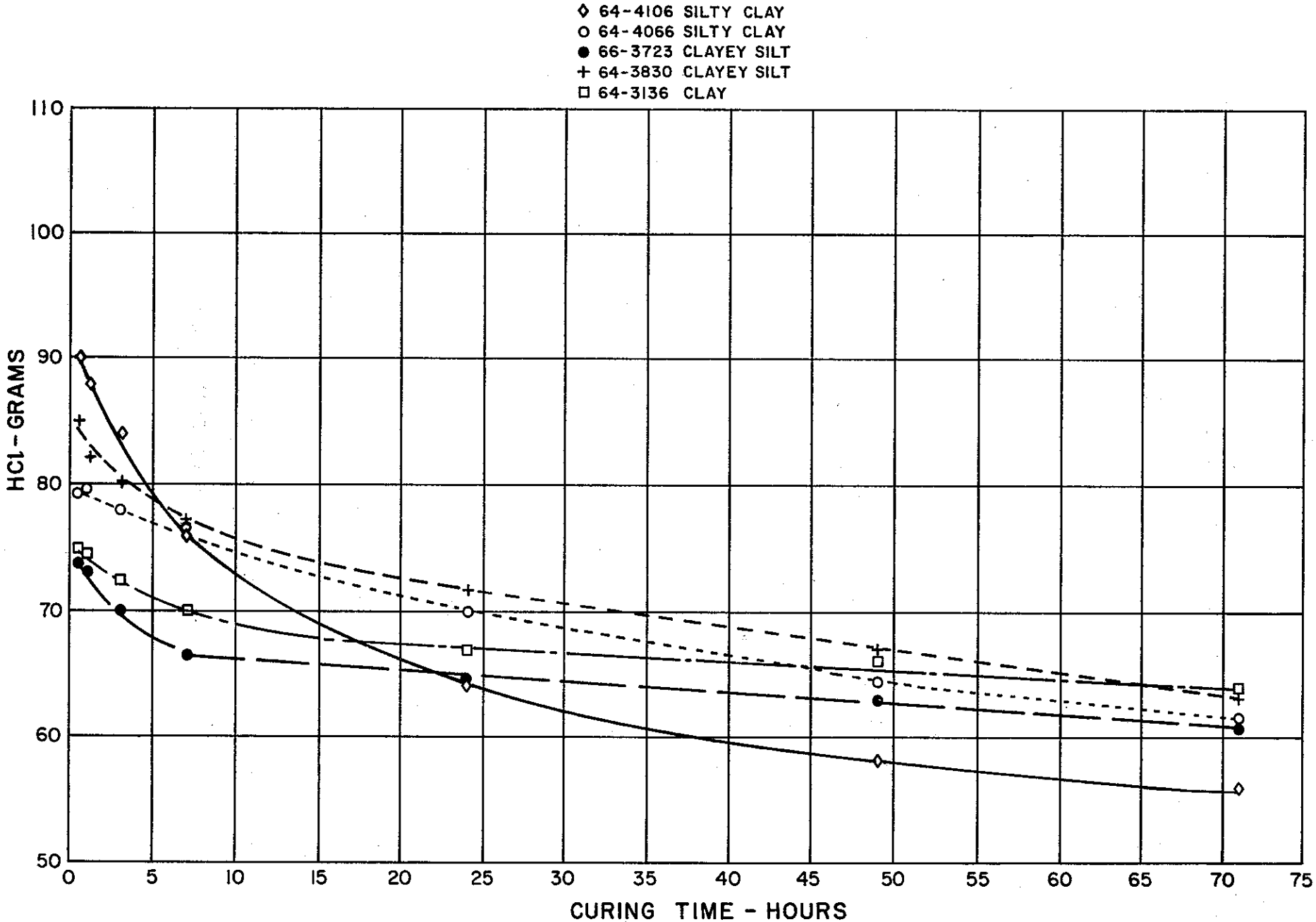

Figure 5

Figure 6

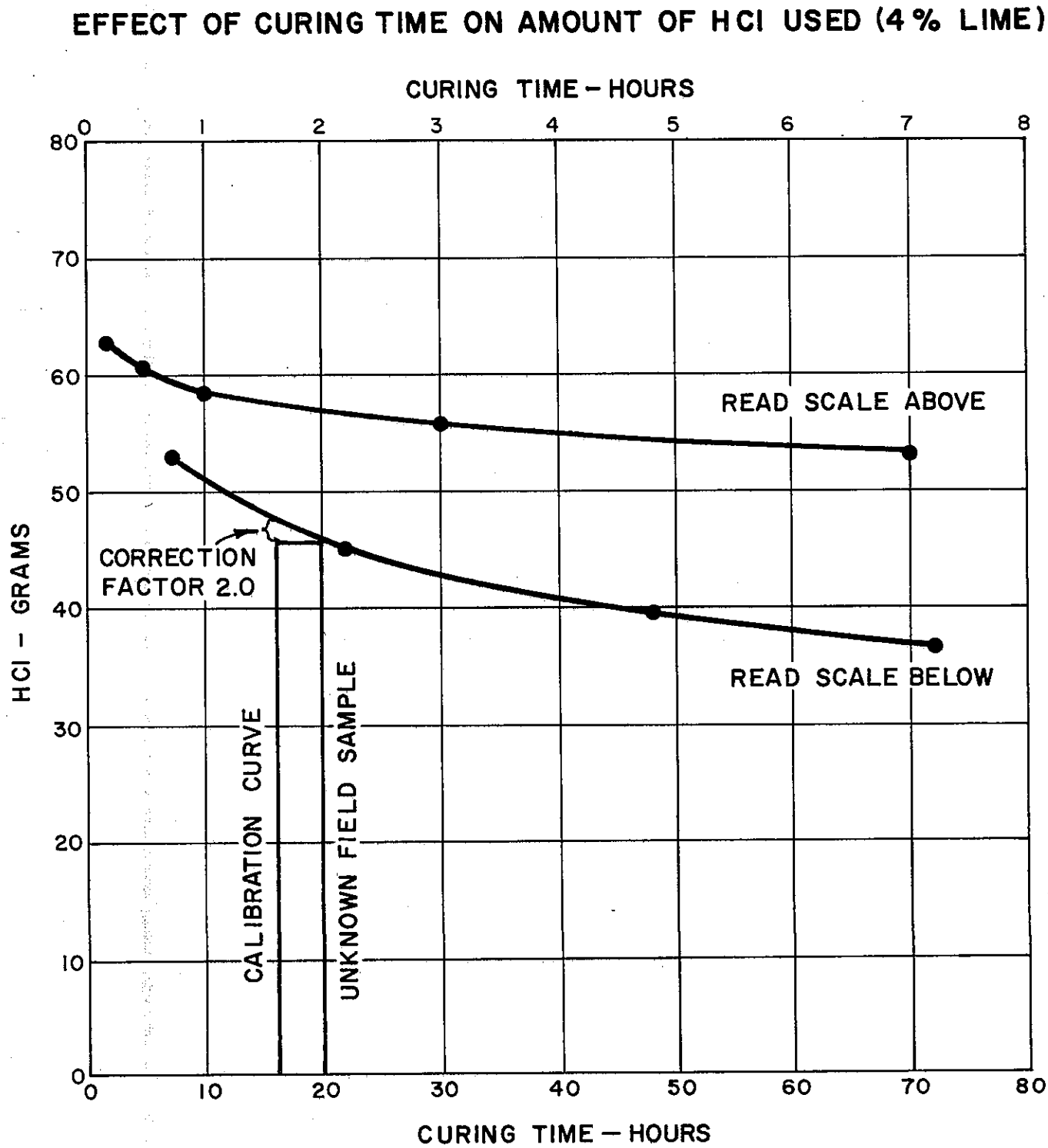

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Figure 7

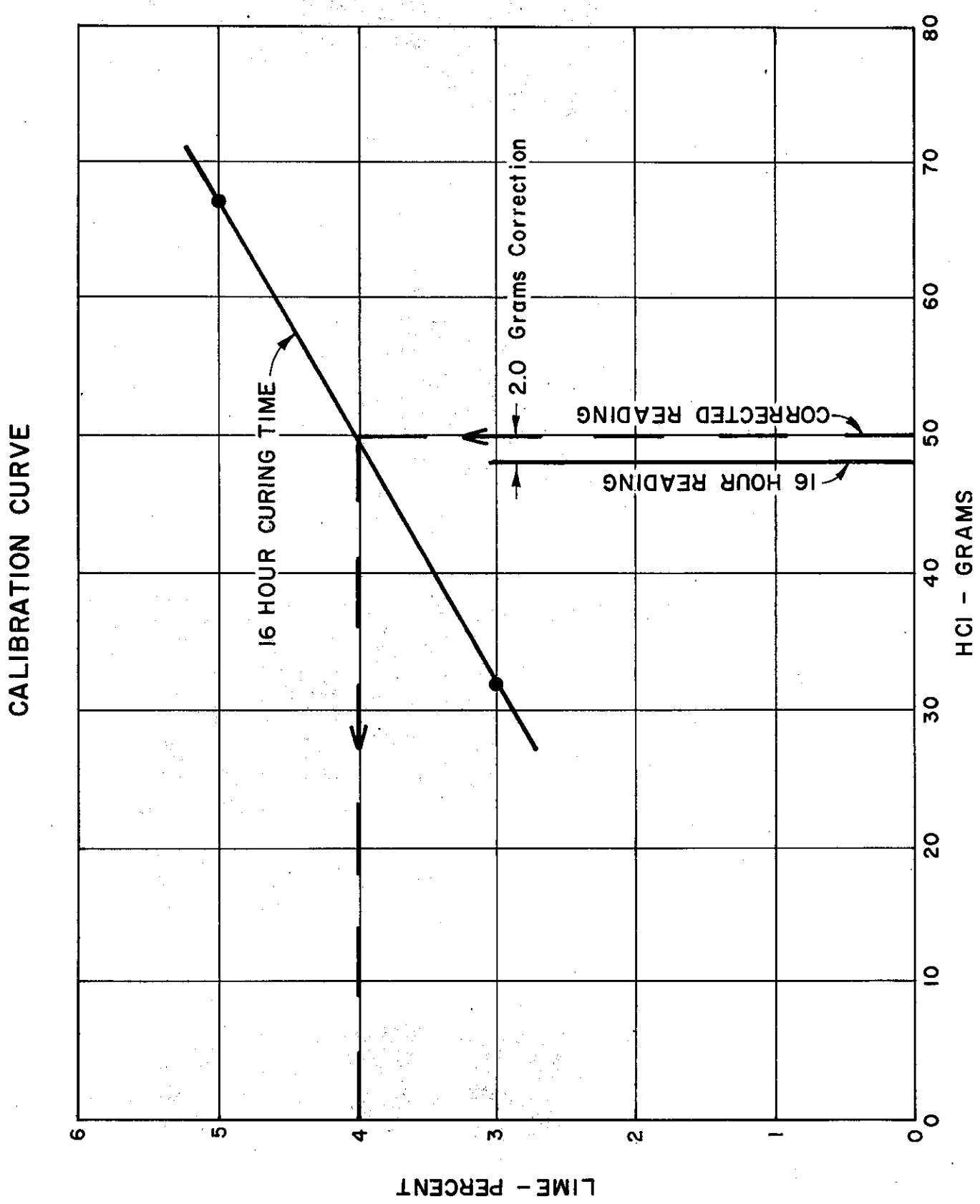

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Figure 8

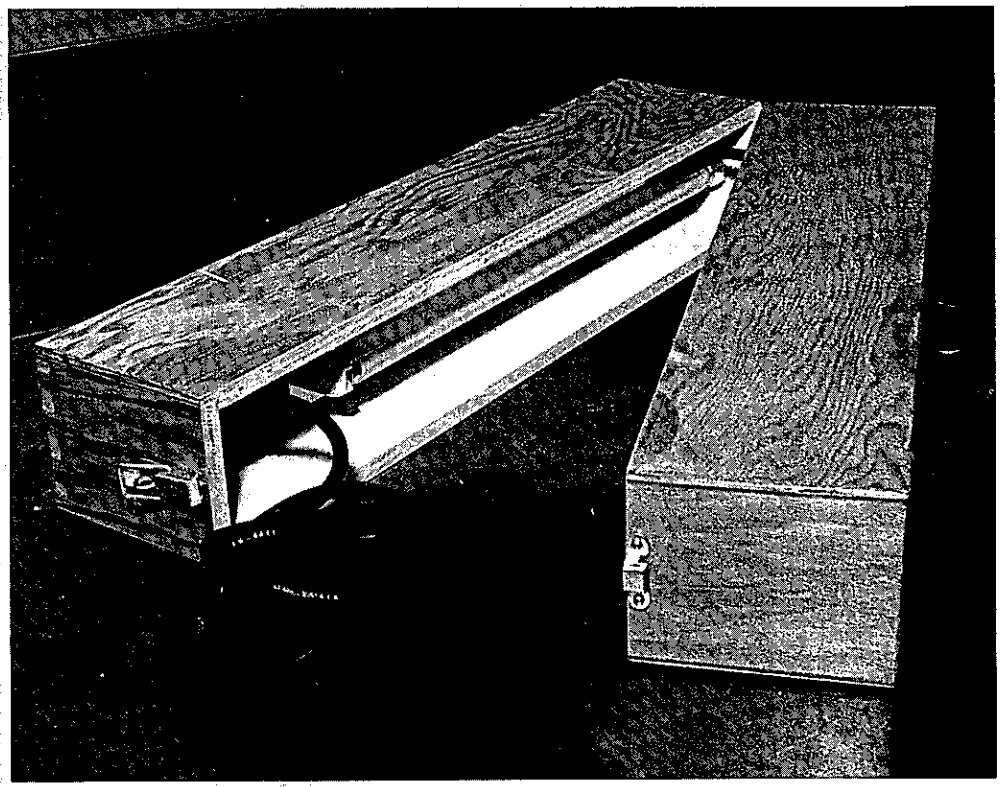

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Figure 9

LIME CONTENT OF 300 GRAM SAMPLE

SILTY CLAY

88.3% $CA(OH)_2$ LIME USED

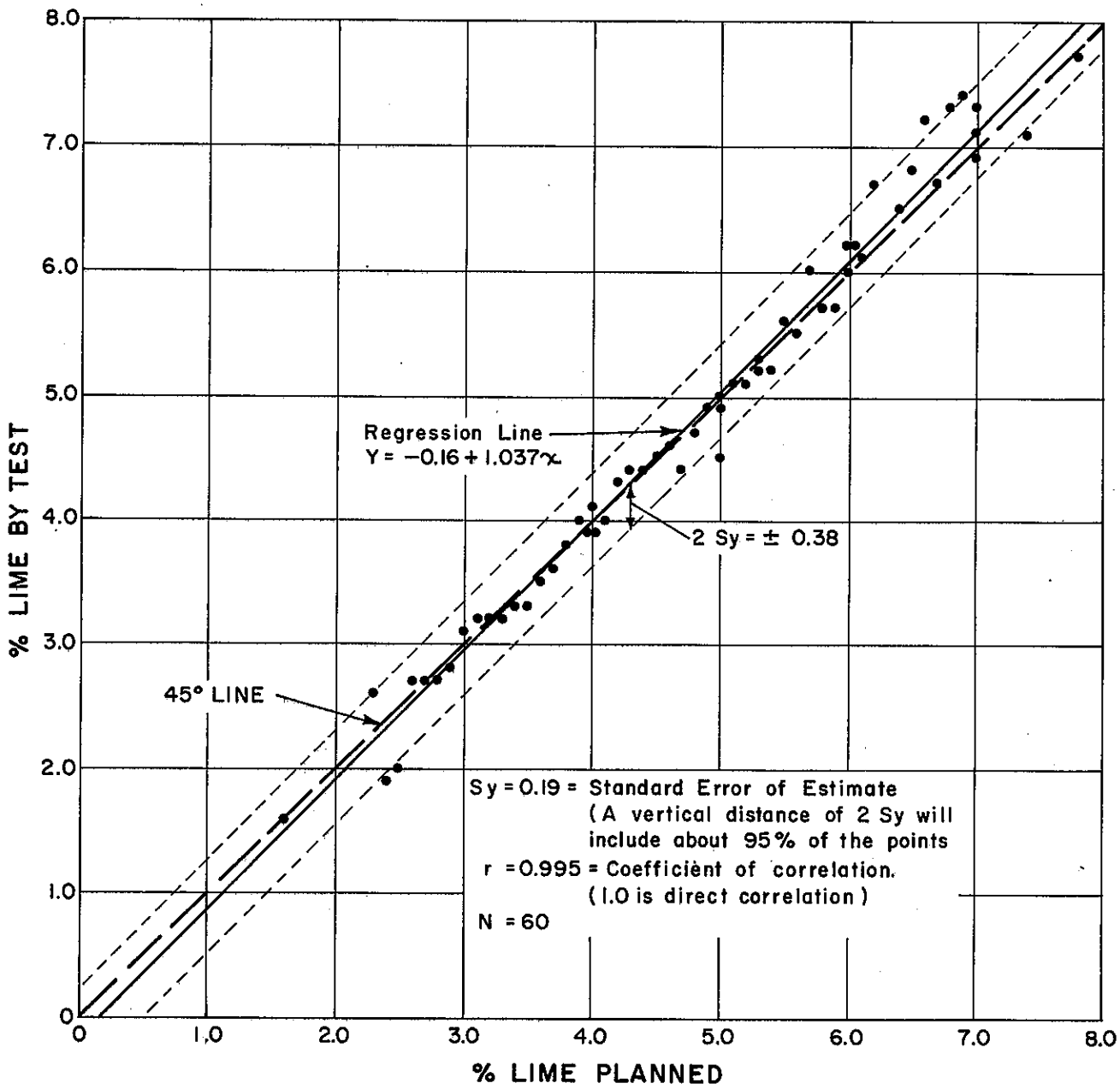

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Figure 10

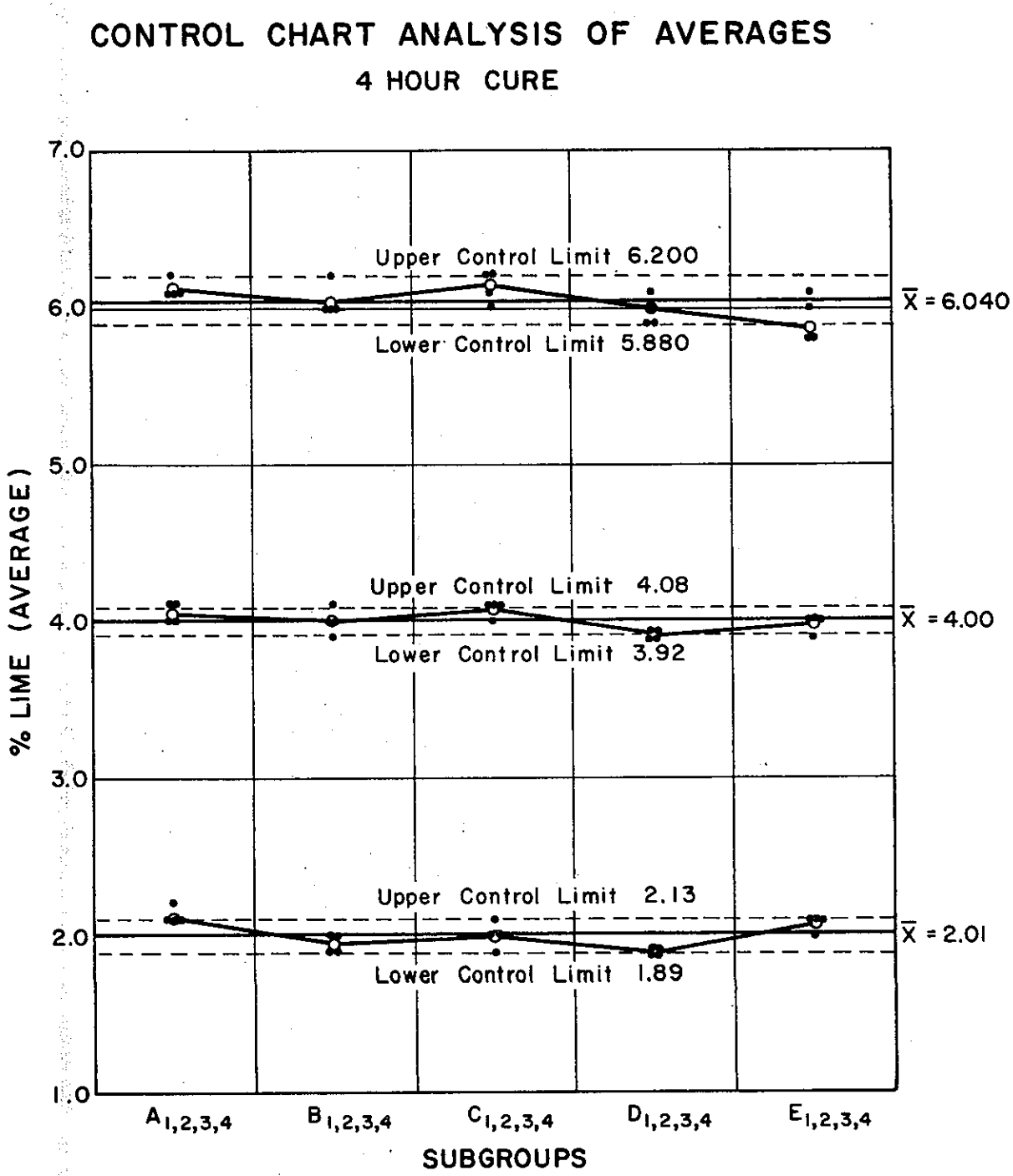

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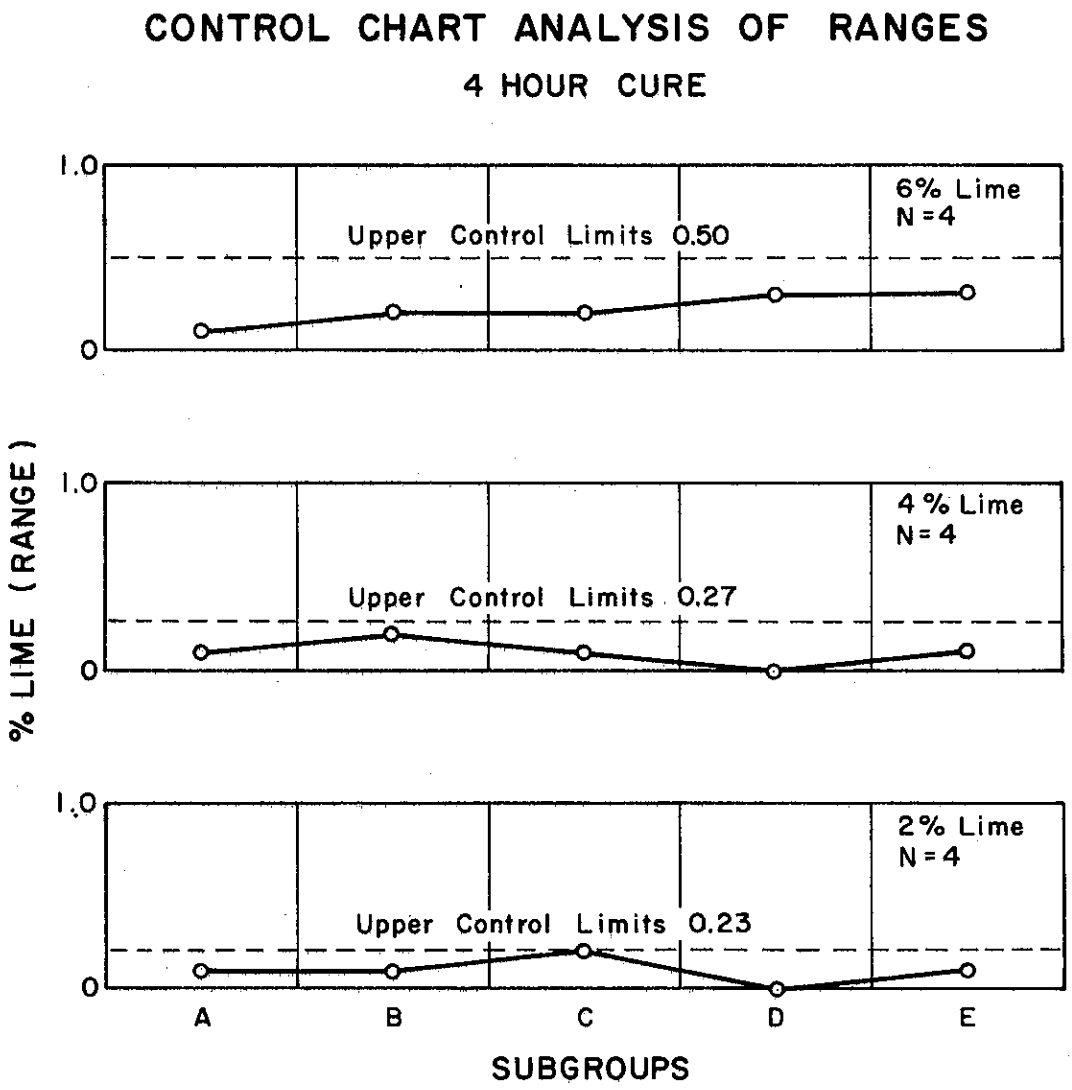
Figure II



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APPENDIX I

MATERIALS AND RESEARCH DEPARTMENT

State of California
Department of Public Works
Division of Highways

Test Method No. Calif. 338-C
August 15, 1967
(11 pages)

PART IV - DETERMINATION OF LIME CONTENT IN LIME TREATED AGGREGATE BY THE METHOD OF TITRATION

Scope

This method of test covers a procedure for determining the percentage of lime in soils or aggregates which have been treated with hydrated lime. The test is based upon the continuous neutralization of an aqueous solution from the lime treated aggregate specimen for a specified time period. This is accomplished by adding sufficient acid to just neutralize the OH ion which is continuously being liberated during the hydration of the lime. The amount of acid used is proportional to the lime content of the treated sample.

Procedure

A. Apparatus

1. 1--100 ml. titrating burette or other device permitting slow, easily controlled addition of acid.
2. 1--burette stand and burette clamp.
3. 10--2 quart wide mouth polyethylene containers (white).
4. 2--5 gal. plastic "carboys" equipped with siphons, neoprene or tygon tubing, hose clamps, etc., for containing acid working solution and water (for safety, do not substitute glass containers for the plastic carboys).
5. Glass dropping bottle.
6. 4--stainless steel stirring rods.
7. 3/8 inch sieve, 12 inch diameter.
8. 1-1/2 inch sieve, 12 inch diameter.
9. A balance graduated to 0.1 gram, and sensitive to 0.1 gram. (ohaus dial-o-gram 1600 gram capacity or equal).
10. A balance graduated to 0.1 gram and sensitive to 0.1 gram. (ohaus model 505, 50 gram capacity or equal).
11. 4 plastic beakers approximately 500 mls. minimum capacity.
12. 1--2,000 ml. glass beaker.
13. 4--50 ml. graduates.
14. Fluorescent light, one 36" long or two 18" long.

B. Reagents

1. Hydrochloric acid (approx. 3N)

Pour the contents of two full standard 6 lb. bottles of concentrated hydrochloric acid C.P. (Service and Supply Stock No. 69010.81) into one of the 5 gal. plastic carboys and dilute with tap water in one gal. increments, to make 5 gal. of solution.

Note: The operator(s) mixing the acid solution are required, for safety, to wear protective gloves, goggles, and aprons.

2. Phenolphthalein indicator solution, 1% solution (Service and Supply Stock No. 69010.85). Dissolve 5 grams of phenolphthalein powder U.S.P. in 250 mls. of ethanol. Dilute with 250 mls. of distilled water.

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3. Separan NP-10

Mix 0.5 grams of NP-10 with 1000 mls. of water in a glass beaker. Stir frequently and allow the solution to stand for one hour. No precipitates should be visible at the end of one hour. Prepare a fresh solution weekly.

C. Test Record Form

Use work card "Field-Laboratory Record of Titration Tests", HMR T-3040, for recording project, calibration and field test data (Figure IV).

D. Determination of a Curing Time Correction Chart

Use the particular aggregate, lime and water from the project and prepare 10 specimens at the planned lime content as follows:

1. Estimate a representative field moisture (%) to be used for the lime treated material on the project.
2. Refer to Table No. 3 and select the weight of aggregate corresponding to the appropriate lime and moisture content. This figure is the dry weight of untreated aggregate required to prepare calibration test specimens of 300 grams. If the samples of untreated aggregate initially contain moisture, then adjust the weight of aggregate plus water to conform to the tabular values.
3. From the project records determine the average percentages of untreated aggregate passing the 3/8 inch sieve and retained on the 3/8 inch sieve. These percentages will be used for proportioning the calibration specimens.
4. Remove and waste any aggregate in the sample of untreated material retained on the 1-1/2 inch sieve and then separate the sample on the 3/8 inch sieve. Recombine for duplicate calibration test specimens with the aggregate weight found in (2) above, and with the 3/8 inch sieve proportions found in (3) above.
5. Pour the weighed aggregate and lime (lime weighed to 0.1 gram) into the two-quart plastic container and dry mix thoroughly with a stainless steel stirring rod.
6. Add the mls. of water given in Table 3 and again mix thoroughly. The time that the water is added is considered the start of the curing period.
7. Test the specimens in pairs at approximate curing times of 1, 3, 7, 24 and 48 hours to establish a HCl versus curing time curve as shown on Figure VI. This curve will be the curing time correction chart for the job. Leave the samples uncovered during the curing period.
8. Measure out 200 mls. of tap water and 50 mls. of NP-10 solution; pour together into a plastic beaker and set one beaker behind each container that has a specimen to be tested. Measure out another 50 mls. of NP-10 solution in the glass graduate and weigh to the nearest 0.1 gram (record net weight) and place behind the plastic beakers.
9. At the end of 1 hour, take the two specimens with a 1 hour curing time, start timer and add the mixed water and NP-10 solution to each test specimen. Allow an interval of 2 minutes between specimens.

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APPENDIX I

MATERIALS AND RESEARCH DEPARTMENT

State of California
Department of Public Works
Division of Highways

Test Method No. Calif. 338-C
August 15, 1967
(11 pages)

PART IV - DETERMINATION OF LIME CONTENT IN LIME TREATED AGGREGATE BY THE METHOD OF TITRATION

Scope

This method of test covers a procedure for determining the percentage of lime in soils or aggregates which have been treated with hydrated lime. The test is based upon the continuous neutralization of an aqueous solution from the lime treated aggregate specimen for a specified time period. This is accomplished by adding sufficient acid to just neutralize the OH ion which is continuously being liberated during the hydration of the lime. The amount of acid used is proportional to the lime content of the treated sample.

Procedure

A. Apparatus

1. 1--100 ml. titrating burette or other device permitting slow, easily controlled addition of acid.
2. 1--burette stand and burette clamp.
3. 10--2 quart wide mouth polyethylene containers (white).
4. 2--5 gal. plastic "carboys" equipped with siphons, neoprene or tygon tubing, hose clamps, etc., for containing acid working solution and water (for safety, do not substitute glass containers for the plastic carboys).
5. Glass dropping bottle.
6. 4--stainless steel stirring rods.
7. 3/8 inch sieve, 12 inch diameter.
8. 1-1/2 inch sieve, 12 inch diameter.
9. A balance graduated to 0.1 gram, and sensitive to 0.1 gram. (ohaus dial-o-gram 1600 gram capacity or equal).
10. A balance graduated to 0.1 gram and sensitive to 0.1 gram. (ohaus model 505, 50 gram capacity or equal).
11. 4 plastic beakers approximately 500 mls. minimum capacity.
12. 1--2,000 ml. glass beaker.
13. 4--50 ml. graduates.
14. Fluorescent light, one 36" long or two 18" long.

B. Reagents

1. Hydrochloric acid (approx. 3N)

Pour the contents of two full standard 6 lb. bottles of concentrated hydrochloric acid C.P. (Service and Supply Stock No. 69010.81) into one of the 5 gal. plastic carboys and dilute with tap water in one gal. increments, to make 5 gal. of solution.

Note: The operator(s) mixing the acid solution are required, for safety, to wear protective gloves, goggles, and aprons.

2. Phenolphthalein indicator solution, 1% solution (Service and Supply Stock No. 69010.85). Dissolve 5 grams of phenolphthalein powder U.S.P. in 250 mls. of ethanol. Dilute with 250 mls. of distilled water.

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3. Separan NP-10

Mix 0.5 grams of NP-10 with 1000 mls. of water in a glass beaker. Stir frequently and allow the solution to stand for one hour. No precipitates should be visible at the end of one hour. Prepare a fresh solution weekly.

C. Test Record Form

Use work card "Field-Laboratory Record of Titration Tests", HMR T-3040, for recording project, calibration and field test data (Figure IV).

D. Determination of a Curing Time Correction Chart

Use the particular aggregate, lime and water from the project and prepare 10 specimens at the planned lime content as follows:

1. Estimate a representative field moisture (%) to be used for the lime treated material on the project.
2. Refer to Table No. 3 and select the weight of aggregate corresponding to the appropriate lime and moisture content. This figure is the dry weight of untreated aggregate required to prepare calibration test specimens of 300 grams. If the samples of untreated aggregate initially contain moisture, then adjust the weight of aggregate plus water to conform to the tabular values.
3. From the project records determine the average percentages of untreated aggregate passing the 3/8 inch sieve and retained on the 3/8 inch sieve. These percentages will be used for proportioning the calibration specimens.
4. Remove and waste any aggregate in the sample of untreated material retained on the 1-1/2 inch sieve and then separate the sample on the 3/8 inch sieve. Recombine for duplicate calibration test specimens with the aggregate weight found in (2) above, and with the 3/8 inch sieve proportions found in (3) above.
5. Pour the weighed aggregate and lime (lime weighed to 0.1 gram) into the two-quart plastic container and dry mix thoroughly with a stainless steel stirring rod.
6. Add the mls. of water given in Table 3 and again mix thoroughly. The time that the water is added is considered the start of the curing period.
7. Test the specimens in pairs at approximate curing times of 1, 3, 7, 24 and 48 hours to establish a HCl versus curing time curve as shown on Figure VI. This curve will be the curing time correction chart for the job. Leave the samples uncovered during the curing period.
8. Measure out 200 mls. of tap water and 50 mls. of NP-10 solution; pour together into a plastic beaker and set one beaker behind each container that has a specimen to be tested. Measure out another 50 mls. of NP-10 solution in the glass graduate and weigh to the nearest 0.1 gram (record net weight) and place behind the plastic beakers.
9. At the end of 1 hour, take the two specimens with a 1 hour curing time, start timer and add the mixed water and NP-10 solution to each test specimen. Allow an interval of 2 minutes between specimens.

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10. Repeat this procedure with the other specimens at the end of 3, 7, 24 and 48 hours curing time.

11. Add two full droppers (approximately 40 drops) of phenolphthalein solution to each container. The water will normally turn red due to the presence of lime.

12. Weigh each plastic container including its contents to the nearest 0.1 gram.

13. Place the fluorescent light behind the plastic containers as shown in Figure V.

14. At 8 minutes after the addition of water to the first specimen, (Step 9), start adding the initial amount of 3N hydrochloric acid with a burette while stirring continuously.

a. The initial amount of acid added is the amount required to cause the red color to just disappear. Repeat this procedure with the second specimen at 10 minutes. Four specimens are normally tested in parts E and F of this procedure and the time is increased to 12 and 14 minutes for the third and fourth specimens, respectively.

b. When the red color starts to reappear after the initial introduction of acid, use the burette to make a second addition of acid in the amount (mls.) necessary to cause the color to just disappear. Subsequent additions of acid are made as necessary to cause the color to just disappear.

Caution: Stir lightly. Vigorous stirring will cause the flocculated particles to disperse making it difficult to see the end point.

15. In addition to the stirring accomplished during each introduction of acid, stir lightly for about 3 seconds of every minute and for about 6 seconds of every fifth minute, whether or not color is present.

a. Perform the stirring in a circular and "zig-zag" pattern across the bottom of the plastic container. Be careful to avoid the possibility of leaving any deposit of unneutralized lime in the center of the container.

b. At the end of 25 minutes after adding the initial amount of water to the first sample, stir the sample vigorously, add the additional 50 mls. of NP-10 solution, (weighed in Step 8) and continue the test. Repeat this procedure with the second specimen at 27 minutes. Four specimens are normally tested in parts E and F of this procedure and the time is increased to 29 and 31 minutes, respectively, for the third and fourth specimens.

16. Continue the procedure of adding acid and stirring until 38 minutes have elapsed since the initial addition (Step 9) of water to the first specimen. Then reweigh the plastic container and contents to the nearest 0.1 gram. Repeat this procedure with the second specimen at 40 minutes. Four specimens are normally tested in parts E and F of this procedure and the time is increased to 42 and 44 minutes for the third and fourth specimens, respectively.

17. Using the difference between the initial (weight determined in Step 12) plus the additional weight of NP-10 (weight determined in Step 8) and the final test specimen weights, calculate and record the total weight of hydrochloric acid used to neutralize the solution.

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18. Plot the grams of HCl acid used against the curing time to establish the correction chart. (Figure VI).

19. The curing time correction curve need be established only once for a project unless there is a change in aggregate or lime source.

E. Determination of a Standard Curve

1. Use the particular aggregate, lime and water from the project and prepare 4 specimens as follows:

a. Prepare two specimens with one percent less than the planned lime content.

b. Prepare two specimens with one percent more than the planned lime content.

c. Estimate a representative field moisture (%) to be used for the lime treated material on the project.

d. Select the proper weight of water, aggregate and lime from Table 3.

2. Determine the curing time for the calibration specimens as follows:

a. Establish a calibration curve with a 4 or 5 hour curing time for use with field samples that are completely mixed and tested during the same day.

b. Establish a calibration curve during the second day after mixing for use with field samples tested on the second day after mixing.

c. The curing time for a field sample begins with the time for mixing and includes the time required for sampling the mixture, for transporting the sample to the testing location and for preparing the 300 gram test specimens.

d. Less correction will be needed and the results will be more accurate when similar curing times are used for calibration tests and field sample tests. The test is sensitive to curing time the first hour after mixing. Therefore, it is advisable to wait until at least one hour has elapsed between mixing and testing.

3. Follow the steps specified in sections D-3 through D-6 and D-8, D-9 and D-11 through 17.

4. Plot the grams of HCl acid used against the percent lime used for the four specimens. Draw a straight line between the average of the tests. This is the standard curve for determining the lime content of field mixed lime treated specimens (Figure VII).

F. Test Procedure for Field Samples

1. Secure one to four field samples of the lime treated aggregate with each sample weighing approximately 3 kilograms. Each 3 kilogram sample will have to be weighed in 2 approximately equal parts of 1500 grams each, since the scale provided for this test has a capacity of only 1600 grams. Obtain weight of sample to nearest gram.

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2. Remove and waste any aggregate retained on the 1-1/2 inch sieve.

3. Separate sample on the 3/8 inch sieve and determine the proportion retained and passing the 3/8 inch sieve on the basis of total sample weight including retained 1-1/2 inch material.

4. Prepare 300 gm. test specimens in the same proportion, retained and passing 3/8 inch material as contained in the original 3 Kg. sample. The retained 3/8 inch material is made up of passing 1-1/2 inch and retained 3/8 inch material to represent all the retained 3/8 inch material including the retained 1-1/2 inch material.

5. Place each specimen in a separate two quart plastic container and line the containers up along the front of the work bench.

6. Follow the steps specified in section D-8, D-9 and D-11 through 16.

7. Record the resulting weight of hydrochloric acid.

8. Determine the correction factor in grams of acid between the curing time for the calibration and field samples from the curing time correction curve (Figure VI). Apply a plus correction factor if the curing time of the field sample is greater than the curing time of the calibration sample. Apply a minus correction factor if the curing time of the field sample is less than the curing time of the calibration samples.

9. Apply the correction factor to the grams of hydrochloric acid recorded in Step 6.

10. From the calibration curve, determine the percent lime (Figure VII).

Example

Assume the following data:

1. The curing time correction chart was established using the planned lime content (Figure VI).

2. The calibration curve was established with a 16 hour curing time (Figure VII).

3. The field sample had a 20 hour curing time.

4. The titration of the field sample required 48.0 grams of acid.

Plot the curing time for the calibration and field sample against the curing time correction curve as shown on Figure VI.

For this field sample, the correction is +2.0 grams (curve values for 47.5 minus 45.5). Apply this correction to the grams of acid required to titrate the field sample (48.0 + 2.0 = 50.0 grams of acid). Apply the 50.0 grams of acid to the calibration curve which indicates 4.0 percent of lime (Figure VII).

G. Precautions

1. This test should not be performed by persons subject to "color blindness".

2. It is very important that the operator vigilantly maintain a neutral solution by repeatedly adding acid as soon as the pink color reappears.

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3. Use only the amount necessary to just eliminate the red color. Excess acid may attack the aggregates, particularly in the later phases when the amount of hydration products from the lime remaining in the mixture may be low.

H. Hazards

1. Exercise extreme care in handling the concentrated solutions of hydrochloric acid (HCl). If this chemical is splashed in the eyes, on the skin or on the clothing, flush the affected areas immediately with liberal quantities of water.

2. Exercise care in the use of the diluted solutions of HCl as this diluted solution can also be harmful to the eyes, cuts, skin or clothing.

3. When handling acids, personnel should wear chemical protective gloves, goggles and aprons.

4. Glass carboys are not to be substituted for plastic carboys described in the test apparatus and all containers having acids or solutions made from them are to be clearly labeled.

5. Phenolphthalein has been known to cause allergic symptoms in humans and has a powerful laxative effect. It can be toxic. Exposed personnel who are sensitive to it should wear protective clothing, if necessary, to avoid bodily contact. Do not ingest.

References
A California Method
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TABLE NO. 3

Quantities of Materials for Calibration Specimens
Containing Various Percentages of Lime

		1% Lime		2% Lime		3% Lime		4% Lime		5% Lime		6% Lime		7% Lime	
% Moisture	Mls. H2O	Dry Wt. Agg. gms.	Lime gms.	Dry Wt. Agg. gms.	Lime gms.	Dry Wt. Agg. gms.	Lime gms.	Dry Wt. Agg. gms.	Lime gms.	Dry Wt. Agg. gms.	Lime gms.	Dry Wt. Agg. gms.	Lime gms.	Dry Wt. Agg. gms.	Lime gms.
5	14	283	2.8	280	5.6	278	8.3	275	11.0	272	13.6	270	16.2	267	18.7
6	17	280	2.8	277	5.5	275	8.2	272	10.9	270	13.5	267	16.0	264	18.5
7	20	277	2.8	275	5.5	272	8.2	269	10.8	267	13.4	264	15.9	262	18.3
8	22	275	2.8	272	5.5	270	8.1	267	10.7	265	13.2	262	15.7	260	18.2
9	25	272	2.7	270	5.4	267	8.0	264	10.6	262	13.1	259	15.6	257	18.0
10	27	270	2.7	268	5.4	265	8.0	262	10.5	260	13.0	258	15.5	255	17.9
11	30	267	2.7	265	5.3	262	7.9	260	10.4	257	12.9	255	15.3	252	17.7
12	32	265	2.7	263	5.3	260	7.8	258	10.3	255	12.8	253	15.2	250	17.5
13	35	262	2.6	260	5.2	257	7.7	255	10.2	252	12.6	250	15.0	248	17.3
14	37	260	2.6	258	5.2	255	7.7	253	10.1	250	12.5	248	14.9	246	17.2
15	39	258	2.6	256	5.1	253	7.6	251	10.0	249	12.4	246	14.8	244	17.1
16	41	256	2.6	254	5.1	251	7.5	249	10.0	247	12.3	244	14.6	242	16.9
17	44	253	2.5	251	5.0	249	7.5	246	9.8	244	12.2	242	14.5	239	16.7
18	46	252	2.5	249	5.0	247	7.4	244	9.8	242	12.1	240	14.4	238	16.6
19	48	250	2.5	247	4.9	245	7.3	242	9.7	240	12.0	238	14.3	236	16.5
20	50	248	2.5	245	4.9	243	7.3	240	9.6	238	11.9	236	14.2	234	16.4
21	52	246	2.5	243	4.9	241	7.2	238	9.5	236	11.8	234	14.0	232	16.2
22	54	244	2.4	241	4.8	239	7.2	236	9.4	234	11.7	232	13.9	230	16.1
23	56	242	2.4	239	4.8	237	7.1	235	9.4	232	11.6	230	13.8	228	16.0
24	58	240	2.4	237	4.7	235	7.1	233	9.3	230	11.5	228	13.7	226	15.8
25	60	238	2.4	235	4.7	234	7.0	231	9.2	229	11.4	226	13.6	224	15.7
26	62	236	2.4	233	4.7	231	6.9	229	9.2	227	11.4	225	13.5	222	15.6
27	64	234	2.3	231	4.6	229	6.9	229	9.1	225	11.3	223	13.4	221	15.5
28	66	232	2.3	229	4.6	227	6.8	225	9.0	223	11.2	221	13.3	219	15.3
29	67	231	2.3	228	4.6	226	6.8	224	9.0	222	11.1	220	13.2	218	15.3
30	69	229	2.3	226	4.5	224	6.7	222	8.9	220	11.0	218	13.1	216	15.1
31	71	227	2.3	225	4.5	222	6.7	220	8.8	218	10.9	216	13.0	214	15.0
32	73	225	2.3	223	4.5	220	6.6	218	8.7	216	10.8	214	12.8	212	14.8
33	74	224	2.2	222	4.4	219	6.6	217	8.7	215	10.8	213	12.8	211	14.8
34	76	222	2.2	220	4.4	217	6.5	215	8.6	213	10.7	211	12.7	209	14.6
35	78	220	2.2	218	4.4	216	6.5	213	8.5	211	10.6	209	12.5	207	14.5

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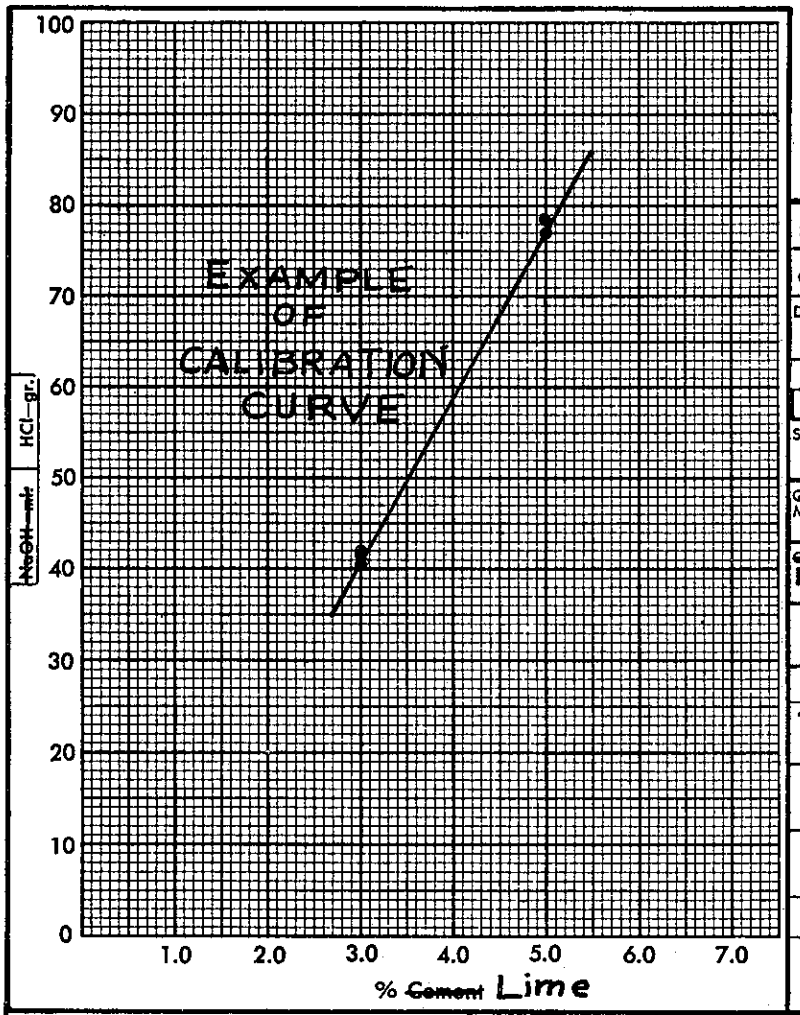

STATE OF CALIFORNIA—DIVISION OF HIGHWAYS

MATERIALS AND RESEARCH DEPARTMENT

Field-Laboratory Record of

TITRATION TESTS

For ~~Cement~~ Lime Content Determination of ~~CTB~~ LT

Sheet 1 of 1 Sheets			Date 6-1-67
Operator John Doe			
Dist.	Co.	Rte.	P. M.
SOURCE	CHARGE	EXPENDITURE AUTHORIZATION	SPECIAL DESIGNATION (USE WHEN APPLICABLE) / OBJECT
Source of Aggregate		~~Cement~~ Brand Lime	
~~Cement~~ Lime Mixer Make:		Road Mix ☐	Batch ☐ Cont. ☐
~~Cement~~ Lime Spreader			
PLANNED	Thickness	~~Cement~~ Lime 4%	Water 13 %

DETERMINATION OF METHOD OF TEST

Item	Specimen Type	Jar No.	NaOH Ml.	Control Values
A	Ottawa Sand			Av. Mls.
B	Aggregate Blank			Lowest Value
C	Av. of A—Lowest Value of B			

If C<6 mls use Acid-Base Proc. (Part I) . . . . ☐

If C≧6 mls use Const. Neut. Proc. (Part II) . . . . ☐

Station	Sample Proportions: Sample Wt. Grams	Pass ⅜ Grams	% Pass ⅜	Jar No.	Gross Jar Wt. Grams: Start	Final	~~NaOH Ml.~~ HCl Gr.	% ~~Cement~~ Lime	Remarks
	300		100	8	746.0	823.0	77.0	5.0	Calibration Curve
	"		"	11	752.9	831.3	78.4	"	Samples. 26 Hours
	"		"	5	787.0	828.8	41.8	3.0	Time Interval Bet-
	"		"	6	789.0	829.9	40.9	"	ween mixing & Testing.
243~5' Lt	3000		100	9	746.0	809.0	63.0	4.2	Field samples with
" 16' Lt.	"		"	7	752.0	813.5	61.5	4.1	same time interval.
245~2' Rt.	"		"	10	746.1	807.3	61.2	4.1	
" 8'	"		"	13	748.9	808.4	59.5	4.0	

HMRT-3040 (REV. 1-65)

Figure IV

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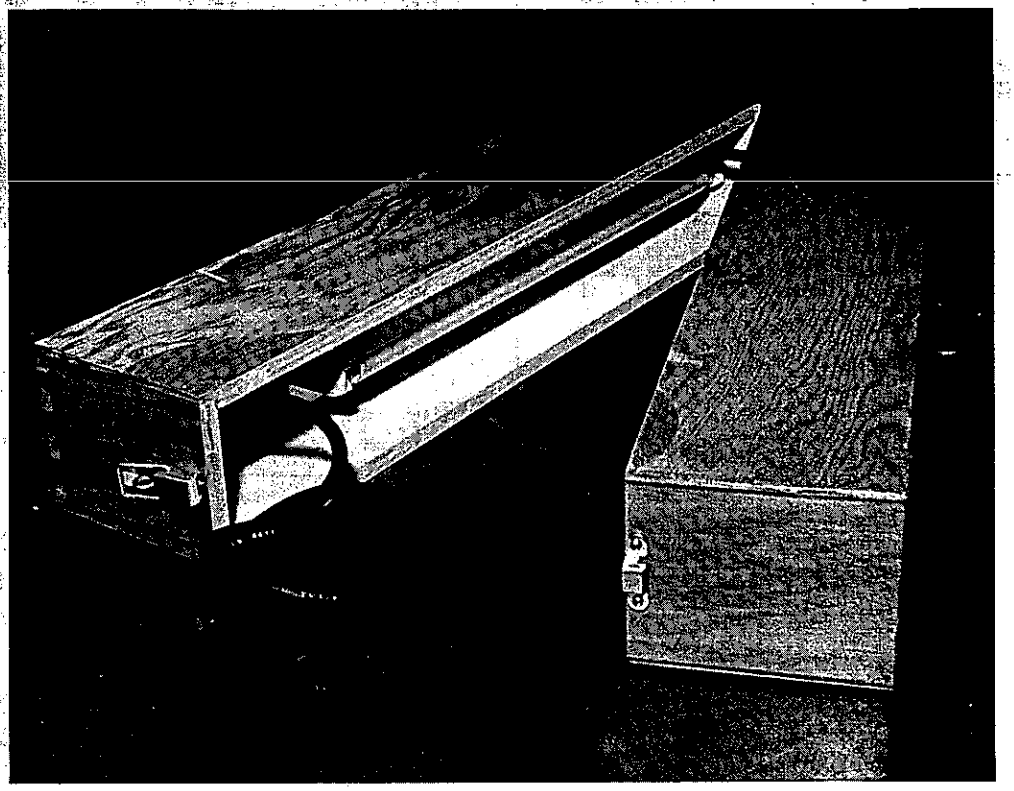

Figure V

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EFFECT OF CURING TIME ON AMOUNT OF HCl USED (4% LIME)

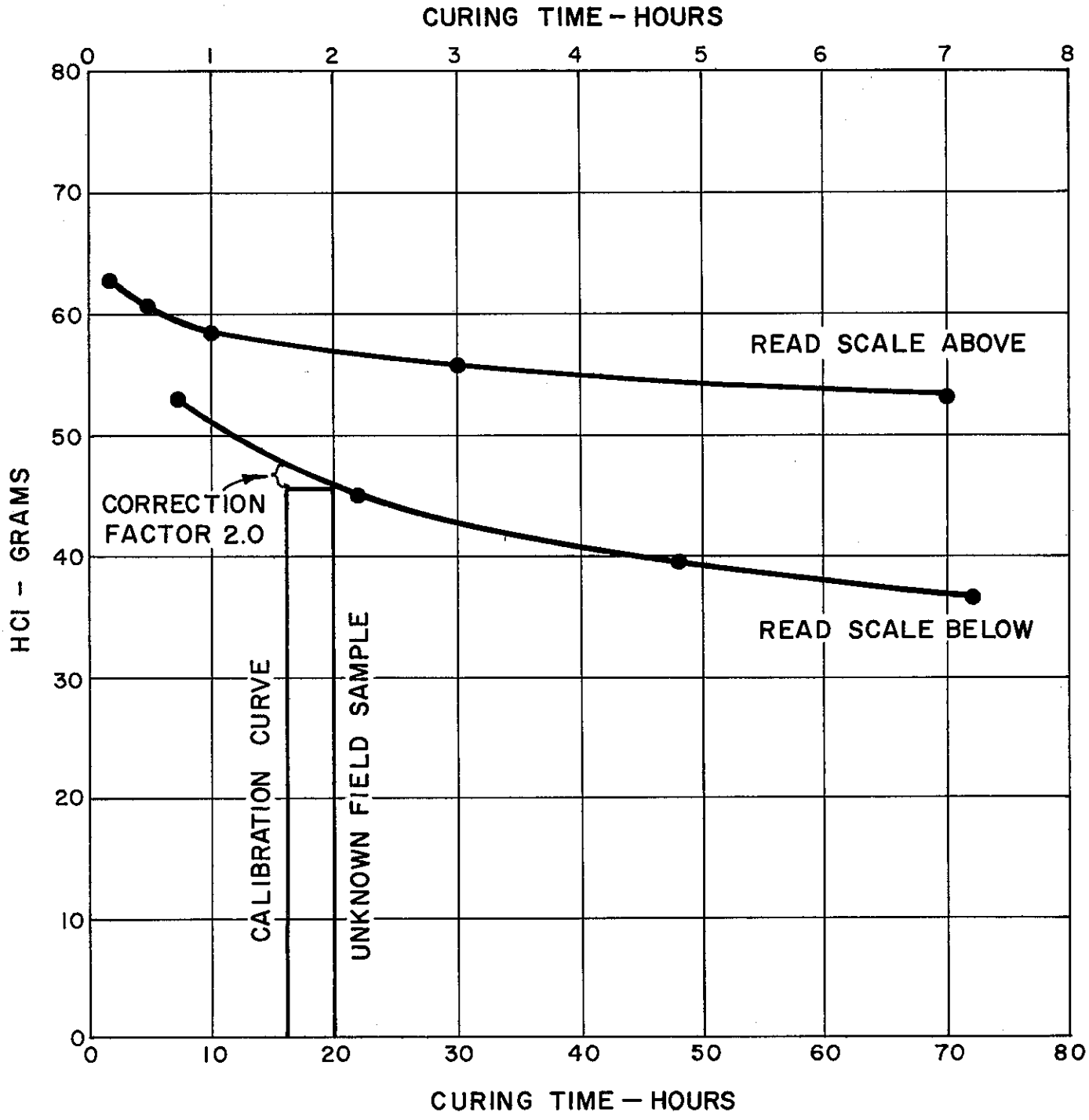

Figure VI

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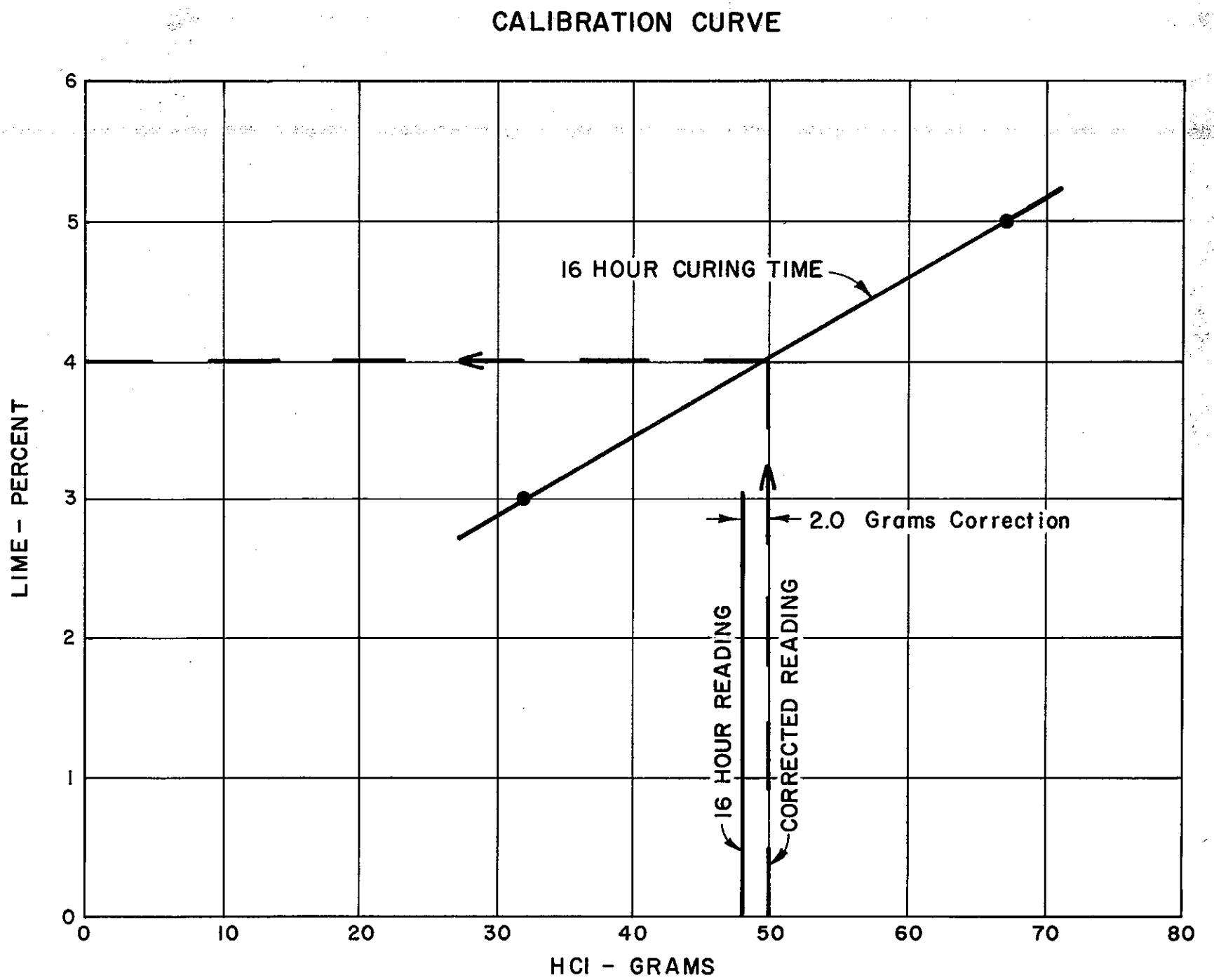

Figure VII