

1963

Abbreviated freeze-thaw test procedures for soil-cement mixtures

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CIRCEO, Jr., Louis Joseph, 1934-
ABBREVIATED FREEZE-THAW TEST PROCE-
DURES FOR SOIL-CEMENT MIXTURES.

Iowa State University of Science and Technology
Ph.D., 1963
Engineering, civil

University Microfilms, Inc., Ann Arbor, Michigan

ABBREVIATED FREEZE-THAW TEST PROCEDURES
FOR SOIL-CEMENT MIXTURES

by

Louis Joseph Circeo, Jr.

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY

Major Subject: Soil Engineering

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1963

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INTRODUCTION

The increased requirement for road and highway construction has resulted in the need for the stabilization of natural or in-place soils to produce a product which will serve effectively as a subbase, base, or surface course. This is especially true in locations where local aggregate sources are scarce or unavailable.

In recent years soil-cement has become one of the most widely used methods of soil stabilization. Soil-cement can be defined as a mechanically compacted mixture of pulverized soil, portland cement, and water which forms a hard, durable, structural material as the cement hydrates. From the first 20,000 square yard scientifically controlled road built near Johnsonville, S. C. in 1935, soil-cement has grown to an annual use in the United States of over 80,000,000 square yards. It has also been successfully used in over 30 other countries.

The minimum cement requirement of a soil is usually determined on the basis of laboratory tests of strength and durability. These tests include unconfined compressive strength evaluation, wet-dry and freeze-thaw tests. The most widely used freeze-thaw test is A.A.S.H.O. Method T 136-57 and A.S.T.M. Method D 560-57 (1, 2).

Compressive strength tests are usually supplementary to

the wet-dry and freeze-thaw tests. An adequately hardened soil-cement mixture will increase in compressive strength with time of curing. Generally an increasing unconfined compressive strength of 300 psi or more at 7 days moist curing will pass the wet-dry and freeze-thaw tests satisfactorily (28).

The wet-dry and freeze-thaw tests are usually considered indicative of the structural competence and durability of soil-cement mixtures. The wet-dry test produces high shrinkage stresses. The freeze-thaw test produces high expansive stresses. These tests were developed to introduce destructive forces which a soil alone could not withstand, but which a structural material would resist. Thus they are more valuable in analyzing a soil-cement mixture as a structural material rather than as a direct criterion of durability. The adequacy of a soil-cement mixture as a structural material would also confirm its ability to withstand weathering (6).

The freeze-thaw test is generally the critical test in determining the required cement content except for mixtures which contain relatively large amounts of silt and clay. For mixtures other than these it is standard practice to mold only one wet-dry specimen at the median cement content, while a freeze-thaw specimen for each cement content investigated is usually molded (28). Thus the freeze-thaw test is the major test in the evaluation of a soil-cement mixture, and

requires considerable time and labor to conduct.

As reported by the "Committee on Soil-Portland Cement Stabilization" of the Highway Research Board, two of the needed areas of soil-cement research and development are better and more practical laboratory tests and the improvement of the freeze-thaw test (17). The present investigation attempts to explore these areas to develop more efficient methods of freeze-thaw testing. The objectives of this investigation are:

1. The development of a relationship which will improve the quality of the freeze-thaw test.
2. The determination of methods to reduce both the number of cement contents to be tested and the number of freeze-thaw cycles to be conducted, while still maintaining the present level of significance of the test.

REVIEW OF PREVIOUS WORK

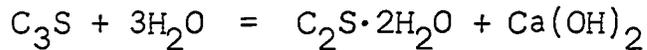
Portland Cement

Portland cement is the product obtained by finely pulverizing clinker produced by calcining to incipient fusion an intimate and properly proportioned mixture of argillaceous and calcareous materials. The major components of portland cement are tricalcium silicate ($3\text{CaO}\cdot\text{SiO}_2$), dicalcium silicate ($2\text{CaO}\cdot\text{SiO}_2$), tricalcium aluminate ($3\text{CaO}\cdot\text{Al}_2\text{O}_3$) and tetracalcium aluminoferrite ($4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{Fe}_2\text{O}_3$), usually abbreviated C_3S , C_2S , C_3A and C_4AF respectively. A typical component analysis of "normal" type I portland cement consists of the following (31):

C_3S	50%
C_2S	25%
C_3A	12%
C_4AF	8%
CaSO_4	3%
CaO	1%
MgO	1%

Type I cement is ground so that at least 90% will pass a #200 sieve. Gypsum (CaSO_4) is intentionally added during grinding to slow the hardening process.

Upon the addition of water and thorough mixing the process of hydration produces crystallite particles of the class $C_2S \cdot 2H_2O$ and $Ca(OH)_2$, as shown by a typical reaction (5):



C_2S produces the same hydration products; but the amount of hydrated lime formed is less, and the rate of hydration is lower. It has been evaluated that C_3S is largely responsible for the strength up to 28 days, and C_2S contributes to the long term strength gain. At the end of one year the strength contributions of C_3S and C_2S are about equal. C_3A also contributes to strength gain; however, because of the undesirable high heat of hydration of C_3A , gypsum and sometimes iron compounds are added to make the C_3A relatively inert during early cement hydration. C_4AF approximates the composition of the ferrite phase as found in commercial clinkers, and is of minor importance to strength.

By adjusting the percentages of various components or adding others, portland cement can be given such various desirable characteristics as rapid hardening (type III), low heat during hydration (type IV), and resistance to sulphates (type V). Types I and III cement are in most general use for soil-cement. Type III cement is made by increasing the C_3S content and the fine grinding so that at least 99.5% of the cement pass a #325 sieve. Type III cements have the dis-

advantage of a high heat of hydration, which makes them unsuitable for many projects.

Hardening Mechanism of Soil-Cement

The addition of portland cement to soil produces definite changes in the properties and structure of the soil. In cohesive soils the first change that occurs is the reduction of plasticity. This is probably caused by the release of calcium ions during the initial hydration stages. The positively charged calcium ions are adsorbed on the negatively charged clay surface. This causes attraction between clay particles and, therefore, flocculation. Recent research (16) has indicated that the high pH and calcium ion concentration liberated during the cement hydration could initiate attack of the clay particles causing the breakdown of amorphous silica and alumina. This could combine with calcium in a pozzolanic-type reaction to form a secondary cementitious material. Thus a clay-cement skeleton and a clay matrix would result. In sandy soils the grains become cemented at points of contact. Catton (6) explains the mechanism as follows:

Study of soil-cement mixtures in the laboratory and field indicates that each cement grain picks up a varying number of soil grains (depending on the grain size of the soil) and as the cement hydrates and crystallizes, a new and larger soil grain or agglomeration is produced. As more and more cement

is added, more soil grains lose their identity to become larger soil grains or agglomerations. These agglomerations of cement grains and soil grains can also be thought of as links in a chain, and when enough cement has been added to link all agglomerations together, with pockets of trapped soil, the mixture becomes a structural material rather than a soil.

Bezruk (4) concluded that the degree of strength and water durability depends basically on the properties and amount of cementing material. The more reactive the cementing material and the greater its amount, the higher will be the strength of the stabilized soil. The interaction of cement with soil may be of a beneficial, detrimental or neutral character. Variation of the colloidal properties of soil favorable to interaction with cement increases the strength of the stabilized soil.

Microscopic studies indicate that individual particles as well as soil microaggregations take part in the reaction of cement with soil as follows (4):

The cementing material becomes distributed in the soil-cement mass as a latticed soil-cement skeleton with thin films enveloping the microaggregations of the soil. In the presence in the soil of a water-resistant microstructure the specific surface of finely dispersed soils become considerably reduced and, consequently, the effectiveness of cement utilization is correspondingly increased. Small admixtures of cement become distributed in the treated soil as separate outcroppings which are not interconnected and do not form a continuous lattice skeleton. In this case the interaction of soil and cement is mainly directed toward increasing cohesion. With increasing cement content a gain in

mechanical strength and impermeability due to the formation of a branched soil-cement skeleton and to filling of the pores between the soil aggregations by the individual hydrating particles of cement is realized.

Moisture is an important constituent in soil-cement mixtures. It is essential both for the compaction of the mixture and for the hydration of the cement. The moisture content used is that which will achieve the desired compacted density. This has been found adequate for the hydration of the cement.

To explain the long term strength gains of soil-cement mixtures, Handy (14) proposed a theory of chemical cementation between the polarized (inert) surfaces of the soil particles and the hydrated cement. Hydroxyl ions from the hydrating cement gel are adsorbed by partially screened silica ions giving initially a weak bond. As time passes the surfaces of the soil particles become depolarized (active), and the original weak bond is improved, thereby gradually increasing the strength.

Development of Soil-Cement Durability Tests

In 1935, an extensive research program was undertaken by the Portland Cement Association to investigate the feasibility of cement as a soil stabilizer. This program was based upon previous experiments by several highway departments (22).

The moisture-density relationships developed by R. R. Proctor in 1929 were found applicable to mixtures of soil and cement. Thus soil-cement mixtures were able to attain the compaction necessary to produce a durable, stabilized material. By 1945 this test was adopted as a standard by both A.S.T.M. and A.A.S.H.O. (1, 2).

In order to properly evaluate the durability of the compacted soil-cement mixture the samples were cured for 7 days in an atmosphere of high humidity. This permitted hydration of a significant portion of the cement before testing. The wet-dry and freeze-thaw tests were evolved from tests used for concrete mixtures. It was found that these tests would reproduce the internal forces caused by moisture changes in the field: the wet-dry test produces high shrinkage forces; however, the high temperatures used in the drying portion of the test tend to accelerate cement hydration. The freeze-thaw test produces high expansive forces in cohesive soils. This test avoids the accelerated cement hydration inherent in the wet-dry test. A brushing procedure to remove the loose material on the sample after each of 12 freeze-thaw or wet-dry cycles was developed to give consistent and reproducible results.

The above tests were developed to determine the minimum cement content required to stabilize a soil adequately, and the following criteria for maximum permissible soil-cement

losses by brushing were established (28):

Soil groups A-1, A-2-4, A-2-5, and A-3, not over 14%

Soil groups A-2-6, A-2-7, A-4 and A-5, not over 10%

Soil groups A-6 and A-7, not over 7%

Research has indicated that the physical and chemical properties of a soil have a great influence on the required cement content (32). These effects are so diverse and inter-related that no simple relationship to determine the minimum cement requirement has been found. However the wet-dry and freeze-thaw tests evaluate the combined effects of these phenomena.

The criteria of the freeze-thaw and wet-dry test to produce an adequate soil-cement mixture were determined on the basis of laboratory test data, outdoor performance of laboratory specimens, and field performance. These data include volume change, maximum moisture content, compressive strength, and soil-cement loss due to brushing (23). The tests were adopted as standards by A.S.T.M. and A.A.S.H.O. in 1945, and the criteria have been validated by the successful field performance of soil-cement mixtures in the last 25 years.

Alternates to the Standard Freeze-Thaw Test

The freeze-thaw test has often been criticized as being too severe and not simulating field conditions. The frequency of freezing and thawing in a moderate climate is considered to have a more severe effect on the durability of soil-cement mixtures than fewer cycles in a colder climate. For example, 40 freeze-thaw cycles per year in Kansas is considered more severe for soil-cement mixtures than 4 cycles per year in Minnesota (30). Soil-cement pavements in tropical areas, not subject to freezing, would not experience the great expansive forces that the freeze-thaw test simulates. The increased temperature of curing in these areas would also produce better quality soil-cement mixtures (7). This could allow a smaller cement content than would normally be used. The present freeze-thaw test does not take these conditions into consideration. Also the freezing temperatures used are not considered representative of actual field conditions.

The British Standard Freeze-Thaw Test (B.S. 1924:1957) determines the change in unconfined compressive strength of cohesive soils when subjected to specified conditions of freezing and thawing (21). No brushing tests are used since this is not thought to simulate field conditions. Also, freezing at a realistic temperature is conducted down from the top of the sample. This introduces a temperature

gradient into the sample which is consistent with actual conditions. The strength loss is compared to a control sample continually immersed in water. A criticism of this test is that there is insufficient control of temperature conditions during the freezing period.

The British Test has been modified in the Soil Research Laboratory of the Iowa Engineering Experiment Station (13). The Iowa State compaction apparatus is used. Climatic conditions were modified to simulate the more severe climatic changes in Iowa. The criteria used were the unconfined compressive strength and the index of resistance to freezing (the ratio of the unconfined compressive strength of the freeze-thaw specimen to that of the immersed control sample). Laboratory results, correlated with field trial base course sections, indicated that the test produced valid results and allowed a smaller cement content than was obtained from standard A.S.T.M. and A.A.S.H.O. tests.

A complementary study (18) concluded that:

1. The Iowa Freeze-Thaw Test is as dependable as the standard A.S.T.M.-A.A.S.H.O. freeze-thaw test.
2. The cement requirements of a soil-cement mixture might eventually be determined by simple strength tests. This is based on a functional relationship between the 14 day unconfined compressive strength and the strength of the same mixture after 7 days

humid curing and 10 cycles of the Iowa Freeze-Thaw Test.

At the present time the Portland Cement Association is conducting considerable research into alternate methods of measuring freeze-thaw resistance of soil-cement mixtures (27). These include length change, unconfined compressive strength, and an accelerated procedure for the standard freeze-thaw test. Results indicate that length change measurements during freeze-thaw cycles might serve as a supplementary indication of durability. Compressive strength results are less conclusive.

The accelerated procedure is perhaps the most interesting development, since the present test requires 6 to 7 weeks to complete. With the use of an inexpensive cabinet with adjustable freeze-thaw periods, an accelerated 12 cycle test of 7 to 10 days seems feasible. Preliminary results indicate that this method of freeze-thaw testing may be developed to a significance equivalent to the standard test.

INVESTIGATION PART I

The freeze-thaw test is generally the critical test in evaluating the required cement content. Three cement contents are usually tested, and the minimum cement content which will meet the specified freeze-thaw loss is usually chosen as the required cement content. However, many soils have recommended cement contents which produce as low as 2% freeze-thaw loss, since the next lower cement content (usually 2% less) tested had a freeze-thaw loss greater than the allowable. Therefore in many cases the recommended cement content is greater than necessary. A cement content which could be selected between these two which would produce the exact specified freeze-thaw loss would result in more economical mix designs.

These problems could be alleviated by a relationship between the cement content and the freeze-thaw loss of a soil-cement mixture. Also, a valid relationship would require testing of only 2 cement contents to establish the relationship. Thus a relationship would introduce the possibility of reducing the number of cement contents required to conduct the freeze-thaw test.

Cement Freeze-Thaw Loss Relationship

In order to obtain results which would apply to as general a case as possible, it was decided to investigate the existence of a relationship using data of a random nature. The data were randomly chosen from the files of the Portland Cement Association¹. These were data sheets of standard freeze-thaw tests which were conducted on soils from actual soil-cement projects throughout the United States during the last 20 years.

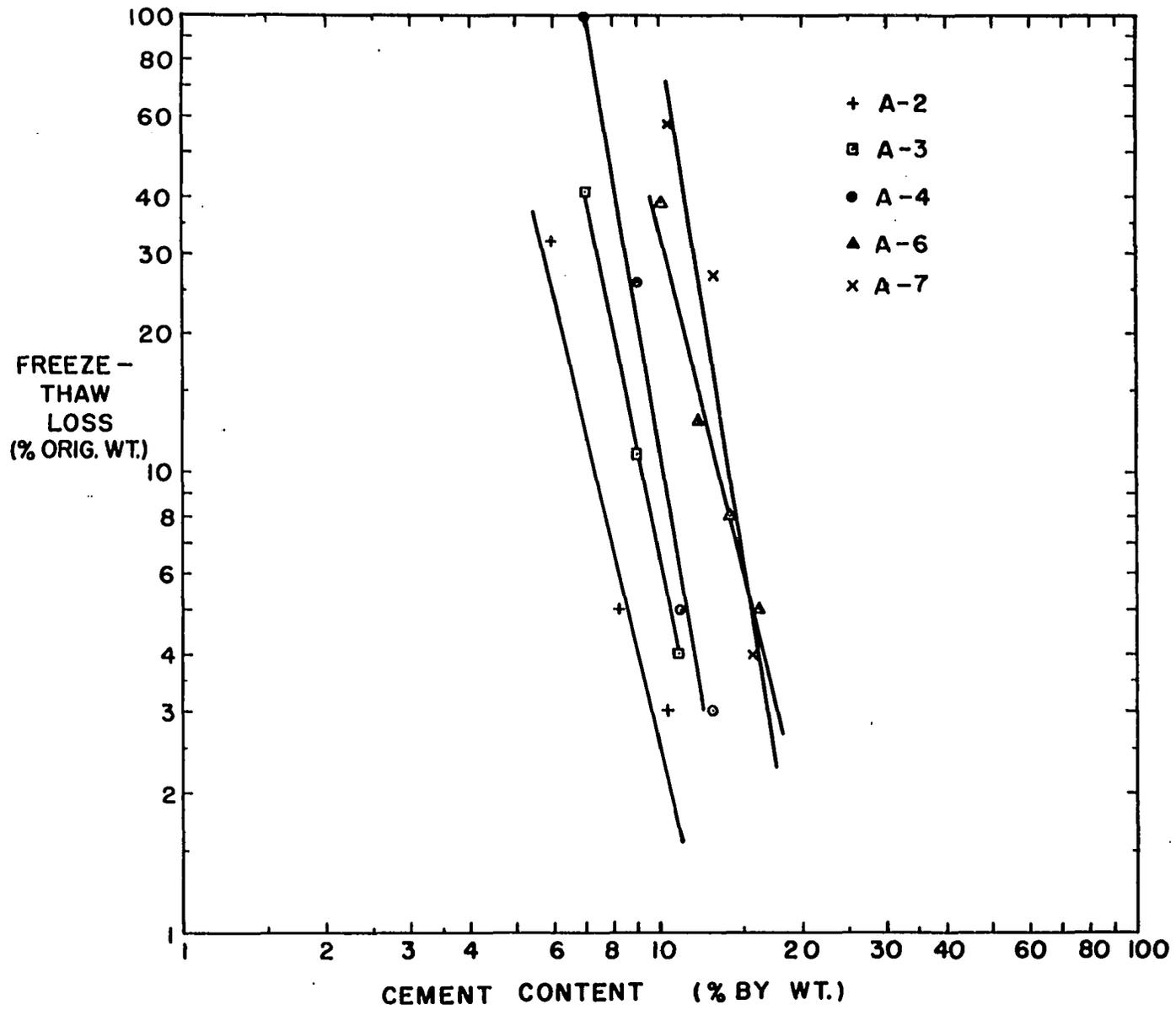
A total of 568 freeze-thaw tests from 172 soil investigations were used. The soils ranged from sandy to clayey, classified as A-2, A-3, A-4, A-6, and A-7 (A.A.S.H.O. designation M 146-49). Any relationship obtained with these data should be valid when applied to the most general situation.

A preliminary graphical investigation indicated that a logarithmic relationship exists between the freeze-thaw loss (% original weight) and the cement content (% by weight) at which the specimen was molded (Figure 1). The cement content by volume was also investigated; however, the cement content on a weight basis gave better relationships.

In order to evaluate the entire mass of data properly, a statistical analysis of the logarithmic relationships was

¹Ray, G. K., Portland Cement Association, Chicago, Illinois. Information on soil-cement test data. Private communication. 1961.

Figure 1. The relationship between the cement content and the freeze-thaw loss of soil-cement mixtures



undertaken. This was done with the use of the "IBM 650" digital computer at Iowa State University of Science and Technology. Closeness of the relationship is best indicated by the square of the correlation coefficient r .

The term r indicates the degree of association or relation between the measured values of one property (cement content) and the corresponding measured values of another property (freeze-thaw loss), for a specified sample population. This term varies from zero, which indicates that a perfect relationship exists and that one property can be accurately predicted from knowledge of the other. In general, a correlation coefficient greater than 0.9 is necessary for the correlation to permit predictions of one value from the other with a reasonable degree of accuracy. In this investigation the proportion of the variance of the freeze-thaw loss that is accounted for by the freeze-thaw loss relation to the cement content is equal to r^2 .

The statistical analysis indicated extremely high r^2 correlations as shown in Appendix A. Thus it appears that the cement content by weight can be related, to within good approximation, to the freeze-thaw loss of a soil-cement mixture. Although A-1 and A-5 soils were not investigated, it would seem logical to assume that these would also follow a logarithmic relationship.

In view of the random sample population used to develop

the relationship, the relationship seems to exist independently of:

- a. The geographical location and type of soil
- b. The technician conducting the freeze-thaw test
- c. The required cement content of the soil

This relationship would also indicate that the freeze-thaw test can be accurately conducted by any trained technician.

The logarithmic relationship is of the form:

$$\log L = A + B \log C$$

where

L = Freeze-thaw loss (% original weight)

C = Cement content (% by weight)

A = Intercept at C = 1% cement

B = Slope

Since soil-cement mixtures with at least three cement contents are usually tested, a logarithmic plot may be constructed with these data. The relationship will reduce the error caused by any possible outliers in the data and reduce the possibility of arriving at erroneous conclusions. By the use of this relationship, the cement content which will give the maximum allowable freeze-thaw loss can be chosen. Economically, the ability to select the exact cement content to produce a specified freeze-thaw loss will result in more

economical mix designs.

Another advantage of such a relationship is the reduction of the number of cement contents necessary to be tested. An experienced tester could conduct the freeze-thaw test with 2 cement contents, and a logarithmic plot connecting these points would establish the relationship. The cement content which will give the required freeze-thaw loss may then be determined. It is best to obtain data which will fall on both sides of the specified freeze-thaw loss; an experienced tester should be able to choose cement contents which produce these results most of the time.

It should be noted that all cement contents below the one that will produce 100% freeze-thaw loss will also indicate 100% loss. In the same manner, all cement contents greater than the one which will give little or no freeze-thaw loss will also produce no freeze-thaw loss. Tests giving 100% freeze-thaw loss and/or 0% loss might therefore lead to erroneous conclusions, and the use of data points between these extremes is recommended.

Slope-Intercept Relationship

Having established a relationship between the cement content and the freeze-thaw loss of a soil-cement mixture, an investigation was undertaken to determine if an overall

correlation exists between the individual logarithmic relationships of the data. A correlation might permit the logarithmic relationship to be established with a decreased number of data points (tested cement contents) and eliminate a great deal of the labor required to conduct the test.

As stated previously, the relationship is of the form:

$$\log L = A + B \log C$$

where A and B are the constants which distinguish one relationship from another. Therefore any correlation of all the cement freeze-thaw equations must involve a relationship between these constants.

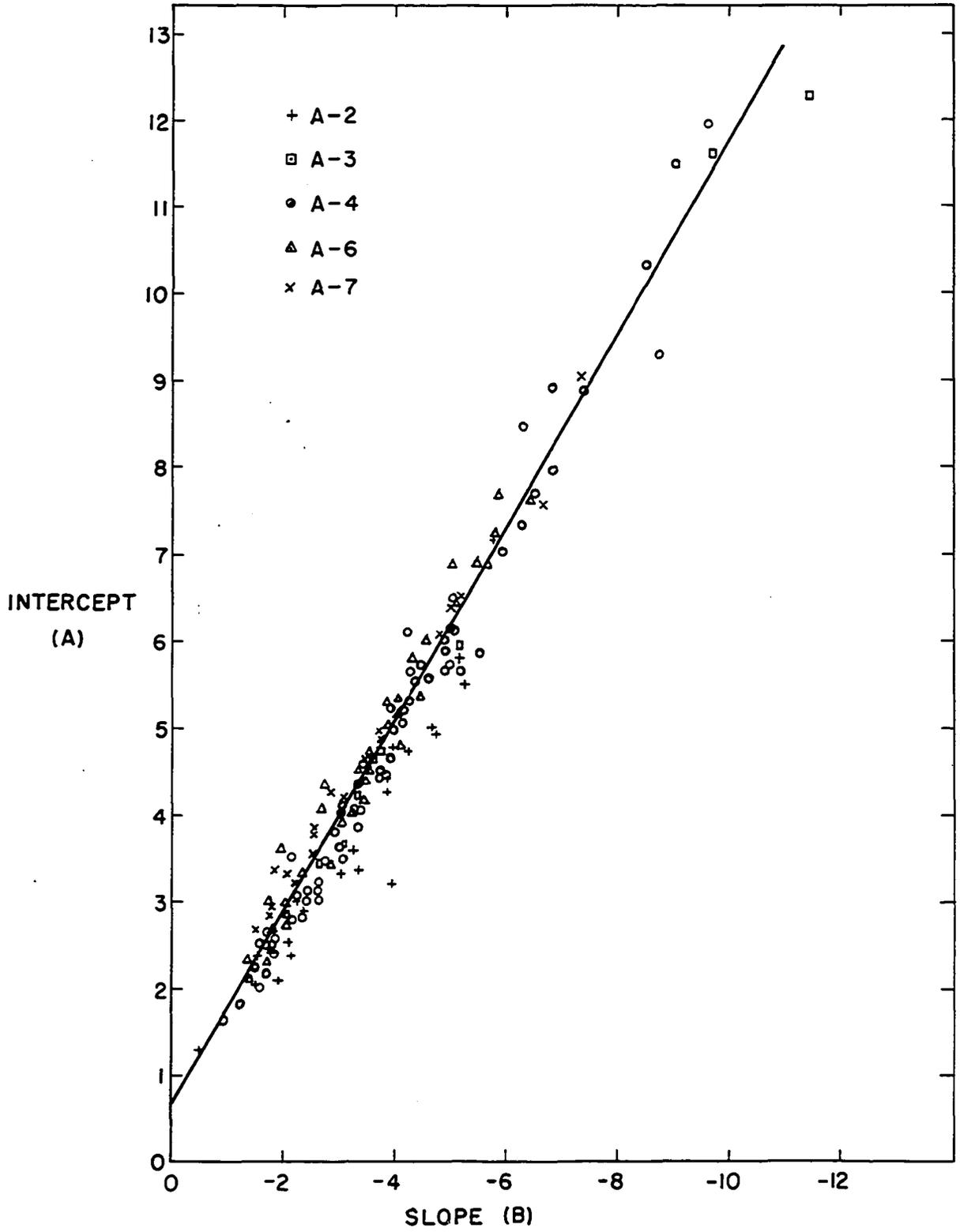
A graphical analysis of these data indicated that an approximate linear relationship exists between the intercepts (A) and the slopes (B) of the equations. This relationship is indicated by the scatter diagram and fitted curve in Figure 2. The granular soils (A-2, A-3) tend to lie below the curve, whereas the fine-grained soils (A-6, A-7) tend to lie above the curve. Thus a difference between the granular and fine-grained soils is apparent in the scatter diagram.

The curve which best represents this scatter diagram can be represented by the equation:

$$A + 1.118B = 0.62$$

From this equation it can be seen that the equation

Figure 2. The relationship between the slope and the intercept of the logarithmic cement freeze-thaw relationships



represented by any point on the curve (an individual cement freeze-thaw relationship) is equal to a constant. Thus:

$$A_1 + 1.118 B_1 = A_2 + 1.118 B_2 = \dots = A_n + 1.118 B_n$$

If the general logarithmic equation is rearranged to:

$$A + B \log C = \log L,$$

it is seen from similarity with the above equation that at $\log C = 1.118$ and $\log L = 0.62$ the equation will be satisfied for all logarithmic relationships. In other words, all cement freeze-thaw relationships theoretically will pass through a common point. This point would be at:

$$L = 4.2\% \text{ freeze-thaw loss}$$

$$C = 13.1\% \text{ cement}$$

Due to the apparent influence of soil type on the scatter diagram, it was decided to investigate each soil type separately to determine more exact relationships. The resulting common intersections of the various soil types are shown in Table 1. It can be observed that as the soil type increases (becomes more fine-grained), the common intersection increases in the "% freeze-thaw loss", whereas the "% cement" remains approximately constant. This is in agreement with the scatter diagram of Figure 2. The individual relationships of the soil types are approximately parallel.

Table 1. Empirical common intersection of the logarithmic freeze-thaw relationships by soil type

Soil type	Common intersection	
	% cement	% freeze-thaw loss
A-2	12.2	2.0
A-3	13.0	2.8
A-4	13.0	2.8
A-6	13.0	5.0
A-7	12.0	10.2

Therefore the slopes (which represent the cement content) would be constant. The transition from granular to clayey soils would produce increasing intercepts (which represent the freeze-thaw loss).

In order to investigate the possibility of the relationships passing through the common intersection, a basic boundary condition must first be satisfied. Since the freeze-thaw loss will decrease as the cement content increases, the slope of the logarithmic relationship must be negative. A soil-cement mixture requiring a cement content greater than the cement coordinate of the common intersection would have to assume a positive slope. This would make a relationship through the intersection invalid.

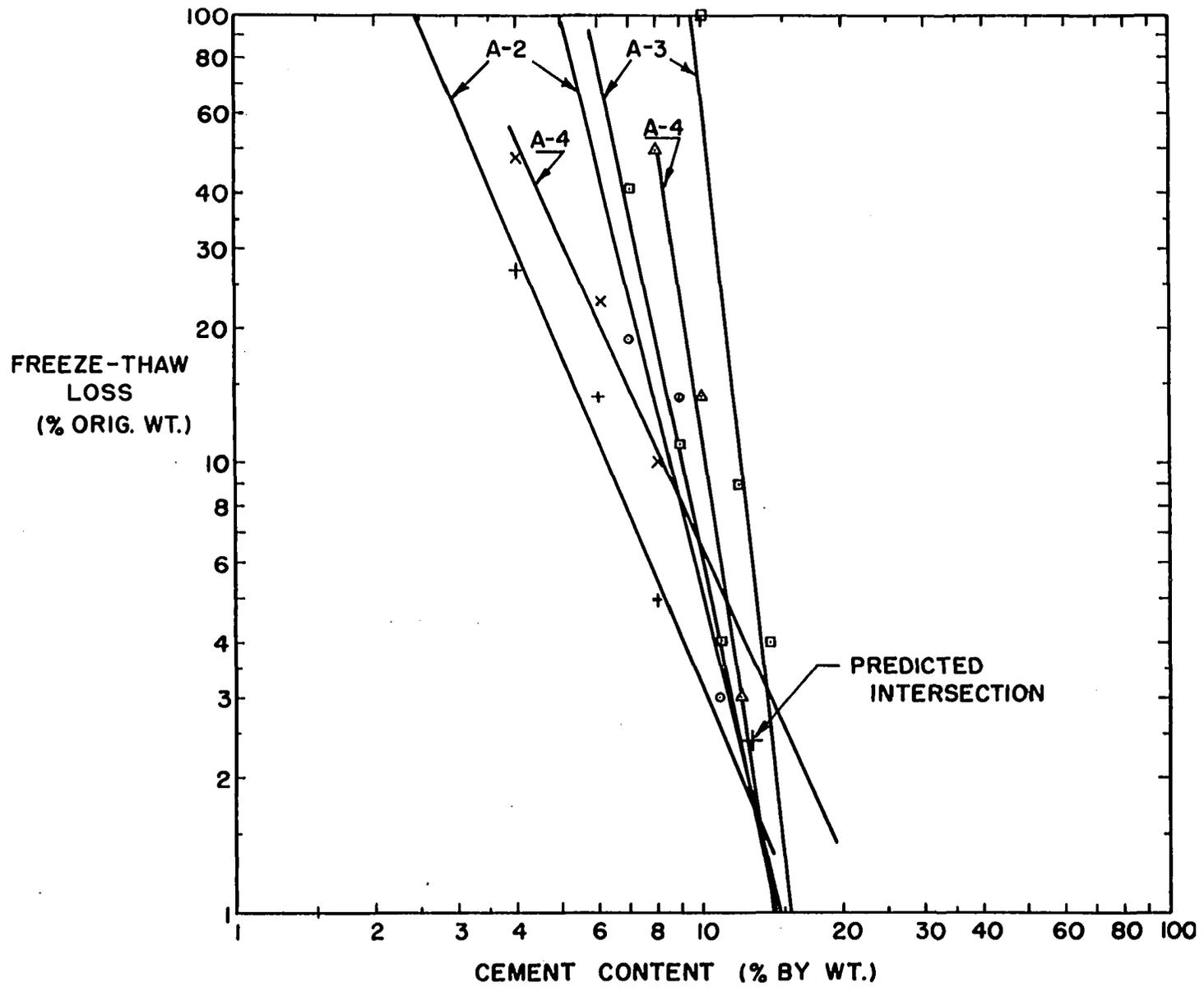
Sandy and silty soils (A-2, A-3, and A-4) have a usual

range of cement requirements from 5% to 12%; clayey soils have a usual range from 9% to 16% cement (28). For the most part this common intersection would not be applicable for A-6 and A-7 soils, and therefore they were eliminated from this investigation. A-2, A-3, and A-4 soils were then investigated to determine if this common intersection was applicable to these data. Since the common intersections for these soil types are similar, an average intersection of $C = 12.6\%$ cement and $L = 2.4\%$ freeze-thaw loss was assumed.

In order to evaluate properly the theory that graphs for these soils would pass through this common intersection, it was decided to plot graphically the cement freeze-thaw relationships using all data points available. Thus the proximity of the relationships to the common intersection could be observed. The graphical analysis of the data indicated that most relationships passed through, or in the vicinity of, the intersection. Typical relationships are shown in Figure 3. An examination of the graph indicates that if any relationship is forced through the intersection, the required cement content (at 10% freeze-thaw loss) will not be greatly affected.

With a common point established through which all relationships of A-2, A-3, and A-4 soils pass, only one cement content would require testing in order to establish the relationship and select the required cement content to produce a

Figure 3. The ability of the logarithmic cement freeze-thaw relationships of granular soil-cement mixtures to pass through the common intersection



durable soil-cement mixture. It would seem plausible that this method would also be applicable to A-1 soils.

Any granular soils which require a cement content greater than approximately 10.5% cement would have to give a nearly vertical or positive slope in order to pass through the intersection. Thus, as in the case of the clayey soils, these would not be adaptable to this method of prediction. An analysis of the data indicated that these soils could be eliminated by placing certain limitations on the result of the freeze-thaw test before applying this method. Other restrictions to increase the validity of the test were also observed. These limitations can be listed as follows:

- a. The cement content of the freeze-thaw specimen should be below 10% cement.
- b. The freeze-thaw loss should fall below 50% soil loss and above the maximum allowable loss.

Granular soils which lend themselves to these criteria should require only one freeze-thaw specimen to evaluate the required cement content.

A reasonable test to evaluate this theory, in keeping with the manner in which this method of prediction would be carried out in practice, is as follows:

1. Determine the required cement content from the logarithmic cement freeze-thaw relationships

using all data points (actual cement requirement).

2. Subject to the prescribed limitations, determine the required cement content from the relationship connecting the data point in each set that had the lowest cement content with the common intersection (predicted cement requirement).
3. Compare the predicted cement requirement with the actual cement requirement.

As a preliminary, 10% freeze-thaw loss was considered the maximum allowable loss for all soil types in order to produce uniform results. Fifty-four sets of data were found to conform to the prescribed limitations. As an added test the actual and predicted cement requirements were compared to the cement requirements recommended by the Portland Cement Association (PCA). However, an absolute comparison is not valid for A-2 and A-3 soils since the PCA cement content for these soils was evaluated at 14% freeze-thaw loss. These results are shown in Appendix A. It can be observed that despite the slightly different criteria, the predicted and actual cement requirements compare favorably with those recommended by the PCA.

Perhaps the best measure of the degree of accuracy of the predicted intersection is the use of the "Standard error of estimate (S_c)". This is a measure of deviation or degree of scatter of the points around the regression equation (in

this case the logarithmic cement freeze-thaw relationship). It provides an estimate of the uncertainty of the prediction of the predicted cement content from the actual cement content. Assuming that a normal distribution of errors is valid, about 66% of the observations will fall within the standard error; about 95% of the observations will fall within two standard errors (25).

The standard error of estimate of the predicted from the actual cement requirements is shown in Table 2. The S_c of the individual soil types ranges from 0.65 to 0.75 with an average value of 0.65. The low average value is due to the large sample population of A-4 soils compared to the other soils. It can therefore be concluded, in light of the existing data, that the use of the predicted intersection will produce an accuracy of $\pm 0.65\%$ cement for 66% of the time, and an accuracy of $\pm 1.30\%$ cement for 95% of the time. It is realized that these percentages may not be entirely accurate, since the variation measured by S_c includes a possible element of bias (inaccuracy), as opposed to error (imprecision), originating from the use of the common intersection point.

It would also be of value to note whether the predictions were on the safe or unsafe side (respectively greater or less than the actual cement requirement). These results are shown in Table 3. It is generally observed that A-2 and A-3 soils are on the safe side while A-4 soils are about

Table 2. The standard error of estimate of the predicted cement requirements from the actual cement requirements by soil type

Soil type	S_c (% cement)	$2S_c$ (% cement)
A-2	0.75	1.50
A-3	0.66	1.32
A-4	0.65	1.30
Average	0.65	1.30

Table 3. Total number of safe and unsafe predictions by the use of the common intersection method by soil type

Soil type	Safe	Unsafe	Exact	Total
A-2	8	4	2	14
A-3	4	0	0	4
A-4	14	18	4	36

evenly distributed. This is what might be expected from the predicted intersections of the individual soil types (Table 1).

An A-2 soil has a predicted intersection at 2.0% freeze-thaw loss. If the relationship were passed through the assumed intersection of 2.4% freeze-thaw loss, the slope of the relationship would be made less negative. Thus the relationship would indicate a higher cement requirement and thereby predict on the safe side. An A-4 soil passed through an intersection lower than the one assumed would be expected to underpredict. However, due to the normally high cement requirements of A-4 soils, a steep slope is expected. Therefore the relationship through the assumed intersection is not greatly altered, and an equal distribution results. A-3 soils would also be expected to underpredict, but perhaps due to the small sample population this was not observed. It is of interest to note that if the A-6 and A-7 soils were passed through the intersection, the invalidity of the line would also cause underprediction.

Therefore it can be concluded that the predicted intersection can be employed to produce significant results. By the use of the standard error of estimate, adequate safety factors can be introduced to produce an accurate estimate of the required cement content. The reduction in the number of cement contents to be tested will significantly reduce the

labor required to conduct the freeze-thaw test. For example, two specimens may be molded at the same cement content, enhancing the accuracy of the test without doubling the labor.

A freeze-thaw test conducted with one cement content, subject to the prescribed limitations, would be sufficient to evaluate the required cement content properly. For any random soil it might be difficult to choose a cement content which would fall within the limitations. However, in the case of a soil series (15) or soils where the cement requirement is approximately known, a cement content can be selected and the method used advantageously. For example, this method could be used in conjunction with the "Short cut test procedures for sandy soils" (24) for major projects in order to determine better the required cement content. Although A-6 and A-7 soils were not investigated, this method might also be applicable for certain of these soils which require low cement contents.

Figure 4 is a graphical representation of the selection of the required cement content with one freeze-thaw specimen at 10% freeze-thaw loss. Similar graphs can be constructed for other allowable losses. Safety factors are easily incorporated into this type of graph.

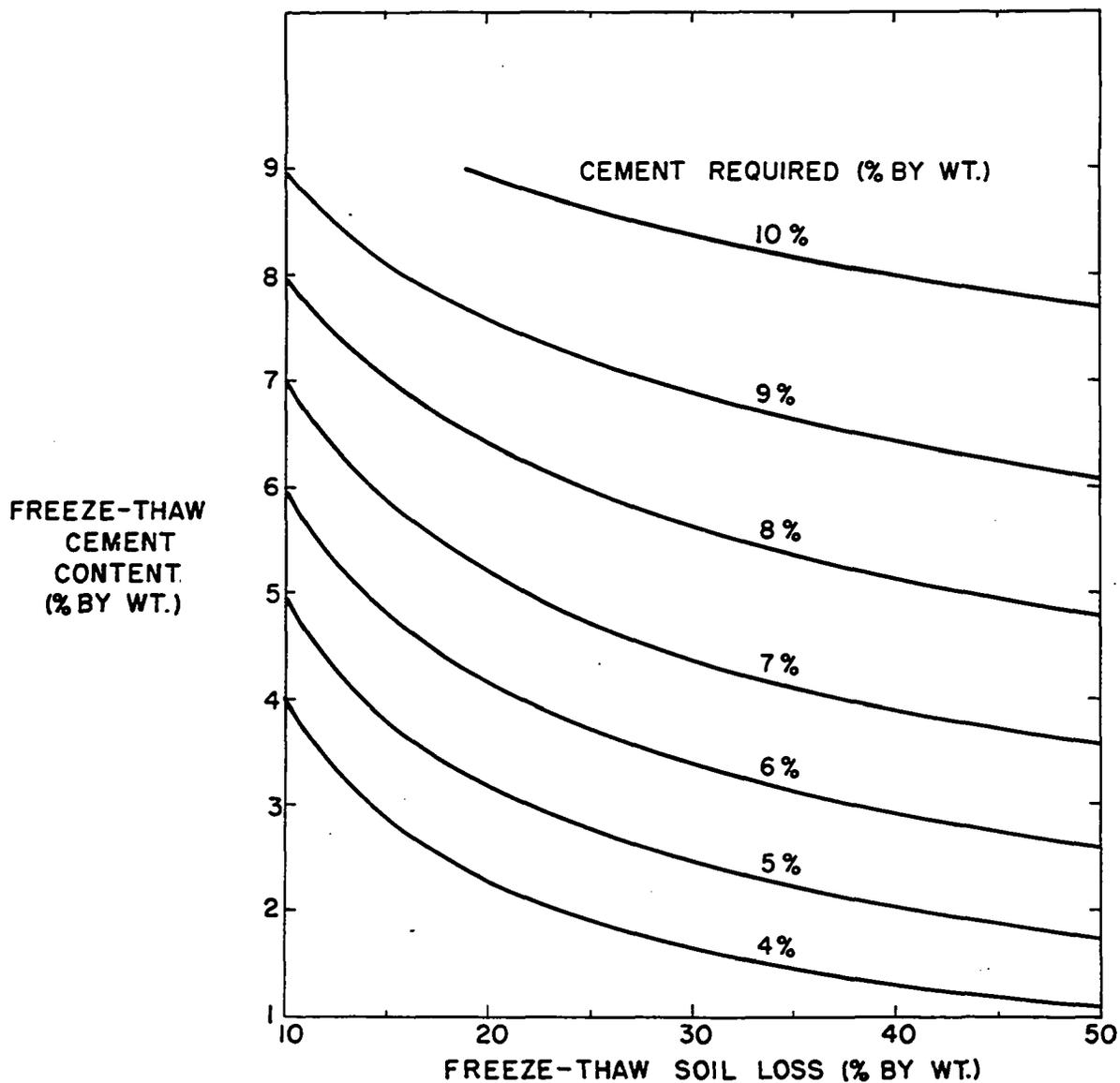


Figure 4. Recommended cement content of soil-cement mixtures at 10% freeze-thaw loss by the common intersection method, applicable to A-2, A-3, A-4, and probably A-1 soils

INVESTIGATION PART II

The number of cycles of freeze-thaw testing was developed from exploratory tests on freezing temperatures, freezing time, thawing time, and soaking time. Twelve cycles were selected since they produced interpretable data and had a practical time limit. The brushing procedure was developed to remove the loosened material, which resulted in more consistent and reproducible results. However, the fact that the standard test requires from 6 to 7 weeks to complete restricts its use in some instances.

Any method either eliminating or reducing the number of cycles involved in the freeze-thaw test would be a major step in accelerating the test. Several correlated short-cut tests have been developed. One example is the "Department of Agriculture Soil Identification System " (19). Soils of the same series, horizon, and texture often require the same amount of cement. This will reduce the number of complete tests necessary when soil survey maps are available. The "Short-cut method for granular soils" (24) correlates data from durability tests to predict the required cement content. The gradation, density, and 7 day compressive strength are the only requirements to determine the cement requirement. The "Glycerol surface area determination" for fine-grained soils containing less than 45% silt has been correlated with

the required cement content from durability tests (10, 17).

The forementioned short-cut methods have been a valuable tool in establishing the required cement content. However, due to the inherent physical and chemical diversity of soils, the only soundly established methods of determining the required cement content of a soil-cement mixture are the standard wet-dry and freeze-thaw tests. Therefore the Portland Cement Association has been developing the accelerated freeze-thaw test (27).

The use of the logarithmic cement freeze-thaw relationship might provide another method to shorten the test without loss in the significance of the test. Twelve cycles of freeze-thaw is not necessarily the only number of cycles which will produce significant results. Other cycles which produce a logarithmic relationship might be useful in altering the test without a loss in its significance.

Influence of the Number of Freeze-Thaw Cycles on the Logarithmic Relationship

Since the logarithmic cement freeze-thaw relationship is valid at 12 cycles of freeze-thaw testing, it would be of interest to investigate the possibility of a similar relationship existing at other numbers of cycles. An extensive investigation was undertaken by Felt (12) to observe the

influence of cement content on the freeze-thaw loss. Freeze-thaw cycles from 12 to 96 cycles were conducted. These data were investigated to observe the influence of the number of freeze-thaw cycles on the logarithmic relationship.

A graphical analysis was first conducted. It can be seen in Figure 5 that the freeze-thaw test conducted at greater than 12 cycles will also produce an excellent relationship. It can also be observed that, as a first approximation, the relationships of each soil appear parallel. In order to confirm these observations, a statistical analysis was conducted with the "IBM 650" digital computer. These results are shown in Table 4.

It is seen that the r^2 correlations are again very high for all cycles. The slopes of the A-2 and A-4 relationships appear to be about the same (3.76 average), while the slope of the A-7 soil increases directly with the number of cycles according to the equation:

$$S = 0.056 C_1 + 2.4$$

where:

$$S = \text{Slope (negative)}$$

$$C_1 = \text{Number of freeze-thaw cycles}$$

In order to investigate the applicability of these relationships below 12 cycles, data of 4 soils from a recent PCA study (26) were used. These soils were classified as

Figure 5. The effect of the number of freeze-thaw cycles on the logarithmic cement freeze-thaw relationship

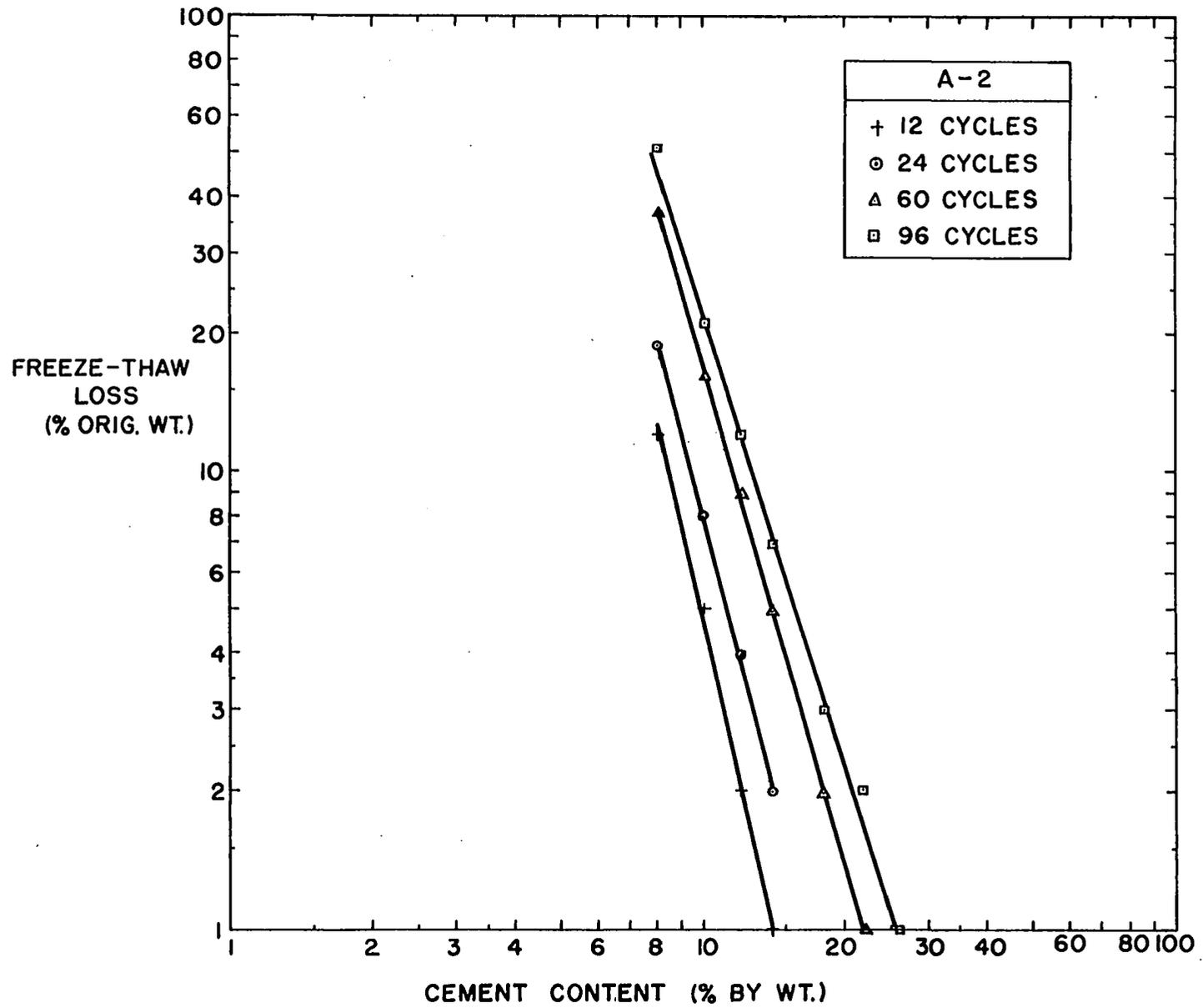


Figure 5. (Continued)

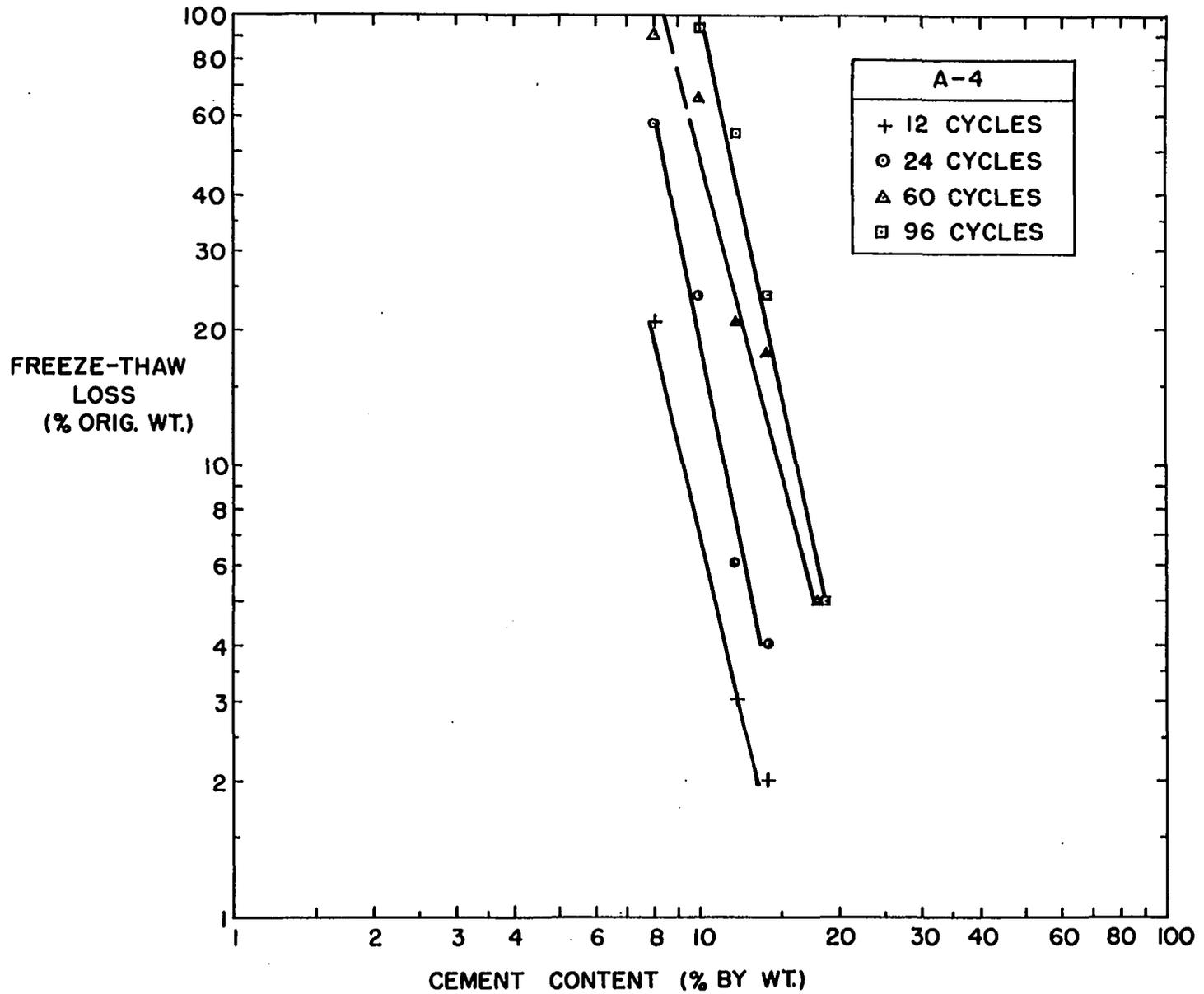


Figure 5. (Continued)

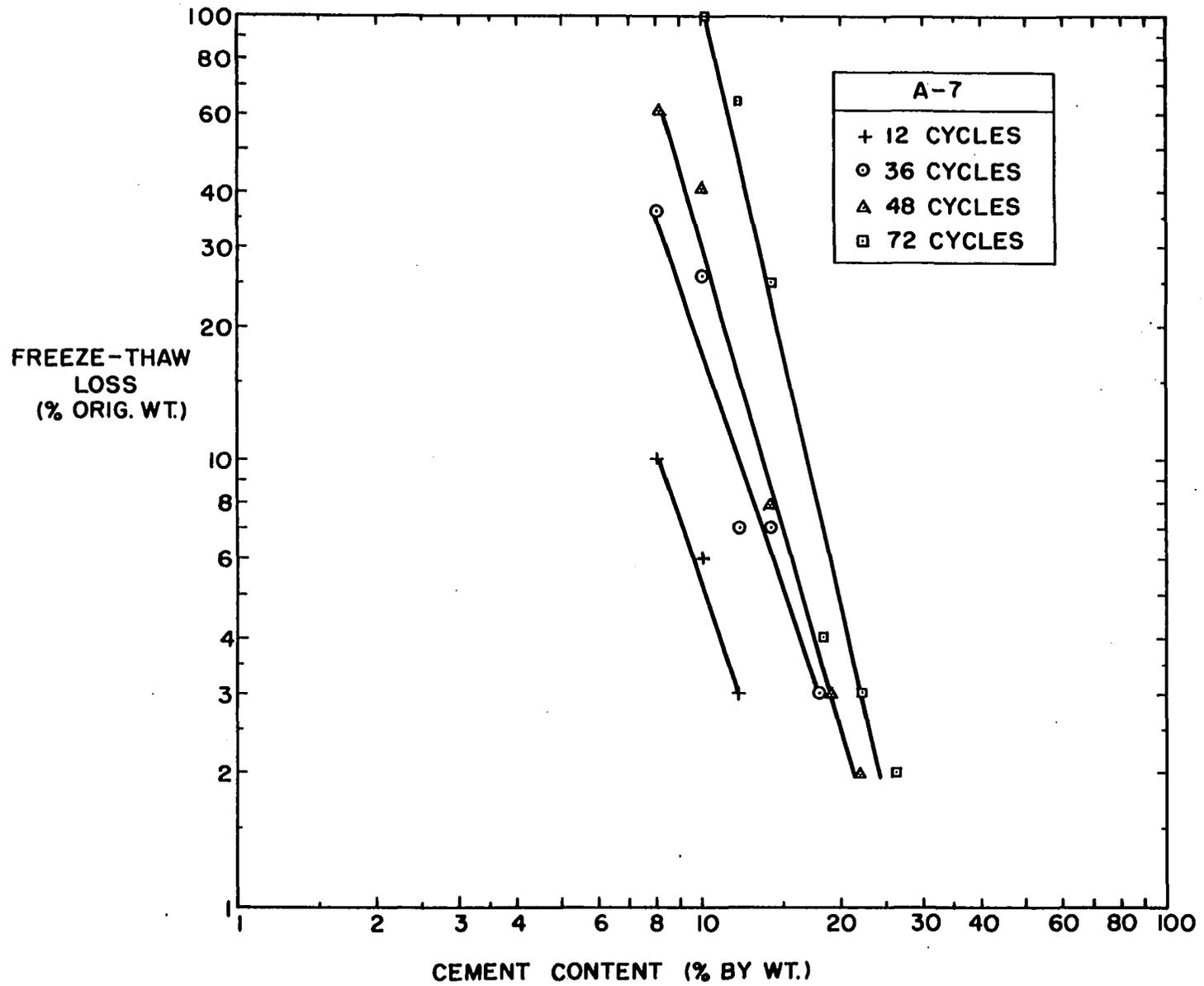


Table 4. r^2 correlation coefficients and slopes of logarithmic cement freeze-thaw relationships at various cycles of freeze-thaw loss

Soil type	Number of freeze-thaw cycles	r^2	Slope
A-2	12	0.997	4.48
	24	0.994	3.70
	60	0.999	3.57
	84	0.997	3.80
	96	0.995	3.24
A-4	12	0.987	4.28
	24	0.960	4.32
	60	0.949	3.22
	84	0.935	3.48
	96	0.917	3.52
A-7	12	0.930	3.10
	36	0.932	4.48
	48	0.954	5.02
	72	0.956	6.51

A-1-b(0), A-4(5), A-4(8) and A-6(10). Freeze-thaw loss at 2, 4, 6, 8, 10 and 12 cycles of testing was evaluated. Either 4 or 5 cement contents were used for each soil.

A graphical analysis indicated that excellent logarithmic cement freeze-thaw relationships exist between 6 and 12 cycles. Below 6 cycles the freeze-thaw loss was almost negligible and the relationships were poor. The A-1-b and A-4 soils produced parallel relationships; the slope of the A-6 soil increased directly with the number of cycles. Thus the phenomena of the relationships of the data of Felt (12) and the Portland Cement Association (26) are similar.

Based on the foregoing analysis of the 7 soils, it is concluded that:

1. A valid cement freeze-thaw relationship exists for all soils between 6 and 96 cycles of testing.
2. The relationships are parallel for A-1 to A-4 soils (the slopes of the relationships are equal).
3. The slopes of the relationships for A-6 and A-7 soils increase directly with the number of cycles.

These observed results would indicate that the logarithmic relationship exists regardless of the number of cycles used to conduct the freeze-thaw test. This would indicate that reproducible and interpretable results might

be obtained at other than 12 cycles of freeze-thaw testing. It is apparent that the specifications for maximum freeze-thaw loss can be altered as the number of cycles are varied. For example, in the case of the A-2 soil, 14% freeze-thaw loss indicates a cement content of 7.9% cement. This cement content corresponds to 50% loss at 96 cycles. Thus if 96 cycles were used as the criteria in the freeze-thaw test, 50% loss would be considered the maximum allowable freeze-thaw loss.

Figure 6 is a plot of the maximum freeze-thaw loss permitted as the number of cycles of the 3 soils of Felt (12) are varied. A transition in the shape of the curves can be observed from the sandy to clayey soils. The A-4 soil assumes a linear relationship. Figure 7 indicates that the A-2 soil becomes linear on a logarithmic plot, while the A-7 soil becomes linear on a semilogarithmic plot. Similar relationships were observed with the PCA data (26).

By the use of these linear relationships it should be possible to choose a maximum freeze-thaw loss at any number of cycles. This would be advantageous in developing a method to conduct the freeze-thaw test at a reduced number of cycles. For example, at 6 cycles of freeze-thaw the required cement content for the A-2 soil could be evaluated at 9% freeze-thaw loss. In the same way the A-4 and A-7 cement requirements could be evaluated at 7% and 5% freeze-thaw loss respectively.

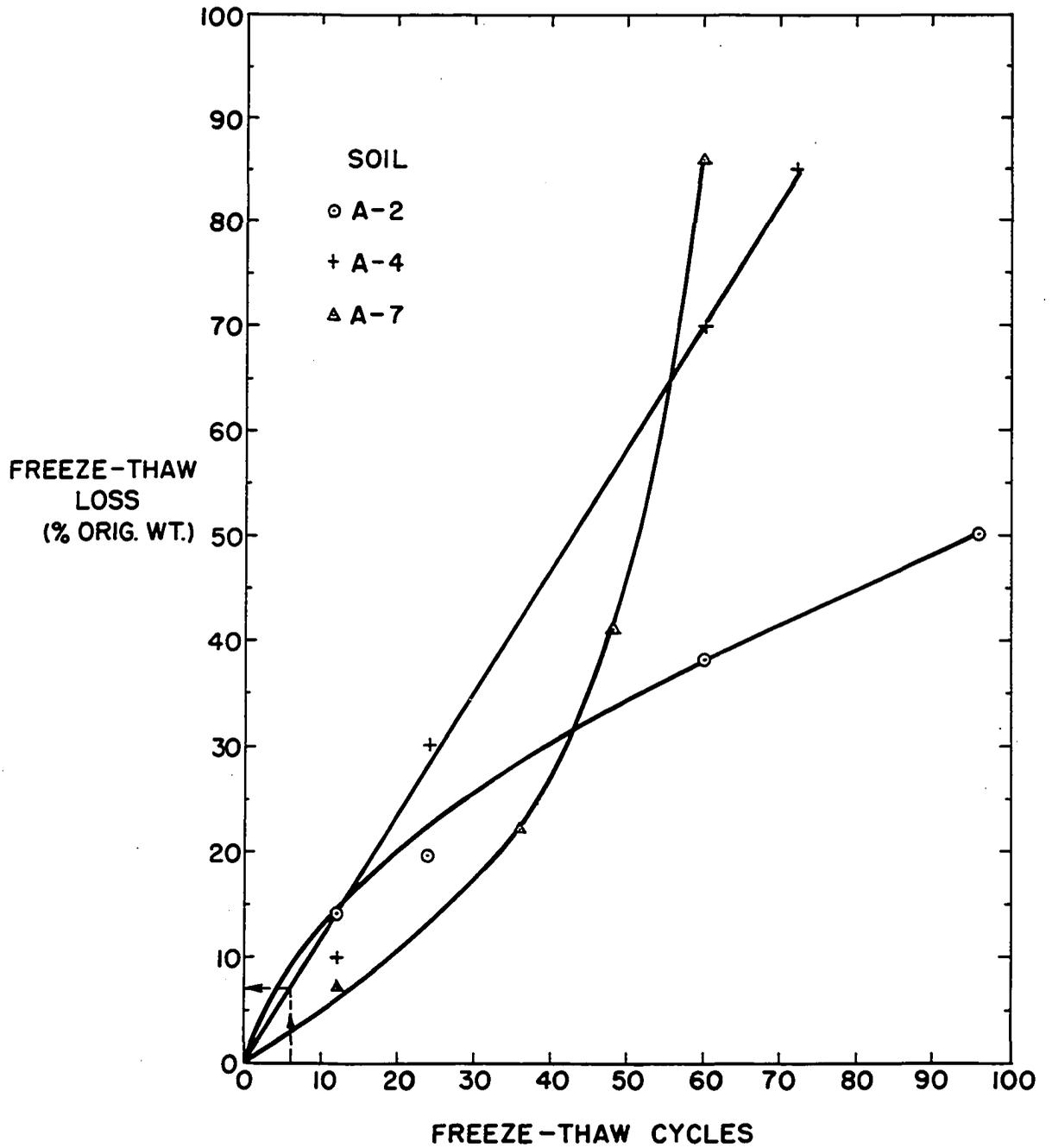
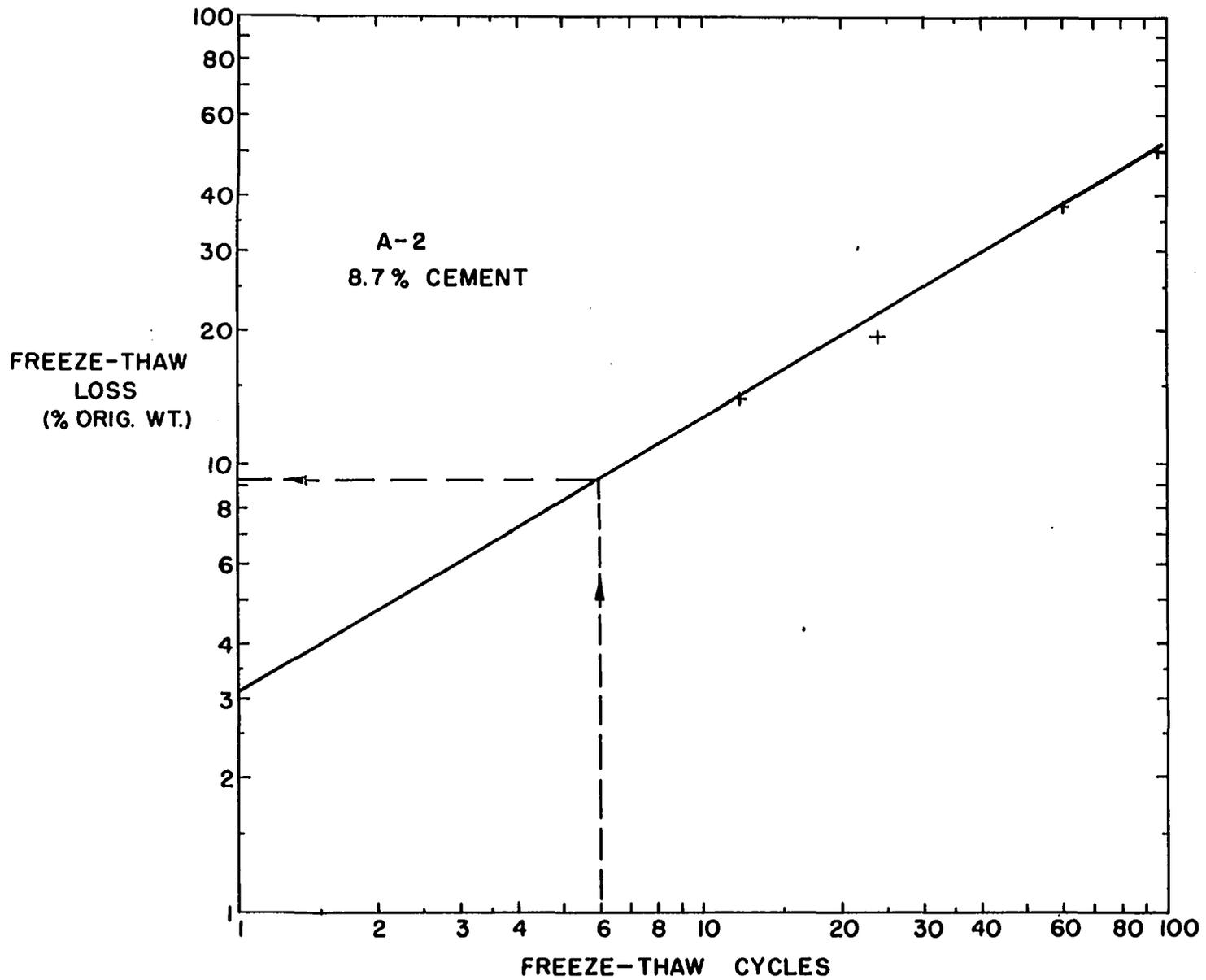


Figure 6. The effect of the number of freeze-thaw cycles on the maximum allowable freeze-thaw loss

Figure 7. Linear relationships of A-2 and A-4 soils required to evaluate the maximum allowable freeze-thaw loss at a reduced number of cycles



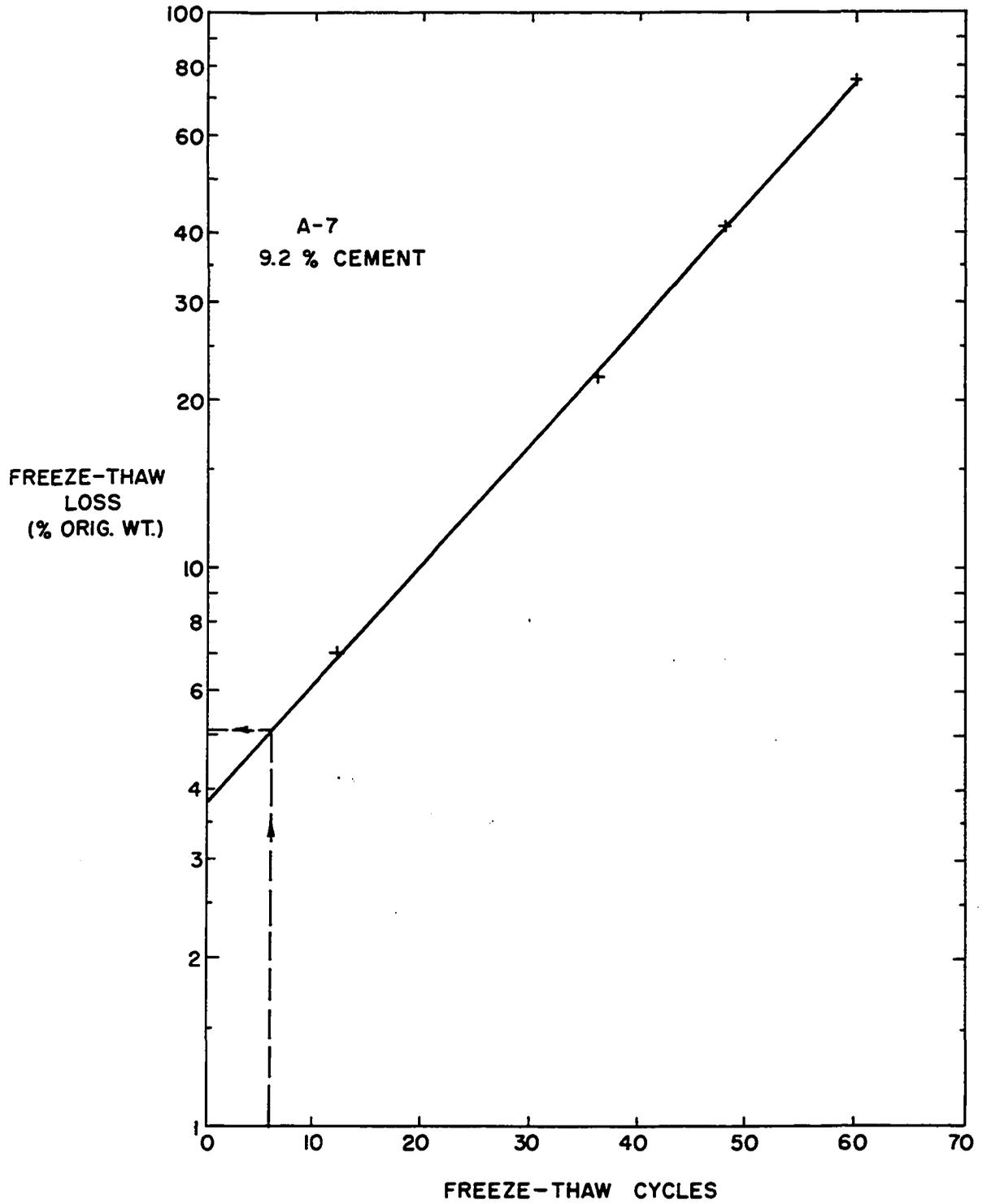


Figure 7. (Continued)

This reduction in the number of cycles would reduce by 50% the time required to conduct the test.

The reduction in the number of cycles was further investigated with data supplied by the PCA¹. Seventy-three sets of data representing most soil types were investigated. Logarithmic relationships at 6 and 12 cycles were determined. The cement content at the specified 12 cycle freeze-thaw loss was chosen. This cement content was then used to evaluate the freeze-thaw loss which would result from 6 cycles of testing. Thus the relationships evolved in the first portion of this investigation have been found useful in selecting the specified freeze-thaw loss required at 6 cycles of testing. The average freeze-thaw loss and the standard deviation at 6 cycles are shown in Table 5. It is seen that the average freeze-thaw loss at 6 cycles to determine the required cement content generally decreases as the soil type increases.

The standard deviations observed are due to both the 6 cycle and 12 cycle logarithmic relationships. Thus the standard deviation due only to imprecision of the 6 cycle interpolation is less than that observed in Table 5. The table is an evaluation of the required 6 cycle loss predicted from a knowledge of the 12 cycle loss. However, due to the

¹Packard, R. G., Portland Cement Association, Chicago Illinois. Information on soil-cement test data. Private communication. 1963.

Table 5. Average freeze-thaw loss after 6 cycles of testing necessary to produce the required cement content of individual soil types

Soil type	Number of soils tested	Freeze-thaw loss at 6 cycles (% original weight)	
		Average	Standard deviation
A-1	7	6.4	2.2
A-2	14	6.5	1.8
A-3	5	5.0	1.1
A-4	22	3.3	1.2
A-6	13	3.2	0.9
A-7	12	2.3	1.2

Table 6. Average freeze-thaw loss after 6 cycles of testing necessary to produce the required cement content of freeze-thaw soil groups

Soil type	Number of soils tested	Freeze-thaw loss at 6 cycles (% original weight)	
		Average	Standard deviation
A-1 A-2 A-3	26	6.0	1.7
A-4	22	3.3	1.2
A-6 A-7	25	2.8	1.0

parallelism observed in the relationships of the granular soils, the predicted standard deviation of the loss at 12 cycles from a knowledge of the 6 cycle loss would be the same on the logarithmic scale. Therefore, the coefficient of variation (the relative standard deviation) of 12 cycles of freeze-thaw testing predicted from 6 cycles would be the same as the coefficient of variation of 6 cycles of freeze-thaw testing predicted from 12 cycles.

These soils have been grouped according to their allowable freeze-thaw loss at 12 cycles; i.e., at 14%, 10% and 7% freeze-thaw loss. The average freeze-thaw loss and the standard deviation of these groups are shown in Table 6. The standard deviation measures the dispersion of a series; the greater the spread of the series, the greater the standard deviation of the series. Assuming a normal distribution of dispersion, 68.27% of the observations will fall within the standard deviation; 95.45% of the observations will fall within two standard deviations. The results in Table 6 indicate that at 6 cycles a reduced freeze-thaw loss is obtained which is fairly consistent for each soil group. Thus it is evident that 6 cycles of testing might produce interpretable and reproducible results from which a valid criteria can be established.

Summary of Part II

The foregoing investigation is based upon data from only a few soils and is not intended to produce any definite conclusions concerning the reduction in the number of freeze-thaw cycles. It was conducted to show that the use of the cement freeze-thaw relationship might provide an effective method of reducing the number of cycles required for the test. It is believed that equally reproducible and significant results can be obtained at a reduced number of cycles.

Future research may utilize this method to good advantage. Further investigations with a great many soils would be necessary to establish the relationships of the slopes to the number of freeze-thaw cycles. This could be accomplished in the course of normal freeze-thaw testing of soil-cement mixtures by the Portland Cement Association. Samples could be weighed after each cycle and the relationships established. The determination of definite trends for various soil types could allow a reduced number of cycles to be established. A minimum number of cycles which would produce interpretable results could be forthcoming. The use of this method in conjunction with the accelerated freeze-thaw test could reduce the entire freeze-thaw test to a minimum period of time.

CONCLUSIONS

Although the freeze-thaw test was originally devised to measure the hardening effect of portland cement on soils, it has become useful as a reliable criterion for the determination of the durability of a soil-cement mixture. The great disadvantage of this test is the large amount of time and labor necessary to complete the freeze-thaw test. This investigation attempts, by correlation analysis, to reduce the amount of testing required to conduct a reliable freeze-thaw evaluation of a soil-cement mixture.

An excellent logarithmic relationship was found to exist between the cement content and the freeze-thaw loss of a soil-cement mixture. This relationship is useful for determining the cement requirement which will produce the exact allowable freeze-thaw loss. This could result in more economical mix design of soil-cement mixtures. Freeze-thaw tests with two cement contents will establish the relationship. When more than two cement contents are used, the relationship will obviate any outliers which might exist in the data. This will reduce any error in testing.

The logarithmic relationships for A-2, A-3, and A-4 soil-cement mixtures were found, approximately, to intersect at a common point. It is conjectured that all granular soil-cement mixtures follow this rule. This can be of great value

when properly applied. A granular soil for which the approximate cement content (below 10%) is known would require a freeze-thaw test with one cement content to establish the relationship and determine, to within reasonable approximation, the required cement content. The standard error of estimate of this method was found to be 0.65% cement.

The logarithmic relationship was found independent of the number of freeze-thaw cycles used in the test. This introduces the possibility of conducting the freeze-thaw test at a reduced number of cycles. Preliminary investigation of this method indicates that the time involved in the freeze-thaw test can be reduced by 50% without loss in the significance of the test.

By the use of these relationships the time and labor required in the conduct of the freeze-thaw test can be greatly reduced.

Suggested abbreviated procedures utilizing both relationships are in Appendix B. Comparison of future results of these procedures and results of the full standard test will further indicate reliability of the abbreviated tests.

RECOMMENDATIONS FOR FUTURE RESEARCH

It is believed that this investigation reveals several opportunities for future research in improving the freeze-thaw test. These may be listed as follows:

1. A study should be made to determine the reproducibility of freeze-thaw loss at both 6 and 12 cycles of testing. This will determine whether the use of 6 cycles of testing will produce interpretable and reproducible results equal to those presently obtained after 12 cycles of freeze-thaw testing.
2. An investigation of the 6 cycle logarithmic relationships might indicate that certain soils will give relationships which pass through a given point, similar to the relationships shown for granular soils at 12 cycles of testing. This would reduce the testing procedure to a minimum.
3. The use of the accelerated freeze-thaw test (27) in conjunction with the abbreviated methods outlined in this investigation would reduce the time required to conduct the freeze-thaw test to a few days. The compatibility of these methods should be studied to determine the applicability of combining these procedures.

4. Field performance of soil-cement mixtures with cement requirements evaluated with 6 cycles of testing should be used to check the validity of the use of a reduced number of cycles in the freeze-thaw test.

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ACKNOWLEDGMENTS

The author is indebted to the late D. T. Davidson, former Professor of Civil Engineering at Iowa State University of Science and Technology, without whose guidance and advice this investigation would not have been possible.

Sincere appreciation is expressed to H. T. David, Associate Professor of Statistics at Iowa State University of Science and Technology, and to R. L. Handy, Professor of Civil Engineering at Iowa State University of Science and Technology, for their guidance in the planning, research, analysis and writing connected with this investigation.

Thanks are also extended to T. Demirel, J. G. Laguros, J. B. Sheeler and other members of the Soil Research Laboratory, Engineering Experiment Station, for the information and advice that was given during the investigation and in the interpretation of results.

Appreciation is expressed for the information furnished by L. T. Norling, G. K. Ray, and R. G. Packard of the Portland Cement Association, Chicago, Illinois.

♦

APPENDIX A

Table 7. r^2 correlation coefficients of logarithmic cement freeze-thaw relationships and comparison of cement requirements

Soil type	r^2 correlation coefficient ^a	Cement requirements (% by weight)		
		Actual ^b	Predicted ^b	PCA ^c
A-2 ^d	0.990	5.0	5.0	6.0
	0.999			
	0.981	6.8	8.0	7.1
	0.999	8.7	8.9	8.2
	0.950	7.6	8.3	7.0
	0.811	12.0	10.7	12.0
	0.999	6.2	6.3	6.1
	0.944	5.9	6.6	6.1
	0.793			
	0.953	7.8	7.8	7.8
	0.798			
	0.823	9.0	8.5	9.3
	0.916			
	0.998	7.8	7.5	7.4
	0.975			
	0.896			
	0.994	4.5	4.8	5.0
0.986	3.8	5.0	4.6	
0.950	6.5	6.4	6.3	
0.996	5.1	5.9	5.4	

^a r^2 correlation coefficients of logarithmic cement freeze-thaw relationships.

^bComparison of the actual and predicted cement requirements by the slope-intercept relationship at 10% freeze-thaw loss.

^cRecommended cement requirements of the Portland Cement Association.

^dCement requirement determined at 14% freeze-thaw loss for PCA and at 10% freeze-thaw loss for actual and predicted cement contents.

Table 7. (Continued)

Soil type	r^2 correlation coefficient ^a	Cement requirements (% by weight)		
		Actual ^b	Predicted ^b	PCA ^c
A-3 ^d	0.992	7.3	7.5	7.9
	0.996	8.0	8.4	9.8
	0.987	9.7	10.5	10.6
	0.951			
	0.999	9.2	9.4	9.3
	0.947			
	0.999			
A-4 ^e	0.999	8.7	8.8	9.0
	0.949			
	0.846	6.8	7.5	8.7
	0.976	9.0	8.5	9.2
	0.975			
	0.907	9.9	10.0	10.3
	0.971			
	0.908			
	0.987	10.4	10.2	10.8
	0.982			
	0.945	7.2	7.2	7.6
	0.950			
	0.987			
	0.938	9.7	9.9	10.5
	0.937	7.5	8.3	8.1
	0.972			
	0.910			
	0.829	9.7	8.4	8.8
	0.948			
	0.878	7.4	8.6	8.3
	0.924			
	0.978			
	0.923	6.9	7.3	8.2
0.905	9.6	9.8	10.5	
0.905	8.2	8.1	9.3	
0.882				
0.978				
0.913				
0.806	11.2	10.0	11.9	
0.965				
0.866				

^eCement requirement determined at 10% freeze-thaw loss.

Table 7. (Continued)

Soil type	r^2 correlation coefficient ^a	Cement requirements (% by weight)		
		Actual ^b	Predicted ^b	PCA ^c
A-4 ^e	0.900			
	0.979			
	0.997			
	0.874			
	0.868			
	0.908	10.0	9.5	11.0
	0.934			
	0.875			
	0.965			
	0.999	9.3	9.5	10.7
	0.932			
	0.950	5.9	5.6	6.1
	0.959			
	0.983	7.6	7.4	8.1
	0.806	10.6	10.8	12.1
	0.994			
	0.894	9.1	9.4	9.8
	0.963			
	0.938	8.7	8.5	9.0
	0.946	8.8	10.1	10.1
	0.934			
	0.994	12.2	10.5	12.6
	0.990	10.0	9.6	10.2
	0.980	10.3	10.3	10.8
	0.962	8.2	8.2	9.0
	0.998	10.2	9.4	10.3
	0.974			
	0.893	8.3	8.6	9.0
	0.993	8.0	7.8	9.3
	0.963	7.7	7.5	7.6
	0.978	7.2	6.6	7.6
	0.891			
	0.873	7.0	6.8	7.6
	0.992			
0.928				
0.878	6.5	7.2	6.8	
0.982	8.2	7.4	8.1	
0.998				
0.806				
0.897				
0.986				
0.977	6.8	6.2	7.2	
0.968	7.2	7.2	7.2	

Table 7. (Continued)

Soil type	r^2 correlation coefficient ^a	Cement requirements (% by weight)		
		Actual ^b	Predicted ^b	PCA ^c
A-6	0.997			
	0.991			
	0.953			
	0.999			
	0.940			
	0.998			
	0.991			
	0.993			
	0.965			
	0.907			
	0.969			
	0.943			
	0.928			
	0.998			
	0.974			
	0.990			
	0.991			
	0.973			
	0.997			
	0.992			
	0.985			
	0.948			
	0.977			
	0.999			
	0.995			
	0.831			
	0.896			
	0.994			
	0.997			
	0.986			
	0.999			
	0.914			
	0.880			
0.997				
0.948				
0.998				
0.999				
0.910				
0.888				
0.992				
0.988				

Table 7. (Continued)

Soil type	r^2 correlation coefficient ^a	Cement requirements (% by weight)		
		Actual ^b	Predicted ^b	PCA ^c
A-6	0.958			
	0.820			
	0.931			
	0.915			
	0.889			
A-7	0.997			
	0.860			
	0.955			
	0.909			
	0.886			
	0.969			
	0.979			
	0.961			
	0.998			
	0.999			
	0.879			
	0.937			
	0.922			
	0.932			
	0.937			
	0.972			
	0.927			
	0.983			
0.979				
0.959				
0.999				
0.909				
0.993				

APPENDIX B

Tentative Abbreviated Freeze-Thaw Tests
for Determining Cement Requirements for Soil-Cement Mixtures

1. For a more accurate method of selecting the required cement content using the present method of freeze-thaw testing:
 - a. Plot the cement contents (% by weight) against the freeze-thaw losses (% by weight) on logarithmic paper;
 - b. Draw the best straight line through these points;
 - c. From this line, select the cement content corresponding to the maximum allowable freeze-thaw loss.
Observation of the relationships will determine whether an increment of cement should be added to arrive at the cement requirement of the soil.
2. If the soil classifies as A-1, A-2, A-3, or A-4 and the cement requirement is below 10% and is approximately known:
 - a. Conduct the standard freeze-thaw test at the approximate cement content;
 - b. If the freeze-thaw loss is below 50% and above the allowable loss, plot the freeze-thaw loss and cement content on logarithmic paper;

- c. Connect this point with the common intersection point (12.6% cement, 2.4% freeze-thaw loss);
 - d. From this line, select the cement content corresponding to the maximum allowable freeze-thaw loss. Alternately a graph similar to Figure 4 will accomplish steps b, c and d.
 - e. The above cement content may be revised, knowing $S_c = \pm 0.65\%$ cement, and keeping in mind that A-1, A-2 and A-3 soils generally predict on the safe side whereas A-4 soils generally have an equal distribution of safe and unsafe predictions.
 - f. If the freeze-thaw loss is above 50%, a higher cement content should be tested; if below the allowable loss, good judgment will determine whether to use this cement content or to retest at a lower cement content.
3. To determine the required cement content after 6 cycles of freeze-thaw testing:
- a. Follow the standard freeze-thaw test method up to 6 cycles of freeze-thaw testing;
 - b. Plot the cement contents against the freeze-thaw losses on logarithmic paper;
 - c. Draw the best straight line through these points;
 - d. Select the cement content conforming to the following criteria for maximum permissible soil-cement losses

by brushing:

Soil groups A-1, A-2 and A-3, not over 6.0%

Soil groups A-4 and A-5, not over 3.3%

Soil groups A-6 and A-7, not over 2.8%

- e. Knowing the standard deviation (Table 6), determine a cement content which should adequately stabilize the soil.
4. To check the reliability of the results obtained by using either abbreviated method, compare to results obtained for each soil by the standard freeze-thaw test. Eventually it might be advisable to alter the criteria to obtain more valid results.