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THE EFFECTS of DEICING SALT on AGGREGATE DURABILITY

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PROGRESS REPORT

FOR

IOWA HIGHWAY RESEARCH BOARD

PROJECT HR-266

THE EFFECT OF DEICING SALT ON AGGREGATE DURABILITY

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ABSTRACT

The Iowa DOT has been using rapid freezing in air and thawing in water to evaluate coarse aggregate durability in concrete since 1962. Earlier research had shown that the aggregate pore system was a major factor in susceptibility to D-cracking rapid deterioration.

There are cases were service records show rapid deterioration of concrete containing certain aggregates on heavily salted primary roads and relatively good performance with the same aggregate in secondary pavements with limited use of deicing salt. A five-cycle salt treatment of the coarse aggregate prior to durability testing has yielded durability factors that correlate with aggregate service records on heavily salted primary pavements. X-ray fluorescence analyses have shown that sulfur contents correlate well with aggregate durabilities with higher sulfur contents producing poor durability. Trial additives that affect the salt treatment durabilities would indicate that one factor in the rapid deterioration mechanism is an adverse chemical reaction.

The objective of the current research is to develop a simple method of determining aggregate susceptibility to salt related deterioration. This method of evaluation includes analyses of both the pore system and chemical composition.

INTRODUCTION

Iowa has 12,800 miles of PCC pavement which includes some roads over 50 years old that have not been resurfaced or rehabilitated. On the other hand some PCC pavement that exhibited high strength and quality immediately after construction deteriorated rapidly and required extensive rehabilitation at an age of less than 10 years.

D-cracking, a type of portland cement concrete (PCC) pavement deterioration attributed to the coarse aggregate in the mixture, was first recognized on Iowa's primary road system in the late 1930s. Generally, the first sign of D-cracking is a discoloration or staining at the intersection of transverse and longitudinal joints. D-cracking will be used in reference to a rapid deterioration characterized by fine parallel cracks along joints, random cracks, or free edges of the pavement slab (Figure 1). Except where noted, references to durability in this report will be in regard to the effect of the coarse aggregate.

Extensive research into the mechanism of and methods of preventing or reducing D-cracking has been conducted since 1960. This research has yielded substantial benefits in identifying the mechanism of D-cracking. Earlier Iowa reserach revealed that most D-cracking in Iowa is a distress that results predominantly from freeze/thaw failure in the coarse aggregate (1). In 1962, based on these findings, Iowa began using ASTM C666 Method B, "Freezing in Air--Thawing in Water" to evaluate the durability of concrete. Continual testing of Iowa carbonate coarse aggregates with a 90 day moist cure modified ASTM, C666 Method B test correlated moderately well with performance of portland cement concrete pavements.

The basic Iowa tests for quality of coarse aggregate for concrete are abrasion by AASHTO T96 and a 16 cycle water-alcohol freeze/thaw test (1).

Since 1978 much research has been conducted in regard to the pore systems of limestones used as coarse aggregate in portland cement concrete and their relationship to freeze/thaw aggregate failure (2) (3). This research has shown that with some exceptions, most nondurable aggregates analyzed exhibit a predominance of pore sizes in the 0.04 to 0.2 micron radius range. The Iowa Pore Index was adapted as a standard test in 1978 (2). It is a very simple test utilizing a modified pressure meter as a test apparatus. A known volume of oven-dried coarse aggregate is immersed in water in the base of the pressure meter. The amount of water injected into the aggregate under a constant 35 psi pressure is recorded. The volume of water injected in the period between one and 15 minutes after application of the pressure is the pore index. A pore index of 27 milliliters or more generally indicates a limestone susceptible to freeze/thaw D-cracking deterioration. Coarse aggregate pore sizes have been determined using a quantichrome scanning porosimeter using pressures from 0 to 60,000 psi.

Current specifications restrict the use of nondurable stone and require higher quality aggregate.

PROBLEM STATEMENT

These tests and specifications have been relatively effective in yielding durable PCC but exceptions continue to occur resulting in rapid deterioration (12-15 years) of recently constructed pavement. These exceptions support the idea that there is at least another major factor other than freeze/thaw that contributed to their rapid deterioration.

The normal concrete mix for Iowa primary pavement construction contains approximately 6.6 bags of cement per yard. The concrete mix normally used for secondary pavement contains approximately 5.2 bags per yard. Earlier Iowa research has shown that with all other factors equal, a higher cement content will produce a concrete with better durability than a concrete with a lesser cement content. A primary pavement with a higher cement content should provide better durability than a secondary pavement with a lower cement factor if the same aggregates are used in both. In Iowa we have some aggregates that do not exhibit consistent service records. A pavement containing a Pennsylvanian crushed limestone from the Exline quarry was incorporated into a pavement on primary highway U.S. 34 in southern Iowa in 1962. Today, it exhibits severe D-cracking. A secondary road containing the same Exline aggregate constructed in 1964 exhibits only staining and no D-cracking deterioration.

A D-cracking susceptible gravel containing 70% carbonate was used in primary highway U.S. 30 in central Iowa in 1965. Today, it exhibits severe D-cracking. A secondary road containing the same gravel aggregate was constructed in 1966 and exhibits no indication of D-cracking today.

Major differences in the winter maintenance practices of secondary roads and primary roads may be the answer for the difference in performance of these two pairs of roadways. A substantial amount of sodium chloride deicing salt is used on primary roads, with limited use of deicing salt on the secondary roads. Earlier research has document the adverse effect of deicing chemicals and salts (4) (5). Additional research is needed to define and explain the mechanism that accelerates the D-cracking deterioration on the primary roadways.

OBJECTIVE

The objective of this research is to develop simple, rapid test methods to predict the durability of aggregate in PCC pavement. The test method includes an analysis of the pore systems and chemical compositions of all aggregate sources.

SALT TREATMENT RESEARCH

A salt treatment preparation was developed in an effort to duplicate the observed accelerated deterioration on primary roads as opposed to secondary raods. In Iowa, historically, the coarse aggregates have exhibited the most influence on the resulting durability of PCC. For that reason, it was theorized that the salt may be adversely affecting the coarse aggregate. A salt treatment of the coarse aggregate was developed to be used prior to addition of the coarse aggregate to the concrete mix. The salt treatment consists of five cycles of drying in an oven at $230^{O}F$ for 24 hours, followed by immersion in a $70^{O}F$, saturated solution of sodium chloride for 24 hours. The $70^{O}F$ salt brine is poured over the aggregate immediately after it is removed from the $230^{O}F$ oven. After the five cycles of salt treatment, the coarse aggregate is rinsed with clean tap water just prior to being incorporated into a concrete mixture.

The 90 day moist cure modified ASTM C666 Method B, "Freezing in Air -- Thawing in Water" testing of concrete mixtures with and without sodium chloride coarse aggregate pretreatment prior to being incorporated into the concrete has correlated very well with field performance. The durability of high quality coarse aggregates such as those from the Alden or Mohs quarries (figures 2 and 3) have not been adversely affected by sodium chloride pretreatment. Even though the resulting durability may be somewhat below the concrete mix without sodium chloride pretreatment, it is not significant. Salt treatment of the limestone fraction of the Ames gravel (figure 4) results in an extremely rapid deterioration of the concrete under freezing and thawing testing. The Smith and Garrison quarries (figures 5 and 6) are others that exhibit extremely accelerated deterioration after salt treatment. This again correlates very well with field performance.

Chemical analysis of the coarse aggregate has been conducted both with and without salt treatment to determine the amount of deicing salt retention. The sodium content increased from 0.05% without treatment to 0.09% after salt treatment on a Stanzel limestone and from 0.05% to 0.44% on a very porous Garrison dolomite.

This salt treatment testing has generated much speculation as to the mechanisms involved. The most immediate reaction is that the salt would render the aggregate hydrophilic. The aggregate would thereby retain the water longer and contain more water at the time of freezing. The salt would also lower the freezing temperature slightly.

The Iowa standard ASTM C666 Method "B" freeze/thaw test on concrete made with a low porosity, fine grained Farmington Stone yielded a durability factor (DF) of 97 and a growth of 0.008 inches. Concrete made with salt treated Farmington stone had a DF of 28 and a growth of 0.164 inches. After completion of the test, a slice was sawed through the 4 inch by 4 inch beams. Multiple fractures in the salt treated aggregate particles are readily identified by a visual observation (figure 7). No cracking occurred in the comparative aggregate without salt treatment. This cracking would appear to be a result of stresses generated by water freezing within the aggregate pore system or salt crystal growth. Apparently the salt treatment alters the water-pore relationship to allow substantial damage from freezing.

In Iowa, the concrete beams are moist cured for 90 days prior to ASTM C666 Method "B" freeze/thaw testing. The average of three beams constitute a sample on a particular concrete mix. Beams have been made where the only variation is the untreated coarse aggregate in comparison with concrete beams made with salt treated aggregate. The nondestructive testing of the concrete is based on the dynamic modulus which according to ASTM C-215 is intended to detect significant changes in the concrete due to freeze and thaw cycles. The initial sonic modulus of many untreated aggregates have been compared to those of treated aggregates. The beams made with untreated aggregates (15 samples) yielded an average initial sonic modulus of 1800 while the beams made with treated aggregate yielded an average modulus of 1750, There was no significant difference in the weight of the beams. This would indicate that even during the cure period, the properties of the concrete made with salt susceptible aggregate were detrimentally affected by the salt treatment.

X-RAY ANALYSIS OF AGGREGATE CHEMISTRY

There is a wide variation in the adverse affect of salt treatment on concrete durability as noted earlier and displayed in figures 2 through 6. This variation did not always relate to the pore system so a further analysis of the aggregate chemistry was conducted using x-ray analysis systems available at Iowa State University. The x-ray fluorescence system has been utilized to evaluated the elemental composition of various aggregates. X-ray diffraction techniques were used to determine the mineral compositions of the aggregates. The scanning electron microscope has been used both to observe the pore sizes and crystal sizes in the aggregates in addition to elemental chemical identification, distribution and analysis. X-ray fluorescence has been used to determine magnesium, sodium, iron, sulfur, silica, calcium, potassium and aluminum contents of most coarse aggregates used in Iowa concrete. These chemical analyses have been compared to service records of pavements constructed with the various coarse aggregates. The sulfur content yielded the best correlation (inverse) with performance of the aggregates that had passed the standard Iowa specifications. The sulfur content correlates very well with aggregate durability in heavily salted primary pavements, especially when magnesium is present.

The scanning electron microscope has been used to further investigate the sulfur content in aggregates exhibiting accelerated deterioration on salted primary roads. Energy plots from the scanning electron microscope (figure 8) of the salt affected aggregates, exhibit similar distribution of iron and sulfur. This would indicate that iron-sulfur particles in the 1 to 2 micron range are uniformly dispersed throughout the nondurable aggregate. The iron and sulfur are very likely present as pyrite or marcasite. These finely divided 1 to 2 micron black specks were identified as pyrite in some nondurable Iowa ledges in 1961 by Donald W. Kohls in a University of Minnesota thesis. The adverse reaction of the pyrite and liberated sulfur has been cited in other research (6) (7) (8) (9) (10) (11).

EFFLORESCENCE ON QUARRY FACE

Salt treatment testing of crushed stone from the Lillibridge Quarry of Cerro Gordo County in northern Iowa would indicate that it would be nondurable in concrete. The Lillibridge stone has an exceptionally high but widely dispersed sulfur content. Whitish efflorescent deposits commonly occur on quarry faces after a lengthy dry period. A chemical analysis of the deposit identifies this material as calcium sulphate and magensium sulphate. It would appear that there is a chemical reaction where the sulfur is released from the iron sulfide. This deterioration of the stone may also occur after being incorporated into concrete and thereby reduce its durability.

ADDITIVES AFFECTING SALT TREATMENT DURABILITIES

After verifying that the salt treatment preparation did cause the ASTM C666 durabilities to correspond to field performance, it was decided to conduct research to determine if various ingredients would either adversely or beneficially affect the durability factor. Two additives and a special cement were used (figure 6) to ascertain their effect on concrete durability. The first was the use of a Type V (sulphate resistant) portland cement rather than the regular Type I. Type V cement (4.8% tricalcium aluminate) did exhibit a significant beneficial effect on the concrete durability after the coarse aggregate was treated with salt.

Fine porous limestone with very low magnesium content was added to a concrete mixture in the amount of 5%. This addition of 5% limestone fines yielded a greater beneficial effect than did the Type V cement.

The addition of 5% porous dolomite fines (high magnesium content) yielded an adverse effect on the salt treated durability beams. The alteration of the durability of concrete produced with salt treated aggregate by the addition of carbonate fines would indicate an affect on the concrete chemistry. From this research it would appear that magnesium may be a substance that promotes an adverse effect in durability of concrete mixes containing porous, pyritic carbonate coarse aggregate.

FINE AGGREGATE AFFECTING SALT TREATED DURABILITY

The Iowa DOT began aggregate durability research with a Conrad automatic freeze/thaw machine in 1962. Aggregate service records at that time indicated that the coarse aggregate in the PCC pavement was a major factor. A limited investigation of the effect of fine aggregate on concrete durability using the ASTM C666 Method "B" procedure produced insignificant differences, so a local sand which was conveniently available was used as a standard.

Recent durability testing after salt treatment of the coarse aggregate has shown that fine aggregate can affect concrete durability. The effect of the fine aggregate has been masked by the normally much greater effect of the coarse aggregate and the limitation of ASTM C-666 method to identify chemical problems. This is a possible explanation of some irregularities in ASTM C666 aggregate durability factors in past years. The Ames Pit in central Iowa, used as the standard fine aggregate until June 1984, can exhibit substantial petrographic variations. The coarse aggregate from this pit may contain as much as 70% carbonate particles, many of which are nondurable. A recent petrographic analysis of the fine aggregate from the Ames Pit identified over 60% carbonate particles in the material passing the #4 screen and retained on the #8 screen.

The more severe salt treatment durability testing has provided an opportunity to reevaluate the effect of fine aggregates. The Ames sand was compared to a Bellevue high quality, predominantly igneous Mississippi River sand. The Bellevue sand yields consistently, significantly higher durability factors. The Bellevue sand is now the Iowa standard fine aggregate for concrete for durability testing. This change should produce greater consistency in coarse aggregate durability testing.

DISCUSSION

Findings from current research have strongly confirmed the fact that there are factors in addition to the pore system associated with the rapid deterioration of PCC pavement. Substantial research has shown that freeze/thaw action on aggregates with susceptible pore systems is a major cause of D-cracking of concrete in Iowa. This does not explain all premature deterioration of concrete as an aggregate may be durable in some pavement and result in rapid deterioration in others.

The five-cycle salt treatment durability relates well to service records of aggregate usage in pavement. It has allowed laboratory studies of other related variables. The study of such variables in actual field trials would require too much time for results.

Research is continuing on many aspects of aggregate durability in PCC pavement. Those currently being studied are:

1. The development of an ice porosimeter to study aggregate pore structures.
2. The effect of fly ash on salt susceptible aggregates.
3. The effect of deicing-salt impurities on concrete durability.
4. The effect of clay type and amount on aggregate durability.
5. The effect of reducing the maximum size of the aggregate when the problem is chemical rather than pore size distribution.
6. The relationship of the total coarse aggregate pore system to the rate of chemical reactivity.
7. The effect of fewer salt treatment cycles.

CONCLUSIONS

This research on aggregate durability supports the following conclusions:

1. Deicing salt accelerates pavement deterioration when salt susceptible aggregates are used.
2. The salt treatment of coarse aggregate prior to freeze/thaw testing yields durability factors that exhibit a definite relationship with field performance.
3. The salt treatment detrimentally affects the quality of concrete prior to freeze/thaw action.
4. The durability of dolomitic coarse aggregate is inversely related to the sulfur content.
5. An adverse chemical reaction contributes to the rapid deterioration of concrete containing porous pyritic dolomite.

ACKNOWLEDGMENT

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FIGURE CAPTIONS

1. A Close-up of D-Cracking at the Intersection of the Transverse and Longitudinal Joints.
2. Alden Durability Factors
3. Mohs Durability Factors
4. Ames Pit Durability Factors
5. Smith Durability Factors
6. Garrison Durability Factors
7. Sawed Cross Sections of Concrete Beams Made with Farmington Stone Showing Multiple Fractures in Salt Treated Aggregates
8. Scanning Electron Microscope Energy Plot of Sulfur and Iron

Figure 1 A Close-up of D-Cracking at the
Intersection of the Transverse and Longitudinal Joints

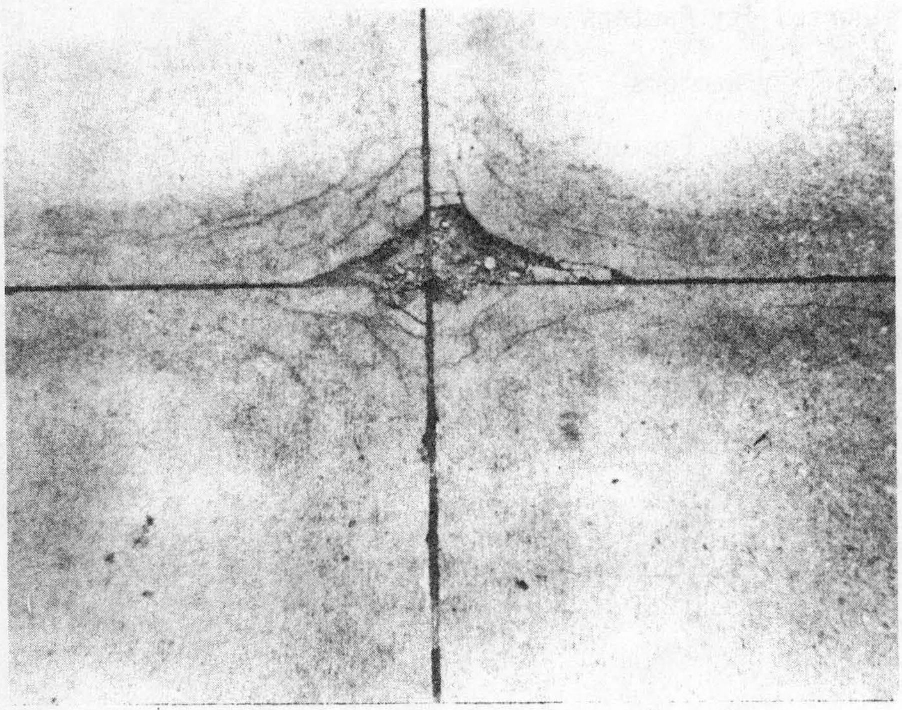

Figure 2. **ALDEN DURABILITY FACTORS**

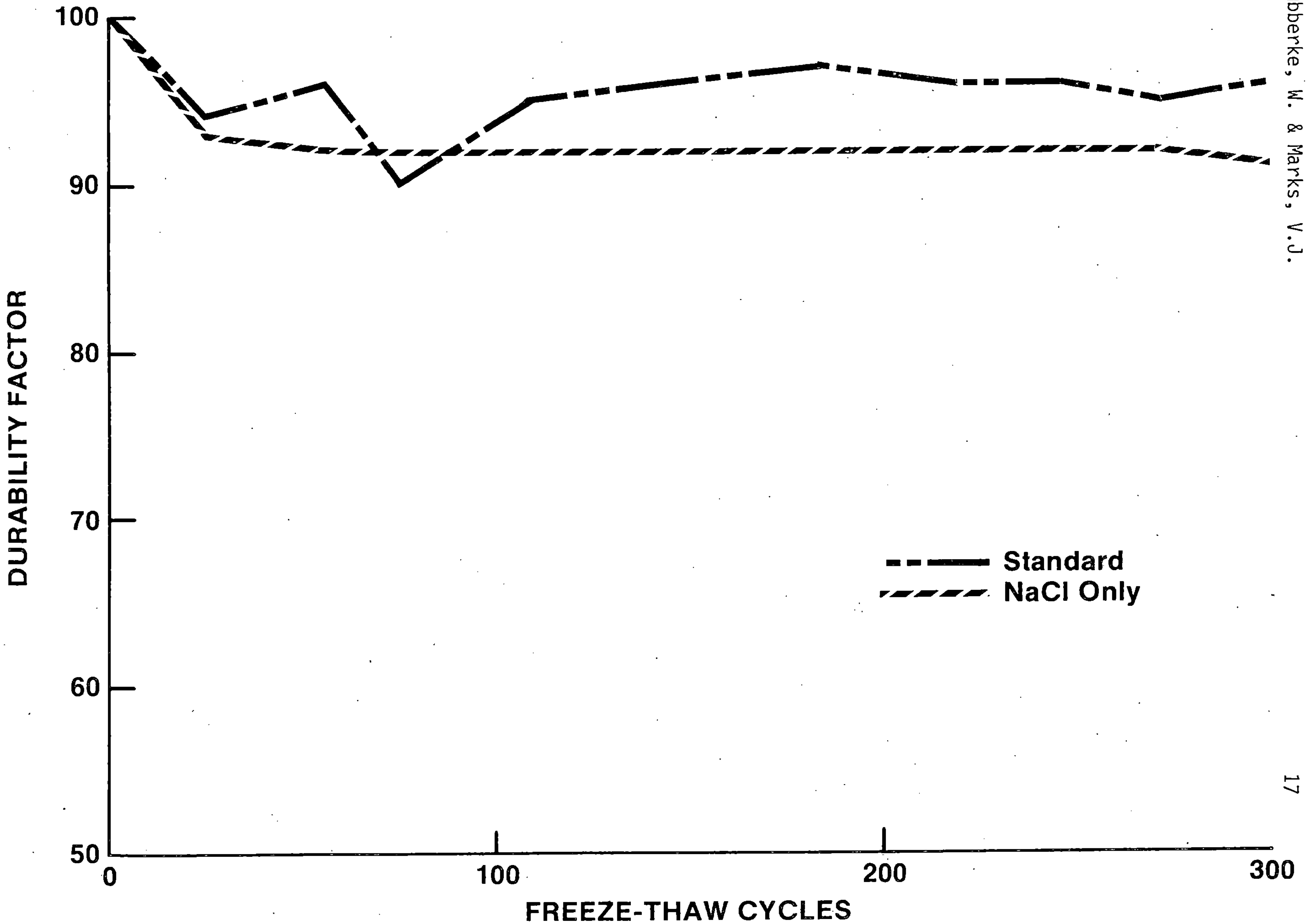

Figure 3. **MOHS DURABILITY FACTORS**

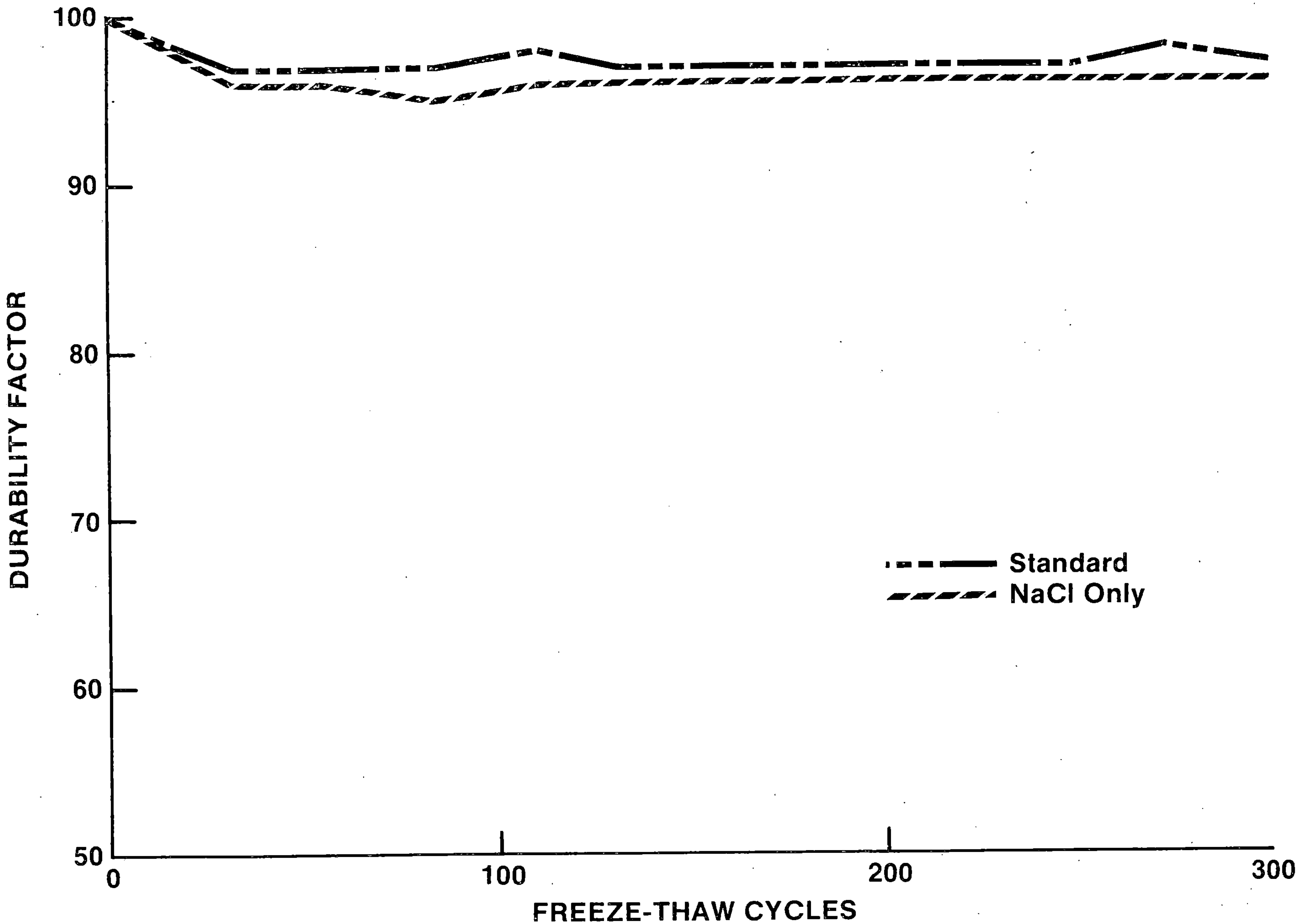

Figure 4. **AMES PIT DURABILITY FACTORS**

(CARBONATE FRACTION ONLY)

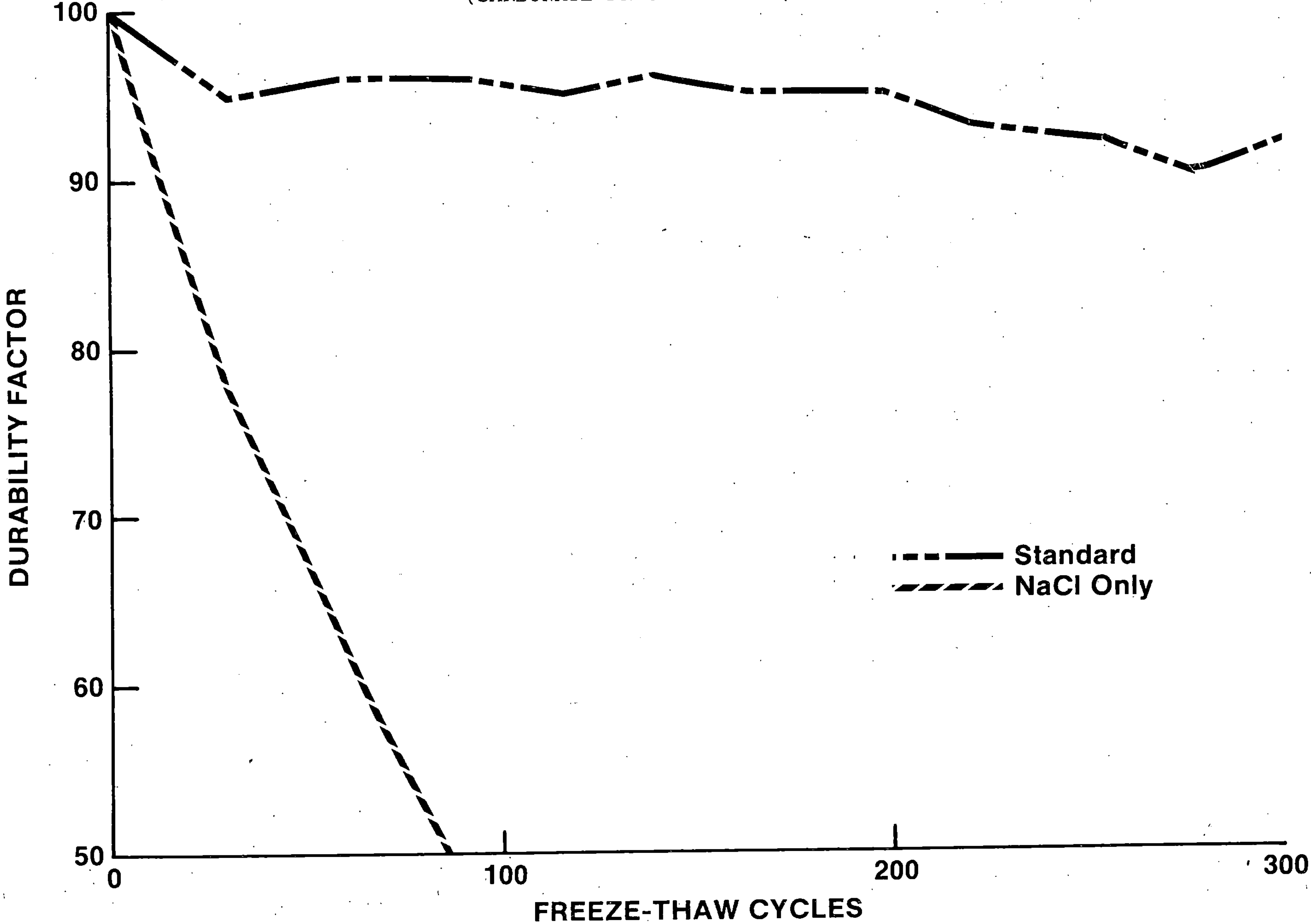

Figure 5. **SMITH DURABILITY FACTORS**

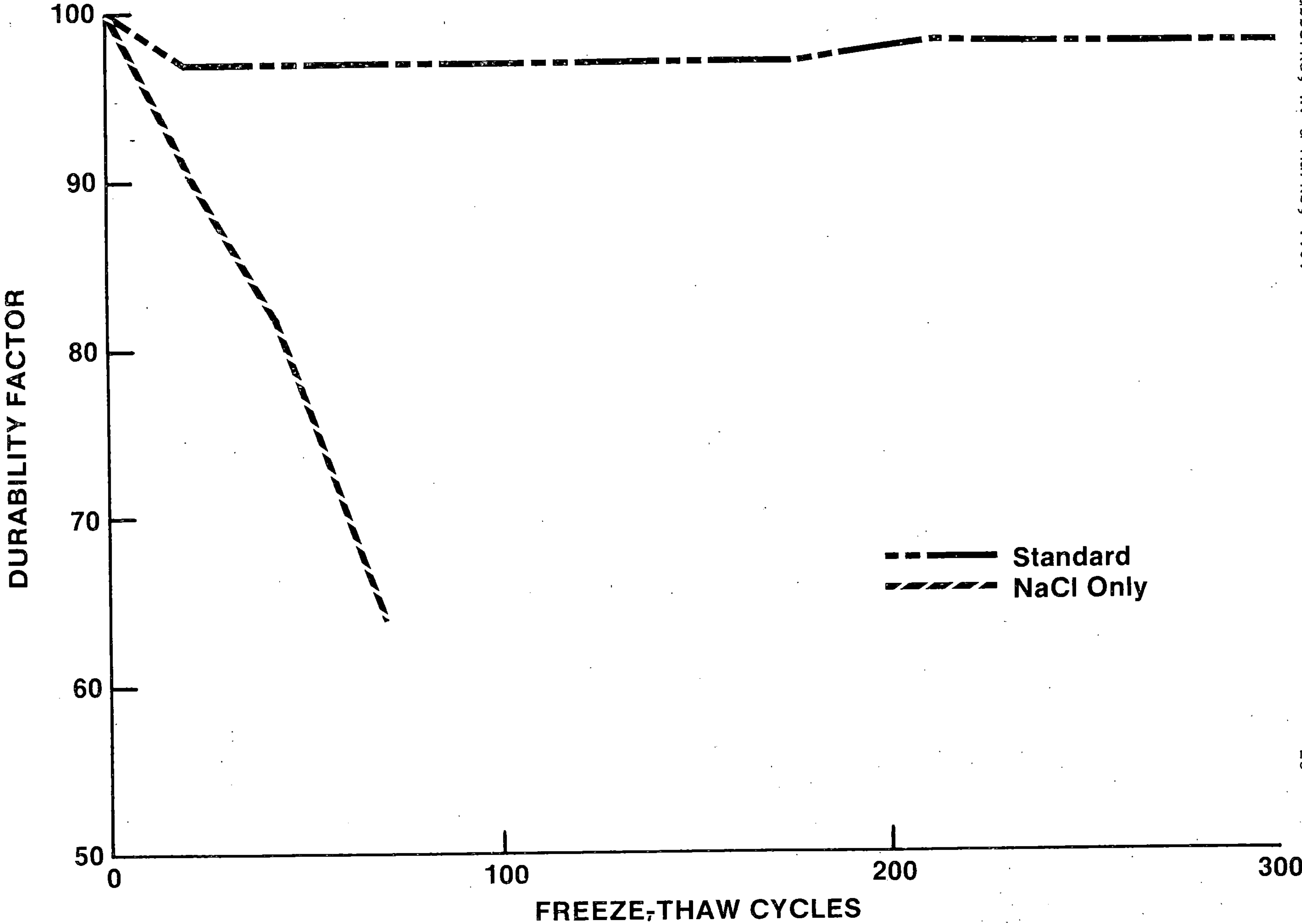

Figure 6. GARRISON DURABILITY FACTORS

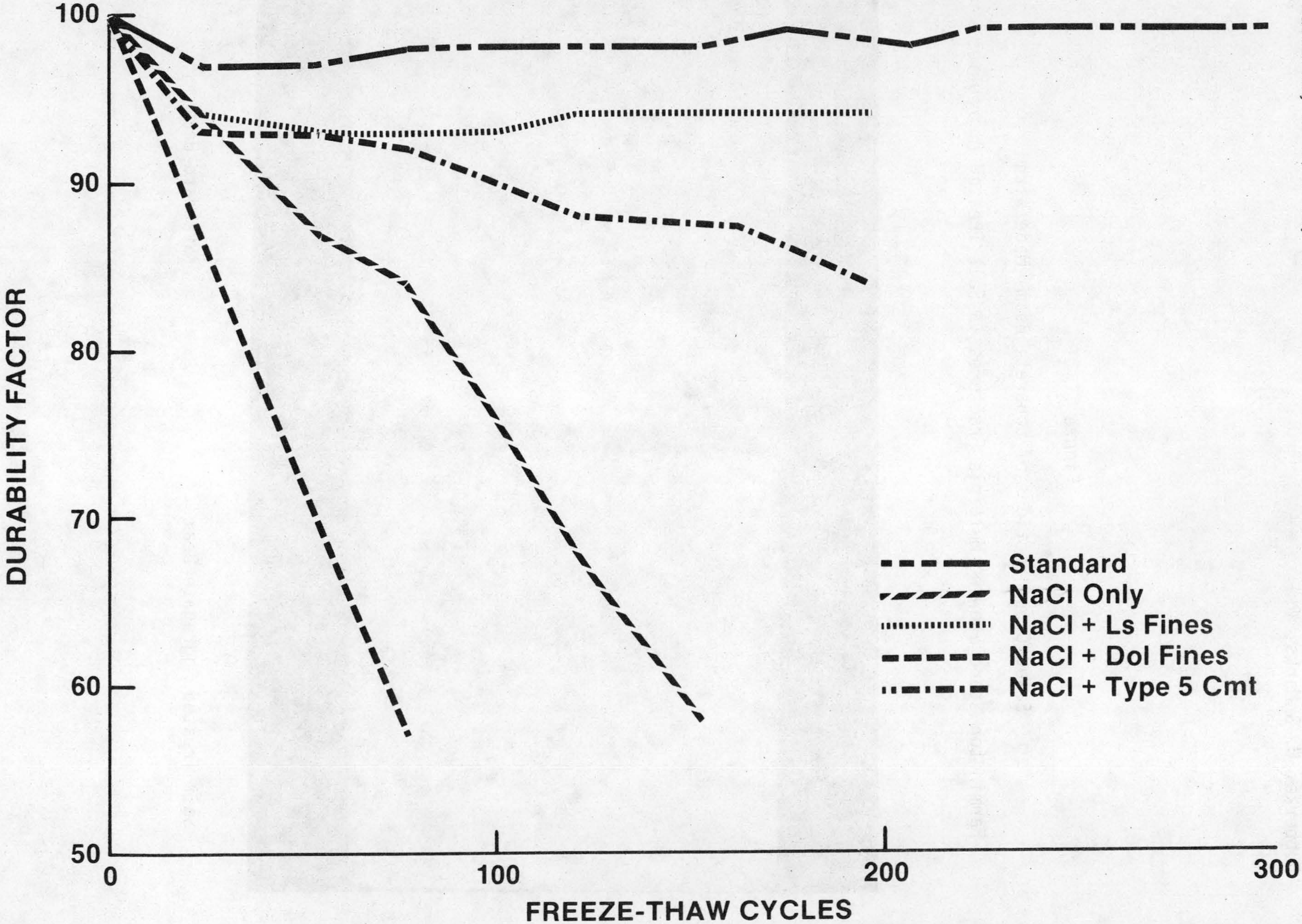

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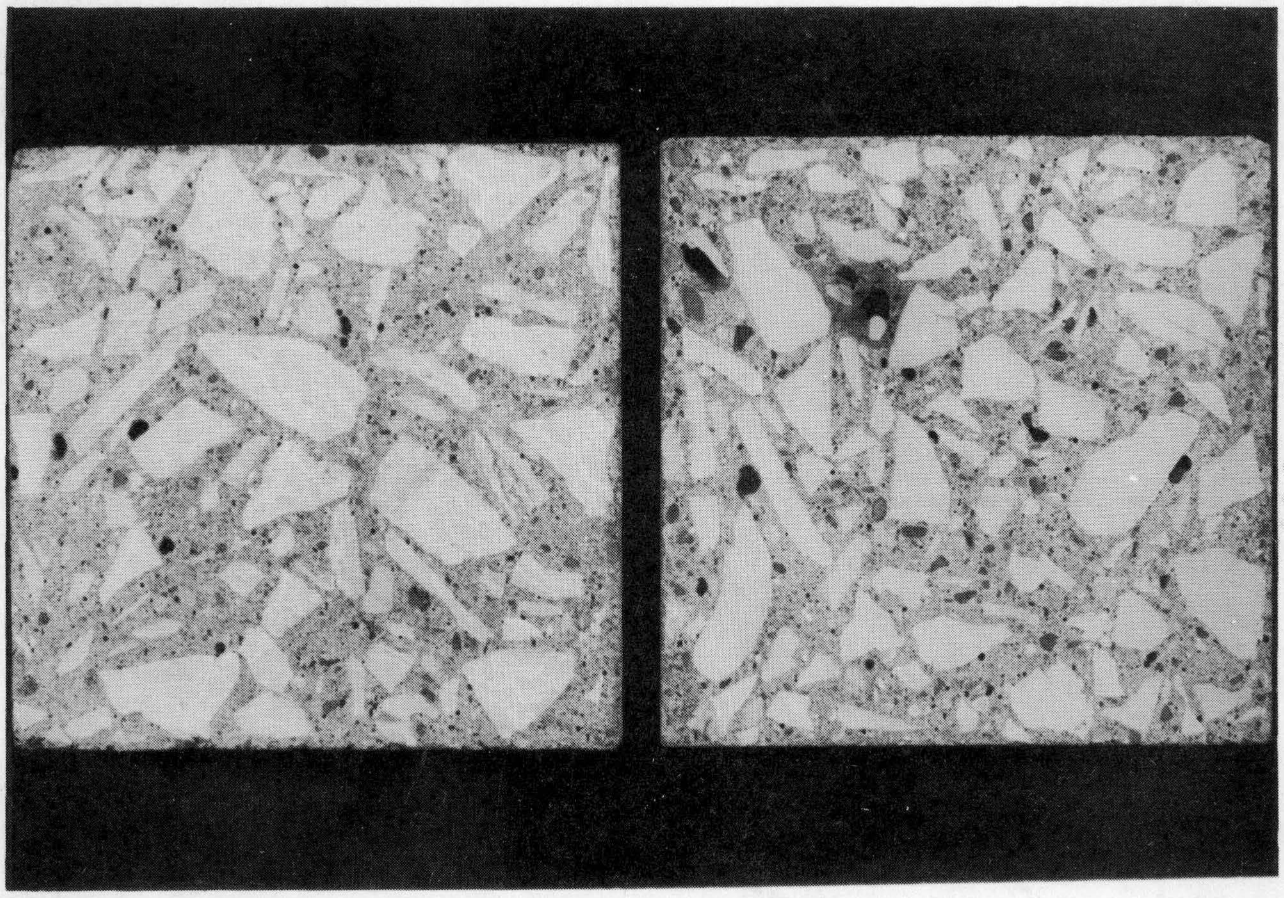
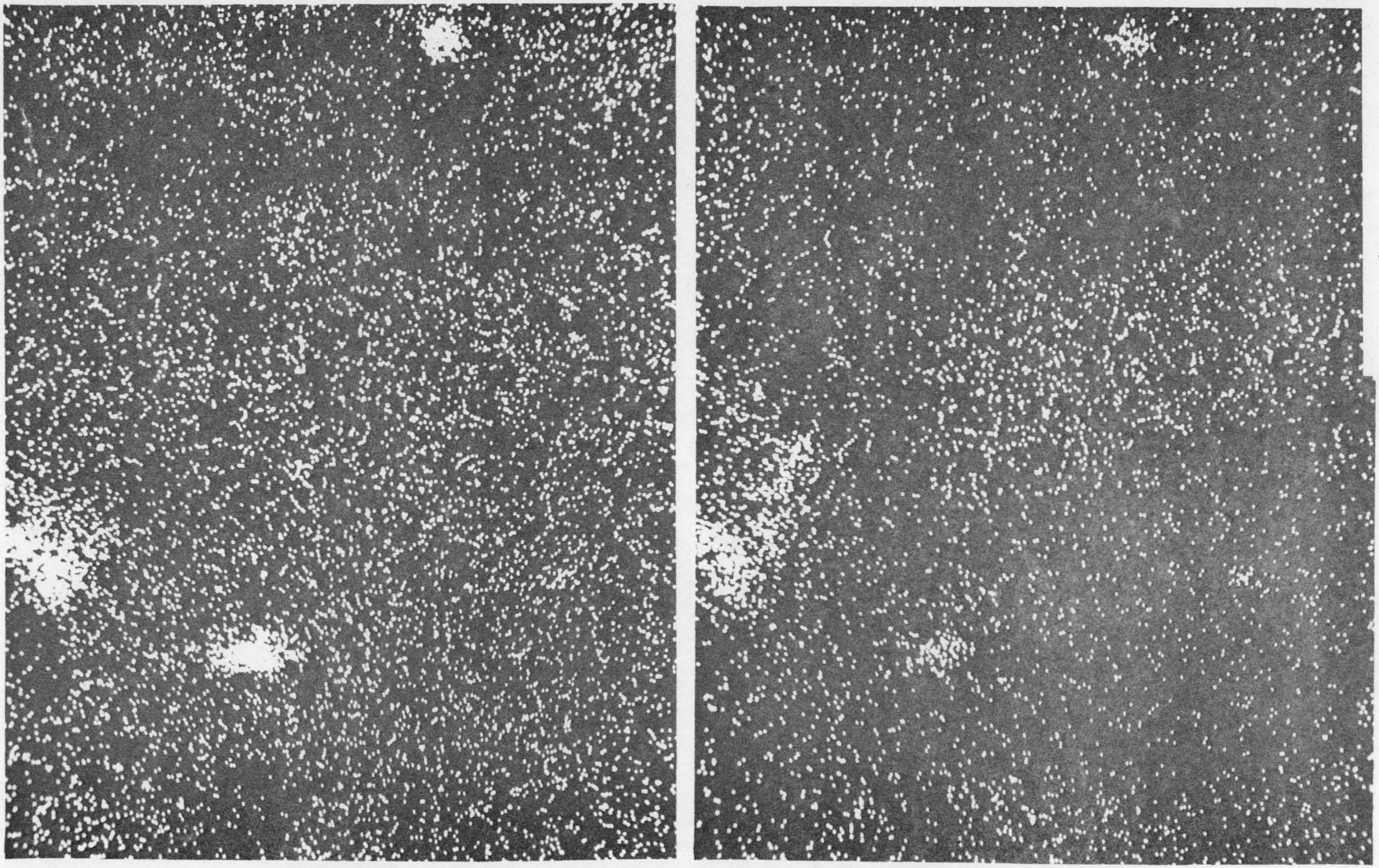
Figure 7

Sawed Cross Sections of Concrete Beams Made With

Farmington Stone Showing Multiple Fractures in Salt Treated Aggregates



Salt Treated Aggregate Beam   Standard Beam



SULFUR 10 Microns **IRON**

Figure 8 Scanning Electron Microscope Energy Plot of Sulfur And Iron