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Final Report

EFFECTS OF SALT AND TRACE MINERALS ON AGGREGATES FOR BITUMINOUS PAVEMENTS

T. R. West
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Indiana
Department
of Transportation

Purdue
University

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T. R. West
Principal Investigator

and

H. J. Park
and
S. E. Grover
Research Assistants

Department of Earth and Atmospheric Sciences
Purdue University

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16. Abstract

Research on the effects of deicing salts and their trace minerals on bituminous highway wearing courses was prompted by early pavement deterioration of federal and state highways in Indiana. Several bituminous surface courses containing slag, slag-dolomite, or dolomite aggregates showed deterioration soon after deicing salts were first applied. Initially, a detailed literature review (Appendix A of report) was conducted. Data on eighteen dolomite sources from a previous study (Bruner, Choi and West, 1995) plus ten additional dolomite sources formed the basis for the brine freeze thaw testing program. Mercury intrusion porosimetry testing conducted on multiple specimens showed that rock texture significantly affects pore size distribution. Petrographic analysis, acid etching of thin sections and electron microprobe studies revealed the distribution of clays on dolomite grains. Additional brine freeze thaw testing on selected dolomite sources, plus one slag sample, were conducted using deicing salts from six Indiana highway districts. Petrographic evaluation of pavement cores from the distressed pavements was accomplished. Brine freeze thaw test results ranged from 0.6 to 27.3% loss for the 28 samples; this test is a good measure of aggregate durability. INDOT has recommended a maximum 30% loss for the brine freeze thaw test. Regression analysis showed that acid insoluble residue content and sodium sulfate loss have the highest correlation with brine freeze thaw loss; elemental magnesium content has a negative correlation with it. It is concluded that dolomite aggregates with a low insoluble residue content have lower sulfate soundness losses, lower brine freeze-thaw losses and greater durability. Evaluation of pavement cores indicated no evidence of deterioration caused by aggregate weathering or degradation. Brine freeze thaw testing is a good measure of freeze thaw resistance of aggregates in bituminous surface courses and no evidence was found indicating that early deterioration of the pavements studied was caused by salt attack.

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I. Introduction

1. Overview

This research on the effects of deicing salts and their trace minerals on bituminous highway wearing courses was prompted by several occurrences of early pavement deterioration on Indiana federal and state highways. Early deterioration of bituminous pavements made with slag, slag-dolomite and dolomite aggregates was observed on three highway projects soon after the deicing salts were applied for the first time. These sites included a section of I-65 in Indianapolis, a section of I-90 east of Gary and a section of SR67 north of Martinsville. Evaluation of these three sites is included in the current study. In addition, a fourth site was evaluated, SR62 north of Charlestown, where pavement deterioration was of a different nature. In the first three situations, aggregate and bituminous binder both came loose from the pavement, whereas for SR62 the coarse aggregate pieces broke away from the binder, leaving aggregate pock marks in the wearing course. This site is also evaluated in the current study.

As a first step in the evaluation process, a detailed literature review was conducted on the effects of salt and trace minerals on aggregates under the direction of Dr. Terry R. West. The detailed annotated review of the pertinent literature (Pittenger and West, 1995) is a 45 page report that is included here as Appendix A. This report focused on two primary subjects: 1) deicing salt and aggregate deterioration of portland cement concrete and 2) moisture damage to asphalt pavement.

A conclusion reached in the literature review was that only limited research on aggregate deterioration in bituminous pavements has been conducted although abundant work on portland cement concrete pavements was accomplished. Some comparison of the behavior between these two pavement types may prove helpful for bituminous pavement deterioration studies. Also it was concluded that many factors are involved in moisture damage (stripping) of asphalt pavements and it would be difficult to isolate the contribution of any specific factor in this study.

Cores were taken in the pavement for the first three cases where deterioration occurred (I-65, I-90 and SR67). A petrographic description of the cores is provided in this report.

Eighteen dolomite aggregate sources were included in the previous study performed under the direction of Dr. Terry West, petrographer and civil engineer (Bruner, Choi and West, 1995). Data on these INDOT approved sources are incorporated in this study. Petrographic analysis, composition based on x-ray diffraction, insoluble residue content, elemental magnesium content and standard laboratory test results were available on these samples. Laboratory physical test results included absorption, specific gravity, sulfate soundness loss and Los Angeles abrasion loss. Statistical evaluation of these data was accomplished.

In the current phase of work, ten additional dolomite sources approved by INDOT were contributed to the data base, yielding a total of 28. Brine freeze thaw testing was accomplished on these 28 samples using the coarse size gradation for the test. Following this a select subset of this group, six dolomite sources, were evaluated using the fine aggregate brine freeze thaw test. A comparison of brine testing results was accomplished.

Mercury intrusion porosimetry testing was performed on multiple specimens of the aggregate sources from the Phase I study materials. Results showed a significant variation in pore size distribution. Subsequently a single quarry with six distinctly different lithologies was investigated using porosimetry methods. Multiple samples of each lithology were examined. For this group of tests the variation in pore size distribution was much reduced. This indicates, that if the lithology or texture is consistent, similar pore size distribution will be obtained with much less variation.

Additional brine freeze thaw testing was performed on 13 aggregate samples, 12 dolomites and one slag. Deicing salts from six different Indiana regions were used for the testing. Amounts of trace elements in the deicing salts were also determined.

Acid etching of thin sections and microprobe studies were performed to evaluate the clay content and its distribution in the carbonate aggregates. The distribution of clays in the carbonate rock was determined.

As a final evaluation pavement cores obtained from the three distressed pavements, were studied using petrographic techniques. Descriptions are provided for these samples. Comparison of data for the 28 sources was accomplished based on regression analysis. Discussion of results and concluding remarks comprise the final evaluation of the sources evaluated.

2. Literature Review

Most research related to pavement degradation in the presence of deicing salts is concerned with the deterioration of portland cement concrete (PCC) and the corrosion of steel reinforcement. That research is motivated in part by the economics of salt deterioration and corrosion of bridge decks, which are costly to replace and are more susceptible to damage. By contrast, much less research has been conducted on aggregate degradation in bituminous pavements caused by deicing salts.

Determination of the causes of aggregate deterioration in the presence of deicing salts is an important area of research. However, development of laboratory test procedures to determine sample response to freezing and thawing in the presence of deicing salts and the correlation of the lab test to field performance have also been investigated. The preferred method for predicting aggregate performance would involve a test that simulates the environment in which the aggregate is exposed. Freeze-thaw testing with immersion in salt solution is frequently recommended.

Development of such a reliable test method has the added benefit that it would be independent of the specific cause of aggregate deterioration.

Most research focused on the determination of the causes for aggregate deterioration, with only a brief concern for the test development related to performance. Therefore, correlation of test methods and aggregate performance is not available. The most detailed correlation of aggregate field performance and laboratory testing is provided by Shakoor (1982).

1) Freeze-Thaw Testing in Combination with Salt Solution

Research by Dubberke and Marks (1985) provides evidence that the use of freeze-thaw testing in combination with a salt solution may be an acceptable method determine field performance of aggregates under freezing conditions involving deicing salt applications. However, it was applied only to aggregates used in PCC concrete, and the freeze-thaw test was performed on concrete beams rather than the unconfined aggregate particles.

Shakoor (1982) performed freeze-thaw tests on argillaceous limestones and dolomites in both plain water and in a 5 percent NaCl solution. It was determined that the salt solution produced a greater loss when the fine material had a more clayey rather than silty composition. He also showed that highly argillaceous rocks are more frost-resistant when the argillaceous mineral is in laminations rather than distributed evenly.

2) Physical-Chemical Relationships of Salt Solutions and Aggregates

Much research has been conducted to determine the physical characteristics of aggregates that are susceptible to freezing and thawing.

(1) Pore Size Distribution

Pore size distribution has been considered as an important factor in an aggregate's susceptibility to freeze-thaw. A theory that there is a critical range of pore sizes detrimental to aggregate durability has been supported. Above a certain critical size, frozen water can easily be expelled from the pores. Below that critical size, no water enters the pore to freeze. Critical pore sizes ranging from 10 angstroms to 10um have been reported, depending on source of the research.

(2) Absorption and Insoluble Residue

According to a study by Dunn and Hudec (1972) the primary reason for aggregate deterioration in presence of deicing salt is the increase in absorption and expansion of the aggregate. This expansion can cause aggregates to fail even without freeze-thaw and NaCl in solution causes significantly greater expansion.

Crumpton, Smith, and Jayaprakash (1989) found that clays in limestone were altered by the salt that caused D-cracking in the pavement. In addition, Shakoor (1982) determined that when a larger percentage of clay mineral was found as the residue obtained in the insoluble residue test it correlated with increased degradation of the aggregate in the salt solution freeze-thaw test.

(3) Alkali-Silica Reaction

Crumpton, Smith, and Jayaprakash (1989) studied the effects of salt on alkali-silica reactivity. Aggregates in their study were highly susceptible to alkali-silica reaction. It was found that the salt solution exacerbated the alkali-silica reaction by keeping the interior of the aggregate wet for a longer period of time, increasing time for reactions to occur. The increased retention time of water in aggregates also exacerbated D-cracking and pavement blow-up.

(4) Aggregate Chemistry

Dubberke and Marks (1985) performed chemical analysis of aggregates and hypothesized that the salt enables the aggregate to retain water longer and therefore contain more water when freezing and that the sulfur content yields the best correlation with performance of the aggregates that had passed the Iowa specifications. Their most recent work (Marks and Dubberke, 1995) determined that deicing salt contributes to the formation of the mineral ettringite located between the aggregate and the PCC paste.

3) Trace Sulfate Compounds in Deicing Salt

Questions have been raised about the detrimental effects of trace compounds in deicing salt, specially magnesium sulfate and sodium sulfate. The explanation for correlation in that both sulfate exposure and saline solutions produces internal stresses within the aggregate that the rock is unable to withstand. Under normal freeze-thaw conditions, this stress is developed from the formation of ice crystals in the pores. When exposed to saline solution, the stress results from increased absorption and under exposure to sulfate, the stress occurs from formation of sulfate crystals.

Pitt et al. (1988, 1992) recommended that the sulfate in the deicing salt be limited to less than 0.28%. But no research included the testing of freeze-thaw effects on aggregates containing various amounts of sulfate in a salt solution.

II. Problem Statement

Deterioration of three bituminous pavement surfaces on Indiana highways was observed following the first year that deicing salts were applied. Concern was that deicing salts had caused aggregate deterioration leading to a loss of bond with bituminous binder. Quality aggregates which passed highway test standards were involved. Some clay-rich carbonate aggregates have undergone deterioration in Portland cement concrete pavements. Evaluation of dolomite and slag used in the bituminous pavement surfaces was deemed necessary to investigate whether a deterioration process caused by salt attack had occurred.

III. Objectives

Deterioration of three pavement surface courses on Indiana highways was observed within one year after placement, that is, following the first year in which deicing salts were applied in the winter months. Both aggregate and binder came loose from the pavement in small pieces yielding holes in the pavement surface. Concern was that aggregate deterioration, owing to deicing salts had caused a loss of bonding ability, leading to the removal of pieces from the road surface.

Deterioration of concrete pavements by salt attack or due to various other chemical mechanisms has been well established in the literature for concrete pavements, but deterioration of aggregates in bituminous surface courses had not been extensively studied. A detailed literature review was conducted to verify that this subject had not already been evaluated and described in the literature. Following the completion of this literature review (found in Appendix A) the research proposal was developed to evaluate the effects of salt attack on aggregates in bituminous surface courses.

The objectives were to obtain aggregate samples from approved INDOT sources consisting of dolomite, used for bituminous surface courses, and evaluate their petrographic and physical properties with regard to deterioration.

The following objectives were planned and accomplished:

1. Incorporate data from the previous study by Bruner, Choi and West, 1995, on 18 dolomite aggregate sources.
2. Augment with ten additional dolomite sources as recommended by SAC committee members which included representatives from INDOT, FHWA and mineral aggregate producers.
3. Conduct brine freeze thaw tests on the 28 aggregate sources using the standard sieve size to evaluate loss and on a larger sieve size for comparison.
4. Mercury intrusion porosimeter method for pore size distribution and determine a) pore size distributions without special consideration of individual textures, b) using sources D-26 and D-27 determine pore size distributions.
5. Evaluate acid insoluble residue amounts and size distributions for the dolomite samples.

6. Evaluate deicing salts used by six highway districts of INDOT, determine composition of trace elements.
7. Freeze thaw testing with salts from six Indiana districts using selected aggregate sources and slag.
8. Acid etching of thin sections to determine distribution of clay minerals.
9. Election microprobe studies of selected aggregate sources to determine distribution and amount of clay minerals.
10. Description of cores obtained from the three pavements.
11. Evaluate results through correlation of data.
12. Present overall results of study, conclusions and recommendations.

IV. Work Plan

Detailed results were available on 18 dolomite aggregate sources from a previous JTRP report by Bruner, Choi and West, 1995, including detailed petrographic and laboratory testing results. Samples remaining from the insoluble residue analyses were still available, so that their grain size distribution could be determined, to provide further information in the current study.

Ten additional INDOT approved sources of dolomite aggregates were selected with the aid of advice from representatives of INDOT, FHWA and mineral aggregate producers on the SAC committee. This yielded 28 aggregate sources for evaluation. Samples were sieved to obtain the appropriate size distribution and brine freeze thaw tests were performed on the samples in the INDOT laboratories, Division of Materials and Tests, Indianapolis, IN.

Regression analysis was performed on the two data sets, the more extensive set for the 18 dolomite sources from the previous study and on the combined 28 sources on which brine freeze thaw testing had been accomplished.

Mercury intrusion porosimetry analysis was performed on the 18 dolomite sources. Brine freeze thaw loss was compared to EDF (Expected Durability Factor) values for the porosimetry study. Correlation was improved when the highest EDF values were excluded from the evaluation.

A second mercury intrusion porosimetry study was conducted on an individual aggregate location consisting of two approved materials, D26 and D27. Detailed petrographic analysis of the dolomite sources was included. This showed that in general, less variation in EDF value occurs among individual sample results that have similar textural characteristics.

Another series of brine freeze thaw tests were conducted in the INDOT laboratories, Division of Materials and Tests. Six deicing salts from different INDOT highway districts were analyzed to determine the trace elements present. Following this, twelve aggregate samples plus one slag sample were evaluated in brine freeze testing using the six different deicing salts.

The grain size distribution of acid insoluble residue samples was accomplished. The results are recorded in two parts, percent greater than No.200 sieve and that percent less than No.200 sieve. The finer portion consists predominantly of clay minerals and the coarser of silt size particles (mostly quartz).

Rock thin sections were prepared for the 28 aggregate sources. Petrographic analysis was performed on the thin sections and descriptions obtained. Glass cover slips typically placed over rock thin sections were omitted so that etching with dilute HCl could be accomplished. A visual view of the distribution of clay particles around the dolomite grains was possible as the dolomite was dissolved but clay and quartz did not. In most cases clay was concentrated around the perimeter of the original grains, whereas in a few situations thin layers of clay mineral were discerned.

Electron microprobe studies were conducted on thin sections of four dolomite samples that represented a range in texture and insoluble residue content. Areas of 0.3 by 0.5 mm up to 1.0 by 1.0 mm were examined. Elemental analysis was accomplished, noting for the constituents that comprise clay minerals.

Megascopic description was performed on the pavement cores taken from the three sites, I-65, I-80 and Indiana St.Rt.67. The nature of the aggregates, both dolomite and blast furnace slag, was noted.

Evaluation of the data obtained during the research was conducted as the research progressed and at the end of all testing procedures. Results, conclusions and recommendations were accomplished as the final work on the project.

V. Analysis of Data

1. Work Performed During the Previous Project by West and Others

In a previous study of Indiana aggregates for bituminous wearing courses, nineteen dolomite sources were evaluated (HPR-2082, Bruner, Choi, and West, 1995). These aggregate sources were also included in the current study.

1) Petrographic Analysis of Dolomites

A detailed petrographic examination was necessary to determine the texture and general mineral composition of the carbonate sources. This analysis included the determination of average grain size, grain shape, percentage of groundmass and void content. Aggregate pieces 9.5 mm (3/8 inch) in size were subdivided using a mechanical splitter so that approximately 200 rock pieces provided a representative sample. Eighteen dolomite sources, approved by INDOT, were included in the study. Megascopic evaluation (unaided eye and hand lens) was accomplished, as well as microscopic, petrographic studies using rock thin sections and a polarizing microscope.

(1) Grain Size

The size of individual grains were determined based on the point count method by advancing a thin section a specified distance using the mechanical stage on the polarizing microscope. After a grain was isolated by the cross-hairs of the calibrated eyepiece, the stage was rotated to measure the grain along its greatest dimension.

(2) Grain Shape

An angularity chart developed by Lees (1964) was used to determine a range of grain shapes. The chart consists of 16 categories of angularity and the scale was modified to measure values between 1 and 16 for simplicity.

(3) Groundmass

Groundmass was subdivided into two categories, cement and matrix. Cement is defined as the fine to coarse material between grains, and matrix as the fine material commonly surrounding floating grains. However it was difficult to distinguish these two materials. Therefore, in this study all were designated as groundmass.

(4) Void Content

Void content was measured based on the observation of voids viewed under cross hairs of the calibrated eyepiece. The percentage of voids was determined as the ratio of voids counted to the total point counts for the sample.

(5) Summary of Petrographic Results

Table 1 is a summary of the petrographic analysis of the dolomite samples evaluated. These parameters were measured in rock thin sections using a petrographic microscope.

Table 1. Petrographic Analysis, Previous Study

Sample I.D.	Grn Size (mm)	Median (mm)	Grain Stand Dev	Matrix (%)	Grnd Mss (%)	Grains (%)	Voids (%)
1	0.13	0.085	9.9	21.5	28.4	59.9	11.7
2	0.079	0.061	3.6	2.8	30.5	59.1	10.4
3	0.059	0.042	3	21.6	42.8	49.3	7.9
4	0.194	0.053	12	26.4	41.9	48.4	9.7
5	0.069	0.052	3.8	30.9	49	45.8	5.2
7	0.074	0.075	3.7	12.3	27.4	64.4	8.2
8	0.087	0.065	4.2	5.5	14.8	71.8	13.4
9	0.066	0.063	4.9	13.2	27.2	63.5	9.3
10	0.07	0.049	3.9	2.8	28.1	61.7	10.2
11	0.058	0.041	3.5	7.9	27.8	64.7	7.5
12	0.082	0.051	4.1	7.5	30.5	64.9	4.6
13	0.057	0.043	4.9	47.5	64.8	31.2	4
15	0.116	0.085	7	24.8	36.9	58.5	4.6
17	0.119	0.114	8.7	9.4	25	68	7
18	0.063	0.058	3.6	47	60.7	35.8	3.5
19	0.059	0.046	7.4	7.4	12.6	77.6	9.8
20	0.085	0.068	4.2	13.2	34.7	55	10.3

2) Chemical Composition

(1) Magnesium Content

Dolomite aggregates meeting INDOT specifications for bituminous surface courses for medium and high traffic volume roads, must be carbonate rocks containing 10.3% or more elemental magnesium. Using an EDTA titration method in accordance with ASTM procedure C 602, Specification for Agricultural Liming Materials, these 18 aggregates were analyzed by INDOT. The relative percentage of dolomite can be calculated based on the percent elemental magnesium.

$$\text{Mg Fraction in Dolomite} = (\text{Molecular Weight of Mg})/(\text{Molecular Weight of } MgCa(CO_3)_2)$$

$$= 24.31\text{g}/184.41\text{g} = 0.1318$$

Therefore, $$\%\ \text{Dolomite} = \frac{\%\text{Elemental Mg}}{0.1318}$$

(2) X-Ray Diffraction

The two minerals comprising the greatest percentage of carbonate rocks are dolomite and calcite. It is difficult to distinguish between these two minerals using traditional petrographic techniques because of their similar properties in polarized light. Determination of the composition of cement and matrix materials also involved a degree of uncertainty. X-ray diffraction was selected as an alternative method to quantify the relative proportion of the two carbonate minerals, which would avoid the difficulties explained in the petrographic section above.

(3) Acid-Insoluble Residue Test

The acid-insoluble test was performed by dissolving approximately 500 grams of the aggregate graded finer than 9.5 mm (3/8 inch) size in 6N HCl. Three tests were performed on each source and the average determined as the percent of insoluble material. The insoluble analysis was accomplished on all of the carbonate samples using ASTM procedure D 3042, Standard Test method for Insoluble Residue in Carbonate Aggregates.

The data evaluated based on the acid-insoluble residue test were divided into two portions. These consisted of the material ≥ 74 μm (No.200 sieve) and that material < 74 μm. This boundary, 74 μm, divides the coarse size from fines for soil-like particles.

(4) Calcite Content

Because of the similar optical properties of calcite and dolomite, it is difficult to distinguish between them using a petrographic microscope. However, for high purity carbonates, like those studied in this research, it is possible to estimate calcite content based on a difference calculation. Essentially all carbonate fraction in these rocks is either dolomite or calcite. Carbonates are also soluble in HCl. Therefore if the dolomite and insoluble residue contents are known the calcite content can be estimated by the following equation:

$$\% \text{ Calcite} = 100\% - \% \text{ Dolomite} - \% \text{ Insoluble residue.}$$

(5) Summary of Compositional Results

Table 2 is a summary of the results for the compositional analysis for the previous study (Bruner, Choi and West, 1995). The elemental magnesium values (column 3) were obtained by INDOT using the EDTA titration method ASTM C602. The x-ray diffraction results for dolomite content (column 2) were obtained at Purdue University. The dolomite contents in column 5 are calculated using the elemental magnesium values in column 3 based on the equation provided previously.

$$\% \text{ Dolomite} = \frac{\% \text{Elemental Mg}}{0.1318}$$

Results for dolomite content in columns 2 and 5 are similar but not exactly the same. Typically the x-ray diffraction value is somewhat higher than the value calculated from elemental Mg content. However, sources 2, 4 and 13 are exceptions to this situation.

Table 2. Compositional Analysis, Previous Study

Sample I.D.	X-ray Diff % Dol	Elem Mg (%)	Sol-Dol % Cal	From Mg % Dol	Insol Res >200, (%)	Insol Res <200, (%)
1	98.3	12.4	4.2	94.1	1.23	0.48
2	93.23	12.9	1.4	97.9	0.02	0.69
3	96.87	11.3	11.1	85.7	2.07	1.06
4	82.72	11.3	12.2	85.7	0.11	2
5	90.18	11.6	2.2	88	3.55	6.28
7	98.66	12.9	0.8	97.9	0.21	1.13
8	99.78	13.1	0.4	99.4	0.03	0.2
9	84.53	10.7	11.4	81.2	4.4	3.02
10	95.84	12.6	0.2	95.6	1.81	2.35
11	93.69	12.2	1.1	92.6	2.71	3.59
12	92.35	11.9	2.1	90.3	3.39	4.26
13	76.4	10.1	17.8	76.6	0.78	4.78
15	99.5	12.8	2.4	97.1	0.27	0.22
17	99.65	12.9	1.8	97.9	0.03	0.32
18	97.85				0.94	1.21
19	87.65				4.45	2.21
20	94.1				3.14	2.76

Column 4 indicates the soluble portion of the rock minus the dolomite content in column 5, which yields the calcite content. This is based on the equation provided above for % calcite. Note: for sample 1, the insoluble residue percentage equals 1.23 plus 0.48 or 1.71%. 100-94.1-1.7 = 4.2% calcite content. Values for calcite content range from 0.2 to 17.8%, with all but one value at 12.2% or less. These aggregates obviously represent carbonates with a low calcite content and a high percentage of dolomite.

The total insoluble residue values are obtained by adding column 6 to column 7. Total insoluble residue values range from 0.3 to 9.93% with most values below 7%. This indicates that the aggregates have high carbonate contents with only minor amounts of impurities present.

Pitt et al. (1988, 1992) recommended that the sulfate in the deicing salt be limited to less than 0.28%. But no research included the testing of freeze-thaw effects on aggregates containing various amounts of sulfate in a salt solution.

3) Laboratory Test Results

Results of standard laboratory tests for the eighteen sources based on INDOT data are provided in Table 3. Absorption, specific gravity, sulfate soundness loss and Los Angeles Abrasion loss are included. Absorption and sulfate soundness loss are results of particular interest in this study. Sample 13 has a high sulfate soundness loss (>11%) and sample 4 has a high absorption value (>5%). Data from Tables 1, 2 and 3 are considered further in a comparison of results for the current study.

2. Work Performed During the Current Study

1) Sampling

(1) Dolomite Aggregates

As dolomite is the bedrock material used for bituminous surface courses for medium and high volume roads in Indiana, twenty-eight crushed dolomite samples were collected for analysis.

Table 3. Laboratory Test Results, Previous Study

Sample I.D.	Absorp (%)	Specif G	Sulf Soun % Loss	L.A.Abra % Loss
1	0.79	2.732	0.36	25.53
2	2.39	2.599		28.53
3	2.64	2.605	9.57	27.05
4	6.25	2.39	9.86	29.36
5	2.2	2.616	7.3	29.19
7	3.05	2.583	4.79	32.43
8	2.38	2.594	0.76	31.73
9	1.21	2.671	5.59	22.47
10	3.76	2.489	3.29	36.98
11	2.02	2.632	5.99	24.75
12	2.74	2.588	2.24	29.5
13	4	2.48	13.18	30.28
15	1.03	2.669	2.56	29.76
17	1	2.718	0.31	25.69
18	2.48	2.619	3.62	27.25
19	1.59	2.648	6.45	30.75
20	2.95	2.59	5.33	28.54

Indiana No. 11 size gradation was used and samples consisting of approximately 50 kg (110 lbs) were collected from each quarry stockpile. The eighteen sources from the previous study were augmented by ten additional sources recommended by INDOT and FHWA.

(2) Slag

Samples of slag retained on 4.75 mm (No. 4) sieve and 9.5 mm (3/8 in) sieve were provided by the Levy Company, Inc. These were tested in brine solution using slag only, but also in combination with a series of different dolomite aggregates.

2) Freeze-Thaw Testing in a Brine Solution

The objective of freeze-thaw testing was to determine resistance of coarse aggregates (both coarse and fine portions) under repeated cycles of freezing and thawing in a brine solution. This method provides information on the soundness of coarse aggregates under weathering conditions. The sulfate soundness test has been used in the past to simulate effects on aggregates during freeze-thaw conditions. A logical extension of the soundness test is to evaluate unconfined aggregate in actual freeze-thaw action (Foster, 1994). The fine aggregate used in bituminous mixes can also be tested under freeze-thaw conditions. This was not part of the current study. The testing procedure is described in Indiana Test Method 209-94, "Soundness of Aggregates by Freezing and Thawing in a Brine Solution". In AASHTO standard T 103, three suggested procedures are provided: Procedure A (total immersion procedure), B (partial immersion procedure with alcohol - water solution) and C (partial immersion with water). Procedure A requires that aggregates be completely immersed in water whereas for procedures B and C the aggregates are immersed in only 6 mm (1/4 inch) of the solution.

The Division of Material and Tests, Indiana Department of Highway concluded that the partial immersion method of freeze-thaw testing does not duplicate the actual in-place conditions of water saturation to which highway materials are subjected during their service period. Because of the common practice on Indiana highways of applying salt (NaCl) to enhance ice and snow removal, actual in-place conditions commonly occur wherein aggregates are frozen while totally immersed in a salt solution (Shakoor, 1982). Therefore, the total immersion procedure (Procedure A above) best depicts the critical in-service condition and it is specified for use in ITM 209-94.

(1) Preparation of Test Sample

a) *Washing and Drying*

The aggregate samples were thoroughly washed on a 150 μm (No. 100) sieve and dried for 24 hours at a temperature of 110.5+5°C (230.5+9.5°F) in a laboratory oven.

b) *Sieving*

Aggregates were sieved to produce coarse and fine aggregate portions in accordance with ITM 209-94, "Soundness of Aggregates by Freezing and Thawing in a Brine Solution" and AASHTO standard T103, "Soundness of Aggregates by Freezing and Thawing". A total weight of about 40 kg of each aggregate sample was sieved to obtain coarse and fine fractions for testing.

The size for fine aggregates in this test are minus 9.5 mm (3/8) in.), and retained on the 300 μm sieve (No.50). Samples were separated into the following portions; 9.5 mm (3/8 in.) to 4.75 mm (No. 4), 4.75 mm (No. 4) to 2.36 mm (No. 8), 2.36 mm (No. 8) to 1.18 mm (No. 16), 1.18 mm (No. 16) to 600 μm (No. 30), and 600 μm (No. 30) to 300 μm (No. 50). Each of the separated fractions contained about 100 g. However, not all aggregate samples collected from stockpiles included sufficient amounts of the fine fraction. Therefore, using the available aggregate samples, freeze-thaw testing in the brine solution was performed on the six samples in which ample fine grained portions were available. The size for coarse aggregate was greater than 4.75 mm (No. 4) sieve. In this test 1000 g of aggregate from 12.5 mm (1/2 in.) and 9.5 mm (3/8 in.) and 300 g of aggregate from 9.5 mm and 4.75 mm were used.

c) *Procedure*

Each of the fractions was completely immersed as an individual sample in a sealed container, in a 3 percent sodium chloride (NaCl) solution. This total immersion procedure is described in ITM 209-94 and as Procedure A, AASHTO standard T103. Alternative freezing and thawing were repeated for 25 cycles in a freezing and thawing chamber in the INDOT laboratory, Division of Materials and Tests. The required freezing period lasting for at least 15 minutes below -26.5 °C (-15.5 °F) and the thawing period between 21.5 °C(70.5 °F) and 24.5 °C(75.5 °F). After completion of the final cycle, each sample was washed and oven dried for 24 hours. Following drying, the fine aggregate was sieved over the original retaining sieve for that sample fraction to determine sample loss. However, in case of the coarse aggregate, aggregates with sizes from 12.5 mm (1/2 in.) and 9.5 mm (3/8 in.) was sieved over a smaller size, the 8.0 mm (5/16 in.) sieve and the aggregates between 9.5 mm (3/8 in.) and 4.75 mm (No. 4) were sieved over a smaller size, the 4.0 mm (No. 5) sieve to determine the weight loss.

For purposes of comparison, for the coarse aggregate samples, following freeze-thaw testing, aggregates were also sieved over the original retaining sieve sizes, the 9.5 mm and the 4.75 mm respectively, for the two portions of the sample. A comparison of results is provided in the subsequent, data analysis section of this report.

3) Pore Size Distribution

Pore size distribution of aggregate pieces was measured because it was believed that pore size distribution has a strong correlation with freeze-thaw durability. The mercury intrusion method was used to obtain this data. The mercury intrusion porosimeter was capable of intruding pores from approximately 500 micrometers to 0.0027 micrometers in size. Pores smaller than 0.0027 micrometers do not affect the durability because any water in these small size pore does not freeze at ordinary freezing temperatures, whereas pores larger than 500 micrometers are not a problem because this water is easily expelled from the pores when water expands on freezing.

The principle of the mercury intrusion technique involves the surface tension of a non-wetting liquid, which opposes the entry of the liquid into a small diameter pore. The non-wetting liquid can be forced into the pores using external pressure, and this pressure is inversely proportional to the pore diameter. Assuming a cylindrical pore, the relationship of external pressure and pore diameter is

$$\mathrm{p} = \frac{-4T_d \cos\theta}{d},$$

where p = external pressure required for intrusion, T_d = surface tension of the intruding liquid, θ = contact angle between the liquid and the solid, d = limiting pore diameter. Because of its low vapor pressure, nonreactivity, and non-wetting characteristics, mercury is the commonly used liquid. The pore size distribution is obtained by measuring the volume of intruded mercury at various pressures, which in turn correspond to different pore diameters. According to studies by

Kaneuji (1978) and Shakoor (1982), values of 484 erg/cm^2 and 125° were used for T_d and θ respectively.

The mercury intrusion porosimeter at Purdue University, manufactured by American Instrument Company, has a maximum capacity of about 415 Mpa (60,000 psi). It was used in this study to evaluate the carbonate aggregates.

(1) Procedure

A total of 22 aggregate samples were tested to obtain their pore size distributions. About 2 to 5 grams of each specimen were used and at least 2 pieces of each sample were tested. Oven-dried samples were evacuated and initially surrounded by mercury. The pressure was raised in small increments and mercury entered the sample in 1 minute after each increment was recorded. With each pressure increment, mercury was forced into the accessible pores of the diameter larger than or equal to that calculated by the above equation. For the cumulative intrusion at different pressures, the corresponding pore diameter was calculated.

(2) Corrections of intrusion data

Two types of correction are needed:

1. Below atmospheric pressure, continued compression of the air in the penetrometer after evacuation must be subtracted from the intrusion reading.
2. At higher pressures, mercury is compressed and its volume decreases providing one effect, whereas the heat generated by the pressure causes the mercury to expand has another effect. The high pressure correction value must be calculated from blank tests.

4) Hydrometer Analysis of Insoluble Residue

The hydrometer analysis was performed to determine the particle-size distribution for insoluble residues, the portion finer than No. 200 sieve size (0.075 mm). The lower limit of particle-size that can be measured by this procedure is about 0.001 mm (1 μm). This test was performed to determine the amount of clay particles present in the smaller than No. 200 sieve fraction of the insoluble residue material. These samples contain both silt and clay. The hydrometer method of analysis is based on Stoke's law, which relates the terminal velocity of a sphere (solid soil particle) falling freely through a fluid, to its diameter. The relation is

$$V = \frac{\gamma_s - \gamma_f}{180\eta} D^2$$

where V = terminal velocity of soil solids, cm/sec

γ_s = density of soil solids, g/cm^3

γ_f = density of water, g/cm^3

η = viscosity of fluid, g/sec/cm^2

D = diameter of the soil particle, mm

It is assumed that Stoke's law can be applied to a mass of dispersed soil particles of various shapes and sizes. This test is used to determine the percentage of dispersed soil particles remaining in suspension. The maximum grain size equivalent to a spherical particle is computed for each hydrometer reading using Stoke's Law. The ASTM procedure D 422, Particle-Size Analysis of Soils, was used and type 152H hydrometer was used.

In the previous project (HPR-2082, Bruner, Choi and West, 1995) performed at Purdue University, the acid-insoluble analysis was conducted on the carbonate samples using ASTM procedure D 3042, Standard Test Method for Insoluble Residue in Carbonate Aggregates. In the current study these insoluble residue samples were analyzed using the hydrometer analysis on the acid-insoluble residue samples obtained during that previous study.

5) Freeze-Thaw Testing with Deicing Salt Solution

In response to concerns regarding detrimental effects of trace compounds in deicing salt, it was decided to perform freeze-thaw tests using the actual deicing salts obtained from several

INDOT highway districts. Six different salts were supplied from the highway districts. Selected for testing were 4.75 mm (No. 4) sieve size aggregates because they were available from the previous sampling process, whereas the coarser sizes had been expended during the first round of testing. In order to include the 9.5 mm (3/8 in.) size, additional quarry sampling would have been required, providing an additional sample process. It was decided not to re-sample the sources, but to use the 4.75 mm size aggregates. The brine tests were performed using the same procedure as that used previously, that is, according to ITM 209-94 and Procedure A of AASHTO Standard T103.

Another concern was the behavior of slag in a brine solution. Because slag is a by-product of steel production from a blast furnace, slags commonly contain iron and other constituents, which could be released to trigger reactions with a dolomite aggregate. Therefore, the aggregate samples were mixed with slag in equal proportions by weight. Using this mixture, freeze-thaw tests were performed in salt solutions made from the six deicing samples, following the same procedure as used in previous tests.

3. Results

1) Freeze-Thaw Testing in a Brine Solution

The objective of freezing and thawing testing in a brine solution was to determine aggregate soundness when deicing salts are applied. It was suspected that a relation existed between aggregate properties and the soundness when immersed in brine solutions. Therefore, based on the results of the brine freeze-thaw tests, correlation between average weight loss of aggregates in the freeze and thaw test and the chemical and petrographical properties of aggregates was studied. The results of brine freeze and thaw tests for the two coarse fractions and the combined total for the 28 aggregate sources are shown in Table 4. For the first fraction, approximately 1000 grams of aggregate was used, which was originally 12.5 to 9.5 mm (1/2 to

Table 4 . Results of Freeze and Thaw Test in a Brine Solution.

Sample I.D.	9.5 mm			4.75 mm			Weighted Average		
	Before T	After T	W.L (%)	Before T	After T	W.L (%)	Before T	After T	W.L (%)
D-001	997.2	959.5	3.78	299.3	291.1	2.74	1296.5	1250.6	3.54
D-002	999.8	988.3	1.15	299.4	294.4	1.67	1299.2	1282.7	1.27
D-003	996.5	951.9	4.48	299	291.7	2.44	1295.5	1243.6	4.01
D-004	999	982.3	1.67	299.4	296.1	1.10	1298.4	1278.4	1.54
D-005	998.2	878.2	12.02	299.4	272	9.15	1297.6	1150.2	11.36
D-007	997.8	909.5	8.85	299.5	279.8	6.58	1297.3	1189.3	8.32
D-008	998.5	991.7	0.68	299.4	298.4	0.33	1297.9	1290.1	0.60
D-009	997.6	972.1	2.56	299.6	290.3	3.10	1297.2	1262.4	2.68
D-010	998.1	965.1	3.31	299.7	296.3	1.13	1297.8	1261.4	2.80
D-011	996.8	944.4	5.26	298.9	291.2	2.58	1295.7	1235.6	4.64
D-012	998	941.9	5.62	299.3	289.4	3.31	1297.3	1231.3	5.09
D-013	997.5	865.9	13.19	298.4	275.4	7.71	1295.9	1141.3	11.93
D-015	998	970.9	2.72	299.5	293.6	1.97	1297.5	1264.5	2.54
D-017	998.4	991.6	0.68	299.6	298.5	0.37	1298	1290.1	0.61
D-018	999	966.1	3.29	299.4	293.1	2.10	1298.4	1259.2	3.02
D-019	1000.4	862.6	13.77	299.8	267	10.94	1300.2	1129.6	13.12
D-020	998.4	865.4	13.32	299.5	264.7	11.62	1297.9	1130.1	12.93
D-021	996	873.3	12.32	299.7	272.5	9.08	1295.7	1145.8	11.57
D-022	1001.3	665.9	33.50	300.4	281	6.46	1301.7	946.9	27.26
D-023	1001.2	803.8	19.72	300.2	246	18.05	1301.4	1049.8	19.33
D-024	999.7	913.6	8.61	299.8	287.7	4.04	1299.5	1201.3	7.56
D-025	1001	995.7	0.53	300.3	298.7	0.53	1301.3	1294.4	0.53
D-026	1001.7	922	7.96	299.9	278.4	7.17	1301.6	1200.4	7.78
D-027	999.9	861.9	13.80	300.6	291.7	2.96	1300.5	1153.6	11.30
D-028	1000.9	997.1	0.38	300.3	295.3	1.67	1301.2	1292.4	0.68
D-029	1000.5	993.6	0.69	300.3	297.6	0.90	1300.8	1291.2	0.74
D-030	999.8	835	16.48	299.7	266	11.24	1299.5	1101	15.28

3/8") in size. After 25 cycles of freezing and thawing the sample was sieved over the 8.0 mm (5/16") sieve. For the smaller sized fraction, approximately 300 grams originally ranging from 9.5 to 4.75 mm (3/8" to No.4). After 25 cycles of freezing and thawing, the sample was sieved over the 4.0 mm (No.5) sieve. A weighted average for the loss was calculated using the weight loss of each fraction of aggregate combined according to the sample weights involved. The average weight loss of coarse aggregates ranged from 0.6% to 27.26% as shown in Table 4. According to Hoover (1994), the maximum allowable, freeze-thaw loss in a brine solution for Class A stone, coarse aggregates should be 30%. This is apparently equivalent to the 12% maximum allowable loss for the water freeze-thaw test and the 12% loss for the sodium sulfate soundness test. Therefore, all 28 aggregates tested pass this requirement as they have losses less than the maximum. However, the value for D-22 approaches 30% with its 27.26% loss.

Table 5 gives the results of the brine freeze-thaw testing on the fine portion of the No.11 gradation for six aggregate sources. The average weight losses ranged from 3.88% to 13.72%. All are well below 30% loss. Recall that for the fine aggregate portion, following freezing and thawing, the sample is sieved over the original retaining sieve, rather than using a somewhat smaller size as is the practice for coarse aggregate testing. This suggests that brine freeze-thaw loss for the fine aggregate portion should be somewhat greater than that for the coarse aggregate portion, all other factors being equal.

Table 5. Results of a Brine Freeze-Thaw Testing Fine Portion, No. 11 Gradation.

Sample I.D	R4.75mm			R2.36mm			R1.18mm			R0.60mm			Averaged W.L
	B.T	A.T	W.L(%)	B.T	A.T	W.L(%)	B.T	A.T	W.L(%)	B.T	A.T	W.L(%)	(%)
D-007	299.5	279.8	6.58	100.6	93	7.55	100	88.8	11.20	100	87.9	12.10	9.91
D009	299.6	290.3	3.10	100.1	85.8	14.29	100.1	92.3	7.79	100	93.9	6.10	7.48
D-010	299.7	296.3	1.13	100.1	98.7	1.40	100.1	98.9	10.19	101.7	98.3	3.34	3.88
D-020	299.5	264.7	11.62	101.1	79.7	21.17	100.4	93.8	6.57	100.5	85.8	14.63	13.72
D-021	299.7	272.5	9.08	101.3	95.2	6.02	100	88.4	11.60	100.2	91.3	8.88	8.89
D-029	300.3	296.2	1.37	100.1	96.8	3.30	100	88.1	11.90	100.1	94.7	5.39	5.47

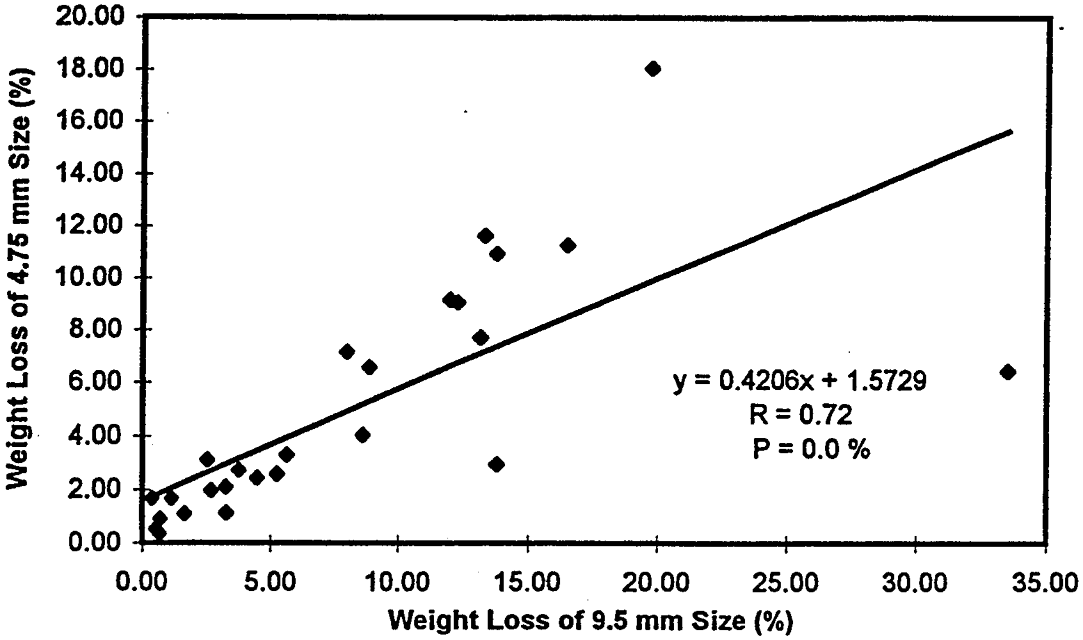


Figure 1 Correlation of freeze-thaw weight loss between 9.5 mm sieve size and 4.75 mm sieve size

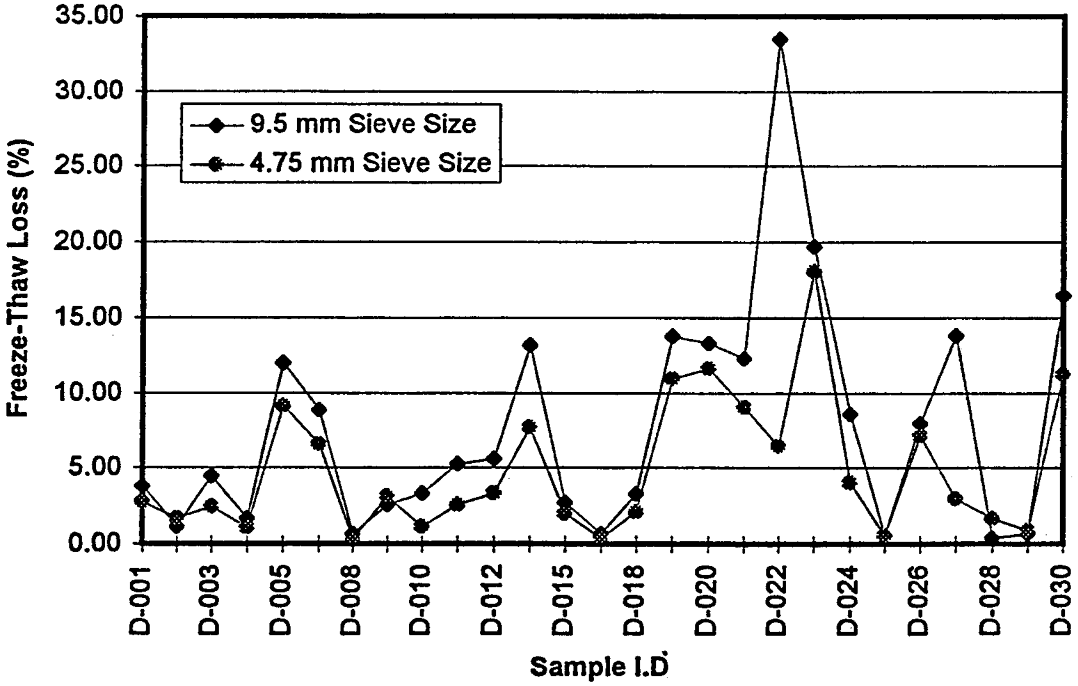


Figure 2 Comparison of freeze-thaw loss and aggregate size

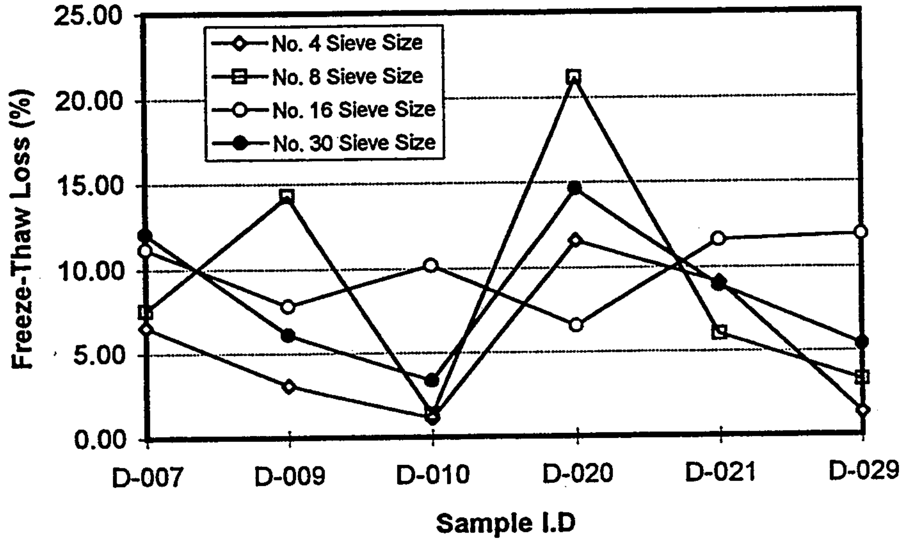


Figure 3 Comparison of freeze-thaw weight loss and aggregate size in fine aggregate

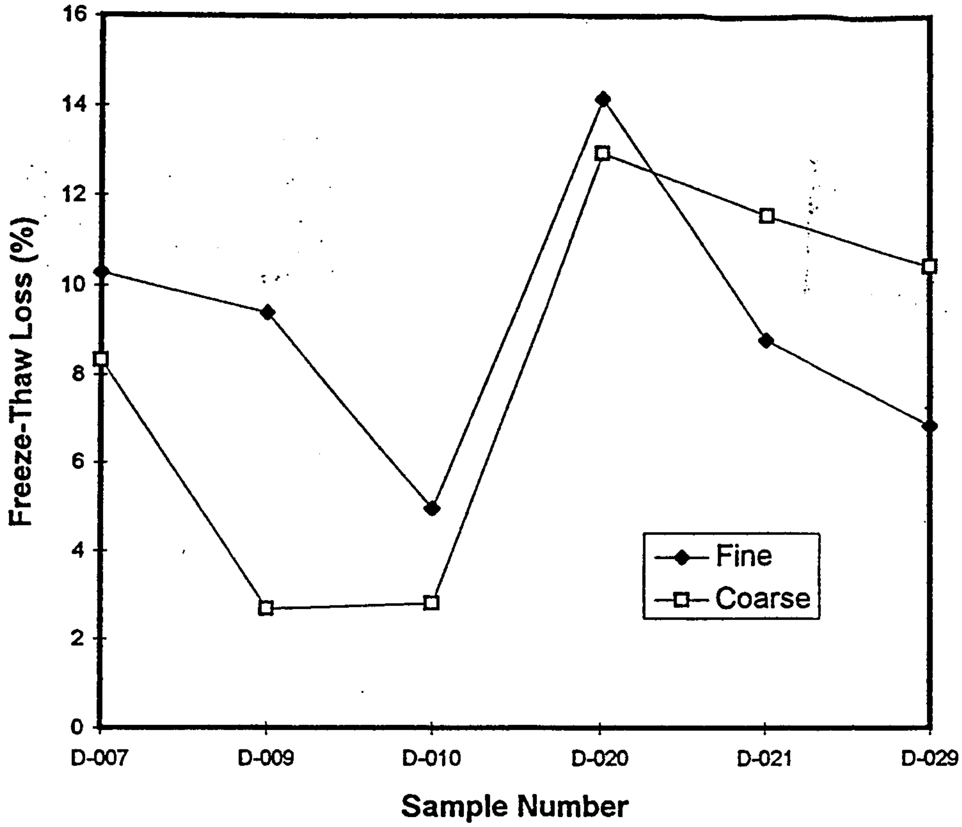


Figure 4 Comparison of percent freeze-thaw loss between fine and coarse aggregates

Referring again to coarse aggregate testing results, there is good correlation for the weight loss between the 9.5 mm (3/8 in.) sieve size samples and the 4.75 mm (No.4) sieve size samples (Figure 1). The correlation coefficient is 0.90 at a significance level P -0.0%. This excellent correlation between the two sieve sizes suggests that the freeze-thaw loss for one size can be used to estimate the weight loss for the other size. Figure 2 is a plot of the comparison of the freeze-thaw loss versus the sieve sizes for coarse aggregates. As can be observed in Figure 2 the freeze-thaw weight loss of the 9.5 mm (3/8 in.) sieve size fraction is almost always greater than the loss for 4.75 mm (No. 4) sieve size.

By contrast, no consistent relationship between weight loss in the brine test versus sieve size for the fine portion of coarse aggregates is observed (Figure 3). Considerable variation in the amounts of loss, does occur, up to nine times the loss from one sieve size to another.

As discussed previously, for purposes of comparison in the analysis of the coarse aggregate portion of the No.11 gradation, the loss from brine freeze-thaw testing was also evaluated using larger sieve sizes. The 9.5 mm (3/8") and 4.75 mm (No. 4 sieve) were used to measure freeze-thaw loss rather than the larger 8.0 mm (5/16") and 4.0 mm (No. 5 sieve) specified in the test method. Table 6 provides the results when samples are sieved over the lower size of the gradation before testing, in order to measure aggregate loss. Only the last nine samples (D22-D30) are included. Losses are several times greater in Table 6 as compared to Table 5. Obviously, using the larger size sieve to measure freeze-thaw loss significantly increases the amount of loss.

Prior to performing the freeze-thaw testing, it was estimated that the weight loss for fine aggregate would generally be greater than the loss for the coarse aggregate of the same sample

Table 6. Results of Brine Freeze-Thaw Testing in a Brine Solution, Coarse Aggregates, Original Sieve.

Sample I.D	9.5 mm			4.75 mm			Weighted Average		
	B.T	After T	W.L	B.T	After T	W.L	B.T	After T	W.L
D-22	1001.3	446.7	55.39	300.4	272.8	9.19	1301.7	719.5	44.73
D-23	1001.2	581.1	41.96	300.2	232.1	22.68	1201.4	813.2	37.51
D-24	999.7	805.8	19.40	299.8	284.4	5.14	1299.5	1090.2	16.11
D-25	1001	935.4	6.55	300.3	297.6	0.90	1301.3	1233.0	5.25
D-26	1001.7	861.8	13.97	299.9	274.4	8.50	1301.6	1136.2	12.71
D-27	999.9	798.4	20.15	300.6	289	3.86	1300.5	1087.4	16.39
D-28	1000.9	961.2	3.97	300.3	294	2.10	1301.2	1255.2	3.54
D-29	1000.5	868.8	13.16	300.3	296.2	1.37	1300.8	1165.0	10.44
D-30	999.8	747.6	25.23	299.7	262.4	12.45	1299.5	1010.0	22.28

source. This is because of the sieves used to determine weight loss are the same as the lower limit of the original gradation rather than a smaller size sieve as is the case for the coarse aggregate. In most cases freeze-thaw loss of fine aggregates did prove to be greater than that for coarse aggregate of the same sample. Samples D-7,9,10 and 20 in Figure 4 show this tendency.

2) Correlation of Brine Freeze-Thaw Loss to Petrographic and Chemical Properties and Physical Tests of Aggregate

As discussed previously, data on petrographic and chemical properties and physical tests of the aggregates performed in previous projects for the same Indiana dolomite sources were available for this project. Therefore, the relationship between aggregate properties and brine freeze-thaw weight loss was evaluated. From the correlation results between aggregate properties and brine freeze-thaw loss, one can evaluate the mechanism for deterioration caused by salt and by trace minerals. Data of petrographic and compositional analysis are provided in Tables 1 and 2 respectively and the data related to physical tests are tabulated in Table 3. Based on this information the coefficient of correlation in calculated between the weighted average, brine freeze-thaw loss and the properties of aggregate (Table 7). Significant correlation is considered P = 5.0% or less because P = 5.0% probability level is the level where relevant correlation begins.

Table 7. Correlations of Petrographic Factors and Percent Brine Freeze-Thaw Loss Weight Average.

		PETROGRAPHIC ANALYSIS						
Sample I.D.	F-T Loss (%)	Grn Size (mm)	Median (mm)	Grain Stand De	Matrix (%)	Grnd Mss (%)	Grains (%)	Voids (%)
1	3.54	0.13	0.085	9.9	21.5	28.4	59.9	11.7
2	1.27	0.079	0.061	3.6	2.8	30.5	59.1	10.4
3	4.006	0.059	0.042	3	21.6	42.8	49.3	7.9
4	1.54	0.194	0.053	12	26.4	41.9	48.4	9.7
5	11.359	0.069	0.052	3.8	30.9	49	45.8	5.2
7	8.325	0.074	0.075	3.7	12.3	27.4	64.4	8.2
8	0.601	0.087	0.065	4.2	5.5	14.8	71.8	13.4
9	2.683	0.066	0.063	4.9	13.2	27.2	63.5	9.3
10	2.805	0.07	0.049	3.9	2.8	28.1	61.7	10.2
11	4.683	0.058	0.041	3.5	7.9	27.8	64.7	7.5
12	5.087	0.082	0.051	4.1	7.5	30.5	64.9	4.6
13	11.93	0.057	0.043	4.9	47.5	64.8	31.2	4
15	2.543	0.116	0.085	7	24.8	36.9	58.5	4.6
17	0.609	0.119	0.114	8.7	9.4	25	68	7
18	3.019	0.063	0.058	3.6	47	60.7	35.8	3.5
19	13.121	0.059	0.046	7.4	7.4	12.6	77.6	9.8
20	12.929	0.085	0.068	4.2	13.2	34.7	55	10.3
	R =	-0.414874	-0.34464	-0.20267	0.231274	0.197139	-0.17752	-0.2067

Table 7 (continued)

		COMPOSITIONAL ANALYSIS						PHYSICAL TESTS			
Sample I.D.	F-T Loss (%)	X-ray Diff % Dol	Elem Mg (%)	Slb-Dol % Cal	From Mg % Dol	Insol Res >200, (%)	Insol Res <200, (%)	Absorp (%)	Specif G	Sulf Soun % Loss	L.A.Abra % Loss
1	3.54	98.3	12.4	4.2	94.1	1.23	0.48	0.79	2.732	0.36	25.53
2	1.27	93.23	12.9	1.4	97.9	0.02	0.69	2.39	2.599		28.53
3	4.006	96.87	11.3	11.1	85.7	2.07	1.06	2.64	2.605	9.57	27.05
4	1.54	82.72	11.3	12.2	85.7	0.11	2	6.25	2.39	9.86	29.36
5	11.359	90.18	11.6	2.2	88	3.55	6.28	2.2	2.616	7.3	29.19
7	8.325	98.66	12.9	0.8	97.9	0.21	1.13	3.05	2.583	4.79	32.43
8	0.601	99.78	13.1	0.4	99.4	0.03	0.2	2.38	2.594	0.76	31.73
9	2.683	84.53	10.7	11.4	81.2	4.4	3.02	1.21	2.671	5.59	22.47
10	2.805	95.84	12.6	0.2	95.6	1.81	2.35	3.76	2.489	3.29	36.98
11	4.683	93.69	12.2	1.1	92.6	2.71	3.59	2.02	2.632	5.99	24.75
12	5.087	92.35	11.9	2.1	90.3	3.39	4.26	2.74	2.588	2.24	29.5
13	11.93	76.4	10.1	17.8	76.6	0.78	4.78	4	2.48	13.18	30.28
15	2.543	99.5	12.8	2.4	97.1	0.27	0.22	1.03	2.669	2.56	29.76
17	0.609	99.65	12.9	1.8	97.9	0.03	0.32	1	2.718	0.31	25.69
18	3.019	97.85				0.94	1.21	2.48	2.619	3.62	27.25
19	13.121	87.65				4.45	2.21	1.59	2.648	6.45	30.75
20	12.929	94.1				3.14	2.76	2.95	2.59	5.33	28.54
	R =	-0.41144	-0.496972	0.283901	-0.49688	0.52007	0.605346	0.078496	-0.09596	0.49007	0.183879

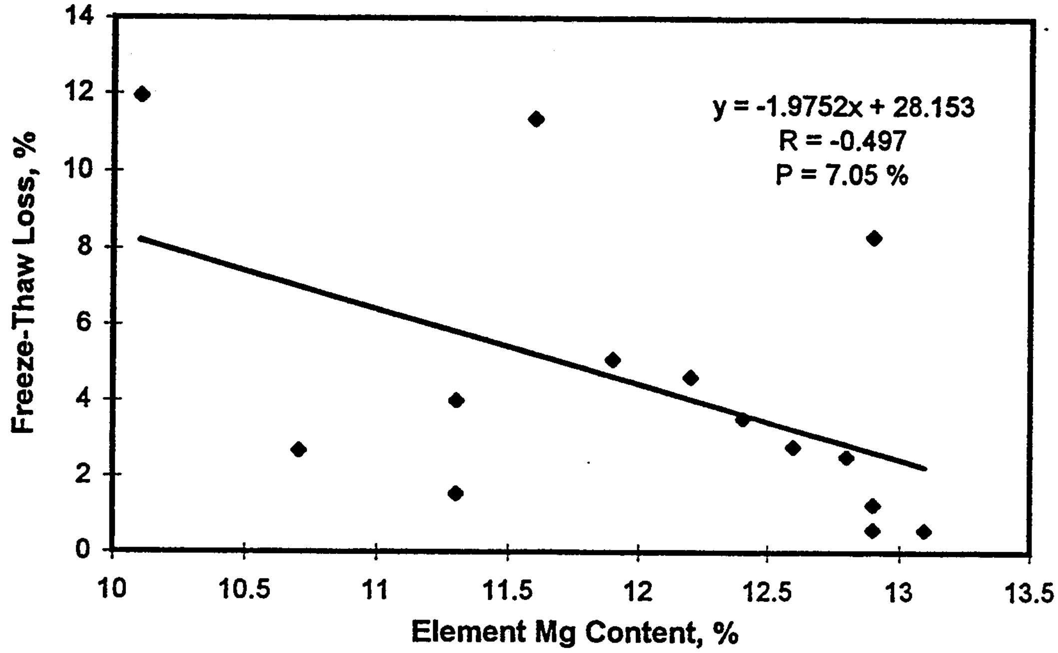


Figure 5 Correlation of element Mg content and percent freeze-thaw loss

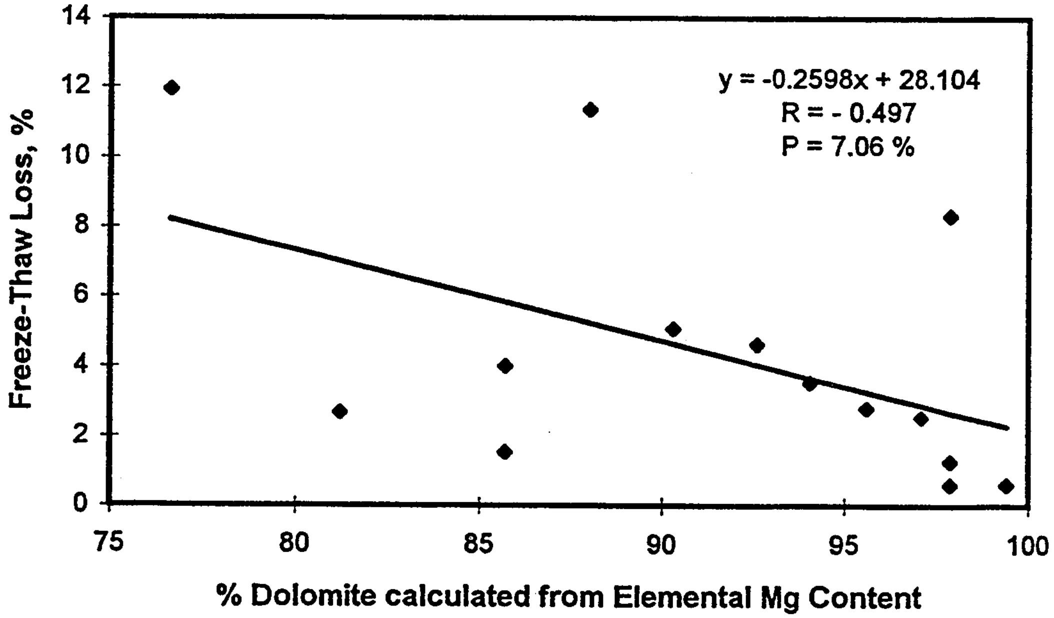


Figure 6 Correlation of percent dolomite (from % elemental Mg) and percent freeze-thaw loss

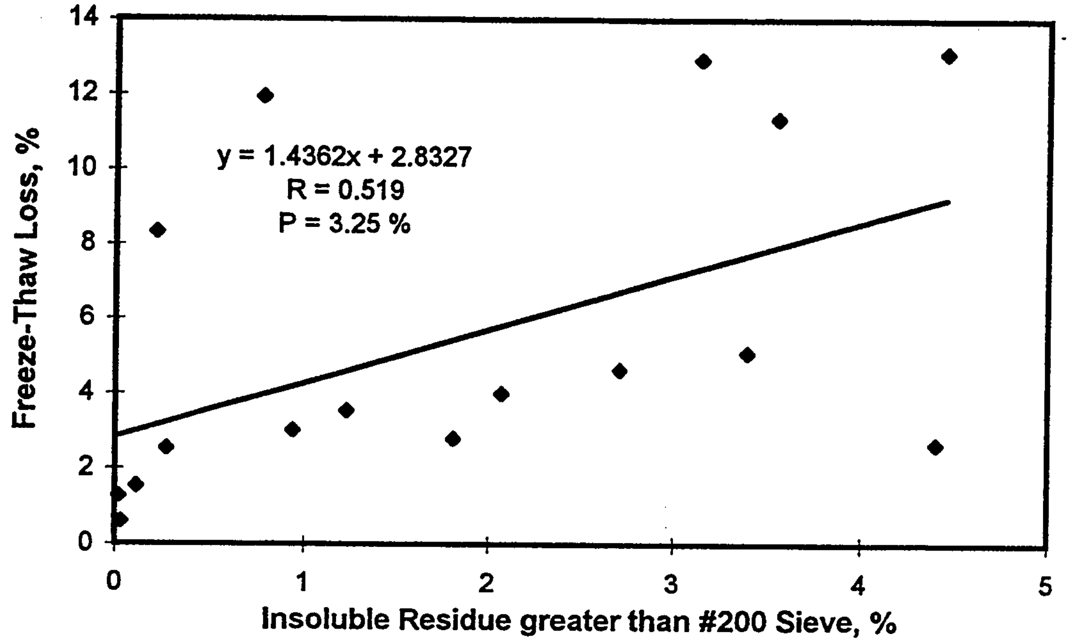


Figure 7 Correlation of acid-insoluble residue >#200 and percent freeze-thaw loss

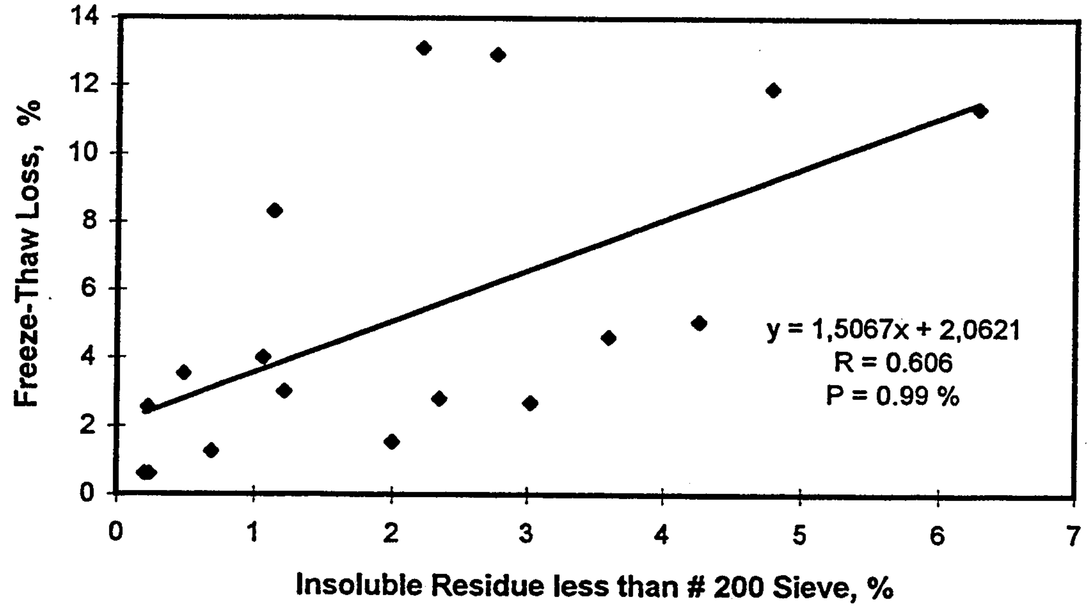


Figure 8 Correlation of acid-insoluble residue < #200 and percent freeze-thaw loss

Significant levels at P = 0.0% are considered highly significant, but a P value greater than 5.0% denotes an insignificant relationship.

The correlation coefficient between the brine weight loss and elemental magnesium content is 0.497 but the P value is 7.05% (Figure 5). This shows that the correlation is not significant because the P value is greater than 5.0%. The correlation coefficient of percent dolomite estimated from percent elemental magnesium is 0.497, but the P level is 7.06% (Figure 6) and also not significant.

The correlation of acid-insoluble residue with the brine solution freeze-thaw test yields the coefficients less than 5.0% P indicating that both relationships are significant. The insoluble residue fraction greater than 0.075 mm (No. 200 sieve) has 0.159 as the coefficient of correlation at P = 3.25%. Also the coefficient for the insoluble residue less than 0.075 mm is 0.606 at 0.99 P significance level (Figures 7 and 8).

According to a memorandum by Hoover (1994) for studies performed at INDOT, the correlation coefficient between sodium sulfate soundness test results and brine freeze-thaw test results in fine aggregates was found to be 0.92 at P = 0.0%. Therefore he concluded that sodium sulfate soundness values had a very good correlation with the brine freeze-thaw test. This is a good indication that the sodium sulfate soundness test can be replaced by the brine freeze-thaw test. However, the value obtained in the current study, 0.49 at 5.41% P value (Figure 9), does not indicate an equally strong relationship between the two parameters.

In Table 8 correlation coefficients are shown for the brine freeze-thaw results for the 9.5 mm (3/8 in.) sieve size portion of coarse aggregate as compared to various aggregate properties. In Table 9, correlation coefficients are shown for the 4.75 mm (No.4) sieve portion versus various aggregate properties. A summary of the correlation coefficients the weighted average, 9.5 mm

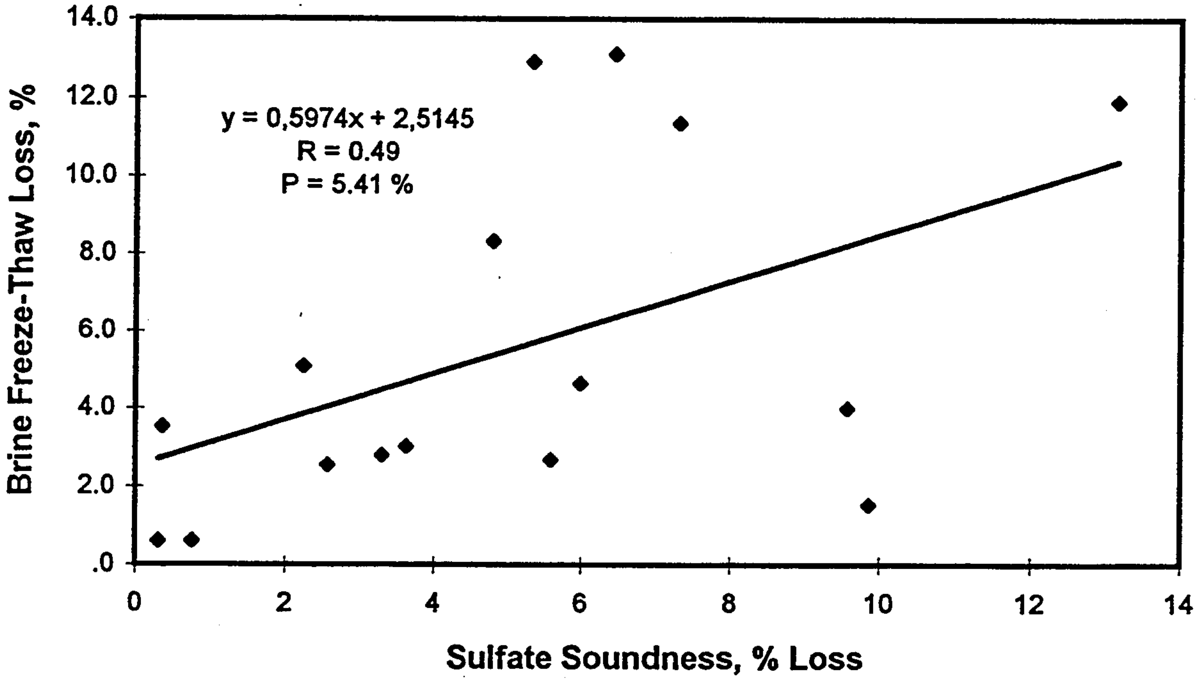


Figure 9 Correlation of sulfate soundness and percent brine freeze-thaw loss

Table 8. Correlations of Sample Properties and Percent Brine Freeze-Thaw Loss.

SAMPLE RETAINED 9.5 mm (3/8 in.) SIEVE

		PETROGRAPHIC ANALYSIS						
Sample I.D.	F-T Loss (%)	Grn Size (mm)	Median (mm)	Grain Stand De	Matrix (%)	Grnd Mss (%)	Grains (%)	Voids (%)
1	3.78	0.13	0.085	9.9	21.5	28.4	59.9	11.7
2	1.15	0.079	0.061	3.6	2.8	30.5	59.1	10.4
3	4.48	0.059	0.042	3	21.6	42.8	49.3	7.9
4	1.67	0.194	0.053	12	26.4	41.9	48.4	9.7
5	12.02	0.069	0.052	3.8	30.9	49	45.8	5.2
7	8.85	0.074	0.075	3.7	12.3	27.4	64.4	8.2
8	0.68	0.087	0.065	4.2	5.5	14.8	71.8	13.4
9	2.56	0.066	0.063	4.9	13.2	27.2	63.5	9.3
10	3.31	0.07	0.049	3.9	2.8	28.1	61.7	10.2
11	5.26	0.058	0.041	3.5	7.9	27.8	64.7	7.5
12	5.62	0.082	0.051	4.1	7.5	30.5	64.9	4.6
13	13.19	0.057	0.043	4.9	47.5	64.8	31.2	4
15	2.72	0.116	0.085	7	24.8	36.9	58.5	4.6
17	0.68	0.119	0.114	8.7	9.4	25	68	7
18	3.29	0.063	0.058	3.6	47	60.7	35.8	3.5
19	13.77	0.059	0.046	7.4	7.4	12.6	77.6	9.8
20	13.32	0.085	0.068	4.2	13.2	34.7	55	10.3
	R =	-0.423461	-0.36282	-0.20944	0.247262	0.216747	-0.19506	-0.22772

Table 8 (continued)

		COMPOSITIONAL ANALYSIS						PHYSICAL TESTS			
Sample I.D.	F-T Loss (%)	X-ray Diff % Dol	Elem Mg (%)	Slb-Dol % Cal	From Mg % Dol	Insol Res >200, (%)	Insol Res <200, (%)	Absorp (%)	Specif G	Sulf Soun % Loss	L.A.Abra % Loss
1	3.78	98.3	12.4	4.2	94.1	1.23	0.48	0.79	2.732	0.36	25.53
2	1.15	93.23	12.9	1.4	97.9	0.02	0.69	2.39	2.599		28.53
3	4.48	96.87	11.3	11.1	85.7	2.07	1.06	2.64	2.605	9.57	27.05
4	1.67	82.72	11.3	12.2	85.7	0.11	2	6.25	2.39	9.86	29.36
5	12.02	90.18	11.6	2.2	88	3.55	6.28	2.2	2.616	7.3	29.19
7	8.85	98.66	12.9	0.8	97.9	0.21	1.13	3.05	2.583	4.79	32.43
8	0.68	99.78	13.1	0.4	99.4	0.03	0.2	2.38	2.594	0.76	31.73
9	2.56	84.53	10.7	11.4	81.2	4.4	3.02	1.21	2.671	5.59	22.47
10	3.31	95.84	12.6	0.2	95.6	1.81	2.35	3.76	2.489	3.29	36.98
11	5.26	93.69	12.2	1.1	92.6	2.71	3.59	2.02	2.632	5.99	24.75
12	5.62	92.35	11.9	2.1	90.3	3.39	4.26	2.74	2.588	2.24	29.5
13	13.19	76.4	10.1	17.8	76.6	0.78	4.78	4	2.48	13.18	30.28
15	2.72	99.5	12.8	2.4	97.1	0.27	0.22	1.03	2.669	2.56	29.76
17	0.68	99.65	12.9	1.8	97.9	0.03	0.32	1	2.718	0.31	25.69
18	3.29	97.85				0.94	1.21	2.48	2.619	3.62	27.25
19	13.77	87.65				4.45	2.21	1.59	2.648	6.45	30.75
20	13.32	94.1				3.14	2.76	2.95	2.59	5.33	28.54
	R =	-0.41899	-0.499975	0.289871	-0.49992	0.509079	0.617926	0.094065	-0.11437	0.506541	0.199014

Table 9. Correlations of Sample Properties and Percent Brine Freeze-Thaw Loss.

SAMPLE RETAINED 4.75 mm SIEVE

		PETROGRAPHIC ANALYSIS						
Sample I.D.	F-T Loss (%)	Grn Size (mm)	Median (mm)	Grain Stand De	Matrix (%)	Grnd Mss (%)	Grains (%)	Voids (%)
1	2.74	0.13	0.085	9.9	21.5	28.4	59.9	11.7
2	1.67	0.079	0.061	3.6	2.8	30.5	59.1	10.4
3	2.44	0.059	0.042	3	21.6	42.8	49.3	7.9
4	1.1	0.194	0.053	12	26.4	41.9	48.4	9.7
5	9.15	0.069	0.052	3.8	30.9	49	45.8	5.2
7	6.58	0.074	0.075	3.7	12.3	27.4	64.4	8.2
8	0.33	0.087	0.065	4.2	5.5	14.8	71.8	13.4
9	3.1	0.066	0.063	4.9	13.2	27.2	63.5	9.3
10	1.13	0.07	0.049	3.9	2.8	28.1	61.7	10.2
11	2.58	0.058	0.041	3.5	7.9	27.8	64.7	7.5
12	3.31	0.082	0.051	4.1	7.5	30.5	64.9	4.6
13	7.71	0.057	0.043	4.9	47.5	64.8	31.2	4
15	1.97	0.116	0.085	7	24.8	36.9	58.5	4.6
17	0.37	0.119	0.114	8.7	9.4	25	68	7
18	2.1	0.063	0.058	3.6	47	60.7	35.8	3.5
19	10.94	0.059	0.046	7.4	7.4	12.6	77.6	9.8
20	11.62	0.085	0.068	4.2	13.2	34.7	55	10.3
R =		-0.36444	-0.25411	-0.16648	0.158173	0.109209	-0.09919	-0.11094

Table 9 (continued)

		COMPOSITIONAL ANALYSIS						PHYSICAL TESTS			
Sample I.D.	F-T Loss (%)	X-ray Diff % Dol	Elem Mg (%)	Slb-Dol % Cal	From Mg % Dol	Insol Res >200, (%)	Insol Res <200, (%)	Absorp (%)	Specif G	Sulf Soun % Loss	L.A.Abra % Loss
1	2.74	98.3	12.4	4.2	94.1	1.23	0.48	0.79	2.732	0.36	25.53
2	1.67	93.23	12.9	1.4	97.9	0.02	0.69	2.39	2.599		28.53
3	2.44	96.87	11.3	11.1	85.7	2.07	1.06	2.64	2.605	9.57	27.05
4	1.1	82.72	11.3	12.2	85.7	0.11	2	6.25	2.39	9.86	29.36
5	9.15	90.18	11.6	2.2	88	3.55	6.28	2.2	2.616	7.3	29.19
7	6.58	98.66	12.9	0.8	97.9	0.21	1.13	3.05	2.583	4.79	32.43
8	0.33	99.78	13.1	0.4	99.4	0.03	0.2	2.38	2.594	0.76	31.73
9	3.1	84.53	10.7	11.4	81.2	4.4	3.02	1.21	2.671	5.59	22.47
10	1.13	95.84	12.6	0.2	95.6	1.81	2.35	3.76	2.489	3.29	36.98
11	2.58	93.69	12.2	1.1	92.6	2.71	3.59	2.02	2.632	5.99	24.75
12	3.31	92.35	11.9	2.1	90.3	3.39	4.26	2.74	2.588	2.24	29.5
13	7.71	76.4	10.1	17.8	76.6	0.78	4.78	4	2.48	13.18	30.28
15	1.97	99.5	12.8	2.4	97.1	0.27	0.22	1.03	2.669	2.56	29.76
17	0.37	99.65	12.9	1.8	97.9	0.03	0.32	1	2.718	0.31	25.69
18	2.1	97.85				0.94	1.21	2.48	2.619	3.62	27.25
19	10.94	87.65				4.45	2.21	1.59	2.648	6.45	30.75
20	11.62	94.1				3.14	2.76	2.95	2.59	5.33	28.54
	R =	-0.36719	-0.464863	0.247259	-0.46462	0.550525	0.532096	0.010647	-0.01565	0.407203	0.118168

Table 10. Summary of Regression Analysis for Each Sample Size.

	WEIGHTED AVERAGE		Samples Retained 9.5 mm Sieve		Samples Retained 4.75 mm Sieve	
Obser Parameter	Correlation Coeff.	P-value (%)	Correlation Coeff.	P-value (%)	Correlation Coeff.	P-value (%)
Grain Size	-0.415	9.82	-0.423	9.03	-0.364	15.04
Median	-0.345	17.64	-0.363	15.23	-0.254	32.5
Grain Std	-0.203	43.64	-0.209	41.98	-0.166	52.31
Matrix (%)	0.231	37.08	0.247	33.87	0.158	54.43
Grnd Mss (%)	0.197	44.75	0.217	40.33	0.109	67.65
Grains (%)	-0.178	49.50	-0.195	45.31	-0.099	70.49
Voids (%)	-0.207	42.63	-0.228	37.94	-0.111	67.16
X-ray diff % Dol	-0.411	10.08	-0.419	9.41	-0.368	0.147
Elem Mg (%)	-0.497	7.05	-0.5	6.87	-0.465	9.4
Slb-Dol Cal(%)	0.284	32.41	0.29	31.48	0.247	39.41
From Mg % Dol	-0.497	7.06	-0.5	6.87	-0.464	9.42
Insol Resi >200	0.519	3.25	0.509	3.69	0.551	2.2
Insol Resi <200	0.606	0.99	0.618	0.82	0.533	2.74
Sulf Sound	0.490	5.41	0.507	4.52	0.407	11.74
L.A. Abra	0.185	46.81	0.199	44.38	0.118	65.15
Absorption	0.079	76.39	0.094	71.95	0.011	96.77
Specif G	-0.096	71.35	-0.114	66.21	-0.016	95.24

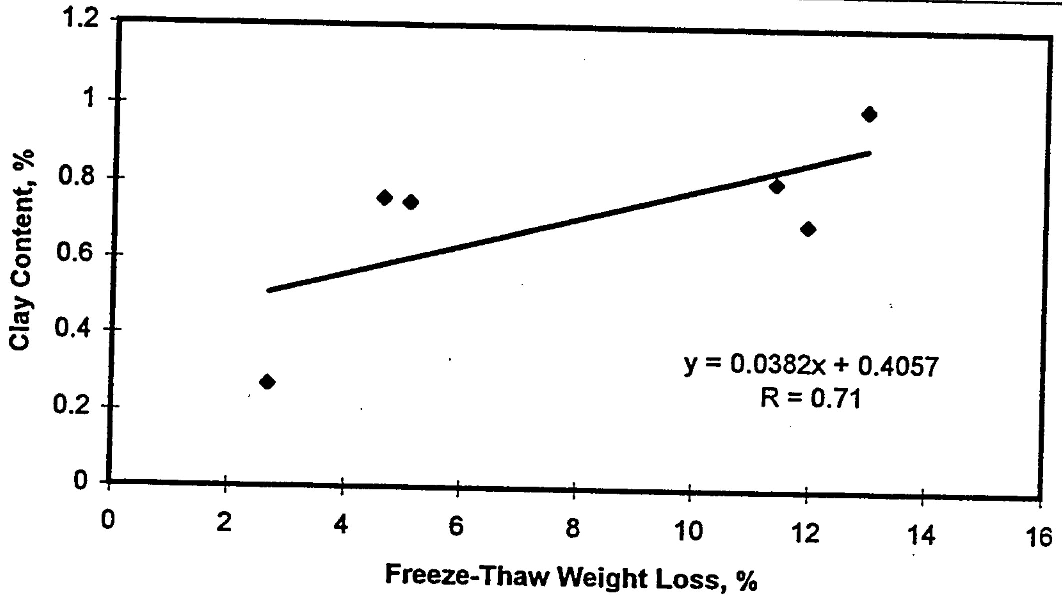


Figure 10 Correlation of percent clay content and percent freeze-thaw loss

and the 4.75 mm sizes are tabulated in Table 10. In most cases, the 9.5 mm (3/8 in) sieve size fraction shows better correlation than does the 4.75 mm (No. 4) sieve size.

3) Hydrometer Analysis

In the above discussion, a good correlation was found between brine freeze-thaw weight loss and acid-insoluble residue less than No. 200 sieve (0.606, P=0.99). Based on this finding it was decided to evaluate the relationship between the amount of clay in the insoluble residue portion less than No. 200 sieve versus the brine freeze-thaw loss. Hydrometer analyses were performed to determine the amount of clay mineral present in the silt and clay size fraction of the residue. Grain size distribution of insoluble residues for each aggregate are shown in Appendix B and the results of hydrometer analysis are tabulated in Table 11. The amount of clay ranges from 0.27 to 1.0%. The plot of correlation between freeze-thaw loss and clay amount is shown in Figure 10. The correlation coefficient is 0.71; this value is improved from 0.606, which is the coefficient for acid-insoluble residue less than No. 200 sieve. Therefore, it is concluded that an increase in clay mineral correlates with increased degradation of the aggregate in a brine freeze-thaw test and the clay mineral percentage in the insoluble residue has a stronger correlation than does the insoluble residue alone. Therefore, the amount of clay in an aggregate affects the freeze-thaw soundness more than does the total amount of insoluble residue less than No. 200 sieve for the aggregate sample.

Table 11. Results of Hydrometer Analysis.

Sample I.D	Brine F-T Weight Loss (%)	Insoluble Residue less than No.200 (%)	Clay Amount (%)
5	11.36	6.28	0.81
9	2.68	3.02	0.27
11	4.64	3.59	0.76
12	5.09	4.26	0.75
13	11.93	4.78	0.7
20	12.93	2.76	1

4) Pore Size Distribution

The size, shape, and distribution of pores in a rock comprise one of the most important properties in determining hydraulic pressure generated in aggregate during freezing (Powers and Helmuth, 1953). The correction factors for 15 intervals above atmosphere pressure are provided in Table 12 (Shakoor, 1982).

Table 12. Correction Factors in Mercury Porosimeter.

Pressure metric	Pressure psi	Correction (ml/ml of Hg in Penetrometer)
Kpa 207	30	0.00000
345	50	0.00000
620	90	0.00002
Mpa 1.03	150	0.00003
1.72	250	0.00005
3.10	450	0.00008
5.52	800	0.00011
8.97	1300	0.00015
15.9	2300	0.00020
27.6	4000	0.00026
48.3	7000	0.00034
82.8	12000	0.00042
138	20000	0.00051
241	35000	0.00063
414	60000	0.00076

The concept of Expected Durability Factor (EDF) was developed by Kaneuji (1978) to predict aggregate performance from porosimeter data. According to Kaneuji (1978), EDFs, based on median pore size and the total volume of mercury intruded, correlated very well with the freeze-thaw durability. The EDF was calculated from pore size distribution using following equation.

$$EDF = 0.579/PV + 6.12\,(MD) + 3.04,$$

where PV = intruded volume of pores

MD = median diameter of pores

Table 13. Results of Mercury Intrusion Porosimeter Test, Phase I.

Sample I.D.	PV	MD	EDF
D-001	0.008	14.90	166.60
	0.027	9.10	80.15
		MEAN	**123.38**
D-002	0.008	0.05	75.69
	0.045	36.61	239.98
	0.003	44.75	469.91
		MEAN	**270.83**
D-003	0.018	0.26	36.77
	0.012	3.58	73.20
	0.015	13.61	124.93
		MEAN	**78.28**
D-004	0.065	2.16	25.17
	0.049	5.64	49.37
	0.007	0.02	85.86
		MEAN	**53.50**
D-005	0.007	0.11	86.42
	0.007	0.31	87.66
		MEAN	**87.04**
D-007	0.022	0.02	29.50
	0.034	0.81	25.03
	0.015	0.09	42.21
		MEAN	**32.25**
D-008	0.018	9.10	90.87
	0.014	5.97	80.93
		MEAN	**85.90**
D-009	0.07	10.22	73.83
	0.041	1.06	23.64
	0.025	0.28	27.89
		MEAN	**41.50**
D-010	0.03	2.31	36.46
	0.019	5.82	69.12
	0.012	1.99	63.47
		MEAN	**56.35**
D-011	0.015	2.79	58.68
	0.035	0.13	20.38
		MEAN	**39.53**
D-012	0.016	0.31	41.09
	0.088	2.42	24.45
		MEAN	**32.77**
D-013	0.033	0.16	21.59
	0.034	0.66	24.08
		MEAN	**22.84**
D-015	0.033	3.18	40.07
	0.017	8.61	89.79
		MEAN	**64.93**
D-017	0.003	11.03	263.54
	0.004	112.00	833.23
		MEAN	**548.39**
D-018	0.023	0.34	30.26
	0.009	0.31	69.28
		MEAN	**49.8**
D-019	0.009	1.59	77.10
	0.003	0.02	196.18
		MEAN	**136.64**
D-20	0.065	4.11	37.08
	0.01	29.80	243.32
	0.024	0.17	28.19
		MEAN	**102.86**
D-21	0.004	15.01	239.66
	0.022	1.59	39.09
		MEAN	**151.32**

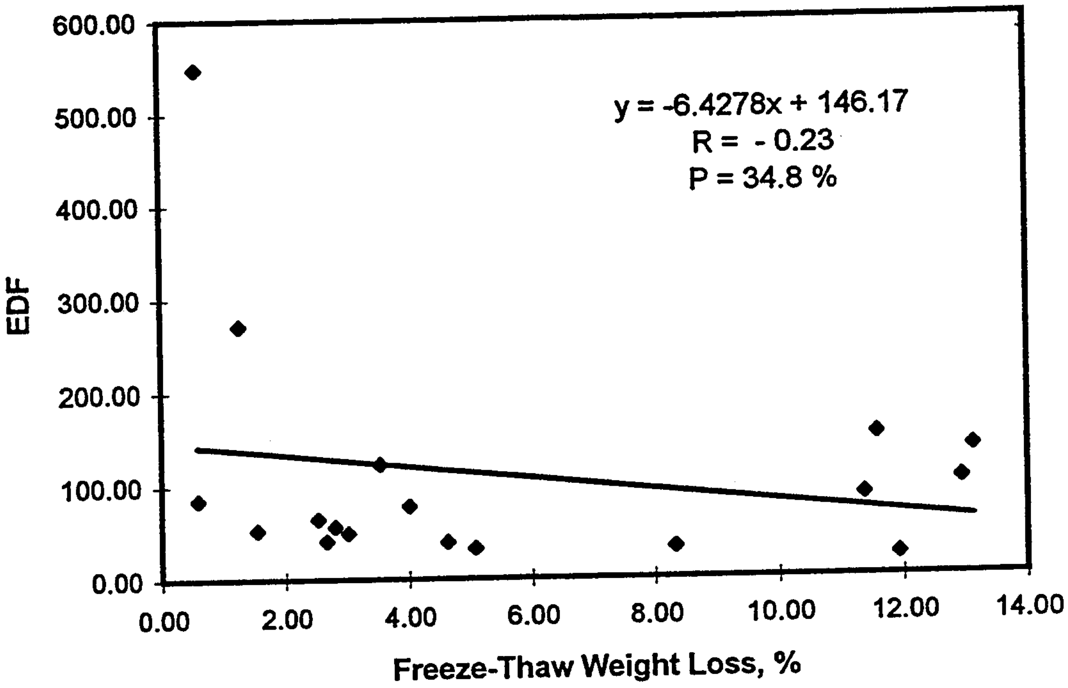


Fig. 11 Correlation of EDF and percent freeze-thaw weight loss

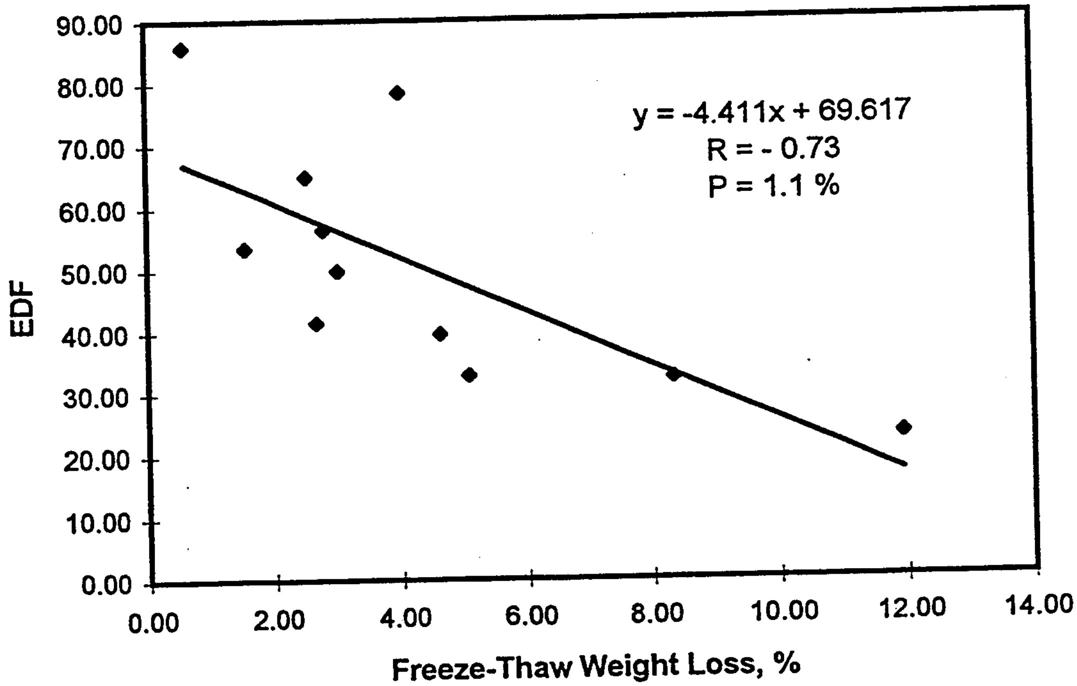


Fig. 12 Correlation of EDF and percent freeze-thaw weight loss after modification

As a result of mercury intrusion porosimeter, the pore size distributions and EDFs for each aggregate are given Appendix C. The summaries of calculation of EDF values in each sample were tabulated in Table 13 and the correlation between mean EDF and a brine freeze-thaw weight loss is shown in Figure 11. The coefficient of correlation is 0.23 at the P = 34.8% probability level indicating insignificant correlation. However, since rock composition and textures of every aggregate piece in a sample are not identical, each aggregate piece may have a significantly different EDF value and, therefore, the reliability of the average EDF is questionable. Whereas some samples have similar EDF values such as from 29.5 to 42.2 in D-07 and 23.6 to 73.8 in D-09, the EDF of others range from 80.15 to 166.60 in sample D-01, from 75.69 to 469.91 in D-02 and from 28.19 to 243.32 in D-20. This problem was pointed out by Hockette (1987) and therefore he concluded that the development of an EDF-based acceptance specification was not recommended.

To reduce the variation in porosimetry data shown in Table 13 and Figure 11, samples with the widest range in EDF values were excluded from the comparison. Seven samples were excluded and the data replotted to yield Figure 12. Note that the seven values excluded from Figure 12 fall on or above the solid line in Figure 11. In effect, the highest EDF values have been excluded, those apparently with the greatest resistance to freeze-thaw deterioration. Therefore, Figure 12 only applies to the EDF relationship for values below 100.

The correlation coefficient was recalculated for Figure 12 yielding a major improvement to 0.73 at a significance level of P=1.1% from 0.23 at P=34.8% in Figure 11. For the seven samples excluded it was assumed that the mean value shown in Table 13 did not properly represent the wide range of individual values. For these it is assumed that extremely different textures were included in the individual porosimetry tests for that sample source.

It is of interest to examine the values for EDF used in previous studies to evaluate pavement performance. Kaneuji (1978) was primarily interested in freeze-thaw resistance of concrete pavements and D-cracking. His results suggest that carbonate aggregates with an EDF below 70 are potentially susceptible to D-cracking when poor drainage for the pavement base course prevails. However, aggregates in a bituminous overlay experience a much less critical situation than that of the coarse aggregates in concrete pavements.

Shakoor (1982) evaluated argillaceous carbonate aggregates in concrete pavements where disintegration of coarse aggregate had occurred after only a few cycles of freezing and thawing. His results indicated that carbonate aggregates with an EDF below 20 are potentially susceptible to this type of disintegration.

Note that in Table 13 the lowest average EDF values are D13 at 22.84, D007 at 32.25, D12 at 32.77, D11 at 39.53, D9 at 41.5, D18 at 49.8, D4 at 53.5 and D10 at 56.35. By contrast D1, D2, D3, D5, D8, D17, D19, D20 and D21 have EDF values greater than 70.

Because of the large variation of EDF in some aggregate samples in Table 13, it was decided to select an aggregate source and subdivide it based on texture to determine whether a consistent EDF value could be obtained for a single texture type. The source selected was D26 and D27. In this quarry Ledges 1 through 3 are placed in one stock pile and Ledges 5 and 6 are placed in another. This is the source that was used in the fourth Indiana pavement that showed deterioration soon after placement. This is a section of SR62, north of Charlestown in southeastern Indiana. This field site is discussed further in a subsequent section of the report.

Samples of Indiana #11 gradations were obtained from each of the two stockpiles. It was decided to perform a megascopic petrographic examination on the two largest portions of the gradation; the 12.7 to 9.5mm (1/2-3/8") size and the 9.5-4.75mm (3/8" -#4) size. Tables 14 and 15 provide the results of the petrographic examination; Table 14 is for the sample of Ledge 1 to 3 material and Table 15 is for the sample of Ledge 5 to 6.

Referring to Table 14, the sample was found to consist of four major rock materials. Particle shape was also determined, designating equidimensional (cube shape) ranging to flat or bladed. Most particles were fairly equal in dimensions. The two categories, light gray dolomite and gray dolomite, comprise most of the sample, 84 to 86% for the two size fractions. The remainder is a darker gray dolomite or a white, coarser material. In all, this sample from Ledges 1 to 3 appears to be quite sound, a high quality material.

Table 14. Petrographic Examination #11 Gradation Stock Pile Sample, Ledge 102-3.

	12.7-9.5mm	9.5-4.75mm
Light gray fine dolomite	(43.5)	(46.1)
Equidimensional	38.0	19.7
Moderately equidimensional	-	14.1
Flat	5.5	11.0
Bladed	-	1.3
Gray fine dolomite	(40.2)	(40.0)
Equidimensional	36.9	21.7
Moderately equidimensional	-	11.0
Flat	3.3	3.4
Bladed	-	3.9
Darker gray, fine dolomite	(8.0)	(2.8)
Equidimensional	8.0	1.4
Modeately equidimensional	-	1.1
Flat	-	0.3
White, coarsely crystalline, somewhat calcareous	(8.3)	(11.1)
Equidimensional	4.9	7.7
Moderately equidimensional	-	1.9
Flat	-	1.5
With stylolites	3.4	-

Table 15, as indicated above, is material from Ledges 5 and 6. A very sizable portion of the aggregate consists of a blue-gray dolomite, 63 to 49% respectively for the 12.7 to 9.5mm and the 9.5 to 4.75mm portions. This is the material providing the darker color as observed in the stockpile. A significant amount of the samples, 23% and 13% respectively for the 12.7 to 9.5mm and the 9.5 to 4.75mm size fractions, contain visible voids. This particular lithology is not present in the Ledge 1 to 3 sample. The same blue gray dolomite without, or with few visible voids, makes up the rest of this blue material, to yield the total of 63 and 49% respectively for the two sizes studied.

Table 15. Petrographic Examination #11 Gradation Stock Pile Sample, Ledge 5-603.

	12.7-9.5mm	9.5-4.75mm
Blue-gray, fine dolomite with visible voids	(23.2)	(12.6)
Equidimensional	20.3	10.4
More voids, weaker	2.2	1.0
Flat	0.7	1.2
Blue-gray, fine dolomite with few to no visible voids	(30.2)	(34.1)
Equidimensional	27.3	25.6
Flat	2.9	8.5
Light gray, fine dolomite	(22.9)	(33.3)
Equidimensional	20.0	22.7
Flat	2.9	10.6
Medium gray, fine dolomite	(22.8)	(18.4)
Equidimensional	20.8	12.1
Flat	2.0	3.7
Bladed	-	2.6
White, green and gray, massive cherty dolomite	0.3	0.9
Fine, weathered, calcareous dolomite	-	0.6
Shale, flat	0.7	0.2

Table 16. Number of Porosimetry Samples Tested for Individual Lithologies, Phase II

Ledge Designation	Subdivision	Brief Description	Number of Samples Tested
102-3	L102	Light gray, fine dolomite	9
102-3	L2-3 Fine	Gray, fine, dolomite	7
102-3	L2-3 Dark	Dark gray, fine dolomite	7
5-603	L5	Light gray, fine dolomite	6
5-603	L6A	Blue-gray, fine dolomite w/o visible voids	3
5-603	L6B	Blue-gray fine dolomite w/ visible voids	6

Table 17. Results of Mercury Intrusion Porosimeter Test, Phase II.

Sample	PV	MD	EDF
L6B	0.021	15.517	125.58
	0.097	44.504	281.37
	0.075	29.669	192.33
	0.072	42.56	271.55
	0.025	18.288	138.12
	0.076	45.683	290.22
L102	0.023	1.948	40.136
	0.025	1.326	34.315
	0.01	3.19	80.463
	0.018	0.2	36.431
	0.02	2.622	48.037
	0.014	0.096	44.985
	0.092	3.647	31.653
	0.036	0.976	25.096
	0.019	0.85	38.716
L6A	0.002	1.77	303.37
	0.021	1.652	40.722
	0.006	0.639	103.45
L2-3 Fine	0.051	2.928	32.312
	0.018	0.033	35.409
	0.025	2.48	41.378
	0.029	1.273	30.796
	0.008	0.355	77.588
	0.043	3.399	37.307
	0.023	0.568	31.69
L2-3 Dark	0.04	0.817	22.515
	0.009	0.0213	67.504
	0.023	0.284	29.952
	0.042	2.196	30.265
	0.064	3.682	34.621
	0.06	2.673	29.049
	0.057	2.273	27.109
L5	0.038	0.533	21.539
	0.03	0.355	24.513
	0.04	0.639	21.426
	0.022	0.779	34.126
	0.024	1.06	33.652
	0.019	2.125	46.519

Light gray and medium gray dolomite comprises most of the remainder of the aggregate evaluated in Table 15, 46% and 52% respectively for the 12.7 to 9.5mm and the 9.5 to 4.75mm portions. This appears to be a high-quality material. Only a small portion of weaker materials is present, 1% and 1.6% respectively for the two portions. This is not sufficiently abundant to yield an aggregate quality problem.

The pore size distribution of each subdivision, measured by the procedure using mercury porosimeter described above, are shown in Appendix D and the results of calculation EDF are shown in Table 17. As can be observed in Table 17, the EDF values in each subdivision are much more consistent than in Table 13. The EDF ranges from 30.8 to 41.4 for Ledge 2-3, fine texture, except for one value of 77.6. It ranges from 21.4 to 46.5 in Ledge 5. Analysis of the EDF values is provided in Table 18. Included is the number of tests, range, mean and standard deviation for each of the different lithologic groups.

Table 18. Statistical Analysis of EDF Values, Phase II

Designation	Number of Samples	Range	EDF Mean, $\bar{x}$	Standard Deviation, s
L102	9	25.1-80.5	42.3	15.0
L2-3F	7	30.8-77.6	40.9	15.3
L2-3D	7	22.5-67.5	34.4	14.4
L-5	6	21.4-46.5	30.3	8.8
L-6A	3	40.7-303.4	149.2	112.0
L-6B	6	125.6-290.2	216.6	64.1

Recall that Kaneuji (1978) was mainly interested in freeze-thaw resistance of concrete pavements with regard to D-cracking. His results suggest that carbonate aggregates with an EDF below 70 are potentially susceptible to D-cracking when poor drainage for the pavement system prevails. Only samples L-6A and L-6B have a mean value of EDF greater than 70. However, the aggregate in a bituminous overlay experiences a much less critical situation than that of the coarse concrete aggregates in concrete pavements.

Recall also that Shakoor (1982) evaluated argillaceous carbonate aggregates in concrete pavements where disintegration of the coarse aggregate had occurred after only a few cycles of freezing and thawing. His results indicated that carbonate aggregates with an EDF below 20 are potentially susceptible to this rapid disintegration. All six of the subdivisions in Table 18 have a mean value of EDF greater than 20. The lowest is L-5 with a value of 30.3, but the lowest individual value obtained for a sample in this group is 21.4, not much above 20.

An overall value of EDF can be estimated for the two designated ledges in the quarry, Ledge 102-3 and Ledge 5-603. Three different lithologies were discerned and evaluated for Ledge 102-3, these are L102, light gray fine dolomite, L2-3 fine, gray dolomite and L2-3 dark, fine gray dolomite. Referring to Table 14 for percentages of the constituents and normalizing the data to exclude the white crystalline material which was not included in the porosimetry study, we obtain the following: L102 = 47.5%, L2-3F = 43.8% and L2-3 Dark = 8.7%.

Using these data, a weighted averaged for the EDF of Ledge 102-3 can be calculated as follows: $\text{EDF} = (0.475)(42.3)+(0.438)(40.9)+(0.087)(34.4) = 41.0$

Three different lithologies were also discerned for Ledge 5-603, these are L5, light gray, fine dolomite; L6A, blue gray fine dolomite without visible voids, and L6B, blue gray, fine dolomite with visible voids. Referring to Table 14 for percentages of the constituents and normalizing the data, we obtain the following: L5 = 46.1%, L6A = 30.5% and L6B = 23.4%. Using these data, a weighted average for the EDF of Ledge 5-603 can be calculated as follows:

$$\text{EDF} = (0.461)(30.3)+(0.305)(149.2)+(0.234)(216.6) = 110.2$$

Based on the above calculation the collective EDF value for Ledge 102-3 is 41.0 and for Ledge 5-603 is 110.2; both are well above 20, the value of concern for rapid deterioration of aggregates from freeze-thaw attack.

Another way to evaluate the aggregate quality is to calculate the percentage of the aggregate gradation that is likely to have an EDF below 20, assuming a normal distribution for the EDF data. This is accomplished by evaluating the standard deviation values provided in Table 18. For L102 with a mean = 42.3 and standard deviation = 15.0 only 6.9% of the distribution lies below EDF = 20. (Note that x = mean and S = standard deviation).

For L2-3F with x = 40.9 and S = 15.3 only 8.6% of the distribution lies below EDF = 20. For L2-3d, x = 34.4 and S = 14.4 only 15.9% lies below EDF = 20. For L-5, x = 30.3 and S = 8.8, only 12.1% lies below EDF = 20. For L-6A, x = 149.2 and S = 112.0, only 14.7% lies below EDF = 20. For L-6B, x = 216.6 and S = 64.1, only 0.1% lies below EDF = 20. Using the same weighted average, for Ledge 102-3 as used above yields the following:

$$(0.475)(6.9)+(0.438)(8.6)+(0.087)(15.9) = 8.4\%;$$

and for Ledge 5-603

$$(0.461)(12.1)+(0.305)(14.7)+(0.234)(0.1) = 10.1\%.$$

If Ledges 102-3 and 5-603 are mixed in equal amounts the EDF value obtained is 0.5(41.0+110.2) = 75.6. In the same way the percent of the aggregate below EDF of 20 is 0.5(8.4+10.1) = 9.3%.

Based on the above calculations 8.4% of Ledge 102-3 would have an EDF less than 20 and 10.1% of Ledge 5-603 would have an EDF less than 20. When the two ledges are mixed equally together the resulting aggregate would have 9.3% of the material registering less than EDF = 20.

From the previous results shown in Table 18 the range of values was small enough to obtain a meaningful value for the averaged EDF. This shows that the variations of EDF for each piece of sample was small. Therefore, it is concluded, if aggregate samples are subdivided based on their texture and lithology, a consistent and representative EDF value for each sample can be obtained. In addition, given the proportion of each textural subdivision present, one can obtain an overall EDF value for the aggregate source. Therefore, reliable correlations between EDF and the brine freeze-thaw weight loss can be developed. Data from this porosimetry analysis are presented in Table 18.

In 1996 a problem developed on a bituminous surface course near Charlestown, Indiana on SR62. The crushed stone used was from source D-26. On the pavement surface, a significant amount of pitting occurred in the overlay where the coarse aggregate pieces were previously located. This yielded an appearance of missing or popped out aggregate pieces. The pavement is a MV or medium volume road. Coarse aggregate used in the pavement is from Ledges 5 to 6 of the quarry.

During the field investigation by Dr. West of the pavement on SR-62, it was observed that considerably more than 10% of the coarse aggregate pieces in the asphalt overlay had been removed by pitting. It was estimated that about one-half of the coarse aggregate pieces were missing, with pits marking the point of their former location. As calculated above, only 10.1% of Ledges 5 and 6 would have an EDF value of 20 or less. This suggests that the pitting problem is caused by some other mechanism than rapid deterioration of the coarse aggregate. Therefore, based on these considerations, it is concluded that both Ledges 102-3 and 5-603 are much higher quality than those aggregates known to disintegrate rapidly during freezing and thawing.

5) Freeze-Thaw Testing with Deicing Salt Solution

Six sources of halite deicing salts were obtained from INDOT districts offices. Their origin, general description and moisture content are provided in Table 19.

Table 19. Description of Halite Deicing Salts, INDOT

Salt Sample	Origin	Description, dry	Moisture Content % by wt.
A	Louisville	Green w/gray chunks	0.251
B	Chicago	Green w/gray chunks	0.341
C	Elkhart	Olivine green	0.362
D	Toledo	White w/gray chunks	0.325
E	Chicago	White w/blue and gray chunks	0.190
F	New Paris	Grayish white w/gray chunks	0.259

Chemical analyses of the halite deicing salts were performed at Purdue University. Salt samples were dissolved in water and diluted with deionized water to accomplish the chemical analysis of the solute. Only cation concentration was evaluated as this was deemed to have the greatest potential for deterioration.

The results of the chemical analysis of the six deicing salt samples are provided in Table 20. The cation composition is shown; the amount of potassium present is 0.06% or less and the magnesium content is 0.007% or less. No obvious indication was found of chemical constituents which would cause accelerated attack of aggregate particles on a bituminous surface.

Deleterious effects of trace compounds in deicing salt have been a concern regarding freeze-thaw resistance. In this research, brine freeze-thaw testing using deicing salt solution was performed to evaluate the effects that trace compounds present in deicing salt have on aggregate durability.

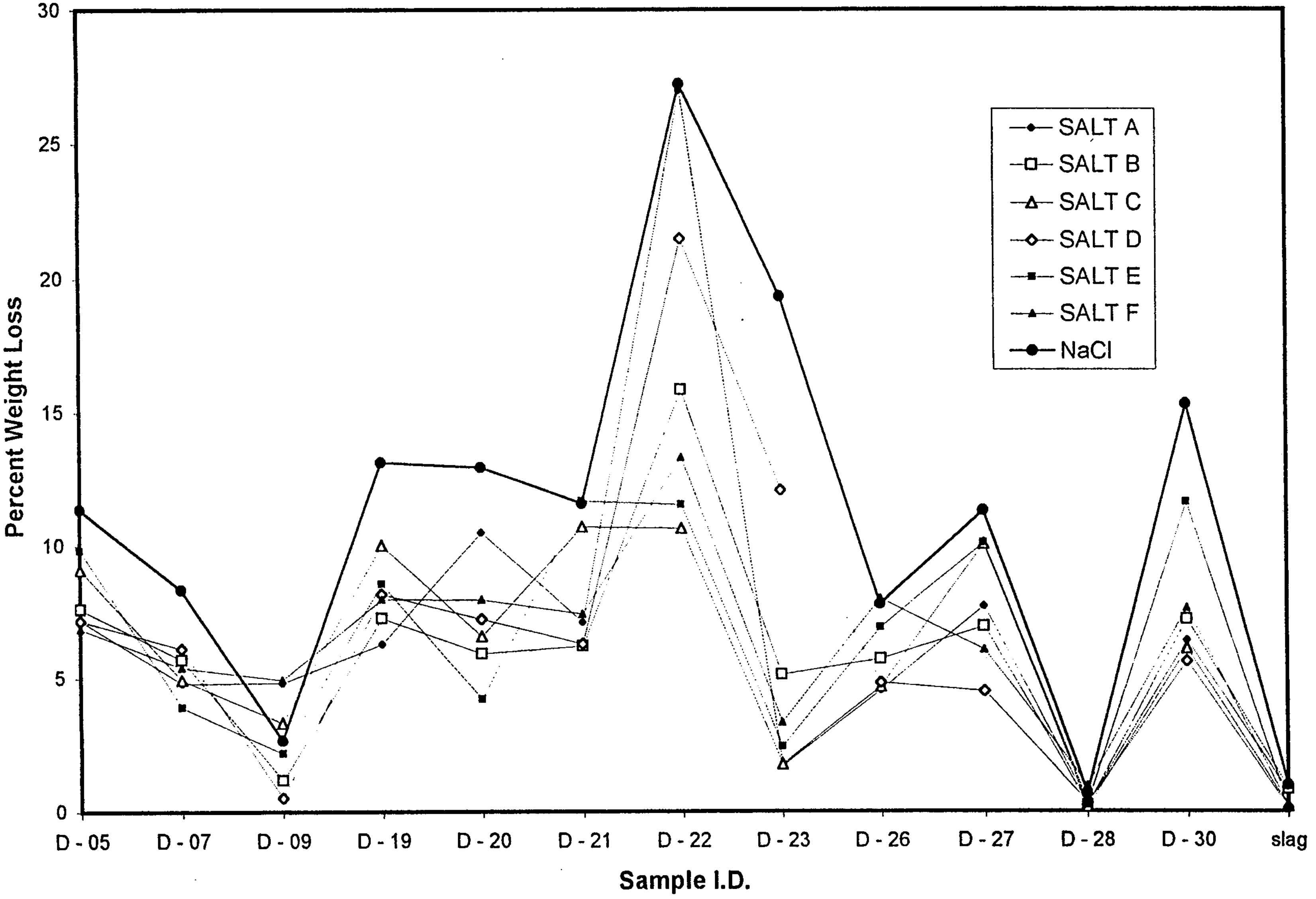
Table 20. Composition of Halite Deicing Salts, INDOT

Salt Sample #	%K	%Mg	%Ca	%Na	%Ba	ppm Fe	ppm Sr	% Insoluble	% Cl^- Assumed	% Accounted For
A	0.017	0.007	0.222	39.2	0.00	269	675	6.4	39.4	85.2
B	0.027	0.004	0.216	40.4	0.00	99	768	4.8	40.6	86.1
C	0.041	0.004	0.285	48.5	0.00	75	488	2.6	48.8	100.2
D	0.047	0.003	0.341	40.2	0.00	131	1044	5.4	40.6	86.5
E	0.055	0.004	0.035	41.1	0.00	170	1723	2.4	41.2	84.7
F	0.059	0.005	0.233	40.3	0.00	159	774	5.1	40.6	86.4

Table 21. Results of Brine Freeze-Thaw Testing with Different Deicing Salts, 4.75 mm Sieve Size.

Sample	SALT A			SALT B			SALT C			Salt D			Salt E			Salt F		
I.D.	Before T	After T	W.L(%)	Before T	After T	W.L(%)	Before T	After T	W.L(%)	Before T	After T	W.L(%)	Before T	After T	W.L(%)	Before T	After T	W.L(%)
5	299.9	278.3	7.20	299.7	276.9	7.61	300.7	273.4	9.08	299.7	278.2	7.17	300.1	270.6	9.83	299.7	279.1	6.87
7	299.8	285.4	4.80	300.2	283.1	5.70	299.7	284.9	4.94	300.1	281.7	6.13	299.7	288	3.90	299.8	283.6	5.40
9	300.4	285.9	4.83	300	296.4	1.20	300.5	290.5	3.33	300.7	299.1	0.53	300.2	293.6	2.20	300.2	285.4	4.93
19	300	281.1	6.30	299.8	278	7.27	300.3	270.2	10.02	299.8	275.3	8.17	299.5	273.9	8.55	299.8	275.9	7.97
20	299.7	268.2	10.51	299.6	281.8	5.94	300.5	280.7	6.59	299.8	278.1	7.24	299.9	287.3	4.20	300.3	276.4	7.96
21	300.6	279.2	7.12	300.7	282	6.22	300.4	268.2	10.72	300.5	281.6	6.29	300.7	265.6	11.67	300	277.7	7.43
22	300	218.9	27.03	300.3	252.7	15.85	300.2	268.2	10.66	300	235.5	21.50	300.6	265.9	11.54	300.6	260.6	13.31
23	300.4	295.2	1.73	300.1	284.7	5.13	300	294.7	1.77	300.5	264.2	12.08	300.8	293.5	2.43	300.7	290.7	3.33
26	300.1	286.5	4.53	300	282.8	5.73	299.5	285.5	4.67	300.1	285.6	4.83	299.7	279	6.91	299.9	275.9	8.00
27	299.8	276.7	7.71	299.7	278.9	6.94	300.2	270	10.06	300.5	287	4.49	300	269.7	10.10	300.2	282	6.06
28	300.2	299.4	0.27	300.7	300	0.23	300	299.5	0.17	300	299	0.33	299.8	298.8	0.33	300.1	297.2	0.97
30	299.5	280.3	6.41	299.7	278.1	7.21	299.8	281.5	6.10	300.3	283.4	5.63	299.5	264.7	11.62	300.2	277.3	7.63
slag	300.1	297.9	0.73	299.4	296.9	0.84	299.6	299	0.20	300.5	300.1	0.13	300.2	299.8	0.13	300	299.5	0.17

Figure 13. Results of Brine Freeze-Thaw Tests Using Different Deicing Salt Sources.



Results of the brine freeze-thaw test using six different deicing salt sources are provided in Table 21. These tests were conducted on the 4.75mm size aggregates. Based on these results a comparison of the weight loss in the different deicing salt solution were plotted against the loss in the standard NaCl brine solution used by INDOT, yielding Figure 13. As can be observed in Figure 13, there is no obvious indication that weight loss for one salt solution is consistently greater than that for another solution. From this it is concluded there is little or no difference in the effects between the different deicing salts on the freeze-thaw soundness. Also, none of them is significantly more destructive to aggregates than is the standard NaCl solution.

However, some sources had freeze-thaw losses in the different salt solutions which were considerably greater than the weight loss in the standard NaCl solution, as in samples D-09, 22, and 27. Specifically, in the case of sample D-22, the weight losses in salts A and D were 27.03% and 21.50 %, respectively. Because the weighed average in Table 4 can be somewhat greater than the value for the 4.75 mm results, these samples could have weighted average values above 30% loss. In Table 4, sample D22 has a value of 29.93% loss. With values near or above the 30% brine freeze-thaw loss this indicates the possibility of an aggregate deterioration problem. Therefore, the freeze-thaw tests in the standard NaCl brine solution may not represent the worst case situation for in-service conditions relative to different deicing salts.

In addition, the iron and other minor constituents in slag were considered relative to detrimental effects on aggregates. Table 22 shows the results of the freeze-thaw test on the slag-dolomite mixture in the different deicing salts. In general the weight losses of mixtures are reduced as compared to the freeze-thaw loss of aggregate alone (Table 21). This reduction ranges from about 2% to 100%, with an average reduction of about 50%. When considering that the mixture consists of equal amounts of aggregate and slag, the 50% reduction shows that the iron and other material in the slag contribute no detrimental effects in the freezing and thawing process.

Table 22. Results of Brine Freeze-Thaw Testing with 50% Slag and 50% Aggregate Using Different Deicing Salts.

Sample	SALT A			SALT B			SALT C			Salt D			Salt E			Salt F		
I.D.	Before T	After T	W.L	Before T	After T	W.L	Before T	After T	W.L	Before T	After T	W.L	Before T	After T	W.L	Before T	After T	W.L
5	299.6	285.6	4.67	300.6	291.6	2.99	300.2	286.4	4.60	300.3	285.1	5.06	299.7	289.2	3.50	300.2	296.7	1.17
7	300	288.6	3.80	300	294	2.00	299.8	292.4	2.47	300.1	295.7	1.47	300.3	292.5	2.60	300.4	295	1.80
9	300.1	295.6	1.50	300.3	294.9	1.80	300.5	298.7	0.60	300.3	297.9	0.80	299.8	298.8	0.33	300	297.3	0.90
19	300.7	291.6	3.03	299.9	288.2	3.90	300.1	276.2	7.96	299.5	290.8	2.90	300.3	291	3.10	299.9	288.6	3.77
20	300.6	280.7	6.62	299.9	286.9	4.33	300.4	289.7	3.56	300.7	285	5.22	300.6	288.9	3.89	300	289.7	3.43
21	299.9	285.2	4.90	299.9	292.5	2.47	300.7	288.4	4.09	300	278.4	7.20	300	299.4	0.20	300.1	294.5	1.87
22	300.2	278.6	7.20	299.9	280.1	6.60	300.4	277.9	7.49	300.4	277.1	7.76	299.8	289.5	3.44	300.2	284.1	5.36
23	300.5	295.4	1.70	300.2	296.5	1.23	300.2	298.4	0.60	300.1	292.1	2.67	300.4	298.7	0.57	300	290.7	3.10
26	300	288.7	3.77	299.9	294.2	1.90	300.1	293.5	2.20	300.3	281.8	6.16	300.2	297.7	0.83	300.1	290.4	3.23
27	299.8	289.2	3.54	300.2	292.4	2.60	299.8	287.7	4.04	299.5	279.9	6.54	300.4	293.9	2.16	300.4	288.4	3.99
28	299.8	299.7	0.03	300.3	300.3	0.00	299.6	299.1	0.17	300.3	299.9	0.13	299.9	299.3	0.20	300.1	299.9	0.07
30	299.9	289.8	3.37	300	289.3	3.57	300	286.3	4.57	300.3	292.5	2.60	300.5	284.9	5.19	299.7	287.2	4.17

6) Etching, Electron Microprobe Studies and Pavement Core Descriptions

(1) Etching Rock Thin Sections

The aggregate thin sections were etched using dilute HCl to determine the content and distribution of clay minerals in the carbonate samples. Inspection of the etched samples indicated that in most cases, the clay was concentrated around the perimeter of the carbonate grains. In a few situations, thin layers of clay minerals were found, producing stripes of lamina, which were visible to the unaided eye.

(2) Microprobe Studies

The samples were divided into three categories based on the relative size of their carbonate grains. Four typical representatives were selected for analysis using the electron microprobe in order to obtain a quantitative and objective evaluation of the clay distribution. A fine grained sample, one large grained sample and two medium grained samples were chosen as typical representatives of the aggregate sources. A range of clay content consisting of high, medium and low was also selected. Samples were prepared by polishing, followed by coating with carbon. The areas of the thin section examined using the electron microprobe varied from 0.5 x 0.3 mm, to 1x1 mm with most sample areas of 0.5 x 0.5 mm.

For the first sample run, the probe was set to distinguish calcium, magnesium, aluminum and iron. For subsequent samples the probe evaluated these same constituents except that silicon was substituted for iron. This change was made for two reasons. First, essentially no iron was found, and secondly a search for the combination of aluminum and silicon made it easier to establish the presence of clay minerals, rather than using aluminum alone. Results from the first sample showed that the sample consisted mostly of dolomite, with small amounts of clay. The clays were found around the perimeter of the grains verifying what was suspected by the etching study. Table 23 presents the results of the microprobe analysis. Microprobe images are provided on the following pages. As can be observed, the aggregates consist almost entirely of dolomite. The image for silicon shows two different shades of particles. On the original image, these two shades appear as two degrees of gray. Using backscatter imaging, it was determined that the brighter pixels consist of quartz, and the duller pixels are illite. This result was confirmed in the image showing aluminum content of the samples.

Table 23. Mineral Content of Thin Sections, Based on Electron Microprobe Analysis.

Sample #	Area Probed	% Quartz	% Illite	% Dolomite
D-005	0.5x0.5 mm	4.4	10-13.6	82.0-85.6
D-008A	1x mm	0	0	100
D-008B	0.5x0.5 mm	<0.2	0.2	99.6
D-010	1x1 mm	0.5	0.1	99.4
D-013	1x1 mm	2	2.4	95.6
D-018	0.5x0.3 mm	1.3	0.3	99.2
D-018	0.5x0.5 mm	<0.1	<0.1	99.9
D-019	0.5x0.3 mm	<0.2	2.8	97.1

Percentages of the different elements were obtained by calculating the number of pixels for each material. This method is somewhat inexact as it relies on a subjective decision regarding the placement of the boundary between the brighter quartz and the somewhat duller illite, or how to distinguish between aluminum and signal noise in the image. However, for the purpose of this study, the precision seems to be adequate. Photographs of the microprobe images are provided in Appendix E.

The images provided in this report were enhanced using Adobe Photo Shop software to add color and to increase the contrast. A difference in shading between gray areas represents different amounts of the elements considered.

(3) Description of Pavement Cores

Deterioration of three bituminous pavement surfaces on Indiana highways was observed following the first year that deicing salts were applied. Pavement cores from these three Indiana highways were examined using a binocular petro-graphic microscope with a magnification of 15x. The cores were obtained from I-80, I-65, and St Rt 67. General descriptions of each core are included in this report. The general description of the cores from each highway are provided. Table 20 is a summary of observed information. Photo micrographs of the cores are provided in Appendix F with an accompanying description of them.

I-65

Evaluation of pavement cores from I-65 was conducted previously by Kreich, 1991, with assistance from the Portland Cement Association. No evidence of salt deterioration of the aggregates was noted. In the current study, the aggregate size in these cores varied greatly from sand to 32x19 mm (1.25x0.75 in.). Sorting varied greatly. Cores showed an average degree of sorting, with a wide range of aggregates from sand size to about 19 mm (0.75 in.). Three of the cores showed poor mixing of aggregate sizes, one area contained large pieces of fractured dolomite. The average percent of slag observed on the bottom of the cores was estimated at about 50%.

The top of the cores indicated that dolomite pieces were more fractured or contain more worn grooves than the slag. Fractures in the bituminous matrix are more common in these samples, and they are frequently found to extend around one side of the aggregate. Some fracturing, however, should be attributed to the coring process and extraction of the core from the drill hole. It is difficult to determine how much of the fracturing in the bituminous material was caused by coring and extraction.

Most of the cores showed small to medium shallow pitting in the bituminous material. Many of these open pits were filled partially with sediment. A small number of pits on the tops of cores appeared to be deep enough to represent locations where small aggregate pieces were ejected.

Table 24. Petrographic Description of Pavement Cores

Core #	Lgst Aggr.	Approx % slag	Comments
I-65	mm (in.)		
SB1	25x13 (1 x 1/2)	40 poor mixing	Dolomite cracked, more cracks in bituminous material. Much of cracking is around the grains.
SB2	25x13 (1 x 1/2)	40 poor mixing	Core much like SB1. Several deep pits observed. Stripping possible.
SB3	25x6 (1 x 1/4)	50 well mixed	Small to medium pitting observed.
SB4	19x13 (3/4 x 1/2)	50 well mixed	Small to medium pitting observed.
SB5	13x13 (1/2 x 1/2)	60 well mixed	Small to medium pitting observed.
NB1	13x13 (1/2 x 1/2)	60	Small to medium pitting observed.
NB2	16x13 (5/8 x 1/2)	60 well mixed	Large cracks in bituminous material, probably artifacts from aggregate removal.
NB3	13x13 (1/2 x 1/2)	65 fair mix	Wide shallow pit located in center of core. Bituminous material shows more cracking than aggregates. Dolomite more likely to be fractured than slag. Grooves in dolomite common on all cores.
NB4	19x10 (3/4 x 3/8)	65 fair mix	Small shallow pits.
NB5	32x19 (1-1/4 x 3/4)	15 very poor mix	Many fractured dolomites observed on non-worn surface. Core has large, shallow pits. Fractured dolomites common on worn surface as is cracked bituminous material.
I-80			
WB1	19x13 (3/4 x 1/2)	90 fair mix	Slag looks worn - More pits than I-65 probably due to natural porosity of the slag-cracking of bituminous and some slag - deep grooves worn around the grains. Some of the pits may be ejected grain sites.
WB2	25x13 (1x1/2)	85, cluster of dolomite	Fractured aggregates - many pits - rocks and slag worn smooth - One large shallow pit may be an ejection site.
WB3	16x13 (5/8 x 1/2)	80	Core looks like WB2
WB4	19x13 (3/4 x 1/2)	85	Grains are larger on average than WB1, WB2, and WB3 - some grooves worn around larger rocks - fractured aggregate - not uncommon to see aggregates with horizontal, parallel fracture pattern.
WB5	13x13 (1/2 x 1/2)	80	Large cracks in one area correspond to rust stain along the edge of one slag rock w/very irregular surface

Table 24 cont.

	mm(in.)		
EB1	19x13 (3/4 x 1/2)	90	Surface less irregular than WB1 through WB5
EB2	25x13 (1 x 1/2)	90 fair mix	Surface irregularities apparently caused by slag porosity. More fractured aggregates present, w/fewer cracks in bituminous material.
EB3	25x13 (1 x 1/2)	90 well mixed	Surface appears grainy - as if dissolved - Fractured bituminous aggregate and slag observed - large shallow pits are common.
EB4	19x13 (3/4 x 1/2)	90 well mixed	One pit shows top of aggregate stripped away leaving bottom portion in the large pit.
EB5	25x13 (1 x 1/2)	well mixed	Many fractured aggregates present - some bituminous material cracked - surface not too irregular - two pits located close in proximity appear to contain parts of shale aggregate.
St.Rd 67			
NB1	19x16 (3/4 x 5/8)	60 well mixed	Very uneven surface with many shallow pits. Cracks in bituminous material common. One dolomite is deeply grooved and may be missing top portion.
NB2	19x13 (3/4 x 1/2)	55 fair mixing	Many dolomites on the non-worn side are fractured. Worn side shows shattered dolomites, very worn slag and cracks in the bituminous material.
NB3	13x6 (1/2 x 1/4)	60 well mixed	Much like NB1 and NB2.
NB4	25x13 (1 x 1/2)	50 fair mix	Very irregular surface.
NB5	19x16 (3/4 x 5/8)	60 fair mix	As in NB4, core has very irregular surface. Deeply grooved, partially missing aggregate is observed which appears to be dolomite.
SB1	13x6 (1/2 x 1/4)	60 well mixed	Much like north bound cores. Bituminous material is fractured and worn around aggregates. Rocks show much fracturing, however, surface less irregular than north bound cores.
SB2	19x6 (3/4 x 1/4)	75 well mixed	Pit contains fractured slag with loose pieces.
SB3	25x19 (1 x 3/4)	60	Similar to other cores from St.Rd 67.
SB4	13x13 (1/2 x 1/2)	60	Worn surface, very uneven.
SB5	25x13 (1 x 1/2)	30	Worn surface very uneven. Large pit in the center is fractured dolomite.

I-80

These cores differ from the I-65 cores in that they contain a much larger percentage of slag. Examination of the bottom of the cores, indicated that as much as 80 to 90% of the mix was slag. Aggregate sizes ranged from sand sized to 25x13mm (1x.05 in.), and the mixing in many of the cores showed larger aggregates on one side of the core with an area of smaller ones along the other side, or in a wide strip down the middle.

The tops of these cores showed a very uneven surface. Some of this is due to the high percentage of slag and its natural porosity, but there are some pits in the surface where it is obvious that the original aggregate was stripped from its position, leaving part of an aggregate still in place. Many aggregates on the surface of the core look worn and polished. It is common to see fractured aggregate pieces, but fractures in the slag as well as fractures in the bituminous material were observed in these cores. The fractures in the bituminous material commonly extend around aggregates, but in some locations they proceed through the aggregates which are also fractured.

St.Rt 67

Most of the cores from SR67 are characterized by a very uneven surface with many wide, shallow pits. In a few instances, the pits contain a piece of aggregate whose top portion has been ejected. Many of the pits, however, are likely related to the porosity of the slag material. The percentage of slag was estimated at about 60%.

Size of the aggregates also ranged from sand size to 25x13mm (1x0.5 in.). The bituminous material shows many fractures, as do many of the dolomite pieces. It is also common to find deep grooves carved into the dolomite, and the slag commonly shows considerable weathering. In regard to these characteristics, cores from SR67 are very much like those from I-80.

VI. Summary and Conclusions

1. Discussion

As a fore-runner to this research, a detailed literature review was conducted on the effects of deicing salts and their trace minerals on aggregates. A 45-page report (Pittenger and West, 1995) is included here as Appendix A. The report focused on two subjects: 1) deicing salt and aggregate deterioration of portland cement concrete and 2) moisture damage to asphaltic pavements.

Based on the literature review it was concluded that although an abundance of research has been conducted on the deterioration of aggregates in portland cement pavements, much less work has been accomplished on aggregate deterioration in bituminous pavements. A comparison of the behavior of these two pavement types may prove helpful in evaluating bituminous pavement deterioration. It was also found that numerous factors are involved in moisture damage (stripping) of asphalt pavements and it is difficult to isolate the impact of specific factors in asphalt pavement deterioration.

Eighteen dolomite aggregate sources, included in a previous study directed by Dr. Terry West (Bruner, Choi and West, 1995), were used as basic information for this research. Data on the INDOT approved sources included petrographic analysis, mineral composition based on x-ray diffraction, insoluble residue content, elemental magnesium content and standard laboratory test results. These included absorption, specific gravity, sulfate soundness loss and Los Angeles abrasion loss. During the current research, brine freeze thaw testing was accomplished on these eighteen dolomite sources. Subsequently, statistical analysis was performed on the petrographic factors and the physical properties. Results are presented in Table 7. A regression analysis shows that 1) insoluble residue %, greater than #200 sieve, 2) insoluble residue %, less than #200 sieve and 3) sodium sulfate loss have the highest correlation with the brine freeze thaw loss.

Magnesium content also shows a higher correlation with brine freeze thaw loss, a negative value, indicating that increasing magnesium content yields a decreasing brine freeze-thaw loss. From this evaluation it is concluded that dolomite aggregates with a low insoluble residue content have lower sulfate soundness losses and lower brine freeze-thaw losses. Clay content was also determined and it also has an indirect correlation with brine freeze thaw loss.

Ten more dolomite aggregate sources, approved by INDOT were added to the data base. Brine freeze thaw testing was accomplished on the 28 samples using the coarse aggregate size, retained initially on the 9.5 mm and 4.75 mm sieves. A weighted average was obtained for these tests and is presented in Table 4. Values ranged from 0.61 to 27.26% loss.

INDOT specifications limit the sodium sulfate loss to 12% for class A aggregates and INDOT has recommended a 30% limit for the brine freeze thaw test. Aggregate 13 (Table 7) has a brine freeze thaw loss of 11.93 and a sodium sulfate loss of 13.18, whereas Aggregate 22 (Table 4) has a brine freeze thaw loss of 27.26%. Both of these sources appear to be of marginal quality based on this information. Aggregate 13 (Table 7) also has a low dolomite content and a high insoluble residue content (5.56%).

Brine freeze thaw testing was conducted on a finer portion of the #11 gradation for dolomite aggregate samples. The purpose was to determine the effect of particle size on the test. Six of the 28 samples were selected for analysis. The average loss ranges from 3.88 to 13.72, all well below the maximum allowable value of 30%. Recall that for the fine aggregate, after completion of brine freeze-thaw testing, samples are sieved over the original retaining sieve, rather than using a somewhat smaller size, as is the practice for coarse aggregate testing. This situation suggests that brine freeze-thaw losses for the fine aggregate portion would be greater than that for the coarse aggregate portion, all other things being equal. A comparison of the last column in Table 4 for the six aggregates in question (7,9,10,20,21 and 29) with the last column in Table 5 shows that for 5 out of 6, the brine freeze thaw loss is greater for the fine aggregate portions. Only for D21 is this not the case.

For nine samples (22-30) of the coarse aggregate fraction, the brine freeze-thaw loss was calculated in a different manner. In Table 6 the losses are tabulated when sieved over the original retaining sieve to determine freeze-thaw loss, rather than a somewhat smaller size being used. A comparison between Tables 4 and 6 for these nine samples shows that a significant increase in % loss occurs when the larger sieve size is used to determine the loss. This result was to be expected and comes at no surprise.

These results indicate that the coarse aggregate, brine freeze thaw test, determined by standard sieves which are somewhat smaller than the original retaining size, is the preferred method for evaluating the percent loss in the test. Also note that all aggregates tested lie below the maximum allowable loss of 30%.

Mercury intrusion porosimetry studies were conducted on the dolomite aggregates in two phases. In the first phase 20 aggregate sources were evaluated (D-1 to D-21 except for D-14). Two or three representative pieces were selected for each source, yielding 43 samples. Pore size distributions were obtained on each sample. The intruded pore volume and median pore diameter were determined for each pore size distribution. The EDF (Expected Durability Factor) according to Kaneuji (1978) was calculated for each distribution based on the equation:

$$EDF = (0.579)/DV + 6.12\ (MD) + 3.04$$

Where PV = pore volume and MD = median pore diameter. The results on this analysis are presented in Table 13. A significant difference in EDF values can be observed for quite a few sample groups for example 1,2,3,4,5,11,15,17,19, 20 and 21. Correlation of EDF versus brine freeze-thaw loss shown in Figure 11 indicates a low R value and poor correlation between the two parameters. Seven sources with the widest range in EDF values were excluded (all had average

EDF values above 100) and the correlation analysis was run again (Figure 12). A major improvement occurred as a 0.73 significance level of P=1.1% was obtained rather than the 0.23 significance and P=34.8% in Figure 11.

Hockette (1987) raised a similar concern about the EDF value and pore size distribution curves based on mercury intrusion porosimetry. Significantly different values were obtained for the same aggregate source and the accuracy of the method had been questioned.

In the second phase of this work, one aggregate source was selected for study that had a range of textures associated with several ledges of the quarry. Six different texture classes were selected from four distinct ledges in the quarry. These are designated in Table 14 (102, 2-3 fine, 2-3 dark, 5, 6A and 6B). Multiple samples of the same texture class were tested using mercury intrusion porosimetry. Typically six to nine replicate samples were run, but in one case, only three were accomplished. A much smaller range in EDF value, mean and standard deviation were obtained (Table 18) when the aggregates were subdivided according to texture type..

Ledge 5 and 6 of this aggregate source (D-26) was used in SR62 near Charlestown in the surface overlay pavement. Pitting of the coarse aggregate of up to 50% of the pieces had occurred on the surface of the pavement. EDF values for Ledges 5 and 6 equal 110.2, well above the value of 20, which indicates a rapid aggregate deterioration level. Calculations indicate that only 10.1% of the sample has an EDF value of 20 or less. Results show that both ledges 102-3 and 5-603 have a much higher quality than aggregates known to undergo rapid deterioration (EDF $\leq$ 20) during freezing and thawing.

Samples from six sources of NaCl deicing salts were obtained from INDOT district offices. Chemical analyses of these salt samples were performed at Purdue University. There is no obvious indication that chemical constituents are present which would cause an accelerated attack of aggregate particles in a bituminous surface courses.

Brine freeze thaw testing was conducted on 12 dolomite sources plus one slag using 4.75 mm size aggregates. No freeze thaw losses greater than 30 were obtained and there is no obvious indication that weight loss for one salt solution is consistently greater than that for another solution. Typically these salts showed less loss that did the standard NaCl solution used for brine freeze-thaw testing. Therefore, a deicing salt used in service is not expected to yield a higher loss than that determined during laboratory testing.

Samples containing 50% slag and 50% dolomite were subjected to brine freeze-thaw testing. The iron and other constituents in the slag did not cause accelerated deterioration to the dolomite, as low losses were obtained. No detrimental affect on brine-freeze thaw loss from the slag was indicated. Loss for the 100% slag sample (Table 21) was less than 1%, indicating that slag is not subject to significant freeze-thaw loss. Also, it does not induce freeze-thaw failure in the adjacent dolomite fraction.

Several companion studies were used to examine the manner in which clays are associated with the carbonate aggregates. Aggregate thin sections were etched using dilute HCl, removing the carbonate minerals, leaving the insoluble particles behind. Inspection of the etched thin sections indicated that the clays had been concentrated around the perimeter of the carbonate grains. In a few instances, thin layers of clay minerals produced lamina that could be discerned by the naked eye.

Electron microprobe studies were conducted to examine further the distribution of the clay minerals in the carbonate aggregates. Clays contain both silicon and aluminum whereas quartz has only silicon. The presence of clay and quartz can be mapped using the electron microprobe by identifying the areas containing silicon and aluminum. Sample D-5 showed a significantly clay content (10-13.6%). Table 2 shows that the insoluble residue content equals 9.8%. By contrast D-

18 has a low clay content indicated in Table 19 and an insoluble residue of 2.15% in Table 2. These results are consistent.

Pavement cores from I-65, I-80 and SR67 were examined petrographically. Cracked aggregate particles and pitting were noted but no major deterioration was found. Aggregate deterioration is not obvious. There is no evidence that pavement deterioration was caused by aggregate weathering or degradation. Petrographic descriptions of the pavement cores are presented in Table 20.

2. Conclusions

1) A detailed literature review (Pittenger and West, 1995 - see Appendix A) identified extensive research performed on aggregate deterioration problems for portland cement concrete pavements. Much less information is available on aggregate deterioration of bituminous pavements. For moisture damage (stripping) of bituminous pavements it is difficult to isolate the factors causing failure.

2) Compilation of data on 18 dolomite sources from a previous study compared to brine freeze thaw results from current research showed insoluble residue content (+200 and -200 sieve sizes) to correlate best with brine freeze-thaw loss. Also, sodium sulfate loss is directly related and elemental Mg content appears to be inversely related to brine freeze-thaw loss.

3) Ten additional INDOT approved dolomite sources were added and brine freeze-thaw testing was performed on the coarse size gradations. Values ranged from 0.61 to 27.26% loss. 30% loss is considered by INDOT as maximum allowable loss for Class A stone.

4) Brine freeze thaw loss was conducted on the finer portion of the No. 11 aggregates. Six sources were included. Average losses ranged from 3.88 to 13.72%, all well below 30% loss. Loss was determined by sieving over the original retaining sieve rather than a somewhat smaller size, as specified by ITM 209-94 and Procedure A, AASHTO, T103, used for the

coarse size tests. Results for the finer portion should therefore show greater losses than for the coarser portion, all other things being equal. Therefore, the fine portion of the #11 gradation had a comparable brine freeze thaw loss to that of the coarse fraction. Manufactured fines from some of the coarse aggregates could exceed 12%.

5) Mercury intrusion porosimetry studies conducted on a variety of aggregates with different textures (the 18 original samples) yielded a considerable range of pore size distributions and EDF values. This raised some doubt concerning the usefulness of porosimetry studies to identify poor quality aggregates.

6) Additional porosimetry studies were performed, (Phase II) this time on aggregates with similar textures and lithologies (D26 and D27). EDF values were much more consistent, yielding a narrow range of variation. This indicates that pore size distributions are directly related to variations in texture, so that rock texture must be considered when analyzing porosimetry data.

7) Coarse aggregate in the bituminous surface course on SR62 near Charlestown was supplied from source D-27. Numerous pits occurred in the pavement where about 50% of the coarse aggregate pieces were missing. EDF values for the aggregate pieces proved to be well above a minimum value of 20, the point where on-set of rapid freeze thaw deterioration occurs. It is concluded that the pitting was caused by some mechanism other than by freeze-thaw damage.

8) Six sources of NaCl deicing salts used by INDOT were used to perform additional brine freeze thaw tests. Chemical analyses of the salts at Purdue University showed no obvious presence of constituents that would cause accelerated attack on aggregate particles in bituminous surface courses.

9) Brine freeze-thaw tests on 12 dolomite sources and one slag were performed using the six NaCl deicing salts plus the standard laboratory NaCl salt. No losses greater than 30% were

obtained. The six salts typically did not yield losses greater than that for the standard laboratory salt. No salt was consistently more damaging than any other.

10) Samples containing 50% slag and 50% dolomite particles were tested using the six deicing salts. The iron and other constituents in the slag did not induce increased deterioration in the dolomite. No significant freeze thaw damage to the slag was noted. The slag is not subject to significant deterioration in brine freeze-thaw testing and does not induce deterioration of the dolomite during the process.

11) HCl etching of thin sections of the dolomite samples was used to examine clay mineral distribution within the carbonate grains. Clays were found to be concentrated around the perimeter of the carbonate grains. In a few cases, thin layers of clay could be discerned.

12) Electron microprobe studies showed the distribution and percentage of clays, quartz and carbonate mineral. Contrasting samples with higher clay contents, D-5 with 9.8% insoluble residue content, and D-18 with a 2.15% insoluble residue were examined. Microprobe results are comparable to the insoluble residue values and other lab test data.

13) Pavement cores from I-65, I-80 and SR67 were examined petrographically. Cracking and pitting of aggregates and cracking of the bituminous binder were noted. No evidence was found that pavement deterioration was caused by aggregate weathering or degradation.

14) Based on this study it is concluded that the brine freeze thaw test is a good measure of freeze-thaw resistance for aggregates in bituminous surface courses. It is also concluded that no significant evidence was found to indicate that early deterioration of the pavements studied was caused by salt attack of the aggregates in the bituminous surface courses.

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VII. Recommendations

1. Continue to collect laboratory data on approved aggregate sources so that a more extensive data base can be obtained. Freeze thaw loss, brine freeze thaw loss, sulfate soundness loss, absorption, specific gravity, Los Angeles abrasion loss, elemental magnesium and acid insoluble residue including size distributions are particularly important. Mercury intrusion pore size distribution and a petrographic description are also of value.
2. Examine further the acceptance criteria for aggregates based on the brine freeze thaw test. A maximum allowable loss of 30% may be too high based on aggregates included in this study. In many instances (8 of 18, Table 7) the sodium sulfate loss exceeds that of the brine freeze thaw loss and a 12% loss is the maximum allowable value for sodium sulfate loss. The original data used to select the 30% loss criteria should be reviewed in light of the findings of this research.
3. Apply these data to other studies of aggregate quality, such as friction resistance of pavement surfaces, D-cracking of concrete pavements and overall evaluation of pavement quality.

VIII. Implementation Suggestions

Follow up on Recommendations provided above. Construct a data base containing the laboratory test data for the approved aggregate sources. This information should prove helpful when other pavement failures occur in the future.

Appendix A

Effects of Salt and Trace Minerals on Aggregates

Part 1
Deicing Salt and Aggregate Deterioration in Portland Cement Concrete

Part II
Moisture Damage to Asphalt Pavement

by Robert Pittenger and Terry R. West

Purdue University
Dept. Of Earth and Atmospheric Sciences
West Lafayette, Indiana 47906

for Indiana Department of Transportation
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Part I
Deicing Salt and Aggregate Deterioration in Portland Cement Concrete

1. INTRODUCTION

At the request of the Indiana Department of Transportation (INDOT), the Purdue University Department of Earth and Atmospheric Sciences has conducted a literature review on the effects of deicing salt and trace minerals on aggregates.

This investigation is prompted by early deterioration of bituminous pavements in Indiana, made with slag, slag-dolomite, and dolomite aggregates, particularly on I-65 near Indianapolis where rapid deterioration was observed following the first application of deicing salt.

Only a limited amount of research on degradation of aggregate by deicing salts has been conducted. The research that does exist frequently focuses on different aspects and causes of aggregate deterioration, or different ways to predict aggregate behavior. There are several variables, some interdependent, that affect how aggregates respond under freeze-thaw conditions with deicing salts. These variables include pore size distribution, amount and type of argillaceous material, and aggregate chemistry and composition.

Articles reviewed for this study are listed in Table 1 based on the topics discussed. Because much of the research is recent and limited in scope, some conclusions have yet to be corroborated by additional studies. This is especially important in areas of aggregate research because a study is frequently limited to aggregate from a specific area, or of a specific rock type, and the information can not be directly applied to other aggregates or regions. Much of the research reviewed here focused both on laboratory investigations as well as case studies.

The vast majority of research relating to pavement degradation in the presence of deicing salt focuses on the deterioration of portland cement concrete (PCC) and the corrosion of steel reinforcement structures. This research is motivated in part by the economics of salt's deterioration and corrosion of bridge decks, which are costly to replace and are more susceptible to freezing. Bridge maintenance can also be more disruptive to traffic flow.

A second area of significant research, indirectly related to this study is the development of alternative deicing mixes that are not as corrosive or destructive as the commonly used NaCl and CaCl.

Determination of the causes of aggregate deterioration in the presence of deicing salt is an important area of research. However the development of laboratory test procedures to determine sample response to freeze-thaw in the presence of deicing salt, and the correlation of the test to field performance, has also been investigated. Logically, the best method to predict aggregate performance would be a test that simulates the environment to which the aggregate is exposed. Frequently mentioned is freeze-thaw testing in conjunction with immersion in a salt solution. Development of such a reliable testing procedure has the added benefit that it is independent of the specific cause of aggregate deterioration.

Most research focuses on the determination of causes for aggregate deterioration, with a brief consideration of test development specific to deicing salt performance. Therefore no detailed correlation of the test with aggregate performance is provided. The most detailed correlation of aggregate field performance and laboratory tests is provided by Shakoor (1982), whose results are summarized in Table 2.

Of final interest, but not a topic of extensive research is the effect of trace sulfate compounds in the deicing salt on the pavement and aggregate. Only two references discussed this problem, and both concluded that sulfate is a significant contributing factor to the degradation of pavement (either aggregate or PCC) in the presence of deicing salt.

2. EFFECTS OF DEICING SALT ON CONCRETE

Although not the primary focus of this literature review, a brief consideration of the current research on the effect of deicing salt on PCC is warranted. This provides background to a discussion of the effects of salt on aggregate and bituminous pavements.

A major detrimental effect that deicing salt has on concrete structures is the corrosion of steel reinforcement. As steel corrodes, it expands in volume up to six times causing internal stresses that exceed the strength of the concrete (Campbell and Detwiler, 1993). To minimize steel corrosion, much attention has been given to reducing the chloride permeability of concrete. Low permeability concrete minimizes migration of the chloride ions to the steel. Research in this area focuses on special mixtures of concrete that may include fly-ash, or silica fume, or different methods of curing. Holland (1987) determined that the use of silica fume helped to reduce steel corrosion because the silica fume plugged internal pores, reducing chloride permeability.

In many cases of damage to concrete caused by steel corrosion and expansion, it was determined that the steel was too close to the surface of the concrete. It has been speculated by some that the cause of corrosion was too thin of a layer of concrete over the steel reinforcements. Miller, Krouskop, Minkarah, and Bodocsi (1993) showed that chloride was able to penetrate and corrode to four inches, deeper than was previously thought. Small fractures and cracks also provided chloride migration pathways to the steel reinforcements.

Although more often a problem with matrix rather than aggregate, scaling of PCC matrix is another common result of deicing salt. Water-cement ratio, amount of air-entrainment, air void spacing factor, and addition of admixtures such as fly-ash or silica fume are all factors that affect the

Table 1. Topics Referenced in Articles

Author	Date	Rating	Aggregate	Concrete	Bituminous	Lab Test	Case Study	Causes of Det.	Proposed Tests	Absorption	Insol. Residue	Pore Size Dist	Osmotic Press.	Porosity	Argillaceous Mat.	Mineral Formation	Expansion	x-ray diff.	de-icing additives	alt. de-icers	Trace minerals	special concrete	conc. cl. perm.	steel corrosion	fly-ash
Marks and Dubberke	1995	3	X	X		X	X	X				X				X	X								X
Dubberke and Marks	1987	3	X	X			X		X									X							
Hudec	1987	3	X						X	X		X	X				X								
Shakoor	1982	3	X	X	X	X	X	X	X		X		X												
Hoebeda and Jacobson	1981	3	X			X			X					X											
Hudec	1978a	3	X			X		X		X		X		X											
Hudec	1978b	3	X			X		X		X		X		X											
Hudec and Rigby	1976	3	X			X		X		X															
Kallas	1963	3			X		X																		
Pitt and Carrazzo	1992	2		X		X			X					X											X
Crumpton et al.	1989	2		X		X		X																	
Pitt et al.	1988	2		X																	X				X
Dubberke and Marks	1985	2	X	X			X	X								X									
Salcedo	1984	2	X		X	X	X	X	X			X			X										
Kaneuji, Winslow, and Dolch	1980	2	X	X		X			X			X													
Dunn and Hudec	1972	2	X			X				X															
Bilodeau et al.	1994	1		X																			X		X
Triano and Frantz	1992	1		X																					X
Zimbleman	1989	1		X												X	X								
Anon	1987	1		X																					
Gillott	1978	1	X	X		X															X				
Aitcin and Laplante	1990	0		X			X															X	X		
Anon	1989b	0	X		X																				
Anon	1989a	0		X	X																	X			
Anon	1987	0			X		X												X						
Callahan	1989	0		X		X																		X	
Campbell and Detwiler	1993	0		X			X															X			
Dagher and Kulendran	1992	0		X		X																		X	
Fansom and Cohen	1991	0		X			X																	X	
Foy, Pigeon and Banthia	1988	0		X		X																X	X		
Gjorv, Tan, and Monteiro	1994	0		X																		X	X		
Grieb, Werner, and Woolf	1962	0		X		X																X	X		
Holland	1987	0		X																		X	X	X	
Lessard et al.	1982	0		X			X															X			
Miller et al.	1993	0		X			X																	X	
Mogawer, Stuart, and Lee	1989	0			X		X												X						
Nadezhdin et al.	1989	0		X		X																		X	
O'Connor and Saiidi	1993	0		X		X	X															X			
Okkenhaug and Gjorv	1992	0		X			X															X			
Palmer	1987	0																		X					
Pigeon, Aitcin, and Laplante	1987	0		X			X															X			
Reagan	1992	0		X																					
Sarkar and Aitcin	1991	0		X			X																X	X	
Virmani	1991	0		X			X																	X	
Whiting and Dziedzic	1989	0		X			X															X	X		

Ratings
3 Highly relevant to proposed study
2 Portions are relevant to the proposed study
1 Portions may be indirectly related to the proposed study
0 Not relevant to the proposed study

Table 2. Summary of Results of Laboratory Testing for Samples from Shakoor (1982)

Sample No.	%Residue	%F-T loss in water (50 cycles)	%F-T loss in 5% NaCl solution (25 cycles)	%Sodium Sulfate Loss (5 cycles)	%Absorption	%Adsorption
T1*	25.8	46.30	90.66	13.46	6.21	3.00
T2*	26.5				6.24	3.58
T3	20.6	14.07	37.40		1.79	1.54
T4	8.3	3.50	37.30	26.49	0.99	0.76
T5*	29.3	61.01	93.46	32.14	5.28	2.29
T7	10.1	3.68	47.37	29.82	0.83	0.36
T8	17.3					
T10	1.9	1.40	1.39	2.68	0.27	0.16
K1*	19.7	9.03	28.15	14.65	4.92	1.41
K3*	23.7	20.56	54.00	34.52	4.48	2.02
K5*	43.7	40.48	99.67	73.13	4.64	2.25
K7*	43.6	62.85	99.40	90.51	4.82	2.20
K9*	45.5	59.72	100.00	91.77	4.83	2.22
K11	8.5	1.51	3.97	5.23	1.98	0.22
K12	11	3.64	15.42	8.70	3.30	0.62
K13	10.1	5.81	18.11	11.02	3.20	0.72
K14	8.4	4.93	2.74	12.23	2.24	0.30
K15	4.9	9.54	16.89	23.63	1.77	0.18
K16	7.4	20.06	36.92	24.18	2.16	0.30
K17	8			25.59	1.24	0.33
K18	17.3			24.11	1.60	0.28
K18B	13.1					
K19*	29.2	83.63	98.02	28.81	3.88	0.52
K20*	37.7	80.66	99.73	93.27	8.88	1.09
L1	13	2.45	28.24	5.06	1.15	0.35
L2	11.6	3.53	43.23	11.02	0.42	0.17
L3*	22	7.86	45.29	9.70	1.51	0.72
L4	10.5	2.60	49.97	6.42	2.86	0.57
L5	10.3	1.56	36.25	7.10		
L6	10.1	3.77	34.01	7.27	2.36	0.98
L7	8.6	2.97	13.75	5.64	0.72	0.35
L8	2.8	2.54	7.42	5.28		

* Performed poorly in field

scaling of concrete in the presence of deicing salts. Foy, Pigeon, and Banthia (1988) determined the critical air void spacing factor to be 750 um, and also noted that a low water cement ratio was helpful in reducing scaling. However, even at water-cement ratios below 0.3, air entrainment was still considered necessary to protect against deicing salts.

Grieb, Werner, and Woolf (1962) did extensive long term testing of a number of concrete slabs and noted that an air content of 3 percent or greater was necessary to prevent scaling for concrete cast in sand. For concrete cast in metal bases, an air content of 7.5 percent or greater was required to prevent scaling. Increased scaling was also noted with the increase in fly-ash and high water-cement ratios.

Pigeon, Aitcin, and Laplante (1987) studied concrete scaling in sidewalks constructed with silica fume concrete. With the addition of silica fume, a bubble spacing factor of 250 um. rather than 500 um was required. With less that 10% silica fume, adequate air-void systems could be produced with proper techniques.

When the research is reviewed collectively, it becomes apparent that a large number of factors affect the durability of concrete in the presence of deicing salts, even when the aggregate is not included in the study. Increasing silica fume content or fly-ash can reduce chloride permeability, which minimizes corrosion of steel reinforcements. However the addition of silica fume or fly-ash requires the addition of more water or the use of a super plasticizer, and make it more difficult to achieve the proper air-void system. Too much water in the concrete mix can cause scaling in the presence of deicing salts. Addition of a super-plasticizer can make it much more difficult to achieve the correct air void system (Pigeon, Aitcin, and Laplante, 1987).

Other circumstances also contribute to the deterioration such as improper curing, freezing to0 early after placement, and poor subgrade drainage (Grieb, Werner, and Woolf, 1962). Anon. (1987b) provides a concise discussion of deicing salt effects on PCC.

There is less extensive research into the effects of deicing salt related degradation on aggregates in concrete matrix. This is discussed in more detail below. Because most case study reports on the degradation of concrete do not discuss the aggregate, it is unknown whether the aggregate was unaffected by the salt, or whether aggregate deterioration also contributed to the degradation of the pavement. It is probably reasonable to assume that the concrete matrix deteriorated before the aggregate. However, factors such as swelling of the aggregate, or formation of different minerals, may have had an effect on the failure of the matrix.

3. AGGREGATE AND DEICING SALT INTERACTION

Of the sources reviewed, only a few focus specifically on the effects of deicing salts on aggregate. Other information comes from research focusing on the effects of freeze-thaw on aggregate, with the discussion of the additional effects of deicing salts, provided in addition. Most of the current research comes from the Iowa Department of Transportation where Vernon Marks and Wendell Dubberke (who helped develop the Iowa Pore Index Test) have developed some new conclusions about how deicing salt contributes to the degradation of aggregate in PCC pavement. Their research is beneficial because of the availability of field studies where pavement made with the same aggregate has been used on salted roads and unsalted roads. This allows the determination of which aggregate deteriorated specifically because of the salt applications.

3.1 Freeze-Thaw Testing in Combination with a Salt Solution

Some of the most useful research directly into the effects of deicing salt on aggregates and prediction of the performance of aggregates in PCC pavement has come from the Iowa Department of Transportation (IDOT). Previous IDOT research supported the conclusion that pore size distribution is related to the aggregate's durability under freeze-thaw but did not account for deicing salt effects. The critical pore size for aggregate failure from this research was 0.04 um to 0.2 um.

Dubberke and Marks (1985) studied aggregates that performed well on unsalted secondary roads, but poorly on salted primary roads. This research has lead to the development of a testing procedure to predict the performance of the aggregate under conditions of freeze-thaw with the applications of deicing chemicals. The salt treatment for the coarse aggregate is conducted before adding the aggregate to the concrete mix. The treatment consists of *"five cycles of drying in an oven at 230 F for 24 hr, followed by immersion in a 70 F saturated solution of sodium chloride for 24 hr."* Freeze-thaw testing was performed on both concrete made with the salt treated aggregate, and concrete made with an untreated aggregate. The concrete made with the salt treated aggregate deteriorated rapidly under the freeze-thaw testing. This correlated well with field performance of the same aggregate samples. However, because the aggregate was exposed to the salt before use in concrete, the methodology of this research does not provide a good simulation of aggregate preformance in field conditions

This research does provide evidence that the use of freeze-thaw testing in combination with a salt solution may be an acceptable method of determining an aggregate's field performance under freezing conditions with deicing salt applications. However, it was applied only to aggregates used in PCC concrete, and the freeze-thaw test was performed on concrete beams rather than the aggregate themselves. Swedish researchers Hoebeda and Jacobson (1981) determined that ten cycles of freeze-thaw in a 1% NaCl solution were sufficient to predict an aggregate's field performance. However, only an abstract was available for this research so no information is available on the aggregates used for the study. The testing procedure was being proposed by the Swedish researchers and was not the focus of the paper. Dubberke and Marks (1985) used several hundred freeze-thaw cycles in their research. From a study of their data, apparently ten cycles of freeze-thaw may not be sufficient to distinguish between degradation related to salt attack versus not related to salt.

Shakoor (1982) performed freeze-thaw tests on argillaceous limestone and dolomite aggregates in both plain water and a 5 percent NaCl solution. It was determined that the salt solution produced a greater loss when the argillaceous material was of clayey rather than silty composition. Shakoor also showed that highly argillaceous rocks are typically more frost-resistant when the argillaceous material is in laminations, rather than distributed evenly throughout the rock. Shakoor's results are summarized in Table 2. Figure 1 is a graph comparing the results for sodium sulfate soundness tests, freeze-thaw in water, and freeze-thaw in 5% NaCl solution. Note that the L series samples have a much higher freeze-thaw loss in NaCl than in water, even with half the number of freeze-thaw cycles. These rocks came from the Huntington lithofacies of the Wabash Formation, the Louisville Limestone, and the Salamonie Dolomite. They all showed significant deterioration after the first year in both concrete and bituminous pavements.

Dubberke and Marks (1985) continued their research by testing special mixes of PCC cement and additives to the NaCl solution. Use of a Type V sulfate-resistant cement improved the performance of concrete made with the treated aggregate. Adding five percent of "*fine porous limestone with very low magnesium content yielded a greater beneficial effect than did Type V cement.*" The addition of 5 percent porous dolomite fines of high magnesium content "*yielded an adverse effect on the salt-treated durability*" of the concrete beams.

3.2 Physical-Chemical Relationships of Salt Solutions and Aggregates

Many researchers have tried to determine the physical characteristics of aggregates that are susceptible to freeze-thaw. Some have also focused on how these physical characteristics relate to freeze-thaw in the presence of deicing salts. On the basis of the available literature, nearly all of the physical characteristics that make an aggregate susceptible to freeze-thaw, become more important in the presence of deicing salts.

3.2.1 Pore Size Distribution

Pore size distribution has long been considered important in an aggregate's susceptibility to freeze-thaw in pure water. A theory that has long been supported, is that there is a critical range of pore sizes detrimental to aggregate durability. Above a certain critical size, frozen water can easily be expelled from the pore, causing little if any damage. Below a certain critical size, no water enters the pore to freeze.

Critical pore sizes range from 10 angstroms to 10 um depending on source of research. Kaneiji, Winslow, and Dolch (1980) developed an equation allowing the determination of durability from a measurement of pore size distribution. Other variables also affect the critical pore size.

Although the effect of pore size distribution on freeze-thaw without deicing salt has been extensively researched, the additional effect of salt solutions has not been accurately determined. This is probably due in part to lack of research, but also on the inconsistencies in the determination of critical pore sizes, and on other variables affecting the critical pore size. Table 3 shows the range of critical pore sizes reported by different researchers.

Author	**Year**	**Maximum Critical Pore Size**	**Minimum Critical Pore Size**
Dubberke and Marks	1985	0.2 um	0.04 um
Various (Shakoor)	1982	10 um	0.01 um
Salcedo	1984	1 to 10 um	0.045 um
Shakoor	1982	0.01 um	--

Table 3. Critical pore size ranges according to different researchers.

A study was conducted by Antonio Marco Salcedo for the Indiana Department of Transportation (Salcedo, 1984). Salcedo focused on the relationship of pore size distribution to aggregate performance under freeze-thaw. He determined that 45 angstroms to about 1 to 10 um is the critical pore size for degradation from freeze-thaw. However, it was concluded that the critical pore size is also dependent on temperature and the rate of temperature change, which helps to explain the wide range of reported values.

Shakoor (1982) also studied the relationship between pore size, insoluble residue, and durability. It was determined that aggregate with greater than 60% of pores less that 0.1 um, were unsound. Rocks with a high insoluble residue content also typically had a high volume of small pores and the more clayey the residue, the higher the volume of small pores.

3.2.2 Absorption and Insoluble Residue

For the past twenty years, Peter Hudec has published research on various aspects of the freeze-thaw durability of aggregates. He has focused on the effects of water absorption by the aggregates. In Hudec's early work, he noted that water absorption was more destructive for shales, siltstones, and argillaceous carbonate rocks than freezing and thawing (Dunn and Hudec, 1972). Hudec also concluded that the primary reason for aggregate deterioration in the presence of deicing salt, is the increase in absorption and expansion of the aggregate. This expansion can cause aggregates to fail even without freeze-thaw. NaCl in solution causes significantly greater expansion. Therefore, it is the expansion due to the salt solution, and not due to the formation of ice crystals, that is the primary cause of failure (Hudec, 1987). He has also stated that rocks tested under salted conditions can absorb 1.13 times the amount of water when unsalted conditions prevail (Hudec and Rigby, 1976).

Crumpton, Smith, and Jayaprakash (1989) studied the effects of salt on weathering of Kansas limestone aggregate without freeze-thaw. They noted that clays in the limestone were altered by the salt. This alteration resulted in D-cracking in the pavement. Even without freeze-thaw, scaling occurred on concrete samples in the laboratory when soaked in saline solution.

Shakoor (1982) focused on the characteristics of argillaceous dolomite that passed acceptance tests but failed in field performance. Insoluble residue testing along with freeze-thaw testing in both water and in 5% NaCl solution were utilized in the research, as well as other laboratory tests. Testing was done on each of 38 samples collected from quarries, and some results are summarized in Table 2 and Figure 1. Shakoor determined that a larger percentage of clayey material in the rock, as determined by analysis of the residue obtained from insoluble residue test, correlated with the degradation of the aggregate in the salt solution free-thaw test. The relationship determined by Shakoor (1982), between insoluble residue and freeze-thaw in 5% NaCl solution is shown in Figure 1. Silt and clay contents ranging from 20% to 45% proved to be unsound in field use. Aggregates with about 10% argillaceous material were normally acceptable.

Physical appearance of carbonate aggregate can be deceiving. Shakoor (1982) also determined that laminated argillaceous aggregate was more durable than aggregate with the same amount of argillaceous material, when the argillaceous material was evenly distributed throughout the rock. Therefore, aggregate that does not appear argillaceous because of a lack of laminations or stratification , may actually have a significant portion of argillaceous material, and may perform poorly in comparison to laminated argillaceous aggregate.

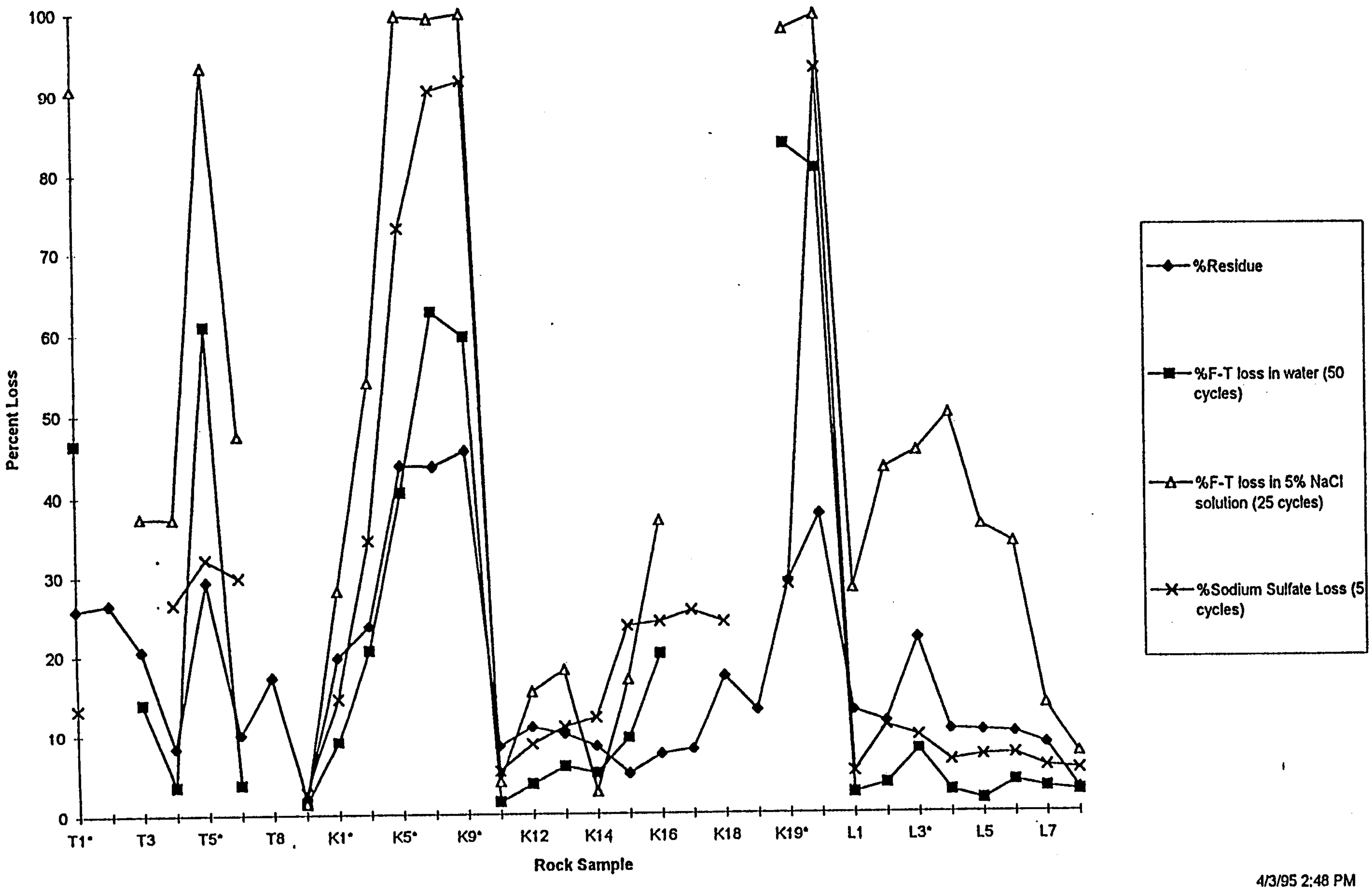
Shakoor concluded that a high degree of absorption (greater that 4%) and a high degree of saturation (greater than 90%) are indicators of probable aggregate failure, although the method was not fool-proof.

3.2.3 Alkali-Silica Reaction

Crumpton, Smith, and Jayaprakash (1989), who noted the scaling of concrete in saline solution without freeze-thaw in Kansas aggregate, also studied the effects of salt on alkali-silica reactivity. The aggregate in their study, as much of the aggregate used in Kansas, was highly susceptible to alkali-silica reaction. It was found that the salt solution exacerbated the alkali-silica

Figure 1

Laboratory durability test losses for samples from Shakoor (1989)



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reaction by keeping the interior of the aggregate wet for a longer period of time, increasing time for reactions to occur. The increased retention time of water in the aggregate also exacerbated D-cracking and pavement blow-up. These and other researchers have noted that D-cracking is the most likely failure of an aggregate under these circumstances. Any aggregate shown not to be susceptible to D-cracking, will not likely fail by any other mechanism.

3.2.4 Osmotic Pressure

Osmosis is a process where a solvent permeates a semi-permeable membrane from a solution of high concentration to the solution of lower concentration, in order to equilibrate the concentration. Osmosis becomes an important factor for a pavement aggregate when pore walls in the aggregate act as a semi-permeable membrane. The addition of deicing salt to aggregates already wet, causes a concentration gradient to develop across pore walls. In an effort to equilibrate concentrations across the pore walls, hydraulic forces may develop within the aggregate that may exceed the strength of the aggregate. It is reasonable to assume that the addition of salt may create the necessary concentration gradient. However it is not so easy to determine under what conditions in aggregates or in pavements a semi-permeable membrane exists. Additionally, if osmotic pressures can develop within the aggregate or pavement, is this pressure a significant factor in the aggregate deterioration in comparison to the other possible effects discussed here?

Although osmotic pressure is mentioned by many researchers as a factor for increased degradation of aggregates in the presence of salt solutions, only Salcedo's (1984) report for the Indiana Department of Transportation, provides a quantitative discussion of this factor. Salcedo cites Verbeck and Gramlich (1955) as a source that cement paste is capable of acting as a semi-permeable membrane. Salcedo also performed a test on three rock samples, but only one, "*a highly argillaceous aggregate with very fine capillary pores*" displayed any osmotic phenomenon. On the basis of these three samples, Salcedo concluded that osmosis occurs whenever there is a significant amount of pore sizes smaller than 0.06 um.

Salcedo does not claim to have determined the significance of osmotic pressure in the deterioration of aggregates, however he does mention one rock sample where the freeze-thaw loss after 50 cycles was less than 4% in pure water, but up to 50% in salt water. It can not be stated for certain that the increased deterioration is caused by of osmotic pressure, and not some other physical phenomenon such as increased absorption and swelling due to the increase in hygroscopy of clay minerals by the NaCl. If this example does not indicate conclusively that some rocks may develop sufficient osmotic pressure to cause deterioration, it does corroborate Shakoor's (1982) data that freeze-thaw tests in pure water may not always accurately predict the rock's behavior in freeze-thaw when deicing salts are present.

In additional testing to determine the contribution of osmotic pressure to deterioration, Salcedo noted that absorption values of these rocks were increased whenever a salt solution was used, which is commonly reported by other researchers. He also noted that the freezing of water began at a lower temperature for salt solutions, and that there was less frozen water. This is consistent with Hudec's observations (Hudec, 1978a) that it is not the formation of ice crystals which damages many aggregates, but instead the absorption of water.

Salcedo's research concludes that there is *"evidence to support the hypothesis that, for argillaceous aggregates, there exists an additional osmotic effect that contributes to the surface failure mechanism."*

Hudec (1987) also mentions that an increase in osmotic pressure caused by a salt gradient in the water of the aggregate yields a build-up of internal forces that can cause aggregate failure.

3.2.5 Aggregate Chemistry

Whereas most researchers have focused on the physical effects of salt solutions on aggregates, such as absorption and pore size distribution, researchers Wendell Dubberke and Vernon J. Marks at the Iowa Department of Transportation are also looking at how the chemistry of an aggregate is correlated with the durability on freeze-thaw conditions in the presence of deicing salts. Dubberke and Marks (1985) performed chemical analysis of aggregates to determine the amount of salt retention. Sodium content increased from 0.05 percent without treatment to 0.09 percent with treatment on one sample to 0.05 percent without treatment to 0.44 percent with treatment on another sample. It was hypothesized that the salt enables the aggregate to retain water longer and therefore contain more water when freezing.

X-ray diffraction and x-ray fluorescence were used in an attempt to correlate aggregate chemistry with performance. *"The sulfur content yielded the best correlation (inverse) with performance of the aggregates that had passed the Iowa specifications."*

Wendell and Dubberke (1987) demonstrated that for a ferroan dolomite exposed to deicing salt, the d-spacing of the dolomite crystals correlated with the durability of the aggregate under field conditions while accounting for poor pore size distributions in the aggregate. They found that by using x-ray diffraction to determine the d-spacing, the maximum intensity dolomite-ankerite peaks, correlated well with aggregate life in pavement. A d-spacing of 2.884 angstroms indicated good aggregate, whereas a d-spacing of 2.914 angstroms showed poor aggregate. Although Wendell and Dubberke obtained good correlations in this study, widespread use of x-ray diffraction may not be within the budget or technical abilities of most transportation agencies, and the study is limited to specific dolomites and their behavior in PCC pavement.

Wendell and Dubberke's most recent work determined that deicing salts contributes to the formation of the clay mineral ettringite between the aggregate and the PCC paste. Ettringite requires a source of sulfur for formation. The formation of the ettringite develops internal stresses between the aggregate and the paste that cause the PCC matrix to fail. When studied in thin section, the ettringite looks very much like the product of alkali-silica reaction, and additional testing is required to verify that the material is ettringite rather than silica gel. After additional exposure to salt solution, the ettringite dissolves leaving only the cracks around the aggregate and paste. Therefore there is only a relatively short time frame for a researcher to determine that ettringite formation was the cause of the aggregate failure.

The concrete section used in this study contained the same aggregate throughout, but was made using five different concrete mixes, four of which were made with fly-ash from various sources. Only the section that did not contain fly-ash showed no deterioration.

To date, Wendell and Dubberke have limited their research to the behavior of aggregates in PCC pavement, however their research suggests possible areas of investigation to pursue in bituminous pavement studies as well.

3.2.6 Additional Physical Effects

In addition to the effects described above, researchers occasionally speculate about other possible factors which may contribute in some fashion to the breakdown of aggregates under freeze-thaw, and in some cases in the presence of deicing salts.

Super Cooling of Water

Salcedo (1984) mentions that Harnik et al., suggest super cooling of salt solution as another negative effect on aggregate durability. When the salt solution reaches a certain temperature below freezing, large amounts of water suddenly freeze. This creates large hydraulic pressures that the aggregate would not be able to withstand.

Effects on the Bituminous Matrix

At low temperatures, a bituminous matrix may become more brittle, and not be able to withstand the expansion of the aggregate, causing the aggregate to fail. Anon (1989b) mentions that the addition of lime may also help prevent the stripping of the bituminous matrix from the aggregate surface.

Heat of Fusion

Because of the heat of fusion, melting of ice and snow caused by the application of deicers, may significantly lower the temperature in the subsurface.

3.3 TRACE SULFATE COMPOUNDS IN DEICING SALT

Questions have been raised about the detrimental effects of trace compounds in deicing salt, specifically magnesium sulfate and sodium sulfate. Some researchers mentioned previously have indicated a relationship between an aggregate's durability under freeze-thaw in laboratory tests with a saline solution, and its durability as defined by the sodium sulfate soundness test. The explanation for the correlation is that both sulfate exposure and saline solutions produces internal stresses within the aggregate that the rock is unable to withstand. Under normal freeze-thaw conditions, this stress is developed from the formation of ice crystals in the pores. When exposed to saline solution, the stress results from osmotic pressure or increased absorption, and under exposure to sulfate, the stress occurs from formation of sulfate crystals.

Pitt et al. (1988, 1992) studied the effect of trace compounds on deterioration of concrete. The research recommended that the concrete be made sulfate resistant with the use of fly ash, although too much fly ash weakens the pavements durability. This is somewhat contrary to the findings of Marks and Dubberke (1995) who studied a concrete section made with the same aggregates and five different PCC mixes. Four of the mixes which contained fly-ash failed in the presence of deicing salts because of the production of ettringite which swelled and stressed the matrix. The one section that did not fail contained no fly ash.

Pitt et al (1988, 1992) also recommended that the sulfate in the deicing salt be limited to less than 0.28%. No research located here included testing the freeze-thaw effects on aggregate in salt solution with various levels of sulfate included. It is reasonable, that to determine how an aggregate will behave under freeze-thaw for a particular highway department's salt source, the aggregate should be tested with salt from that source before use in the pavement.

Gillot (1978) performed the most extensive research into the effects of deicing compounds and sulfate. The research was performed on quartzites and limestones in cement blocks, soaking samples in this solution for a two year period. The resulting dimensional change was then measured for the blocks. Limestone showed changes and the sodium chloride and calcium chloride solutions both caused surface morphology changes in the samples. The research falls short in that it does not assess the effects of mixtures of salt and sulfate solutions and does not expose the samples to freeze-thaw. Also, like many of the other researchers, this study was performed only on concrete samples.

3.4 CONCLUSIONS

On the basis of the research reviewed here, the following conclusions can be draw:

1. No single study of the effects of deicing salt on aggregates in bituminous pavement was located. No single study was located on the effects of deicing salt on bituminous pavement matrix.

2. Although several researchers have used freeze-thaw testing in saline solutions to determine an aggregate's performance, the scope of the research is limited. There is no extensive correlation between this type of testing, and prediction of field performance. Among these researchers are:

   a. Shakoor (1982) compared freeze-thaw testing on argillaceous limestone aggregates in saline solution with the same test in pure water and compared the results to field performance.

   b. Dubberke and Marks (1985) used such a test on concrete slabs made with limestone aggregate and correlated the test results to field performance.

   c. Gillot (1978) measured dimensional changes in concrete blocks soaked in saline solutions and sulfate solutions, however correlation with field performance was not evaluated.

3. Absorption, adsorption, pore size distribution, porosity, degree of saturation, sulfate soundness, insoluble residue composition, distribution of argillaceous material, and mineral structure have all been studied in connection with aggregate durability under freeze-thaw with deicing salt, or aggregate durability in concrete under freeze-thaw with deicing salt.

4. It is theorized that for some argillaceous aggregates, it is the increased absorption caused by salt that causes degradation, rather than the formation of ice within the aggregate. It would be of benefit to develop a test capable of eliminating poor aggregate using absorption cycles only and without performing freeze-thaw cycles.

5. Much research has been devoted to the failure of concrete matrix under conditions of freeze-thaw in the presence of deicing salt, without mention of the aggregate. From the focus of the research it could be interpreted that in concrete pavement, the matrix durability is the limiting factor in resistance to freeze-thaw in the presence of deicing salt, and in bituminous pavement, the aggregate durability is the limiting factor. However some research has shown that aggregate-matrix interaction in PCC pavement may result in different modes of degradation than in bituminous pavement.

6. The effect of deicing salts on certain aggregates has been investigated, and the effect of sulfate solution on aggregates has been investigated, but research results found on the effect of a combined salt and sulfate solution is limited to Gillot (1978). If trace levels of sulfate in deicing salt

are suspected to be an additional factor in the degradation of aggregate, the development of a test using freeze-thaw in salt and sulfate solutions should be considered.

7. In PCC pavements, the effect of adding fly-ash is uncertain. Pitt et al. (1988, 1992) recommend adding fly-ash to make the concrete sulfate resistant, although too much fly-ash can reduce pavement durability. Pigeon, Aitcin, and Laplante (1987) recommend adding fly-ash to reduce chloride permability, but this results in the need for a higher water-cement ratio, and increases the possibility of scaling. Marks and Dubberke (1995) cite a case where a pavement section constructed from the same aggregate source, using five different concrete mixes, failed prematurely under freeze-thaw in the presence of deicing salt in the four sections that contained fly-ash in the mix. Only the fly-ash free pavement did not fail.

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Part II
Moisture Damage to Asphalt Pavement

1. INTRODUCTION

There has been extensive research done on the causes, treatment, and testing of moisture related stripping in asphalt pavement. Much of the research has been funded by the Federal Highway Works Administration and has resulted in comprehensive and detailed investigation reports. These reports are excellent sources of practical information from actual case studies of moisture damage in asphalt pavements.

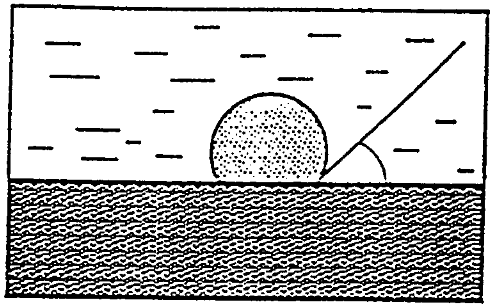
The most common areas of research are evaluation or comparison of test methods, evaluation of causes of stripping, and evaluation of treatment technologies or additives. Specific research into the effects of deicing salt, or trace constituents of deicing salt on asphalt pavement stripping is more limited. However there is abundant research into the relationship between aggregate type and stripping potential, the effects of deicing salt on specific aggregate types in asphalt pavement is not widely studied.

Many properties of asphalt pavement can influence the pavement's susceptibility to moisture damage. This makes it difficult to isolate one particular cause as the primary cause of deterioration. Asphalt-aggregate properties such as aggregate mineralogy, aggregate shape and porosity, aggregate moisture content, dust coatings, and gradation are all important to consider when evaluating pavement for moisture damage potential. Asphalt mix type, viscosity, and additives are also important factors. There is also disagreement on the benefits of the use of surface seals.

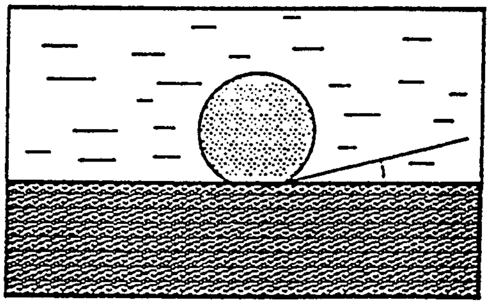
Environmental effects during and after construction are important considerations. As would be expected, construction of an asphalt pavement in a wet environment is more likely to result in moisture damage. Good subsurface drainage is important to reduce the buildup of pore pressures and to allow the removeal of water to prevent scouring. Traffic load and compaction of the pavement are also factors influencing the pavement's susceptibility to moisture damage.

2. STRIPPING AND FACTORS INFLUENCING MOISTURE DAMAGE

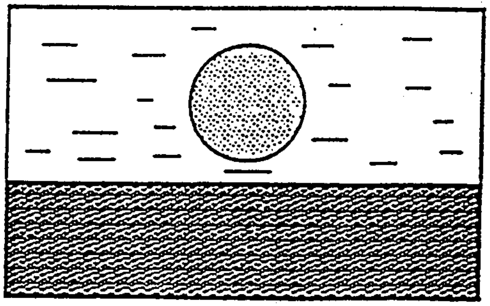
Stripping of asphalt from aggregate in bituminous pavements can be thought of as the reduction in the contact angle of the asphalt-aggregate interface (Hicks, 1991). This is illustrated in Figure 2. If the aggregate is not completely covered by the asphalt, moisture may come in contact with the aggregate at the asphalt-aggregate boundary. Over time, the moisture will displace the asphalt cement. This is because of the greater affinity of the aggregate for water than for the oil-based asphalt cement. If the aggregate is of a type susceptible to moisture damage, once in contact



(a) The moment at which the aggregate, with the drop of bitumen, is immersed in water. The contact angle is less than 90°.



(b) The water begins to remove the bitumen drop from the aggregate surface and the contact angle decreases.



(c) Finally, the stage is reached where the contact angle is 0° and the bitumen loses contact with the aggregate surface.

Figure 2. Scemeatic of the stripping process (from Hicks, 1991).

with the moisture, the aggregate may deteriorate causing failure of the pavement surface (Hicks, 1991).

To prevent stripping, it is necessary to completely cover the aggregate with the asphalt cement. Kennedy, Roberts, and Anagnos (1984) state that mixtures that retained less than 70 percent of the asphalt on the aggregate are susceptible to moisture damage. Stripping can also be minimized by changing the chemical-physical properties of the aggregate surface to reduce the affinity for water over asphalt cement, or reducing the ability of water to displace the asphalt cement. The is the primary function of chemical additives or lime.

Hicks (1991) lists four factors commonly related to moisture damage in asphalt. These include asphalt concrete characteristics (including aggregate, asphalt cement, and type of mixture) weather during pavement construction, environmental effects, and drainage of the pavement and subsurface. These are summarized in Table 4.

2.1 Asphalt-Aggregate Properties

Aggregate characteristics important to stripping include surface texture, porosity, pore size, and surface moisture, and chemical composition. Using quality aggregate can help to minimize stripping. Aggregates should have low porosity of approximately 0.5% or less and a clean surface (Krebs and Walker, 1971). A rough texture is also beneficial. It is speculated that a greater energy is required to strip the viscous asphalt from a rough aggregate because the asphalt cement interlocks with the surface contours. However it may be more difficult to get adequate coverage of the aggregate when the cement is more viscous. Some additives reduce viscousity temperarily during the mixing process to allow adequate coverage. Viscosity is then regained over time allowing the asphalt cement to better rip the aggregate (Khandal, 1992).

Some researchers indicate that the presence of iron, magnesium, or calcium on the aggregate surface is beneficial to prevention of stripping, while sodium and potassium are detrimental (Hicks, 1991). Dust on the aggregate is also considered by many to be detrimental. The surface of the aggregate must also be able to accept or donate electrons, or form hydrogen bonds, acid or base pairs, or insoluble salts (Hicks, 1991). Table 5 summarizes characteristics of aggregates and minerals and their relationship to stripping as defined by Stuart (1990). Factors listed by Khandal (1992) include inadequate pavement drainage, inadequate compaction, excessive dust on aggregate, and inadequate drying of the aggregates.

Yoon and Tarrer (1988) tested the stripping potential of several aggregates by use of the boiling water test. They found that aggregates with a high surface electrical potential and those that impart a high pH on water in contact with the aggregates are more susceptible to stripping.

Factor	Desirable Characteristics
1) Aggregate	
a) Surface Texture	Rough
b) Porosity	Depends on pore size
c) Mineralogy	Basic aggregates are more resistant
d) Dust Coatings	Clean
e) Surface Moisture	Dry
f) Surface Chemical Composition	Able to share electrons or form hydrogen bonds
g) Mineral Filler	Increases viscosity of asphalt
2) Asphalt Cement	
a) Viscosity	High
b) Chemistry	Nitrogen and phenols
c) Film Thickness	Thick
3) Type of Mixture	
a) Voids	Very low or very high
b) Gradation	Very dense or very open
c) Asphalt Content	High
4) Weather Conditions (During or Immediately Following Construction)	
a) Temperature	Warm
b) Rainfall During Construction	None
c) Rainfall Following Construction	Minimal
d) Freeze-Thaw Following Construction	Minimal
5) Traffic Loading	Low traffic

Table 4. Summary of factors influencing moisture damage (from Hicks, 1991).

Slight Stripping	Moderate Stripping	Severe Stripping
	a) Minerals	
Biotite Hornblende Feldspars: • Labradorite • Bytownite • Anorthite Chlorite Sericite Muscovite Diopside Olivine Pyroxenes Augite Calcite	Biotite Hornblende Feldspars: • Oligoclase • Albite • Anorthite Garnet Quartz Muscovite	Biotite Hornblende Feldspars: • Oligoclase • Albite • Anorthoclase • Microcline • Perthite • Andesine Chalcedony Quartz
	b) Igneous Rocks	
Gabbro Basalt Greenstone (Basalt) Quartz Dolerite Diabase Scoria, Slag Peridotite	Biotite Granite Basalt Olivine Dolerite w/Analcite Quartz Diorite Andesite Diabase	Granite Biotite Granite Aplite Granite Pegmatite Granite Soda Granite Granite Porphyry Granodiorite Obsidian Albitised Olivine-Diorite Diorite Rhyolite Trachyte Pumice Dacite Syenite
	c) Metamorphic Rocks	
Siliceous River Sand Siliceous Sand w/Iron Oxide Coat Serpentine	Biotite Feldspar Gneiss Feldpathic Quartz-Sercite Gneiss Granitic Quartz-Feldspar Gneiss Biotite-Muscovite Schist Diabase-Hornfels Hornblende-Gneiss Biotite Schist	Quartzite Granitic Gneiss Quartz-Sericite Schist Feldspathic-Quartzite Biotite Schist Muscovite Schist
	d) Sedimentary Rocks	
Limestone Dolomite Graywacke Limerock	Limestone Dolomite Limerock Reef Coral Calcareous Sandstone	Iron Oxide-Rich Arkose Chert Flint Breccia Feldspathic Sandstone Sandstone Chalk Oolitic Limestone Argillaceous Sandstone

Table 5. Summary of aggregates and minerals according to the degree of stripping assoiated with them (from Hicks, 1991).

Because of the more significant influence of other factors, and the variety of asphalt mixes, it is difficult to characterize features of an asphalt mix that are beneficial or detrimental to stripping. It is generally considered beneficial to use a viscous asphalt as it is less likely to be displaced by water.

A viscous asphalt may have decreased wetting ability and therefore not provide a complete cover of the aggregate. Schmidt and Graf (1972) note that if asphalt mixes have the same viscosity, the chemical characteristics of the asphalt are negligible, however other researchers have found evidence to the contrary. Carboxylic acids and certain sulfooxide compounds have been found to be susceptible to moisture damage (Petersen, Ensley, and Barbour 1974, and Petersen, Plancher, Ensley, Veneble, and Miyake 1982). Damage to these compounds may increase the brittleness of the asphalt causing cracking under stress and increasing moisture penetration.

2.2 Type of Mixture

Dense-graded hot mix asphalt is generally thought not to strip unless there is excessive air voids or insufficient asphalt cement (Brown, Sparks, and Marsh, 1959). The amount of voids in a dense-graded mix is generally thought to be the most important factor contributing to stripping (Hicks, 1991). Shatnawi and Van Kirk (1993) also recommend lowering the air voids and reducing the ratio of fines to asphalt to minimize stripping.

Proper compaction during pavement placement is important in reducing the air voids. Liu and Kennedy (1991) determined that sufficient compaction was the reason pavement samples in California showed no signs of stripping 48 months after construction.

Amount of moisture in the aggregate at the time of mixing is an important factor. Parker and West (1992) measured moisture contents of hot bin aggregates and freshly mixed hot asphalt concrete. They found moisture levels likely to affect the susceptibility of the mix to damage. They also stated that the amount of moisture retained in the plant produced mix is highly dependent on ambient temperature and the moisture content of the aggregate stockpiles.

Parker and West (1992) also state that mixes containing limestone as the dominant aggregate were not adversely affected by residual moisture. They conclude that residual moisture may explain some of the discrepancies noted between laboratory testing, which is always performed on thoroughly dried aggregate, and field results.

Although most researchers indicate that dense-graded pavements are necessary to minimize moisture damage, other researchers studied open-graded mixes. Shuler and Hanson (1990) worked with open graded asphalt mixtures and noted that stripping could be minimized with the use of anti-stripping agents to the asphalt or aggregate and after polymer modification of the binders.

2.3 Environmental Effects

As would be expected, cool and damp weather during construction will significantly increase the susceptibility of asphalt pavement to stripping (Hicks, 1991). This is also more likely to increase the moisture content of aggregates in stockpiles, which is less likely to be completely dried during the mixing process. Khandal (1992) states that the drying of aggregate is important in reducing the susceptibility to stripping.

Climate and traffic loadings after pavement construction play an important role in asphalt stripping. For weather variations, temperature fluctuations, freeze-thaw, and wet-dry cycles can influence the amount of stripping in asphalt pavements. Pore pressure buildup and scouring are the two major mechanisms through which water causes stripping in asphalt. Cyclic traffic loading of the asphalt increases the effect of these conditions, and therefor stripping is more likely in pavements with higher traffic loads (Hicks, 1991).

2.4 Subsurface Pavement Drainage

As moisture builds up in the pavement, two important mechanical processes promote stripping of the aggregate. Pore pressures build up within the pavement layers causing the water the permeate into areas wear the asphalt cement may not completely cover aggregate surfaces. This may lead to displacement of the asphalt surface by the water. The second method is mechanical scouring. With scouring, repeated traffic loading causes water movement within the pavement layers that will tend to erode away the asphalt covering of the aggregate. Water in contact with the aggregate surface will displace the asphalt causing stripping. With increased amounts of traffic, there is an increased in the frequency of movement of water in the pavement and the scouring effect is exacerbated.

Subsurface drainage is important in minimizing stripping. Proper drainage can reduce the pore pressures and remove water that would otherwise scour the asphalt-aggregate interface. Proper subsurface drainage has been considered a significant contributing factor in mitigating moisture damage. These conclusions are based on work by academic researchers and substantiated by practical experience in a variety of state highway departments (Hicks, 1991).

3. CORRECTIVE TREATMENTS

Hicks (1991) gives three methods to reduce stripping. These include surface seals, use of quality aggregate, and pre-treatment of the aggregate. Yoon and Tarrer (1988) also recommend pre-coating the aggregate, and curing the asphalt-aggregate mixture to minimize stripping.

3.1 Surface Seals

Surface seals are used to minimize migration of water into the pavement layer. They are used only with dense graded mixtures. The most common type is "fog sealing," which consists of spraying a light application of liquid asphalt onto the pavement surface. Other commercially available sealants are also used. Some researchers report that surface seals increase the amount of stripping in a pavement. This may occur if the source of the water is beneath the pavement. Krutz and Stroup-Gardiner (1990) noted that sealing the surface accelerated moisture damage of pavements in Nevada by trapping moisture that would otherwise escape through the pavement layers. This would indicate that the use of sealants and proper drainage have a symbiotic relationship in the prevention of moisture damage.

3.2 Aggregate Pre-Treatment

Pretreatment of aggregates consists of removing or replacing aggregate surface ions that are likely to be removed by water or cause weak bonding to the asphalt. Sodium, potassium, or calcium ions may react with acids to form salts at the surface of the aggregate increasing the likelihood of stripping (Hicks, 1991). Aggregates with iron or magnesium at the surface are less susceptible to stripping.

Ramaswamy et al. (1990) discuss the use of sprinkle treatment to reduce moisture damage. With this method, a thin layer of highly polish-resistant pre-coated aggregates is applied to the surface of a freshly placed hot mix wearing surface, and is embedded into the surface during the rolling operation. This improves skid-resistance, improves durability by reducing the hardening process of the bitumen, and reduces stripping tendency. It also increases skid-resistance in areas where supplies of polish-resistant aggregate may be limited.

3.3 Additives

Additives are frequently used to minimize moisture damage in asphalt pavements. Additives are generally divided into two groups; chemical additives and lime. Chemical additives are usually commercially manufactures of proprietary formulas, but consist predominatly of amines. Chemical additives can be mixed in with the asphalt cement or coated over the aggregate. Lime is applied to the aggregate and has been shown to be very effective. The difficulty with lime is ensuring adequate coverage of the aggregate surface with the lime.

3.3.1 Chemical Additives

Chemical manufactured additives or lime can be used to increase bonding of the asphalt to the aggregate. There are a large number of chemically manufactured additives that are available, and each is purported to be effective based on the manufacturer's testing. Most chemical additives work by reducing the surface tension in the asphalt and thereby improving the wetting characteristics (aggregate surface coverage) of the mix. The chemical additives may also increase the adhesion of the asphalt to the aggregate.

Although the chemical makeup of the additive is proprietary information, most chemical additives consists of amines, which are primarily basic compounds. Amines increase the ability of the asphalt to wet the surface of the aggregate. Chemical additives can be added to the liquid asphalt mix, or applied directly to the aggregate surface before addition of the asphalt cement. The latter is more efficient because all the additive is utilized at the asphalt-aggregate interface where it is most effective. Yoon and Tarrer (1988) indicated that the effectiveness of some anti-stripping additives may be sensitive to pH of water in contact with the aggregate. Ping and Kennedy (1991) found that most anti-stripping additives were effective on gravel but less effective on limestones and sandstone.

Tarrer (1987) indicates that the effectiveness of a chemical anti-stripping agent can be determined by observing the contact angle of an asphalt drop immersed in water. Also reported in the same source is that additive dosage level was more important than the type of additive, and 0.5 to 1.0% was the typical dosage for desired performance.

3.3.2 Lime Additives

State highway department surveyed in Hicks (1991) report that the use of lime additives is a very effective way to minimize stripping. Lime can be added in many ways, however, the lime becomes effective only after moisture is introduced to activate the lime (Ishai and Craus, 1977). It is thought that lime produces a sharp decrease in the interfacial tension between the water and the aggregate. Hoever some researchers have speculated that lime works with the carboxylic acid to produce insoluble residues that adhere to the surface of the aggregate. Lime also produces calcium ions that can replace hydrogen, sodium, and potassium ions at the aggregate surface. For protection against stripping, the lime must be in its oxide (CaO) and hydroxide ($Ca(OH)_2$) forms. Carbonate lime ($CaCO_3$) is not effective in preventing stripping.

Methods of applications include treatment of the aggregate with 1) dry hydrated lime, 2) hydrated lime slurry, 3) dry hydrated lime with moist aggregate, and 4) hot (quicklime) slurry (Hicks, 1991). Each of these procedures has been shown to be effective in reducing stripping.

Adding dry lime to the aggregate is generally considered to be the least effective of the methods (Hicks, 1991). This is because it is difficult to keep the lime on the surface of the aggregate until it is covered with the asphalt. Hudson et al. (1990) concluded that even though addition of lime through wet methods resulted in higher strengths than dry methods, the difference was not statistically significant to support a conclusion that wet methods are better than dry methods.

Hydrated lime slurry can be used to cover the aggregates with lime. The disadvantage of this method is that the aggregate must then be dried and this increases energy costs. Ping and Kennedy (1991) found hydrated lime applied in slurry form to effectively reduce stripping susceptibility on all rock aggregate types they tested.

Adding dry hydrated lime to moist aggregate is another alternative. A water content of 3 to 5 percent in the aggregate is desirable. The lime is then added using a positive mix pugmill or tumble mixer (Hicks, 1991).

Coating the aggregate with a hot (quicklime) slurry is also a possible treatment method. This produces an endothermic reaction which elevates the temperature and increases drying of the aggregate. The disadvantage of the method is increased hazards for workers (Hicks, 1991).

Some researchers note that hydrated lime may adversely harden the asphalt cement. On tests of six pavement sections made with no additives, hydrated lime, and chemical additives, Maupin (1987) concluded that the correlation between the hardening of the asphalt cement and the age of the pavement was poor for five of the sections. Only one project showed a correlation of hardening with age, and that section showed no difference in hardening between sections made with hydrated lime, chemical additives, or no additives.

4. TESTING FOR MOISTURE DAMAGE POTENTIAL

Most available research focuses on developing and evaluating tests to predict susceptibility of pavement mixes to moisture damage. A variety of tests are available to test asphalt for potential for stripping. Hicks (1991) reports that a major area of research in state highway departments is in determining which tests are appropriate for which asphalt mix combinations, and correlating laboratory results with field results.

Tests can be separated into two general categories. The first type of test are those that coat aggregate with asphalt cement and immerse the sample in water at either room temperature or boiling. The effect on the asphalt-aggregate mix is then recorded visually. The second group uses compacted specimens either from the laboratory or from field cores. The samples are conditioned and a comparison is made between conditioned and unconditioned samples. Usually the samples are compared by diametral resilient modulus, diametral tensile strength, or both (Hicks, 1991).

For any of the tests, there is no definable criteria that results in 100% effective prediction of the potential for moisture damage. Ping and Kennedy (1992) found reasonable correlations between various testing procedures to determine susceptibility of asphalt-aggregate mixtures to stripping. Test methods studied included the original Lottman method, the modified Lottman method, the Tunniclif-Root method, and the boiling test (Tex-530-C).

Dickinson, Blachly, and George (1990) note that the IRS (Index of Retained Strength) test was considered to be a valid and useful stripping test. The IRMR (Index of Retained Modulus of Resiliency) was determined to have the greatest potential for future improvement.

Parker and Gharaybeh (1988) evaluated different test methods for use in Alabama. The boil and indirect tensile test were found to be the most promising. However neither these tests nor the stress pedestal test accurately predicted the performance of all mixes. The results were consistent although not always correct. They also noted a good correlation between the boil test results and indirect tensile test values. This improves credibility of these tests as predictors of stripping when applied to different mixes. They recommend against using any test for all mixes and indicate that each test should be applied only to specific mix combinations. It is not indicated how the test should be selected based on the type of mix used. They acknowledge that variability in aggregate drying, gradation, and asphalt content may be important in affecting stripping potential.

Parker (1987) evaluated the freeze-thaw pedestal test, the boil test, and the indirect tensile test for assessing stripping potential. The freeze-thaw pedestal test was recommended against because of test complexity and weak correlation with stripping performance. It was recommended that the boil and indirect tensile tests be used together, even though the results were imperfect.

Kennedy, Roberts, and Lee (1983) recommend the Texas boiling test for initial, short-term screening because it is simple and easy to conduct. The Texas freeze-thaw pedestal test is recommended for final and long-term evaluations. Mixtures with high air void content should be evaluated using the indirect tensile test on both dry and wet specimens.

5. CONCLUSIONS

Abundant research is available on moisture damage in asphalt concrete. The research is primarily focused on the causes, treatment, and testing of moisture related stripping in asphalt pavement. Much of the research has been funded by the Federal Highway works Administration and has resulted in comprehensive and detailed investigation reports. These reports are excellent sources of practical information from actual case studies of moisture damage in asphalt pavements. From this research, the following conclusions can be drawn:

1. Moisture damage to asphalt pavement can be caused by any one of a number of factors including, aggregate chemical and physical properties, asphalt mix, gradation, additives, placement environment, drainage, traffic load, and construction practices. Combinations of these factors may make determination of the actual cause of pavement failure difficult or impossible.

2. There are a variety of tests useful in predicting the susceptibility of pavement to moisture damage. However no single test method has been shown to be 100% effective for all mixes. Without a 100% effective test method, it is inevitable that an asphalt-aggregate combination susceptible to moisture damage will be mistakenly put into use.

3. Some construction practices such as sealing have bee shown to be beneficial in preventing moisture damage, and increasing susceptibility of pavement to moisture damage. This contradictory information makes it even more difficult to assess causes of pavement deterioration and develop preventative practices.

4. Little research has been done on the contribution of deicing salt or trace constituents of deicing salt to moisture damage in asphalt pavement. The large number of other variables that contribute to susceptibility to moisture damage makes assessment of the importance of de-icing salt more difficult. From research done on deicing salt damage to aggregates in PCC, it is evident that salt will accelerate moisture damage of the aggregate. It is reasonable to assume that once water comes in contact with the aggregate in asphalt-cement pavements, the deicing salt will also accelerate damage to susceptible aggregate.

5. The most common areas of research are evaluation or comparison of test methods, evaluation of causes of stripping, and evaluation of treatment technologies or additives. Specific research into the effects of deicing salt, or trace constituents of deicing salt on asphalt pavement stripping is more limited. Although there is abundant research into the relationship between aggregate type and stripping potential, the effects of deicing salt on specific aggregate types in asphalt pavement is not widely studied.

6. Environmental effects during and after construction are important considerations. Constructing a pavement in a wet environment is likely to be detrimental to the pavement. Good subsurface drainage is important. Traffic load is also a consideration. The compaction of the pavement is also a factor influencing the pavement's susceptibility to moisture damage.

6. RECOMMENDATIONS

The most common areas of research are evaluation or comparison of test methods, evaluation of causes of stripping, and evaluation of treatment technologies or additives. There has been only limited research into the effects of deicing salt, or trace constituents of deicing salt on asphalt pavement stripping. It is thought that salt may increase the brittleness of asphalt cement and this has been the primary area of research on the salt effects on asphalt cement. Although there is abundant research into the relationship between aggregate type and stripping potential, the effects of deicing salt on specific aggregate types in asphalt pavement is not widely studied.

Some of the research reviewed for the salt effects on concrete and aggregates also addressed asphalt cement (Shakoor, 1992). Aggregate types which are more resistant to moisture and salt degradation in PCC, would also be more likely to be resistant to moisture and salt degradation in asphalt cement. However, as noted here, although aggregate type is important in reducing the susceptibility of asphalt cement pavement to moisture damage, stripping in asphalt can have many causes not related to the aggregate.

One difficulty in studying the causes of stripping and other moisture damage in asphalt concrete is the variety of factors all capable of causing stripping. These include aggregate chemistry and mineralogy, aggregate residual moisture content, asphalt mix design and additives, grading, compaction, sub-pavement drainage, construction techniques, traffic loading, and environmental conditions during and after construction. Many of the investigations provide somewhat inconsistent results because of the difficulty researchers have in isolating each of these variables. Any further investigation into the effects of de-icing salt and stripping, would need to consider each of these other variables. With the isolated number of cases proposed for investigation, it may be impossible to precisely determine that one single factor was the predominant cause of stripping in the pavement.

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Ping, W.V. and T.W. Kennedy (1991) *Evaluation of stripping and moisture damage in asphalt pavements treated with lime and anti-stripping agents, Interim Report*, Texas University, Austin: Center for Transportation Research, FHWA/TX-92+441

Ramaswamy, S.D., E.W. Low, and T.F. Fwa (1990) *A study in the use of sprinkle treatment to improve bituminous surfacings*, Proceedings, Sixth Converence, Road Engineering Association of Asia and Australia, March 1990.

Schmidt, R.J. and P.E. Graf (1972) *The effect of water on the resilient modulus of asphalt treated mixes*, Proceedings, Association of Asphalt Paving Technologists, Vol. 41, pp 118-162.

Shatnawi, S.R., and J. Van Kirk (1993) *Premature Asphalt Concrete Pavement Distress Caused by Moisture Induced Damage*, Transportation Research Board 2101, 1993.

Shuler, S. and D.I. Hanson (1990) *Improving durability of open-graded friction courses*, Transportation Research Record 1259.

Stuart, K.D. (1990) *Moisture damage in asphalt mixes -- A state of the art report*, Report No. FHWA-RD-90-019, Federal Highway Administration, Washington DC, March.

Tarrer, A.R. (1987) *Stripping of asphalt concrete - Chemical testing, Final Report*, Auburn University Engineering Department and Alabama State Highway Department.

Yoon, H.H. and A. R. Tarrer (1988) *Effect of aggregate properties on stripping*, Transportation Research Record 1171.

DESCRIPTIONS OF RELEVANT ARTICLES FOR PART I

Anon. (1987), *Don't let deicers scale your concrete,* Concrete Construction, Vol. 32, No. 11, November.

This article lists 13 tips to prevent scaling of concrete by deicers. Heavily emphasized is the use of air-entrained concrete (5.5 to 7.5 percent air). Also listed are tips for aggregate, pouring, curing, and sealing the surface with linseed oil. A brief but very informative article.

Bilodeau, A., V. Sivasundaram, K.E. Painter, and V.M. Malhotra (1994), *Durability of concrete incorporating high volumes of fly ash from sources in the US,* ACI Materials Journal, Volume 91, Number 1, Jan-Feb (American Concrete Institute)

Concrete with a water and cement contents of about 115 and 155 kg/m3 respectively. Proportion of fly-ash in the total cementicious material is about 55 to 60 percent. Freeze-thaw, deicing salt scaling resistance, and chloride-ion penetration tests were conducted on the samples.

Performance was satisfactory for the freeze-thaw and chloride ion penetration tests. The concrete performed poorly for the deicing salt scaling resistance test. Results varied for this test depending on the fly-ash source. More testing was recommended in this area. Extensive quantification of results is provided.

Crumpton, Carl F., Barbara J. Smith, and G.P. Jayaprakash (1989), *Salt weathering of limestone aggregate and concrete without freeze-thaw,* Transportation Research Record, No. 1250.

This article discusses the effect of salt on limestone aggregate in Kansas. Much of the aggregate available in Kansas is highly alkali-reactive. Sand and gravel used in Kansas commonly contains glassy volcanics, chert, opal, or chalcedony. Shale is also used as aggregate. Clays in the limestones are altered by the salt, and quartz is altered by electric currents in the paste. D-cracking is a common result. Significant deterioration of pavement did not occur until Kansas began using deicing salt about 30 years ago.

The testing was done on concrete cups filled with salt water. A silane sealer used on three cups did not prevent migration of the chloride through the concrete. Salt scaling of unreinforced concrete occurred without freeze-thaw. It was concluded that salt exacerbates the alkali-aggregate reaction, D-cracking, and pavement blow-up by keeping the concrete interior wet for longer periods of time, increasing time for reactions to occur.

Dubberke, Wendell and Vernon J. Marks (1985), *The effect of deicing salt on aggregate durability,* Transportation Research Record, No. 1031.

Case studies in Iowa have shown that aggregates on heavily salted roads do not perform as well as the same aggregates used in unsalted secondary roads. A five cycle chloride treatment performed before freeze-thaw tests allowed for improved prediction of an aggregates performance on heavily salted roadways. X-ray fluorescence and the scanning electron microscope showed that high levels of sulfur, especially when accompanied by magnesium, accelerated deterioration of aggregate in the presence of salt. The sulfur is likely present in the aggregate as pyrite or marcasite. It is mentioned that the adverse reactions may not take place until after the aggregate has been used in concrete.

Dubberke, Wendell, and Vernon J. Marks (1987), *The relationship of ferroan dolomite aggregate to rapid concrete deterioration*, Transportation Research Record, No. 1110.

A study of Iowa pavements made with aggregate that has exhibited inconsistent performance was implemented. The aggregate performed well in PCC pavement on secondary roads that received little to no salting, but poorly on primary roads that were heavily salted. The research was conducted to develop a simple test to determine the durability of the aggregate. Aggregates with known pore structure problems were accounted for in the study.

The study showed that the d-spacing of the maximum intensity dolomite-ankerite peaks was a good indicator of the durability of the aggregate. It was shown that d-spacings of 2.884 angstroms provided good aggregate while d-spacings of 2.914 angstroms were poor aggregate. This showed that the d-spacing obtained from x-ray diffractometry was a good indicator of aggregate durability under salting conditions. Iron substituting for magnesium in the dolomite crystalline structure of coarse aggregate correlated well with performance history when NaCl deicing salts are used.

Dunn, R. R., and P.P. Hudec (1972), *Frost and sorption effects in argillaceous rocks,* Highway Research Record, Highway Research Board
From TRIS search

No study of deicing salt was performed, however it was determined that water absorption is more destructive of shales, siltstones, and argillaceous carbonate rocks that freezing and thawing.

Gillott, J. E. (1978), *Effect of deicing agents and sulphate solutions on concrete aggregate*, Quarterly Journal of Engineering Geology, Volume 11, pp. 177-192.

Samples of quartzites and limestones were soaked in solutions of sodium chloride, calcium chloride, and magnesium sulfate. Over a period of two years, precise measurements were taken to measure changes in size of the samples. The paper concludes that adverse effects on concrete may be a results of attack on limestone aggregate as a well as on cement paste. Quartzites showed no dimensional change in the solutions. Limestones did show change and were further investigated with scanning electron microscopy. Calcium chloride and sodium chloride were responsible for a variety of surface morphology changes in the limestone including etching, cleavage step removal, cleavage widening, block removal, and deposition.

Harvey, Richard D., James W. Baxter, Gordon S. Fraser, and Craig B. Smith (1978), *Absorption and other properties of carbonate rock affecting soundness of aggregate*, Geological society of America, Engineering Geology Case Histories Number 11.

These researchers studied how properties of a carbonate rock related to the sodium sulfate soundness loss. The rock samples were separated by rock type and alumina content. For low alumina limestones, water absorption accounted for 89% of the variation observed in the soundness test data. For high-alumina limestones, absorption accounted for 78%. Equations are provided to predict soundness loss based on rock type and other tests, however it is admitted that the equations do not predict durability with sufficient accuracy. In each case, absorption is a factor in the equation related to the sulfate soundness loss. No testing was performed with salt solution and this topic is not mentioned.

Hoebeda, P. and T. Jacobson (1981), *The influence of deicing chemicals on the frost resistance of aggregates,* National Swedish Road and Traffic Research Institute.
In Swedish, from TRIS search

Methods of investigating the resistance of aggregates including direct freeze-thaw tests and other indirect methods are discussed. Laboratory tests concluded that porosity is an important factor in salt degradation. Ten cycles of freezing and thawing were enough to give a sufficiently great disintegration of non-resistant aggregates. Development of a freeze-thaw testing procedure in combination with deicing chemicals, preferable a 1% NaCl solution is recommended.

Hudec, P. (1978a), *Standard engineering tests for aggregate: What do they actually measure?,* Geological society of America, Engineering Geology Case Histories Number 11.

Rocks with frost sensitivity tend to saturate by adsorption ad the water contained in the rocks is largely adsorbed water. This water is virtually unfreezeable. Frost susceptible rocks tended to expand upon wetting even at room temperature. Seismic velocity studies were performed on 45 specimens. "*The sound rocks showed a uniform* decrease *in the transverse wave velocity in the saturated state when compared to the dry state. The unsound rocks showed an* increase *in the transverse wave velocituy in the saturated state compared to the dry state*". It was concluded that the sorptive rocks became more "*rigid*" upon saturation, with the rigidity caused by the strong forces of surface sorption. Also sound aggregate "*did not saturate critically (greater than 91% saturation), that is, it has some pores open*". The unsound aggregate achieved greater than 91% saturation. However, this appears less important when it is considered that the vast majority of water in unsound aggregate remains unfrozen.

In small enough pores, rigid water is able to fill the entire pore and exert pressure on the wall of the pore. Alternate wetting and drying cycles will add and remove this pressure in the same way as alternate freeze-thaw cycles. This continued addition and removal of stress is able to break down the rock.

Hudec claims that only a small fraction of unsound carbonates are truly "frost-sensitive", that is break down because of the formation of ice crystals within the pore structure. However the standard freeze-thaw tests are based on the theory that ice formation is the cause of unsound rock deterioration.

Hudec, P. (1978b), *Rock weathering on the molecular level,* Geological society of America, Engineering Geology Case Histories Number 11.

This paper presents the statistical relationship between the internal surface area of 26 rock specimens and their physical properties. Both freeze-thaw loss and magnesium sulfate loss were shown to be reliable predictors of a rocks susceptibility to deterioration, the magnesium sulfate test the better predictor. When relating internal surface are to freezing of pore water, internal surface area was shown to be more important in durability than the amount of water in the pores that actually froze. It was also shown that a higher absorption correlated with high magnesium sulfate loss and high freeze-thaw loss. The degree of saturation is related to the size of the surface area. "*The higher the internal surface area the greater the degree of saturation, that is, the percentage of pores filled*".

Hudec, P (1987), *Deterioration of Aggregates - The underlying causes. Concrete durability,*
Katherine and Bryant Mather International Conference, Atlanta GA
American Concrete Institute
P.O. Box 19150
Redford Station

Detroit, MI 48219

The same material susceptible to freeze-thaw damage also deteriorate significantly under repeated wetting and drying cycles. NaCl solution causes significantly greater expansion, therefore ice formation in the pores is not the primary cause f failure. Increase of osmotic pressure due to the salt is also a contributing factor. Methods that determine pore size distribution, surface sorption characteristics of the pore walls, and volume changes on wetting as more definitive measures of aggregate durability.

Hudec, Peter P. and Stephen G. Rigby (1976), *The effects of sodium chloride on water sorption characteristics of rock aggregate*, Bulletin of the Association of Engineering Geologists, Volume 13, No 3.

Crushed carbonate aggregate from 57 samples was tested to determine the effect of common deicing salt on the sorption of the rock under low and high humidity condition, and under immersion.

The sorption of the rocks was compared under normal, untreated conditions and after immersion in 3% sodium chloride solution. At 45% RH, 30 degrees C, no significant change was observed. At 98% RH, 30 degrees C, the salted rocks adsorbed approximately twice the amount of water. The water intake under immersion conditions was 1.13 times greater for the salted rocks. The degree of saturation increased approximately 13%.

The enhanced water content of aggregates that have been exposed to deicing salts increases the rock's susceptibility to deterioration.

Kallas, B. F. (1963), *Performance of asphalt pavements subjected to deicing salts*, Highway Research Record, Highway Research Board, No 24, pp 49-61, 1963
From TRIS Search

This paper describes the effects of common deicing salt solutions on the performance of asphalt pavement. No loss or damage was noticed on the pavement and the study concluded that well-designed and well-constructed asphalt pavements are not damaged by sodium and calcium chloride salts.

Kaneuji, M., D.N. Winslow, and W.L. Dolch (1980), *The relationship between an aggregate's pore size distribution and it's freeze-thaw durability in concrete*, Cement and Concrete Research, Vol. 10 pp. 433-441.

Testing was done on 14 aggregates and the porosity determined by mercury intrusion. The aggregates were then tested to determine the durability factor from standard laboratory freeze-thaw tests. The results showed that both the total pore volume and the median pore diameter influenced the durability. An equation was developed allowing the determination of durability from a measurement of pore size distribution. Results from several Indiana highways verified the predictions. This is from a Purdue University Ph.D. thesis.

Marks, Vernon J., and Wendell G. Dubberke (1995), *Investigation of PCC pavement deterioration -- A few facts are worth more than 100 opinions*, Iowa Dept. of Transportation.

Cracking deterioration was identified on a three-year old pavement in Iowa. The coarse aggregate had a excellent history of performance. Examination showed that cracks were primarily in the cement matrix and not through the aggregate. The deterioration had been identified as ASR.

When the concrete was further investigated with a low-vacuum Scanning Electron Microscope. This showed that there was no silica gel but there was sulfur, aluminum and calcium in the air voids (assumed to be ettringite). It was determined that exposure to NaCl causes the ettringite to form, swell, and crack the matrix. Subsequent exposure to NaCl causes the ettringite to dissolve and disappear. Cracks radiated out from the ettringite filled voids, but there was no ettringite in the cracks, leading to the conclusion that the voids were filled with ettringite before the formation of the cracks. Elemental maps showed that the white material in the voids contained almost no silica. Laboratory tests confirmed that ettringite could grow in air voids. This ettringite can also be shown to dissolve in the presence of chloride ions, cause swelling and then could dissolve within 48 hours. Although this study is in concrete, it indicates results should also be investigated for bituminous pavements.

Pitt, J.M., R.A. Carnazzo, J. Vu, and M. Seshardi (1988), *Control of concrete deterioration due to trace compounds in deicers, Phase I Report,* Iowa Dept. of Transportation Highway Division
826 Lincoln Way
Ames IA 50010
Abstract only was available from TRIS search

The incfluence of trace compounds from rock salt deciers on PCC mortar and concrete. An evaluation of the deicers in stock throughout Iowa showed that about 95% had enough sulfate to cause accelerated deterioration of concrete. Sulfate compounds of calcium and magnesium were found to be equally deleterious. Magnesium chloride was found to be innocuous. Two approaches to limiting deterioration included limiting of sulfate to 0.28 percent and mixing fly-ash with concrete to increase sulfate resistance.

Pitt, J. M. , R.A. Carnazzo, J. Vu, and M. Seshardi (1992), *Control of concrete deterioration due to trace compounds in deicers, Final Report,* Iowa dept. of Transportation Highway Division
826 Lincoln Way
Ames IA 50010
Abstract only was available from TRIS search

Three aspects of rock salt deicer action on freeze-thaw resistance of Portland cement concrete. The first demonstrated that a 10 to 15% replacement of cement with fly ash doubled the life of the concrete by reduction of porosity and stabilization of calcium hydroxide. Excessive fly ash countered the benefit.

The second aspect was the behavior of aggregates displaying different service lives in concrete subjected to deicers. Freeze-thaw in water produced failure in the aggregate, but in deicers, damage was exclusive to the aggregate-mortar interface. Porosity was a good although not perfect predictor of aggregate service life with low porosity the best.

The third aspect was to develop a test to predict service life. The researchers developed a predictive model of a pavement subject to different deicing materials as follows: no deicer -- life = 25 years, low sulfate NaCl -- life = 19 years, and high sulfate NaCl -- life = 14 years.

Salcedo, Antonio Marco (1984), *Identification of frost susceptible aggregate and their use in concrete or bituminous pavements,* Joint Highway Research Project Report JHRP-84-23, Final Report.

The performance of aggregates is correlated with the pore size distribution using discriminant Analysis. A rating system is developed to distinguish poor aggregate from excellent aggregate. The study only focuses on the two extremes of performance. However extensive background research of freeze-thaw studies is presented. It is indicated that pore diameters ranging from 45 A to 1-10 um are most susceptible to freeze-thaw damage. However this range is also dependent on the lowest temperature achieved, and the rate of temperature decrease. It is also noted that bituminous pavements may be more susceptible to damage from freezing of argillaceous aggregate because at lower temperature, the bituminous matrix is less accommodating of the expansion of the aggregate. Naturally, if the application of deicing salts exacerbates the aggregate expansion, the deterioration would be more severe.

Shakoor, Abdul (1982), *Evaluation of methods for predicting durability characteristics of argillaceous carbonate aggregates for highway pavements*, Purdue University PhD Thesis.

A study was performed on argillaceous dolomite which had passed acceptance tests, but failed severely in field performance. The study focused on which tests were best able to predict the performance of the aggregate. Freeze-thaw and failure mechanisms are summarized in the report.

For the analysis, approximately 200 pounds of sample was collected from 38 locations at three quarries. Petrographic examination of the samples included megascopic examination of hand specimens and polished slabs, microscopic examinations of polished slabs, and thin sections, acid etching of polished slabs and thin sections, analysis of pavement core samples, insoluble residue tests, and specific gravity and absorption as well as a number of other standard aggregate tests.

It was found that the more durable aggregate had specific gravity greater than 2.5 and absorption less that 4.0%. Freeze-thaw tests were performed with both water and 5% salt solution. Loss from the 5% solution was much more pronounced when the argillaceous material was clayey in composition rather than silty. Insoluble residues of > 20% or EDF < 20 performed poorly in the field.

Mechanisms for freeze-thaw failure and failure from deicing salts are summarized although not explicitly studied. From other researchers, it was theorized that salt causes a build up of osmotic pressures forcing water to move from low salt concentrations to higher salt concentrations. The salt at the top of the slab causes the water to move towards the top of the slab. The critical concentration for salt is 2% to 4% for the most deterioration of the aggregate.

Damage is more severe when the deicer is applied shortly after the placement of the pavement for concrete. Damage is also more severe when there are cycles of freeze-thaw with salt water on the pavement and no opportunity for the salt solution to be washed off with fresh water. Melting of ice and snow by salt may also reduce the subsurface temperature significantly because of the heat of fusion of the ice.

Triano, James R. and Gregory C. Frantz (1992), *Durability of MSW fly ash*, Journal of Materials in Civil Engineering, Volume 4, Number 4, Nov.
624.1805 J826

The durability and leachate properties of concrete made with fly ash from solid waste mass-burn and refuse derived fuel plants is studied. The paper concludes that MSW fly ash has little potential for use in structural quality concrete. High doses of air-entraining mixture were required to produce 6-7% air contents in concrete with MSW fly-ash. Concrete containing mass-burn fly ash had "outstanding" resistance to surface scaling when exposed to deicing salts in a freeze-thaw environment. Concrete containing RDF fly ash had poor scaling resistance. MSW fly ashes and the concretes containing fly ashes had very high chloride levels.

Zimbleman, Ruprecht (1989), *Resistance of concrete to frost/deicing salt in the light of new findings*, Beton Stahlbetonbau, Volume 84, Number 5, May.
In German

The author indicates that frost and deicing chemical destruction in concrete is caused by the aggregates being surrounded by a porous crystalline system that forms around each aggregate grain. "This system spreads into the dense cement paste and differs from the point of view of its structure and chemical composition." No additional information is provided in English.

ADDITIONAL ARTICLES REVIEWED FOR PART 1

Aitcin, P. C., and P. Laplante (1990), *Long-term compressive strength of silica fume concrete*, Journal of Materials in Civil Engineering, Volume 2, Number 3, Aug.
624.1805 J826

Concrete cores from silica fume and non-silica fume concrete were studied for compressive strength. The cores were 4 to 6 years old and had been exposed to freeze-thaw and deicing salts. The results showed that the silica fume-concrete had not experienced any strength loss when compared to the non-silica fume concrete. Silica fume mixes did seem to suffer more from field placing and curing conditions than non silica fume concrete. The concretes seemed to exhibit very low chloride-ion permeability.

Anon. (1989a), *The 3 most often asked questions about salt*, Better Roads, Volume 59, Number 10, Oct., pp. 33-34.
625.705 B459

This article is questions about salt answered by an admitted proponent of salt use. The need for quality air-entrainment of concrete is emphasized. He sates that salt is not a problem for bituminous pavements because it is a petroleum mix. No mention of the effect on aggregate is made.

Anon. (1989b), *Asphalt research moves ahead*, Better Roads, Volume 58, Number 10, Oct. p 22-24.
625.705 B459

This article discusses some areas of study funded by the Federal Highway Administration. These include the stripping of asphalt aggregate and additives such as lime to prevent this occurrence. Also discussed are polishing of aggregate, where it is suggested that using aggregates of different hardness is beneficial, synthetic asphalt, and the addition to the mix of deicers Vermiglit and PlusRide. CMA is also discussed as a deicer.

Anon. (1987), *Performance of two ice-retardant overlays*, Public Works, Volume 118, Number 7, July.
620.5 P96

This article discusses the performance of two asphalt mixes which contained pellets of Vermiglit, a calcium chloride deicer. As the pavement wears, the deicer is released and this prevents snow and ice buildup. The article evaluates the effectiveness of the mix on preventing ice buildup, but the effects on pavement degradation are not discussed. This article is not relevant to the study.

Callahan, Mark R. (1989), *Deicing salt corrosion with and without inhibitors*, Transportation Research Record, Number 1211

Different deicing solutions were tested on steel coupons to determine their effect on corrosion rates. Samples of each solution were also ponded on reinforced concrete blocks. Corrosion was measured by electrical potential readings.

Deicers tested were calcium magnesium acetate (CMA), CMA + NaCl, Quicksalt + PCI, and CG-90, a polyphosphate solution being developed by Cargill. All deicers were less corrosive than NaCl, however only pure CMA significantly inhibited corrosion of steel embedded in concrete.

Campbell, Glenn M. and Rachel J. Detwiler (1993), *Development of mix designs for strength and durability of steam-cured concrete*, Concrete International: Design and Construction, Volume 15, Number 7, July.
693.505 c749

This article states that corroded steel can increase in volume by 6 fold. Corrosion damage in beams of a bridge in San Francisco is studied after 17 years of exposure. Some beams were made of steam-cured concrete and others were cured at ambient temperatures. The steam-cured beams had to be repaired because of corrosion damage, while the other beams showed no signs of damage. Previous work showed that curing at elevated temperature coarsens the cement paste pore structure and increases penetration of chloride ions. Reducing water cement ratio is of limited effectiveness in improving the concrete. Silica fume and slag can be used to improve durability.

Dagher, H.J. and S. Kulendran (1992), *Finite Element modeling of corrosion damage in concrete structures*, ACI Structural Journal, Volume 89, Number 6, Nov.-Dec.
691.5 Am3pas

This article discusses the process of deterioration of concrete by corrosion and expansive stresses from steel rebars and decks. No information is provided on deicing salts and concrete deterioration.

Fansom, James and Edward Cohen (1991), *Lincoln Park Lagoon bridge restoration*, Concrete International: Design and Construction, Volume 13, Number 6, June.

The restoration of a concrete bridge built in 1936 is discussed. Much of the concrete had suffered significant deterioration. An extensive description of problems with the bridge is provided. Six core samples were taken to design the restoration. Methods of restoration to provided the best aesthetic appearance are described. Little information on salt effects of the concrete is provided.

Foy, Christiane, Michel Pigeon, and Nemkumar Banthia (1988), *Freeze-thaw durability and deicer salt scaling resistance of a 0. 25 water-cement ratio concrete*, Cement and Concrete Research, Volume 18, Number 4, July.
620.13 C332

Samples of 0.25 water-cement ratio concrete with various air-void spacing factors were tested for freeze-thaw durability and deicing salt scaling resistance. A critical air void spacing factor of 750 um was determined. All specimens showed a very good resistance to deicer salts showing that low water concrete may be useful in preventing deterioration from deicing salts. After 150 cycles, 0.6 kg/m2 of scaled off particles was measured. The results indicate that even at water cement ratios below 0.3, air entrainment still is necessary to protect against deicing salts.

Gjorv, Odd E., Kefang Tan, and Paulo J.M. Monteiro (1994), *Effect of elevated curing temperature on the chloride permeability of high strength lightweight concrete*, Cement, Concrete, and Aggregates, Volume 16, Number 1, June
620.130287 c331

Testing was done to determine how high curing temperatures and use of either wet or dry aggregate affects compressive strength and chloride permeability. For both the wet and dry aggregate, an increase in chloride permeability was observed with increasing curing temperature. The increase in permeability occurred primarily above 60 degrees C.

Grieb, W.E., G. Werner, and D.O. Woolf (1962), *Resistance of concrete surfaces to scaling by deicing agents*, Public Roads, 32(3), Aug. pp. 64-73

Concrete slabs were subjected to freeze-thaw (154 cycles over five years) and deicing with CaCl2. The effect of various protective coatings and admixtures was evaluated. Different air-entraining admixtures were effective to various degrees. There was less scaling when concrete was cast with a sand base rather than a water-tight metal base. No scaling was shown if the concrete was cast in sand with an air content of greater that 3 percent. Some scaling was found on concrete cast in metal bases with air contents as high as 7.4 percent. Above 7.5 percent, there was no scaling regardless of the base type. Low-alkali Portland cement was more resistant than high-alkali Portland cement. Use of slag cement did not result in a difference in scaling when compared to Portland cement. Small amounts of low-carbon fly ash did not increase scaling but larger amounts of fly ash, or high carbon fly ash, resulted in increased scaling. Moist curing seemed to increase the amount of scaling. Membrane curing gave some protection as long as the membrane remained intact. Use of vacuum treatment on the exposed surface helped to reduce scaling when compared to similar concrete without vacuum treatment. Protective surface treatments applied 14 to 28 days after the concrete was cast, generally delayed but did not prevent scaling. An aqueous silicone solution was effective in reducing scaling while increasing compressive strength, but increased setting time. An emulsion of polystyrene latex as an admixture reduced scaling but the mix contained over 10 percent air. Of the deicing agents tested, calcium chloride was found to be the most effective and caused the least scaling. It was also concluded that scaling can be reduced by eliminating the use of waterproof coatings on the subgrade, reduction in the water-cement ratio, and permitting concrete to dry before freezing.

Holland, Terrance (1987), *Working with silica fume concrete*, Concrete Construction, Volume 32, Number 3, March.
624.18305 C 749

Use of silica fume helps reduce rebar corrosion because the reaction products of the fine silica fume plug internal pores. This helps to keep chloride out of the concrete. Use of silica fume can not replace good construction practices. Much of the article is a thorough discussion the working, placement, finishing, and curing of silica fume concrete. The need of superplasticizers with silica fume concrete is also discussed.

Lessard, M., S.L. Sarkar, D.W. Ksinsik, and P.C. Aitcin (1982), *Long-term behavior of silica fume concrete*, Concrete International: Design and Construction, Volume 14, Number 4, April.

This paper reports on the results of a ten-year study of the use of silica-fume in concrete in sidewalks in Quebec. Silica fume was added near the job-site. The mixes contained no superplasticizers and slump was controlled by adding water. The samples therefore have a high water-cement ratio.

Air contents ranged from 2.1 to 8.3 percent and the bubble spacing factor ranged from 220 to 490 um.

Miller, R. A., B. Krouskop, LA. Minkarah, and A. Bodocsi (1993), *Chloride penetration and the effect of porosity in a pavement*, Transportation Research Record, Number 1392

A 9" thick concrete pavement from Chilocothe, Ohio was studied for chloride penetration and porosity. Cores were taken through sound areas, sealed joints, and cracks. Chloride levels were measured a four depths in the concrete. Steel mesh was located at depths of 3 to 4 inches in the pavement.

The sound cores showed chloride below corrosion limits at a depth of 3 inches. No corrosion of the steel mesh was observed. Chloride in the crack and joint cores were above corrosion limits in most cases. The mesh had completely corroded. Flexural cracks may be enough to transmit corrosive levels of chloride. There was not correlation between porosity and chloride content. Damage was the most important factor in chloride penetration.

A petrographic study was performed on the aggregate. The concrete used well-graded aggregate. Air-entrainment varied from 2 to 12 percent but most samples were near the average of 7 percent. There was not sign of alkali-aggregate reaction or chemical attack.

Mogawer, Walaa S., Kevin Stuart, and K. Wayne Lee (1989), *Evaluation of effects of deicing additives on properties of asphalt mixtures*, Transportation Research Record, Number 1228, pp 41-53
625.7 N214hr c.2

This paper evaluates the performance of Vermiglit and PlusRide on asphalt properties. These additives are deicers which, as the pavement wears, are released and inhibit the forming of ice on the pavement. The Verglimit improves the temperature susceptibility and increases its susceptibility to moisture. PlusRide increased resistance to low-temperature cracking but decreased resistance to rutting.

Nadezhdin, A., D.A. Mason, B. Malric, D.F. Lawless, and J.P. Fedosoff (1988), *Effect of deicing chemicals on reinforced concrete*, Transportation Research Record, Number 1157.

Spalling of concrete is caused by the growth of crystals. Air-entrainment acts as a cushion to absorb the increased volume. Testing was done to determine the effects of various deicer formulations on concrete subjected to freeze-thaw. Corrosion was measured as electrical potential in rebar. The difference in freezing temperatures between concrete pore solution and an outside deicer solution is shown to be one of the important factors in the spalling process.

O'Connor, Daniel N. and M. Saiidi (1993), *Compatibility of polyester-styrene polymer concrete overlays with Portland cement concrete bridge decks*, ACI Materials Journal, Volume 90, Number 1, Jan.-Feb.

Polymer concrete overlays are used to prevent the penetration of deicing materials into reinforced concrete bridge decks. This article presents a comparison of basic engineering properties for polymer and Portland cement concretes followed by a study of two representative bridge deck/overlay finite element models subjected to a variety of temperature loads to determine internal stresses.

This paper focuses on a polyester-styrene polymer concrete as a bridge deck overlay. The compressive strength of polymer concrete is approximately twice the strength of the concrete in the deck, but drops by 25 percent as temperatures approach 120 degrees F. The coefficient of thermal expansion for polymer concrete is more than twice that of Portland cement concrete.

No information on the penetration of deicing materials into the polymer concrete is provided.

Okkenhaug, Knut and Odd E. Gjorv (1992), *Effect of delayed addition of air-entraining admixtures to concrete*, Concrete International: Design and Construction, Volume 14, Number 10, Oct.

This paper indicates that only about half of structures studied with intentionally air-entrained concrete had adequate air voids for frost resistance. The research was limited but concluded the following: 1. Adding air-entraining mixture with mixing water simultaneously produced a higher total air content than when AE mixture addition was delayed, but the air void system obtained was poorer. The best air-void system was obtained when the AE was added at the end of the mixing process. 2. when the air-entraining mixture was used in combination with a liggno-sulfate water reducer, a poorer and less stable air void system was produced. 3. When the AE mixture was used in combination with a naphthalene-based superplasticizer, a very poor and unstable void system was obtained. Using a melamine-based superplasticizer appeared to produce a much better and more stable air void system. 4. The best air void system was obtained when used without any water reducers or super plasticizers.

Palmer, David A. (1987), *Formates as alternative deicers,* Transportation Research Record, Number 1127.

The use of sodium formate as an alternative to sodium chloride is discussed. It is much less toxic than previously thought. It can be spread as a concentrated solution or as a slurry. The freezing-point curve is similar to that of sodium chloride. Use of corrosion inhibitors is briefly discussed and determined to be unsatisfactory with higher corrosion rates than organic salts. Advantages and disadvantages of CMA are discussed and CMA was also determined to be an unsatisfactory replacement of sodium chloride.

No actual testing of formates is done for this article. It is a review of current information. Sodium formate solutions have been tested at airports and several disadvantages are listed that require further investigation. Early testing showed that both calcium and sodium formates caused spalling in concrete. Later studies showed that sodium formate does not spall concrete or injure asphalt. It has been tested as a deicer with sand in Minnesota, and Celenese Chemical Company is working on improving its performance.

Pigeon, Michel , Pierre-Claude Aitcin, and Pierre Laplante (1987), *Comparative study of the air-void stability in a normal and condensed silica fume field concrete,* ACI Materials Journal, Volume 84, Number 3, May-June.

This paper reports the findings of a research program studying freeze-thaw durability and the stability of the air-void system in a specific CSF mix from batching through placement. Additional test data show shrinkage and compressive strength.

The data indicates that CSF concrete without the right bubble spacing factor performs very poorly, but is satisfactory with the right bubble spacing factor. A bubble spacing factor of 250 um was required versus 500 um for normal concrete. Severe scaling was reported in poorly controlled CSF concrete in sidewalks in Quebec. This was the result of inadequate air-void systems. A comparative study of sidewalk with normal and silica fume (silica fume-to-cement ratio of 8 percent) was performed. The article concludes that with less that 10 percent CSF, concrete with satisfactory air void spacing factor can be produced with proper techniques. It is noted that the use of CSF increases the water dosage so that water-cement ratio is on the order of 0.5 when no superplasticizer is used.

Reagan, Frank (1992), *Performance characteristics of traffic deck membranes,* Concrete International: Design and Construction, Volume 14, Number 6, June.

This paper contains a qualitative discussion on the use of membranes to prevent deicing salt from penetrating into traffic decks. Use and physical properties of the membranes are described, such as permeability, adhesion, elongation, tensile strength, tear strength, elasticity, and other factors. No information is provided on the physical effects of deicing salts on concrete or aggregates.

Sarkar, Shondeep L. and Pierre-Claude Aitcin (1991), *Phenomenological investigation of concrete deterioration in a median barrier,* Cement and Concrete Research, Volume 21, Number 5, Sept.

A deteriorated reinforced median barrier in Quebec was studied. The barrier was constructed of gray cement in 1972. The most prevalent deterioration was map cracking, longitudinal cracking, rebar corrosion, scaling, and spalling. Freeze-thaw action followed by leaching of constituents led to a significant increase in porosity. This increased the concretes vulnerability to chemical attack. The embedded metal can deteriorate due to deep penetration of Cl ions. The paper also described the corrosion of rebar by carbonation, and the corrosion process. Little information is provided on preventing degradation by deicing salts.

Virmani, Y. Paul (1991), *Technical Summary. Salt penetration and corrosion in prestressed concrete members,* Materials Performance, Volume 30, Number 8, Aug.

This article discussed the effect of roadway deicing salts and marine spray on corrosion of prestressing steel in prestressed bridge components. It is suggested that cracks in the concrete caused by the prestressing operation may impair the effective power of the concrete cover and help promote the corrosion of the steel members. Corrosion was most often observed in bridge elements adjacent to leaking joints and faulty drains. Distress was more pronounced in the deicing environment than in a marine environment. Routine inspection and proper maintenance were considered very important in protecting steel from water. Shotcrete repairs, and fiberglass jackets did not appear sufficient to protect steel components, however epoxy-based mortar may be an effective temporary remedial measure.

Concrete studied in deicing environments was air-entrained and had low water-cement ratio. None of the concrete examined was of poor quality. A summary of 13 reasons identified as causes of steel corrosion in identified.

Vitaliano, Donald. F. (1992), *Infrastructure costs of road salting,* Resources, Conservation and Recycling, Volume 7, Number 1-3, Oct.

This article is written by an economist and provides examples of cost determination of road salting. However there is no technical information on how salting affects pavement.

Whiting, D. and W. Dziedzic (1989), *Chloride permeabilities in rigid concrete bridge deck overlays*, Transportation Research Record, Number 1234.

Bridge deck overlays constructed of latex-modified concrete, superplasticized dense concrete, or condensed silica-fume concrete were tested for chloride permeability. Silica-fume concrete was judged to be the most impermeable, followed by latex-modified and then superplasticized-dense concrete. Over long periods of time, chloride eventually penetrated all mixes and built up to substantial levels.

DESCRIPTIONS OF RELEVANT ARTICLES FOR PART II

Aschenbrener, T., R. Terrel, and R. Zamora (1994), *Comparison of the Hamburg wheel-Tracking Device and the Environmental Conditioning System to Pavements of Known Stripping Performance, Final Report*, Colorado dept. of Transportation.

Moisture damage in hot mix asphalt is studied in Colorado. This research determined that the Hamburg wheel-tracking device has very severe moisture conditioning, and by modifying the specification, the results could accurately identify most sites of known performance. The ECS test procedure has a mild moisture conditioning. By making the specification more severe, the results could also identify areas of known filed performance.

Aschenbrener, T., R. Terrel, and R. Zamora (1993), *Investigation of the Modified Lottman Test to Predict the Stripping Performance of Pavements in Colorado, Final Report.*, Colorado Dept. of Transportation.

Moisture damage in hot mix asphalt in Colorado is studied with the modified Lottman test. Two levels of severity for laboratory conditioning were identified that correlated well with field conditioning. For mixtures placed under high traffic, high temperatures, high moistures, and possible freeze, the severe laboratory conditioning defined in the report should be used. The milder conditioning defined in this report is suitable for low traffic sites.

Bruce, B. (1990) *Evaluation of asphalt stripping tests in Montana*, Montana Department of Highways.

The actual field moisture susceptibility of 10 bituminous mixtures was compared with that predicted by laboratory testing by the modified Lottman procedure and the Root-Tunnicliff procedure as well as immersion and compression testing.

Dickinson, L.L., A.T. Blatchly, and A.J. George (1990) *Evaluation of asphalt stripping tests in Oregon, Final Report*, Oregon department of Transportation.

Four test methods were compared to establish a standard for evaluating moisture susceptibility of asphalt concrete design mixes. The tests included the Index of Retained Strength (AASHTO T165), the Index of Retained Modulus of Resiliency (OSHD TM315), the modified Lottman (AASHTO-T283), and the Root-Tunniclliff (NCHRP 274). None of the four tests predicted the same degree of asphalt stripping across the range of asphalt and aggregate tested. The IRS test was determined to be a valid and useful stripping test. The IRMR test was determined to have the greatest potential for future use.

Hudson, S.W., F.N. Finn, J.H. Treybig, J.A. Epps, and V. Anderson (1990) *AC stripping problems and corrective treatments, Final Report*, ARE Incorporated.

The study determined the most effective method of introducing lime into asphalt mixtures and to improve the reliability of the laboratory test methods to identify moisture susceptible asphalt-aggregate combinations. The addition of lime through wet methods resulted in higher strengths than dry methods, however the statistical difference was not significant enough to conclude that wet methods are better than dry methods.

Kandhal, P.S. (1992) *Moisture susceptibility of HMA mixes, Identification of problem and recommended solutions.* Auburn University: National Center for Asphalt Technology. Report #92-1.

Factors inducing premature stripping in HMA pavements are described such as; inadequate pavement drainage; inadequate compaction of HMA pavement; excessive dust coating on aggregate, inadequate drying of aggregate, and overlays on concrete pavements. The report recommends an investigative methodology and reviews the current practices of specifying moisture susceptibility tests across the United States.

Kennedy, T.W., and F.L. Roberts (1984) *The economics of considering moisture susceptibility during mixture design* -- Proceedings of the fourth conference on asphalt pavements for South Africa

This paper summarizes the results of a laboratory study to develop tests for moisture susceptibility of asphalt pavement, and the consequences of not evaluating the moisture susceptibility of asphalt mixes. Three tests, the indirect tensile test on wet and dry cylindrical specimens, the Texas freeze-thaw pedestal test, and the Texas boiling test showed that all three could differentiate between stripping and non-stripping asphalt mixtures. The boiling and pedestal tests can also be used to evaluate the individual components of mixtures, and was more accurate than the indirect tensile test.

Kennedy, T.W., F.L. Roberts, and J.N. Anagnos (1984) *Boiling test for evaluating moisture susceptibility of asphalt mixtures,* Texas University Austin: Center for Transportation Research

This report contains a description of the development and use of theTexas boiling test to evaluate stripping of materials susceptible to moisture damage.

Kennedy, T.W, F.L. Roberts, and K.W. Lee (1983) *Evaluation of moisture effects on asphalt concrete mixtures,* Transportation Research Board 2101.

This report summarizes conclusions drawn by the researchers in evaluating different test methods. It is recommended that the Texas boiling test be used or initial short term screening, and the Texas freeze-thaw pedestal test be used for final and long-term evaluation. The Indirect tensile test on both wet and dry specimens is recomended for mixtures with a high air voids content.

Krutz, N.C. and M. Stroup-Gardiner (1990) *Relationship between permanent deformation of asphalt and moisture sensitivity,* Transportation Research Record 1259.

Severe rutting and stripping was observed in Nevada pavements during the first warm weather following a chip seal application. Sealing the surface appears to accelerate the moisture damage by trapping moisture that would otherwise escape the pavement layers. Samples were tested for moisture relation to deformation. It was concluded that moisture conditioning plays a significant role in permanent deformation.

Lui, M. J. and T.W. Kennedy (1991) *Field eveluation of stripping and moisture damage in asphalt pavements treated with lime and anti-stripping agents, Final report.* Texas University Center for Transportation Research.

Results of field evaluation of the effectiveness of lime and various anti-stripping additives is summarized using the Modified Lottman Test and the Boiling Test. There was very little evidence of stripping 48 months after construction. Most liquid anti-stripping additives were found to be effective on the gravels but less effective on limestones and sandstones. Hydrated lime applied in a slurry form was found to be effective for all aggregates tested.

Maupin, G.W. Jr. (1989) *Assessment of stripped asphalt pavement, Final Report.*, Virginia Department of Transportation

This report attempts to develop a quantitative test to evaluate pavement layers to determine if pavement needs to be removed or overlain. The indirect tensile test was used to evaluate data based on three field projects. Criteria defining minimum strength necessitating removal are suggested.

Maupin, G.W. Jr. (1989) *Assessment of stripped asphalt pavement*, Transportation Research Board 2101.

The indirect tensile test is used to determine the extent of stripping in pavement layers based on three field projects. The procedure makes possible the evaluation of pavement layer strength by in-situ methods. Criteria for minimum strength necessitating removal are suggested.

Maupin, G.W. Jr. (1987) *The use of hydrated lime as an anti-stripping additive, Final Report,* Virginia Department of Transportation

This investigation evaluated the performance of six test sections of asphalt concrete that contained no additive, hydrated lime, and a chemical additive. There was generally less stripping in pavements with hydrated lime than in pavements with no additive or with chemical additives. The tensile strength test correlated well with the amount of stripping that was observed. There was poor correlation between hardening of the asphalt and pavement age, or with asphalt mix.

Parker, F. Jr. and R.C. West (1992) *Effects of residual aggregate moisture on stripping potential of asphalt concrete mixtures*, Transportation Research Board 2101.

This report investigates the poor correlation of field stripping performance and stripping test prediction in Alabama pavements. It is concluded that laboratory tests are performed in thoroughly dried aggregates whereas aggregates used in pavements may contain residual moisture that would lead to stripping. Moisture measurements of hot-bin aggregates and freshly mixed hot asphalt concrete occasionally confirm the presence of residual moisture at levels that are likely to have an effect on the moisture damage susceptibility of the mix. The amount of moisture retained in plant-produced mix is highly dependent on ambient temperatures and the moisture content of the aggregate stockpiles.

Parker, F. Jr., and F.A. Gharaybeh (1988) *Evaluation of tests to assess stripping potential of asphalt concrete mixtures*, Transportation Research Board 2101

Stress pedestal, boil, and indirect tensile tests were evaluated for assessing the stripping potential of asphalt concrete mixtures in Alabama. The boiling and indirect tensile tests were most promising, although neither they nor the stress pedestal test accurately predicted the expected performance of all mixes. They did produce consistent although sometimes incorrect results.

Variability in aggregate drying, gradation, and asphalt content may be an important factor in affecting stripping potential.

Ruth, B.E. (1985) *Evaluation and prevention of water damage to asphalt pavement materials*, American Society for Testing and Materials, Report Number STP 899.

This is a collection of papers which provide insight into the severity of stripping, test methods for evaluations, and relative effectiveness of using antistripping additives to minimize in-service stripping. The papers empahsize the effectiveness or ineffectiveness of antistripping agents as evaluated by various laboratory test methods.

Shatnawi, S.R., and J. Van Kirk (1993) *Premature Asphalt Concrete Pavement Distress Caused by Moisture Induced Damage*, Transportation Research Board 2101, 1993.

Causes of premature pavement distress in California are evaluated. It was shown that road sections that showed more severe stripping were those that received a chip seal treatment or had an asphalt concrete overlay over an existing chip seal, or were constructed with pavement reinforcing fabrics. It was concluded that using a lime slurry form or using a liquid anti-stripping agent in combination with lowering the air voids and reducing fines could minimize stripping. The moisture induced damage test (AASHTO T283) had the best potential to identify moisture susceptible mixes.

Shuler, S. and D.I. Hanson (1990) *Improving durability of open-graded friction courses*, Transportation Research Record 1259.

Open graded friction course mixtures used in New Mexico were evaluated in the laboratory to determine the potential for stripping with the boiling test. Results of this study indicated that optimum asphalt content of open graded friction course mixtures varies depending on the asphalt, anti-stripping agent type, and quantity, and whether the binder is polymer modified. Adding anti-stripping agents to the aggregate and polymer modification of the binders significantly reduced the stripping as measured by the boiling test.

Tarrer, A.R. (1987) *Stripping of asphalt concrete - Chemical testing, Final Report*, Auburn University Engineering Department and Alabama State Highway Department.

The objectives of this research was to develop a better understanding of the nature of adhesion and stripping, to relate stripping propensity with aggregate characteristics, and to identify the fundamental properties of additives that are primarily responsible for effectiveness, and develop criteria for selection and use of anti-stripping additives. The boil test was used to study the stripping process. It was concluded that aggregates that impart a higher pH on the water are more susceptible to stripping. The effectiveness of anti-stripping agents can be determined by observing the contact angle of an asphalt drop on an aggregate immersed in water. Additive dosage level was more important than the type of additive and 0.5 to 1.0% was the typical dosage for desired performance.

Taylor, M.A. and N.P. Khosla (1983) *Stripping of asphalt pavements: State of the art*, Transportation Research Board 2101.

This is a comprehensive survey of literature regarding moisture damage to asphalt pavements since 1954. Topics covered include mechanism of stripping, factors influencing stripping, use of anti-strip additives, and tests to predict moisture damage.

Yoon, H.H. and A. R. Tarrer (1988) *Effect of aggregate properties on stripping*, Transportation Research Record 1171.

Various aggregates were characterized based on physical properties such as pore volume and surface area, and chemical and electrochemical properties. Susceptibility to stripping was evaluated using the boiling test. A higher propensity for stripping was shown in aggregates that have a relatively high surface electrical potential or impart a high pH to water. The effectiveness of some types of anti-stripping agents was sensitive to the pH of water in contact with the aggregate. Curing the asphalt-aggregate mixture and pre-coating the aggregate improved stripping resistance considerably.

Appendix B

Insoluble Residue Grain Size Curves

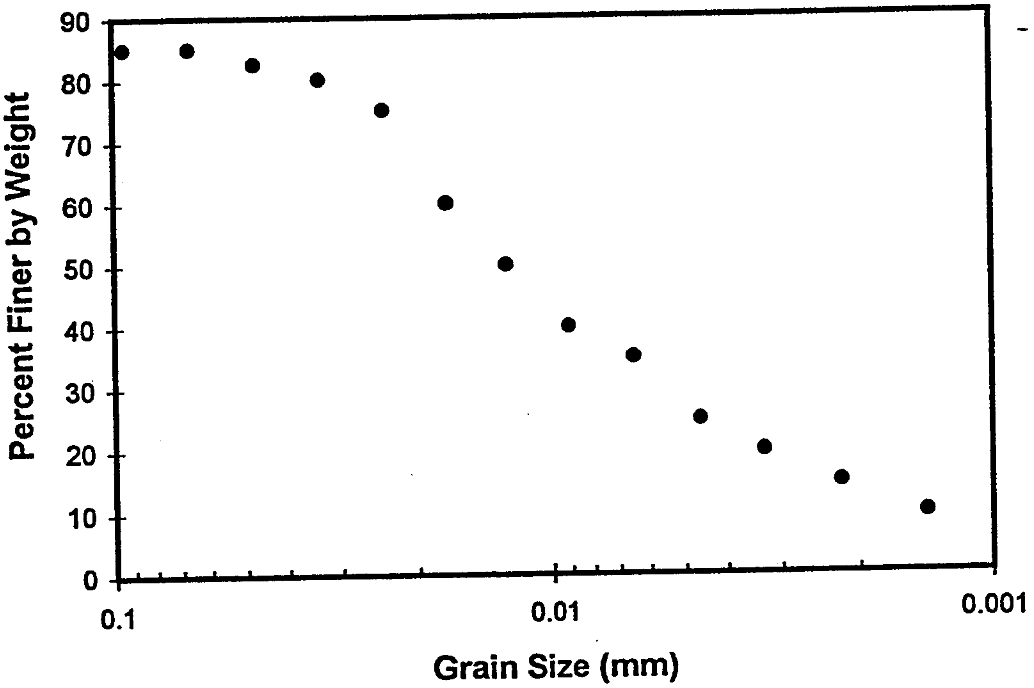


Figure B - 1 Grain size distribution of insoluble residue less than No. 200 sieve in sample D-05

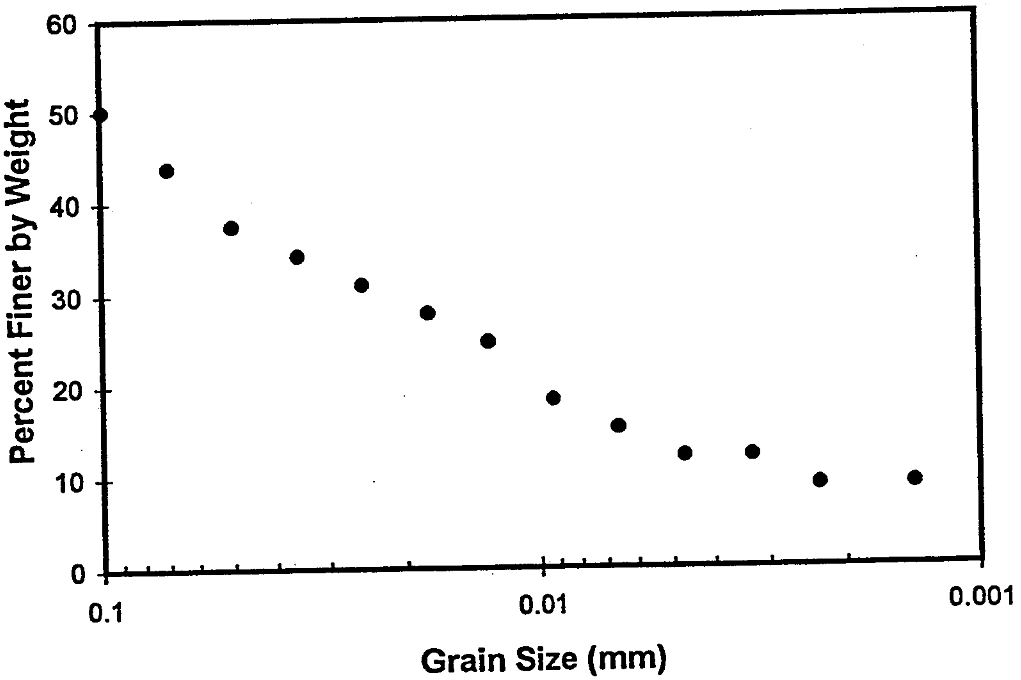


Figure B - 2 Grain size distribution of insoluble residue less than No. 200 sieve in sample D-09

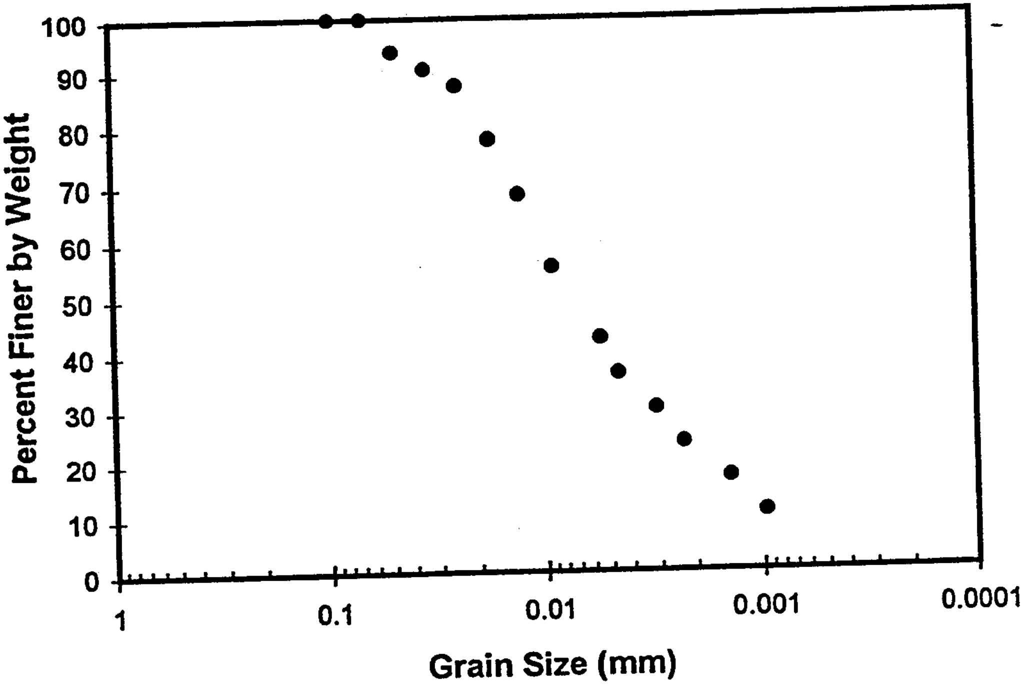


Figure B - 3 Grain size distribution of insoluble residue less than No. 200 sieve in sample D-11

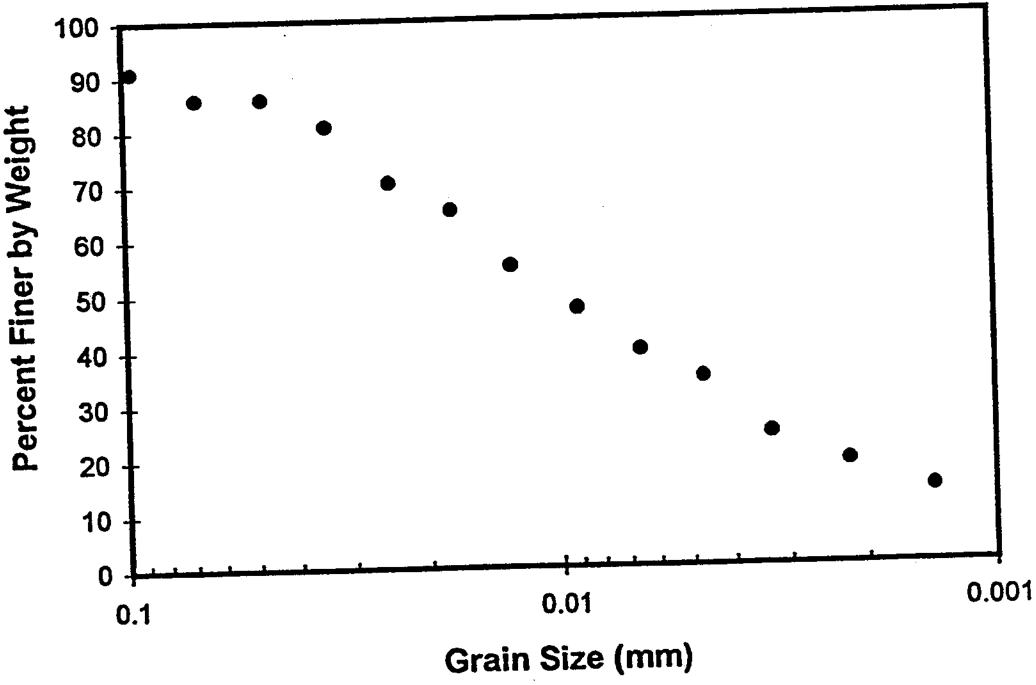


Figure B - 4 Grain size distribution of insoluble residue less than No.200 sieve in sample D-12

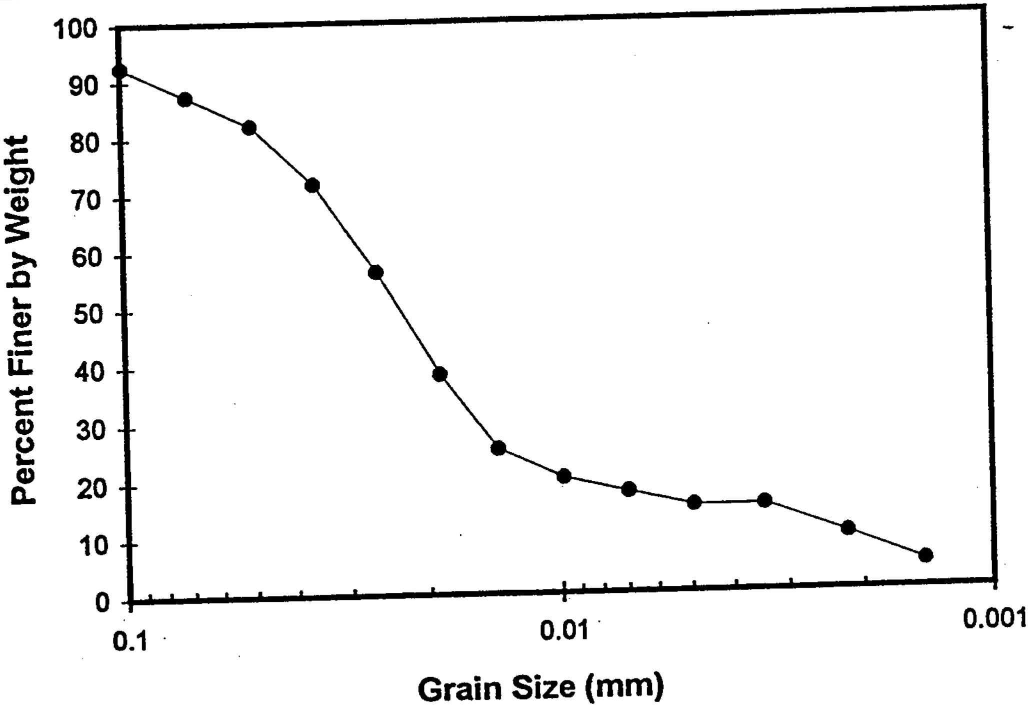


Figure B - 5 Grain size distributionof insoluble residue less than No. 200 sieve in sample D-013

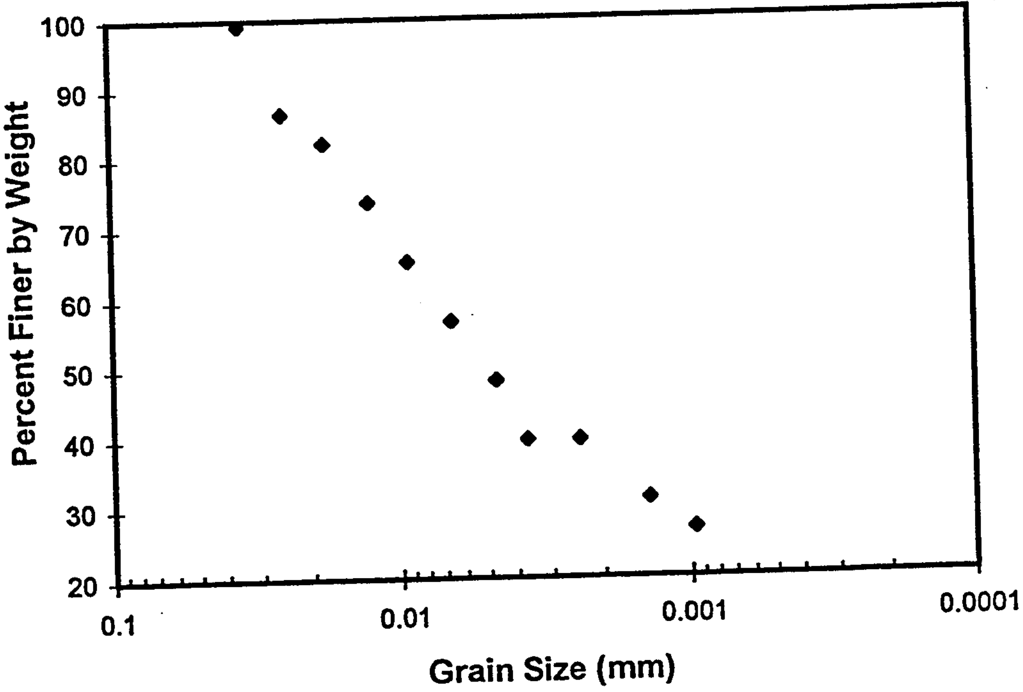


Figure B - 6 Grainsize distribution of insoluble residue less than No. 200 sieve in sample D-020

Appendix C

EDF and Pore size Distribution Curves,
Dolomite Aggregates, Phase I Analysis

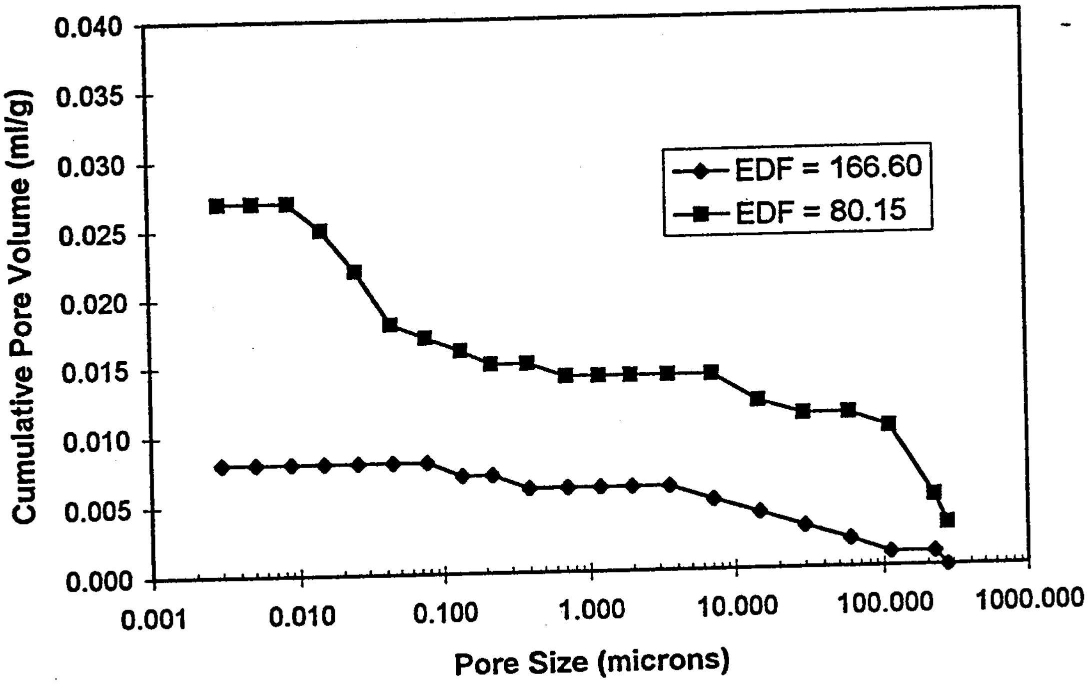


Figure C - 1 Pore size distribution for sample D - 01

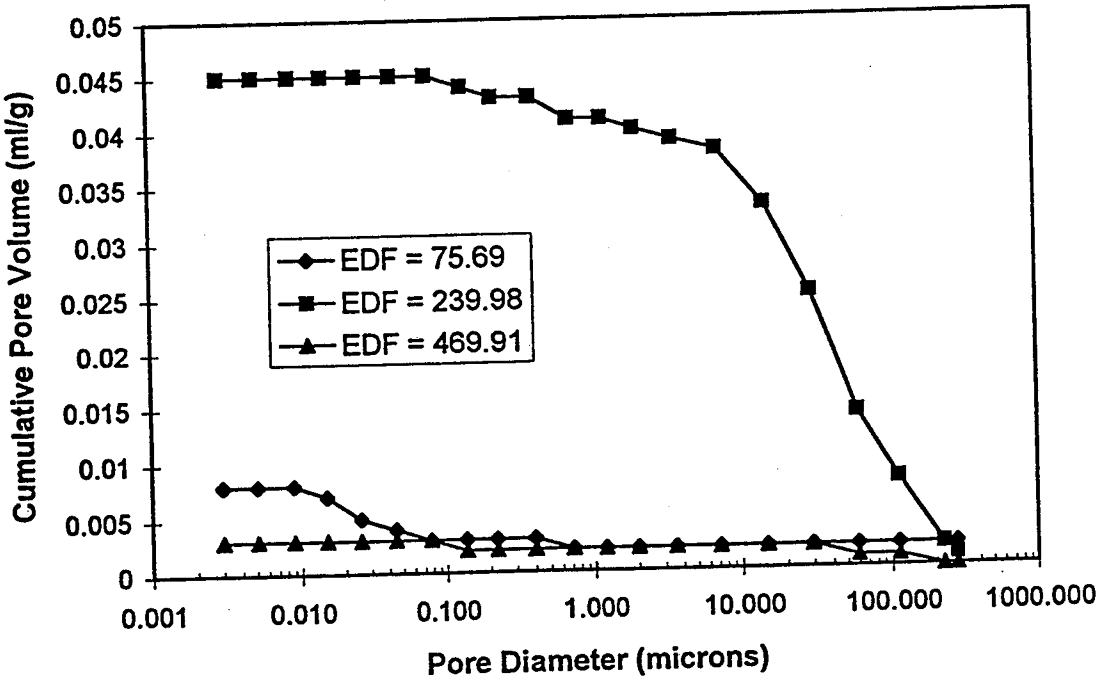


Figure C - 2 Pore size distribution for sample D - 02

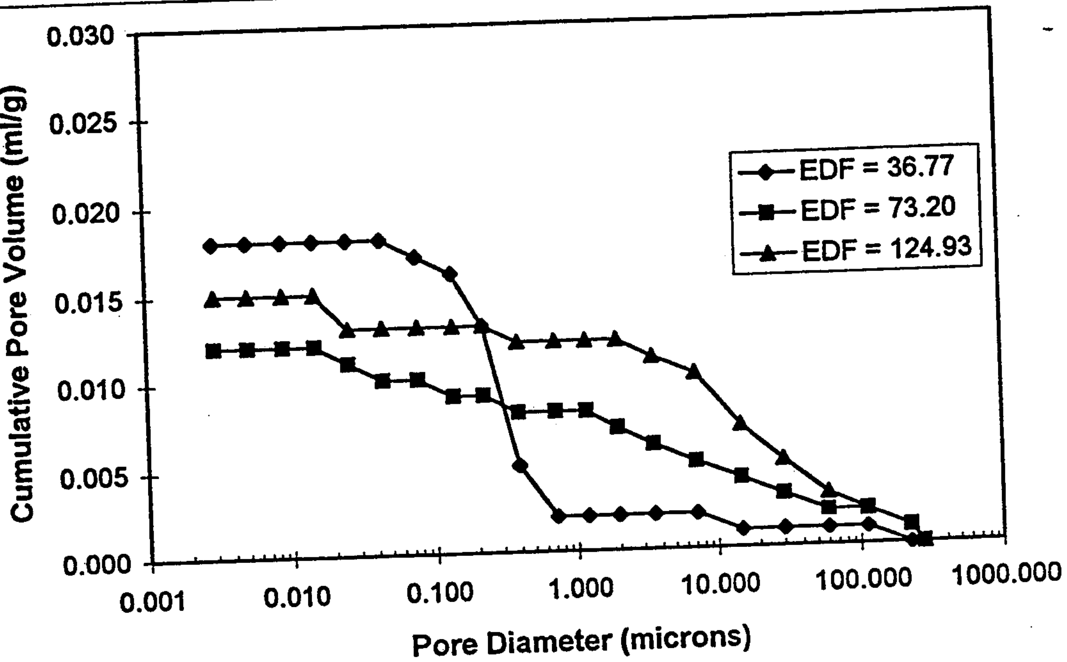


Figure C - 3 Pore size distribution for sample D - 03

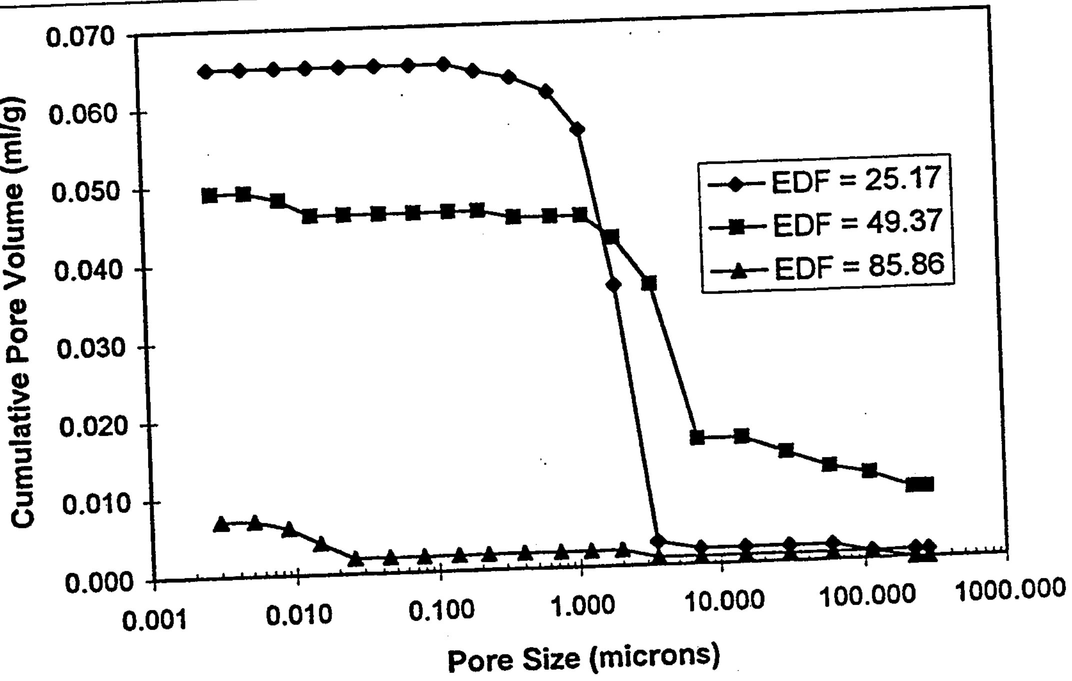


Figure C - 4 Pore size distribution for sample D-04

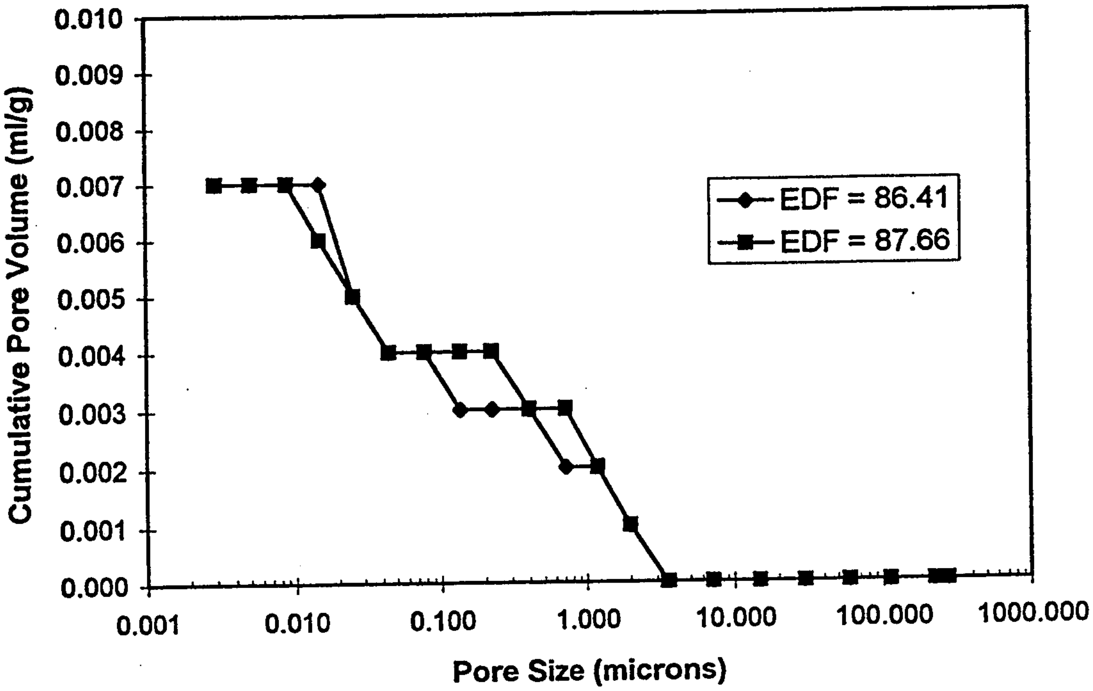


Figure C - 5 Pore size distribution foe sample D - 05

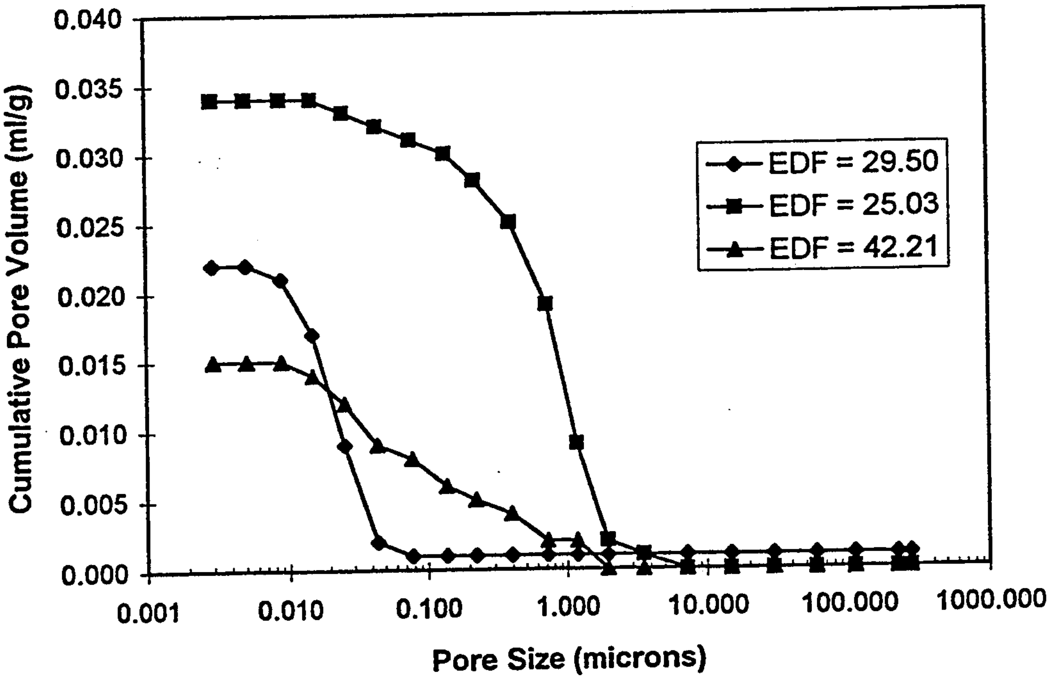


Figure C - 6 Pore size distribution for sample D - 07

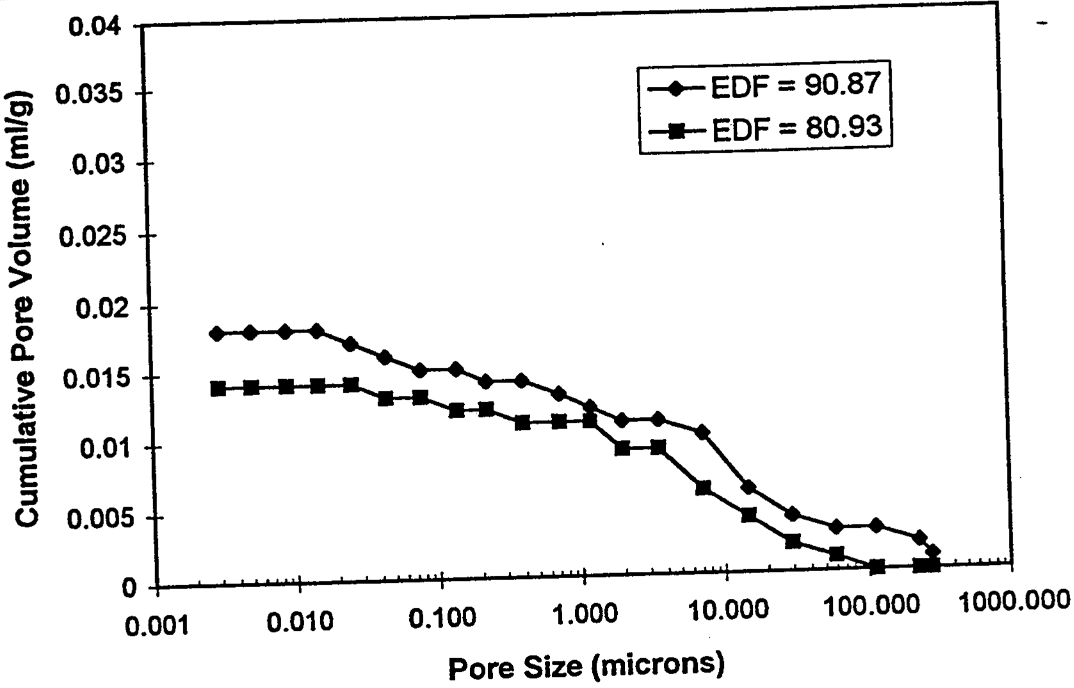


Figure C - 7 Pore size distribution for sample D - 08

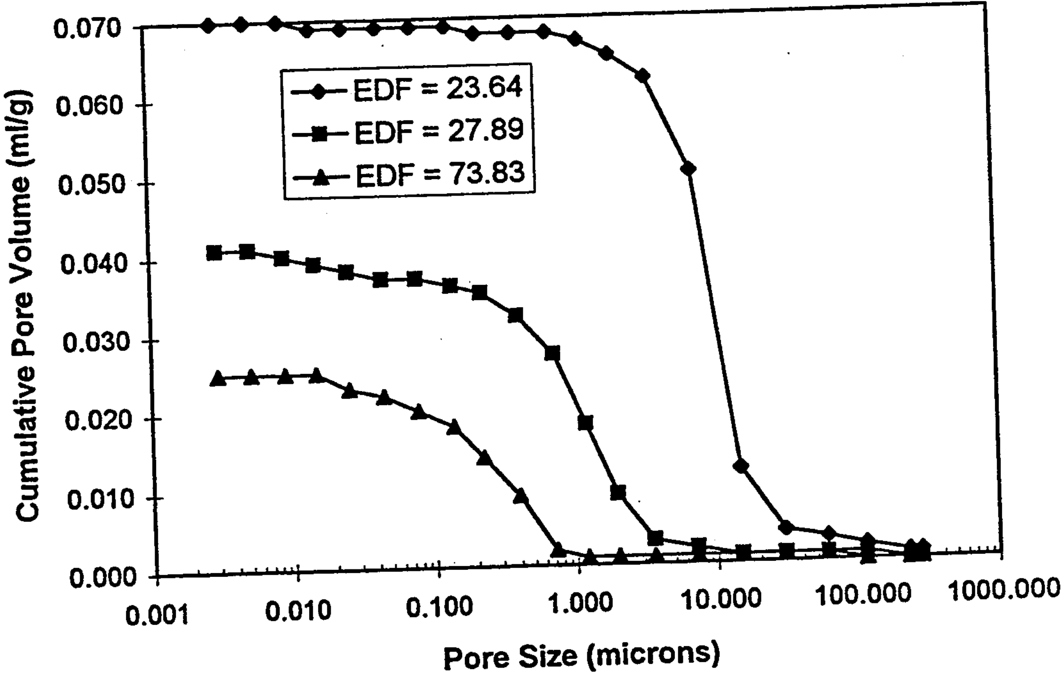


Figure C - 8 Pore size distribution for sample D - 09

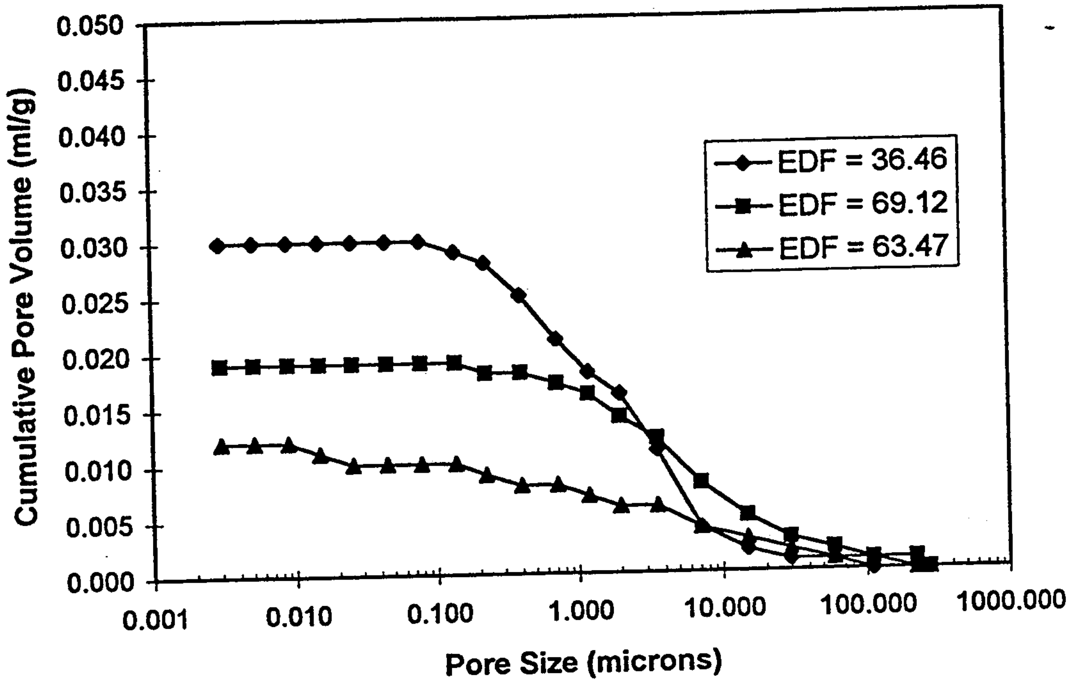


Figure C - 9 Pore size distribution for sample D - 10

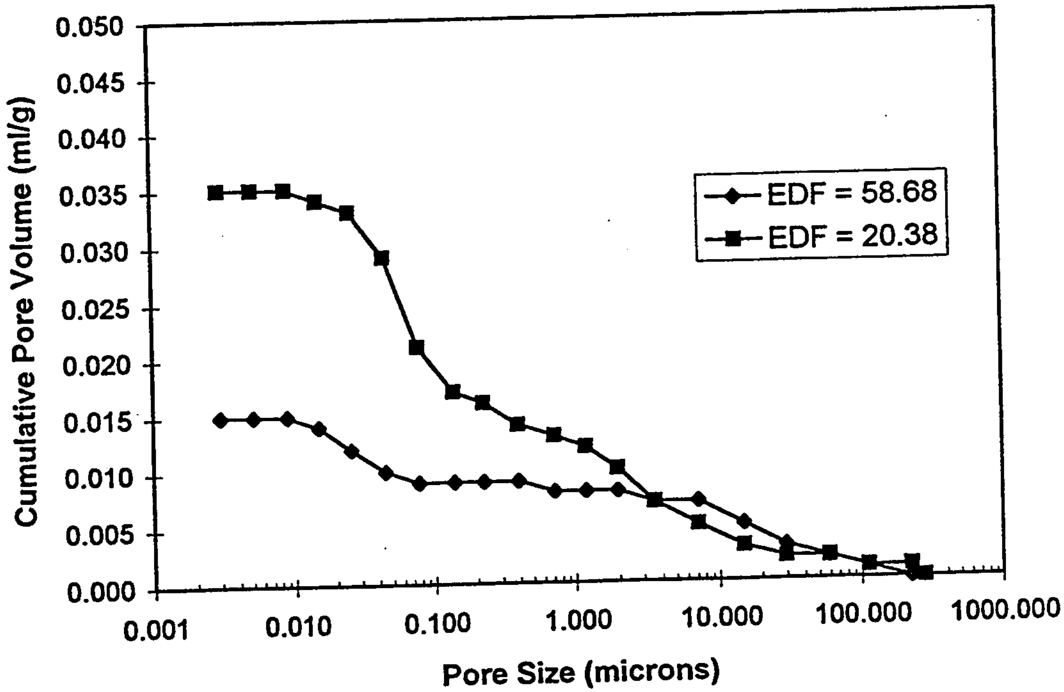


Figure C - 10 Pore size distribution for sample D - 11

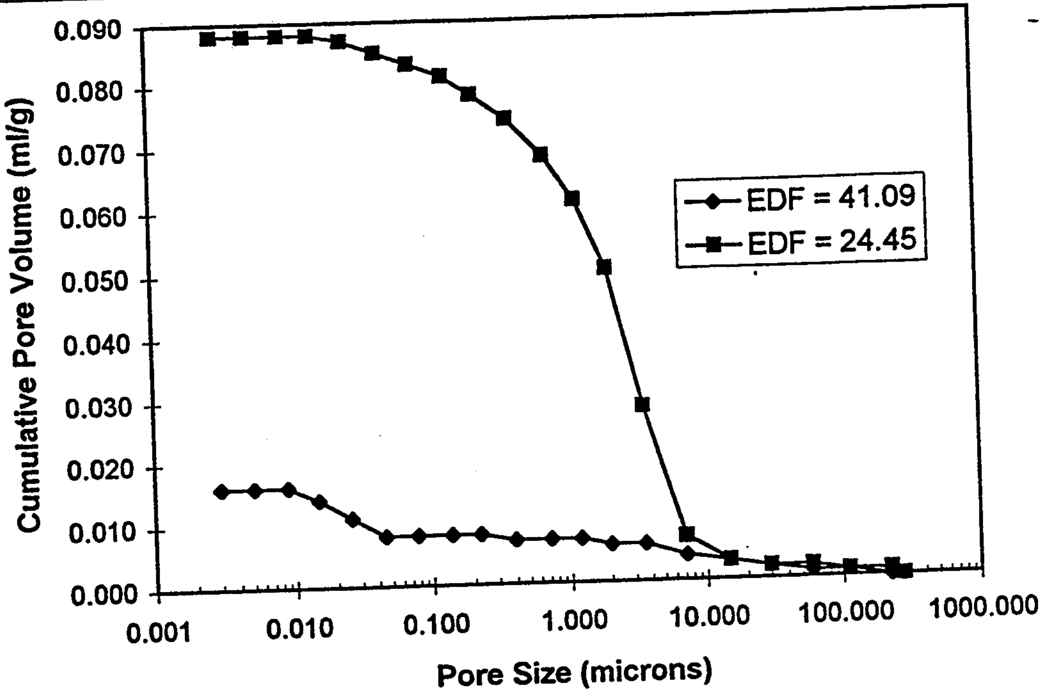


Figure C - 11 Pore size distribution for sample D - 12

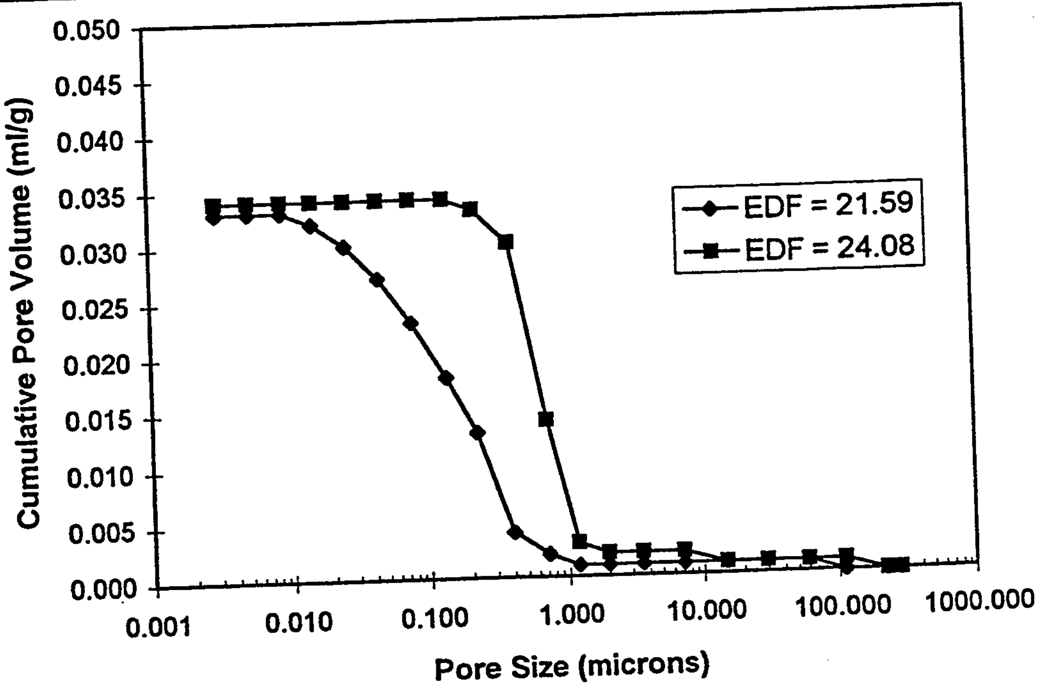


Figure C - 12 Pore size distribution for sample D - 13

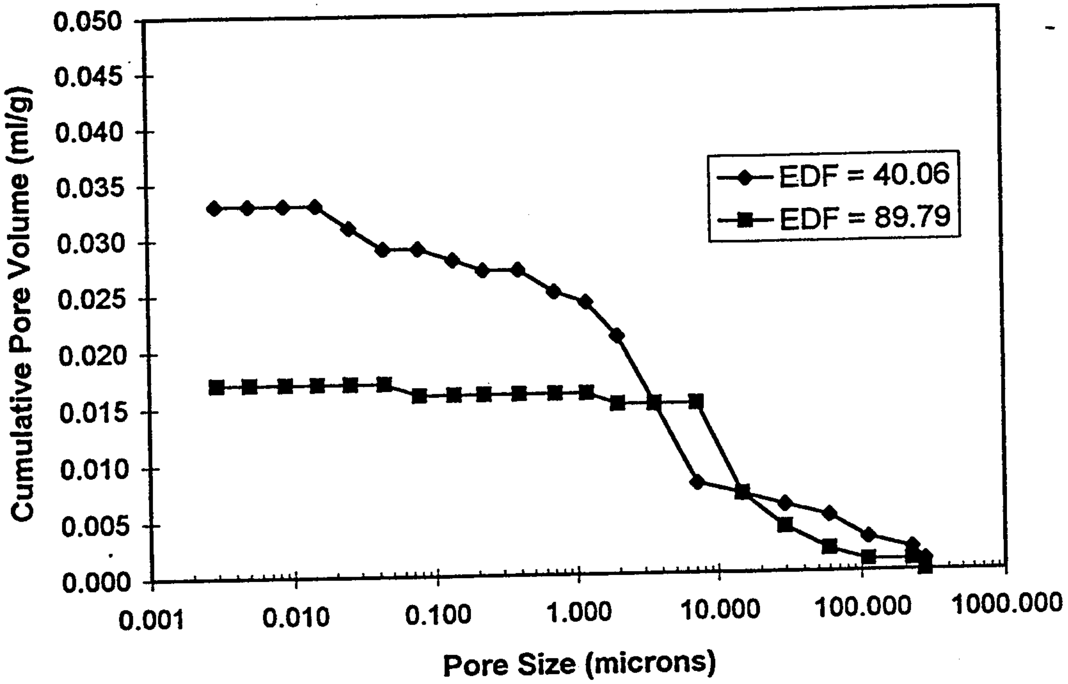


Figure C - 13 Pore size distribution for sample D - 15

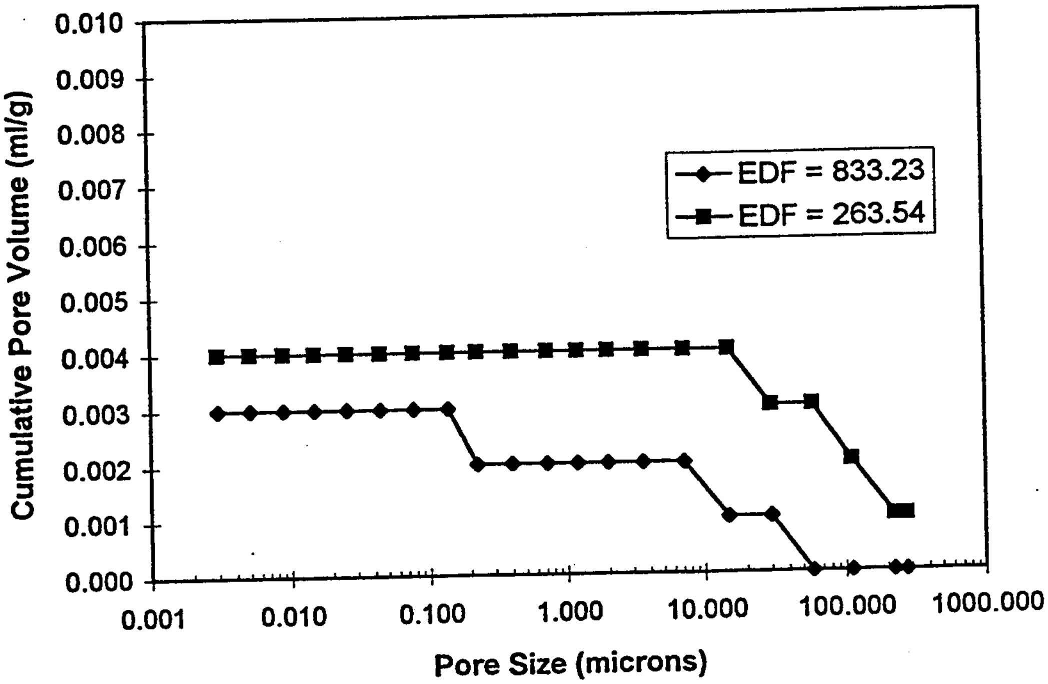


Figure C - 14 Pore size distribution for sample D - 17

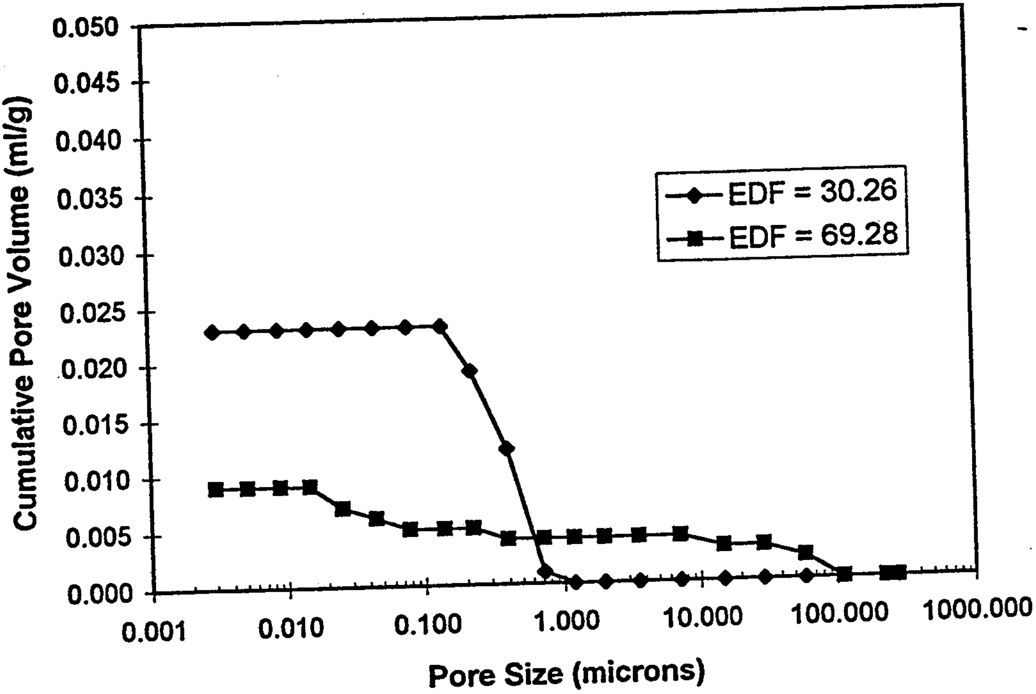


Figure C - 15 Pore size distribution for sample D - 18

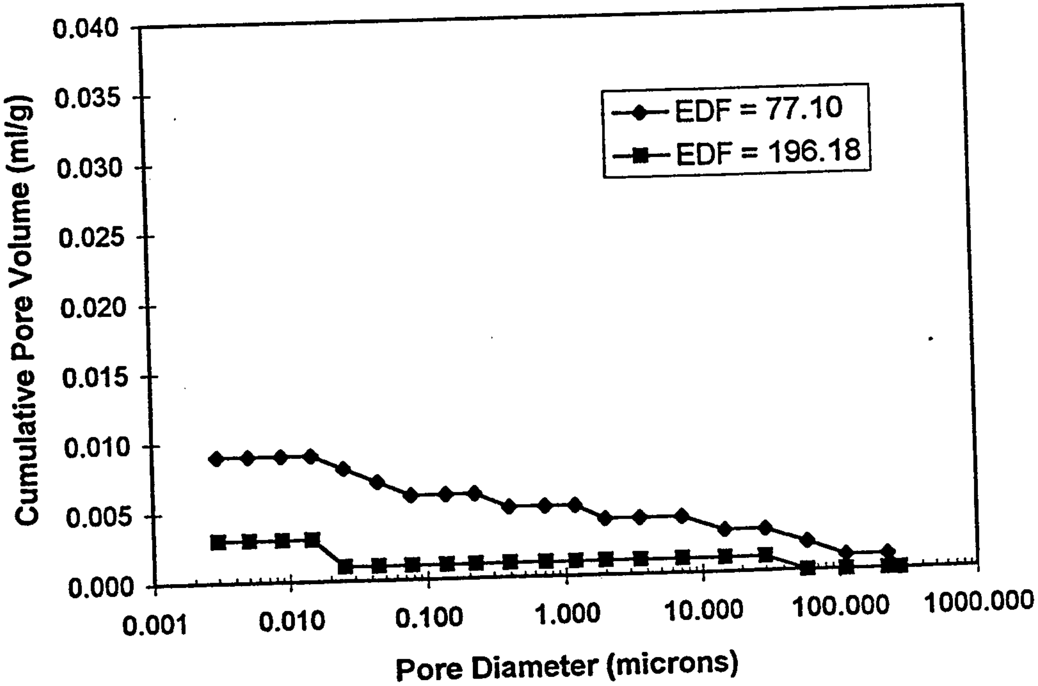


Figure C - 16 Pore size distribution for sample D - 19

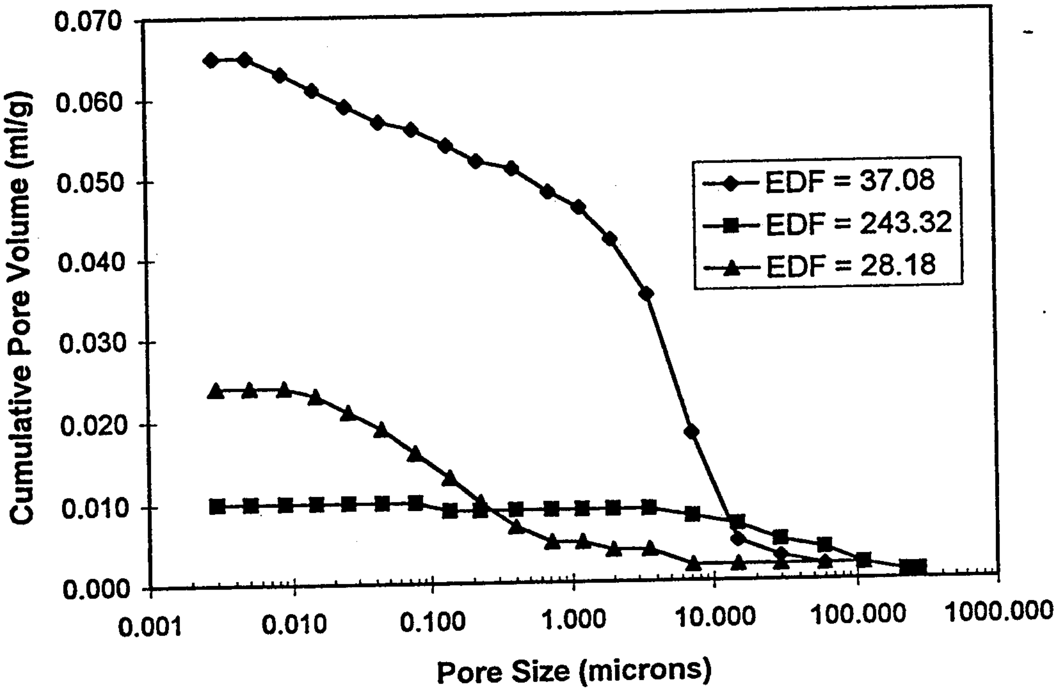


Figure C - 17 Pore size distribution for sample D - 20

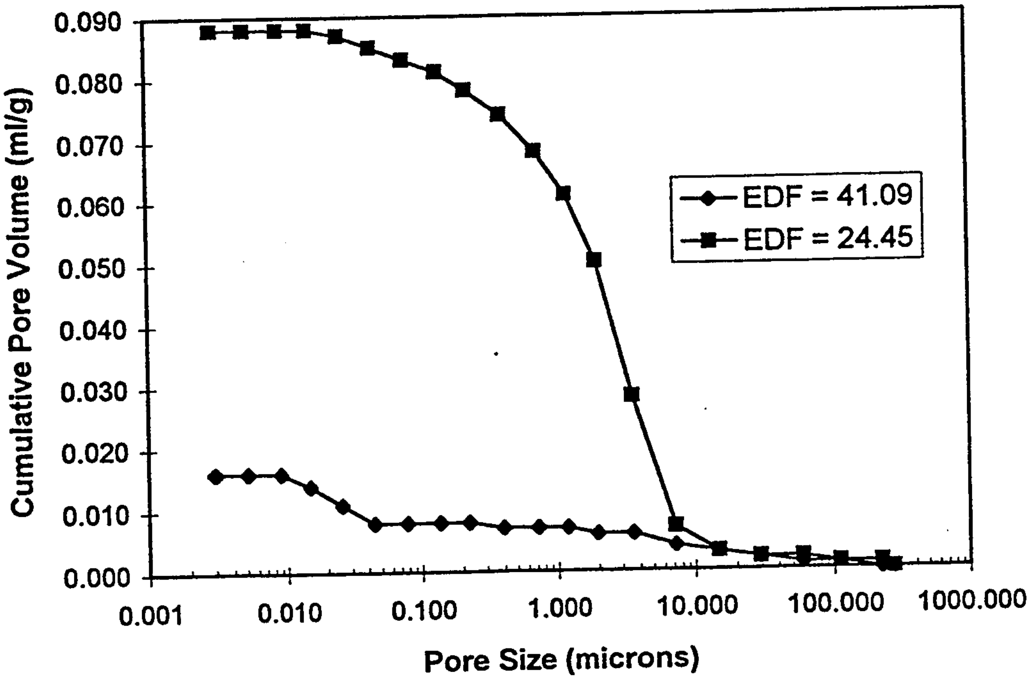


Figure C - 18 Pore size distribution for sample D - 21

Appendix D

EDF and Pore Size Distribution Curves,

D-26 and 27 Different Texture Samples

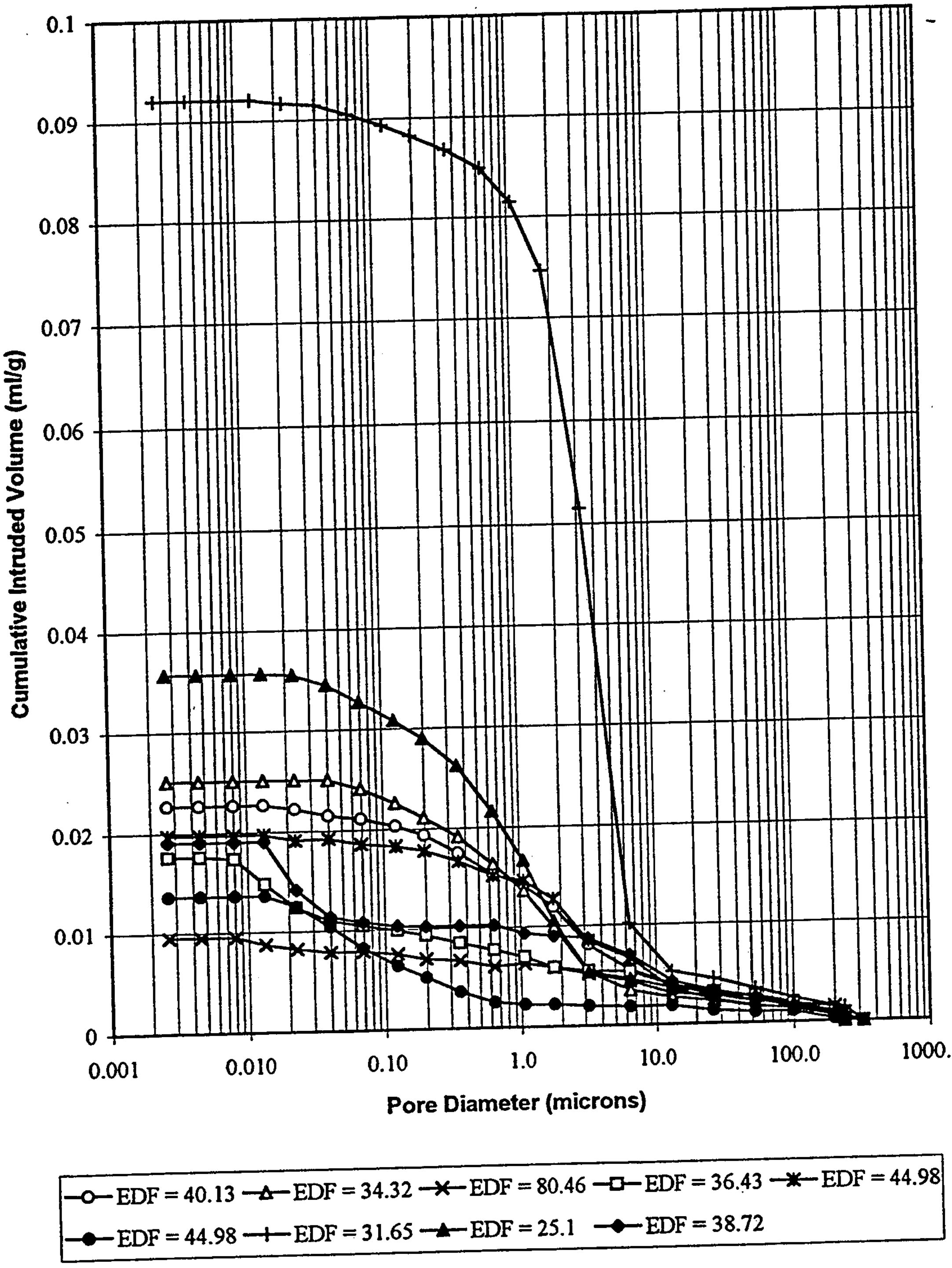


Figure D-1. Pore Size Distribution of Samples From Ledge 102.

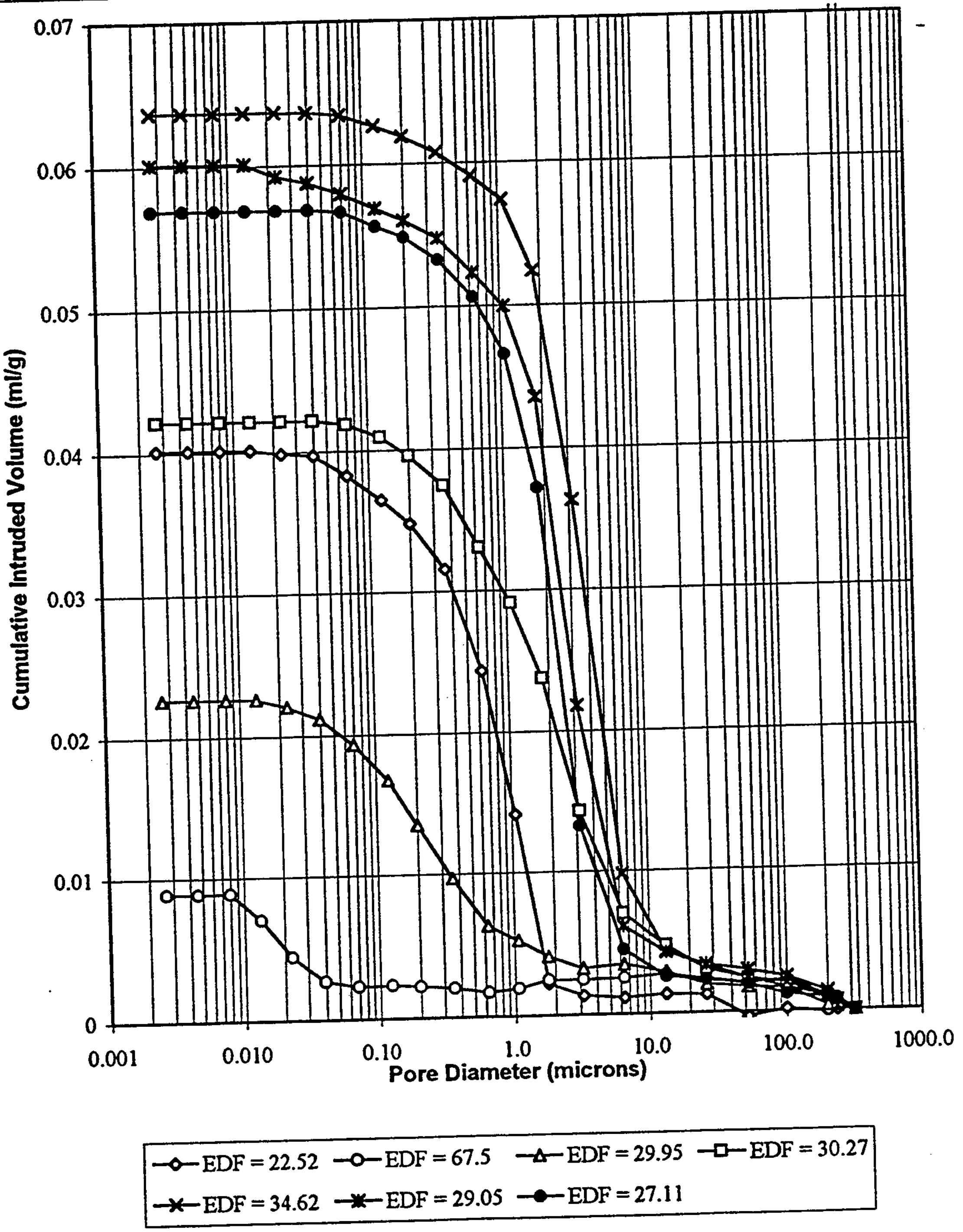


Figure D-2. Pore Size Distribution of Samples From Ledges 2-3 (dark grained).

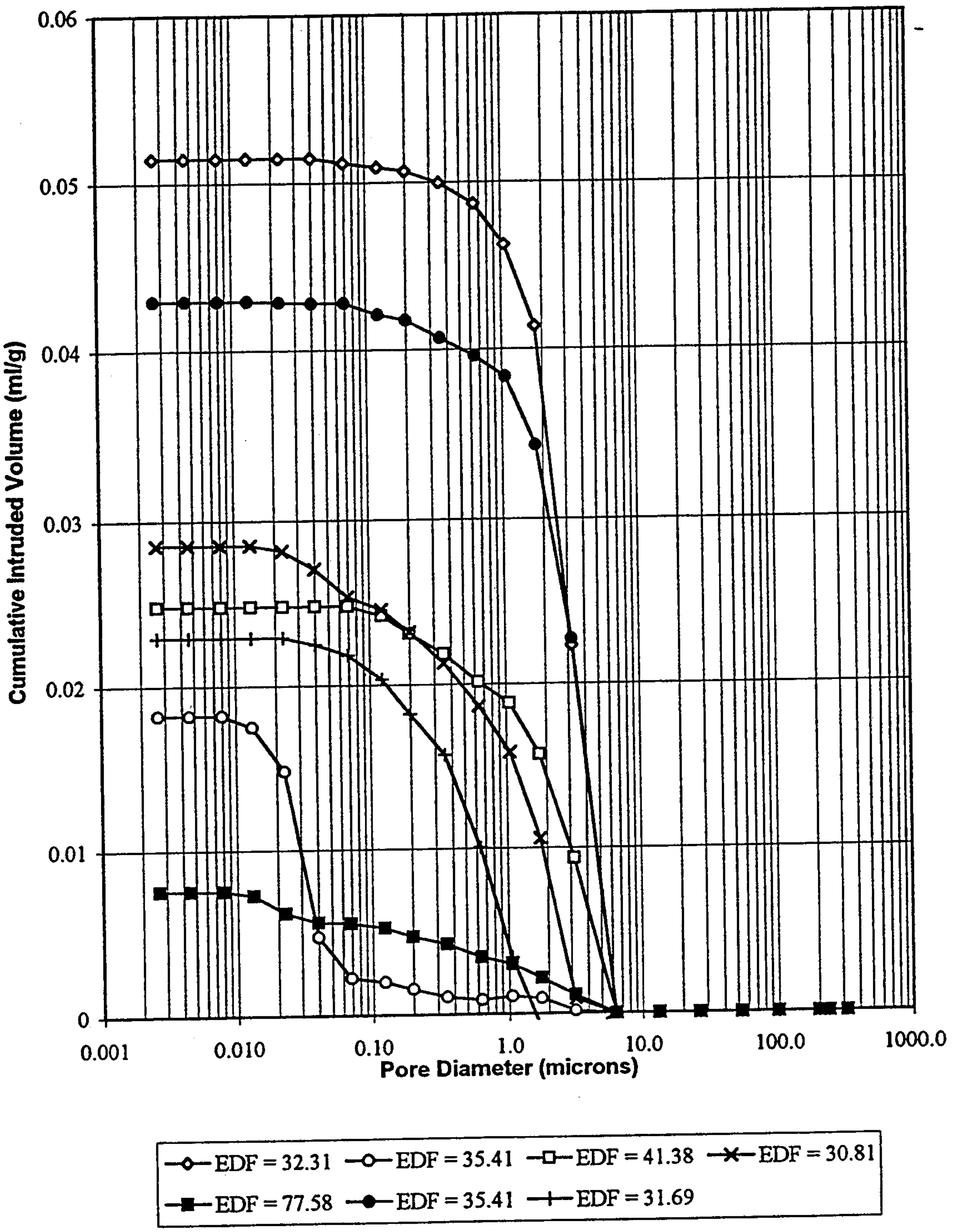


Figure D-3. Pore Size Distribution of Samples From Ledges 2-3 (fine grained).

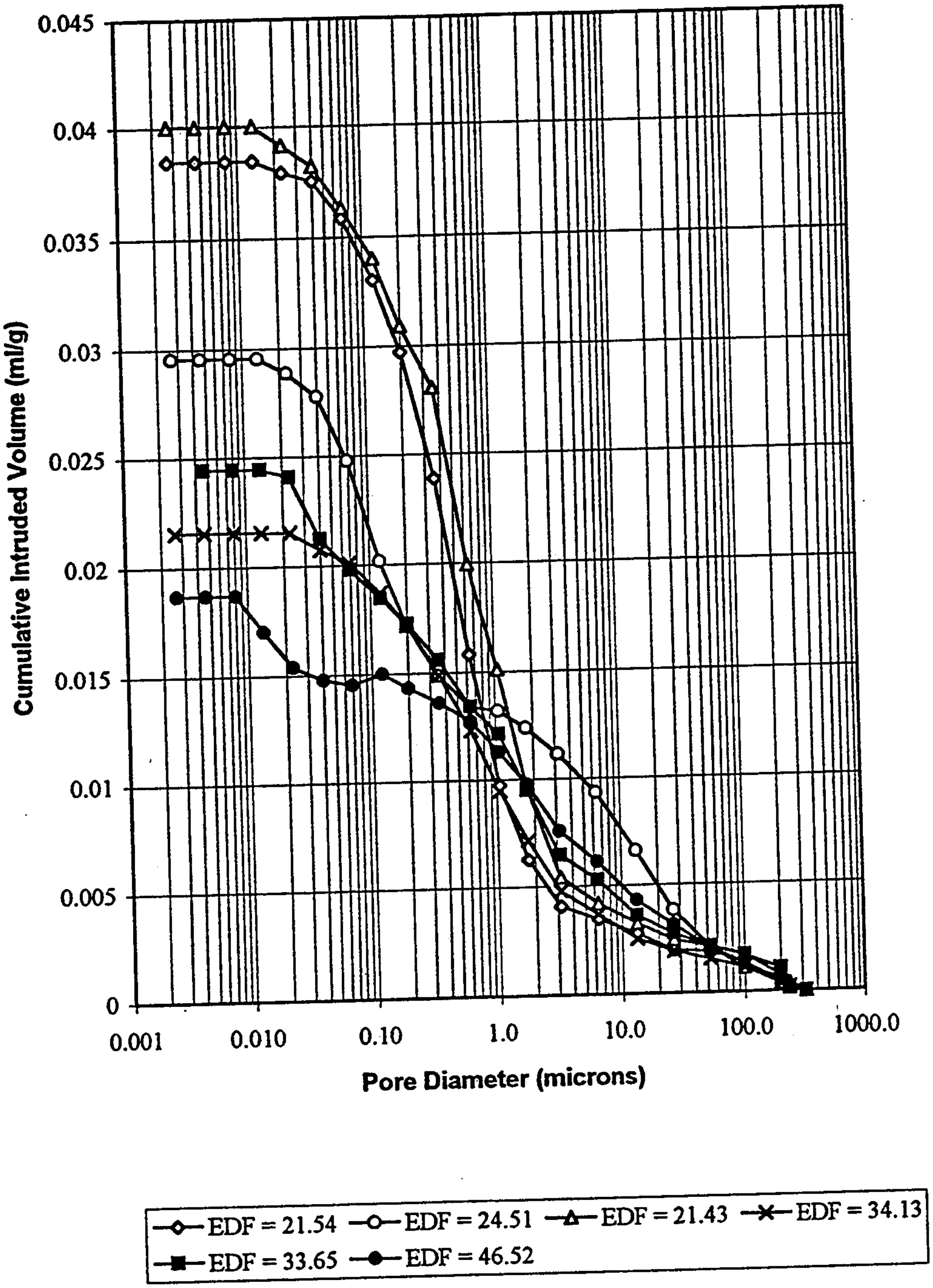


Figure D-4. Pore size distribution of samples from ledge 5.

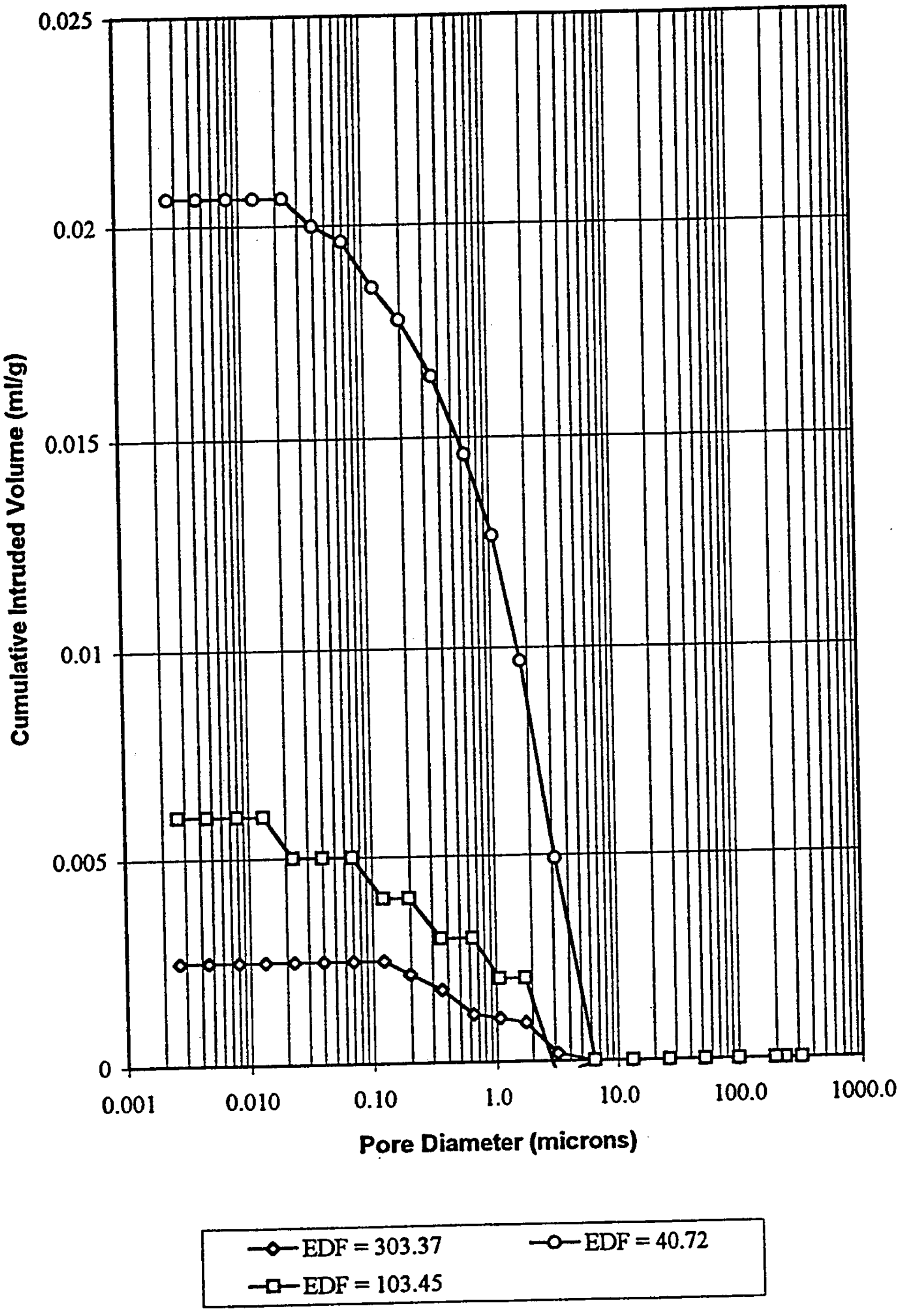


Figure D-5. Pore size distribution of samples from ledge 6A.

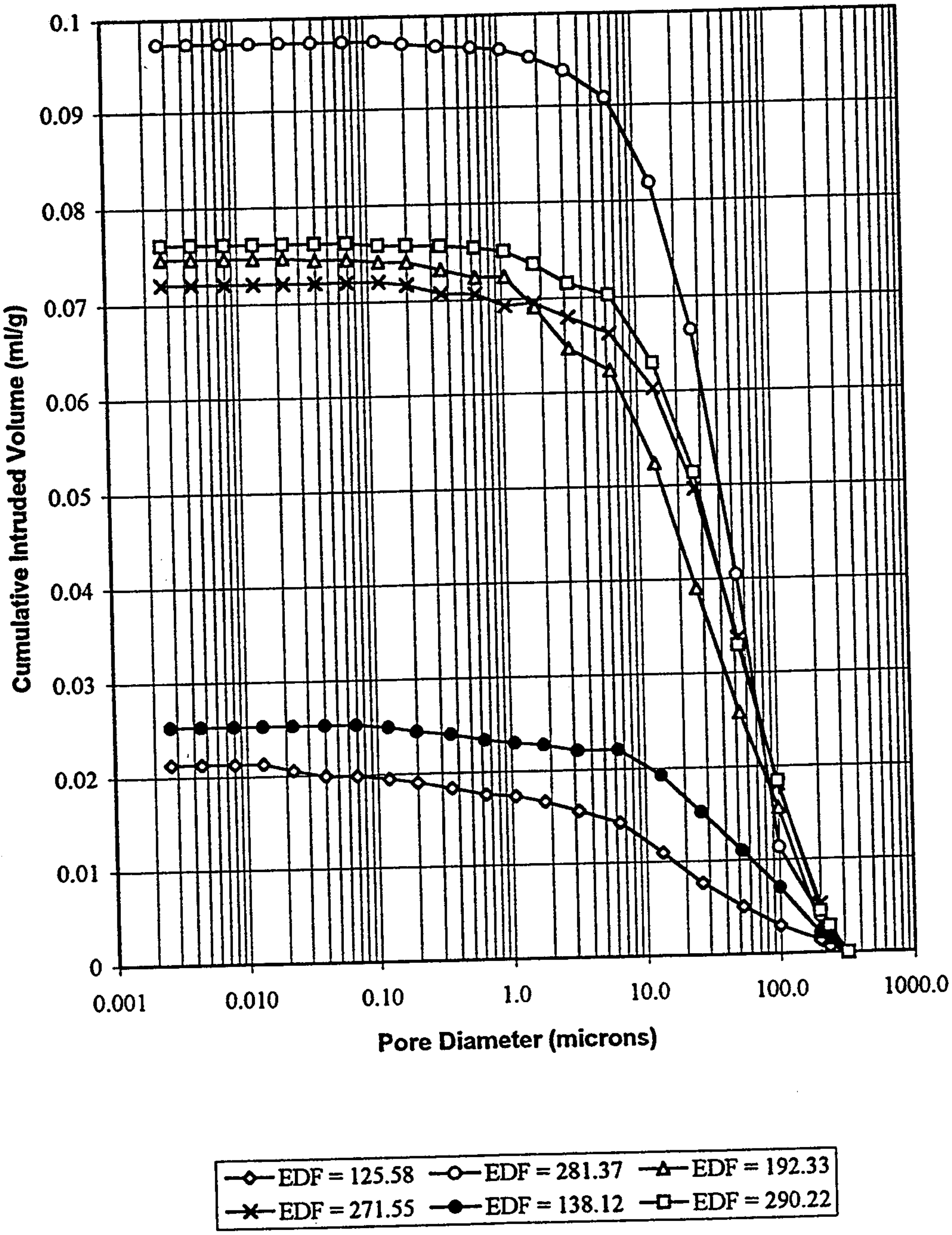


Figure D-6. Pore size distribution of samples from ledge 6B.

Appendix E

Photographs of Electron Microprobe Analysis

Figure E-1. Microprobe Photo D-05, Silicon

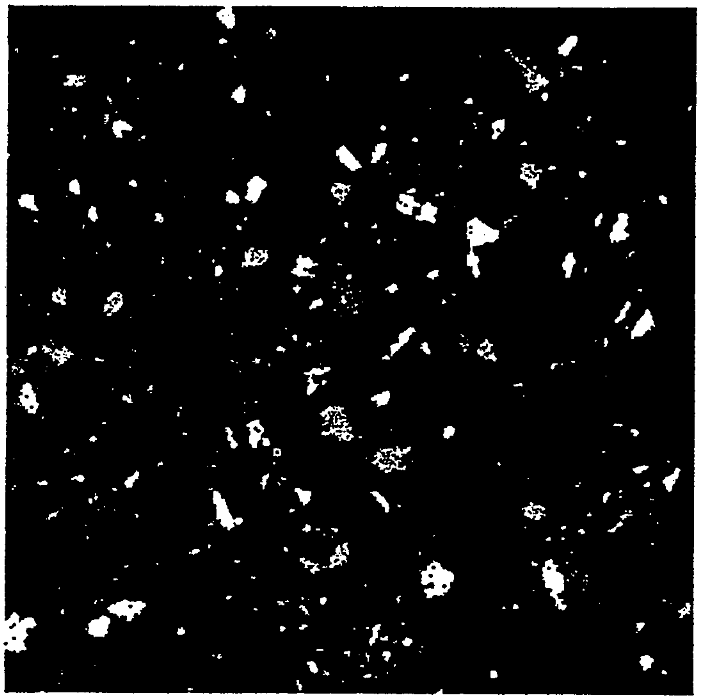


Figure E-2. Microprobe Photo D-05, Magnesium

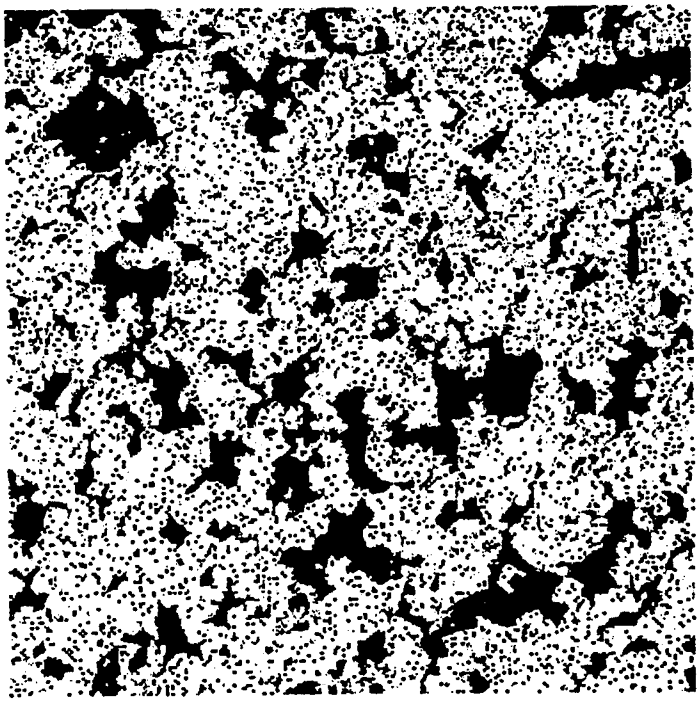


Figure E-3. Microprobe Photo D-05, Calcium

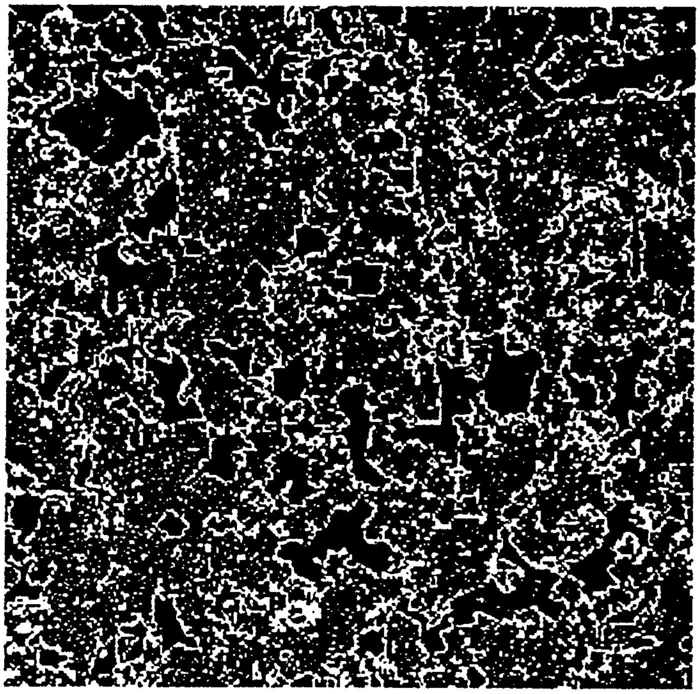


Figure E-4. Microprobe Photo D-05, Aluminum

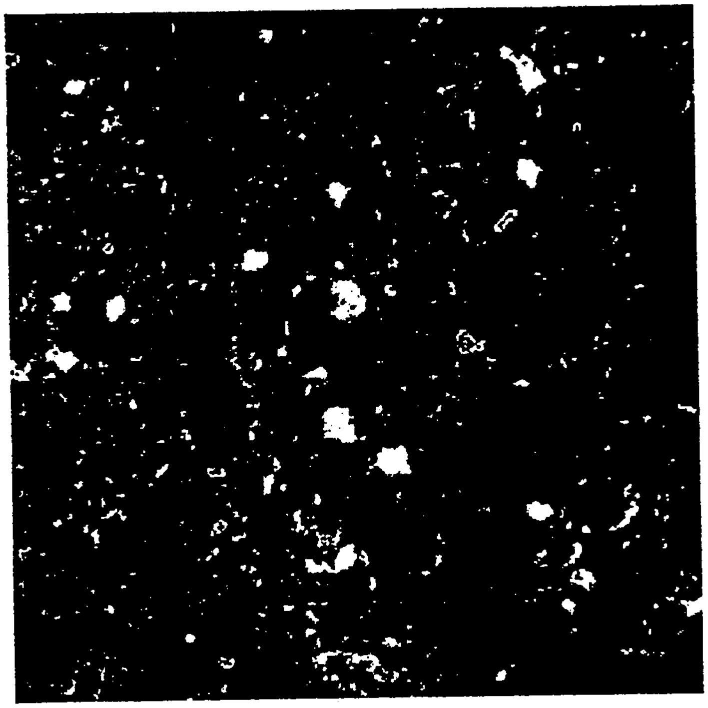


Figure E-5.   Microprobe Photo D-10, Magnesium

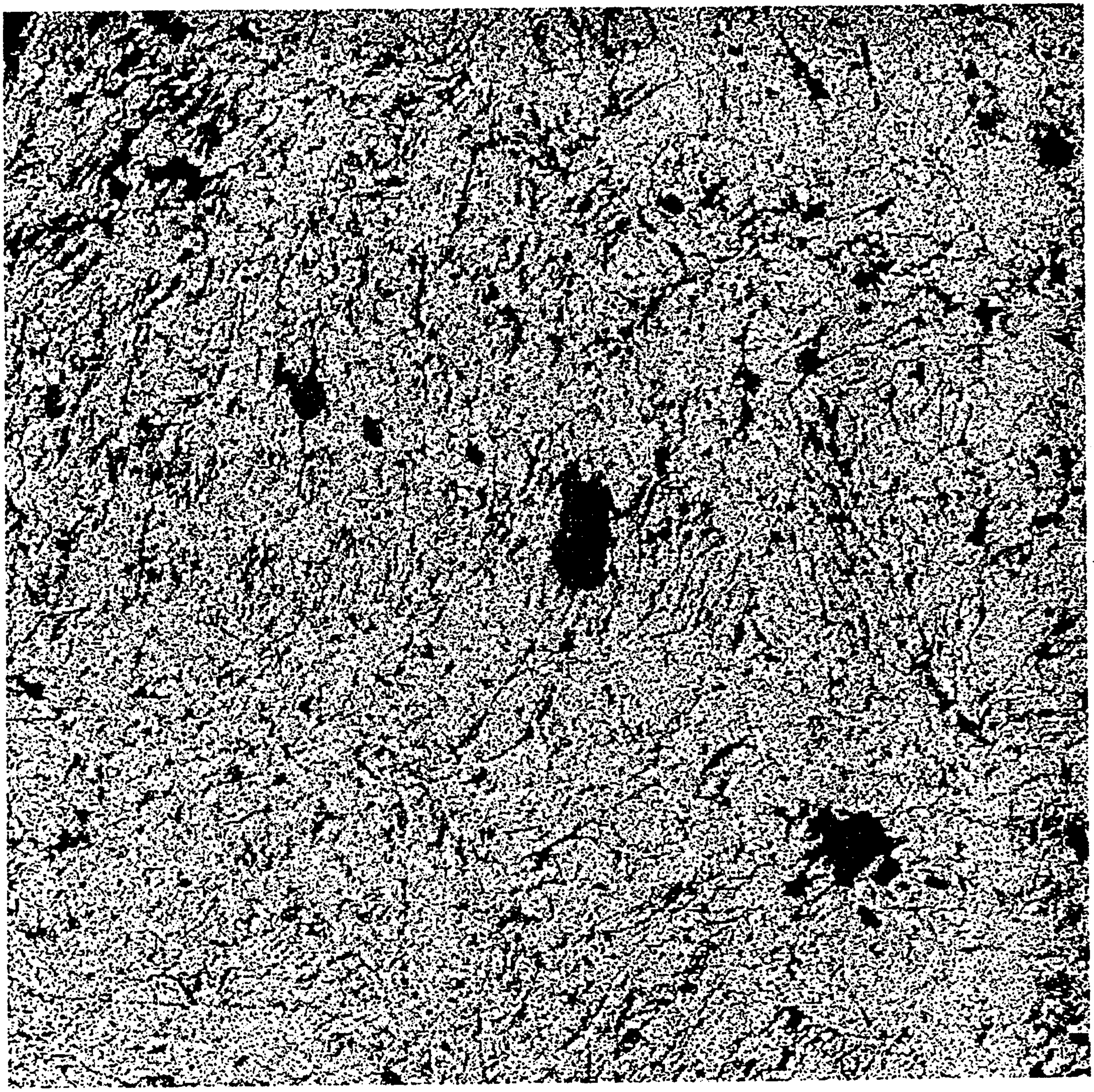


Figure E-6. Microprobe Photo D-10, Calcium

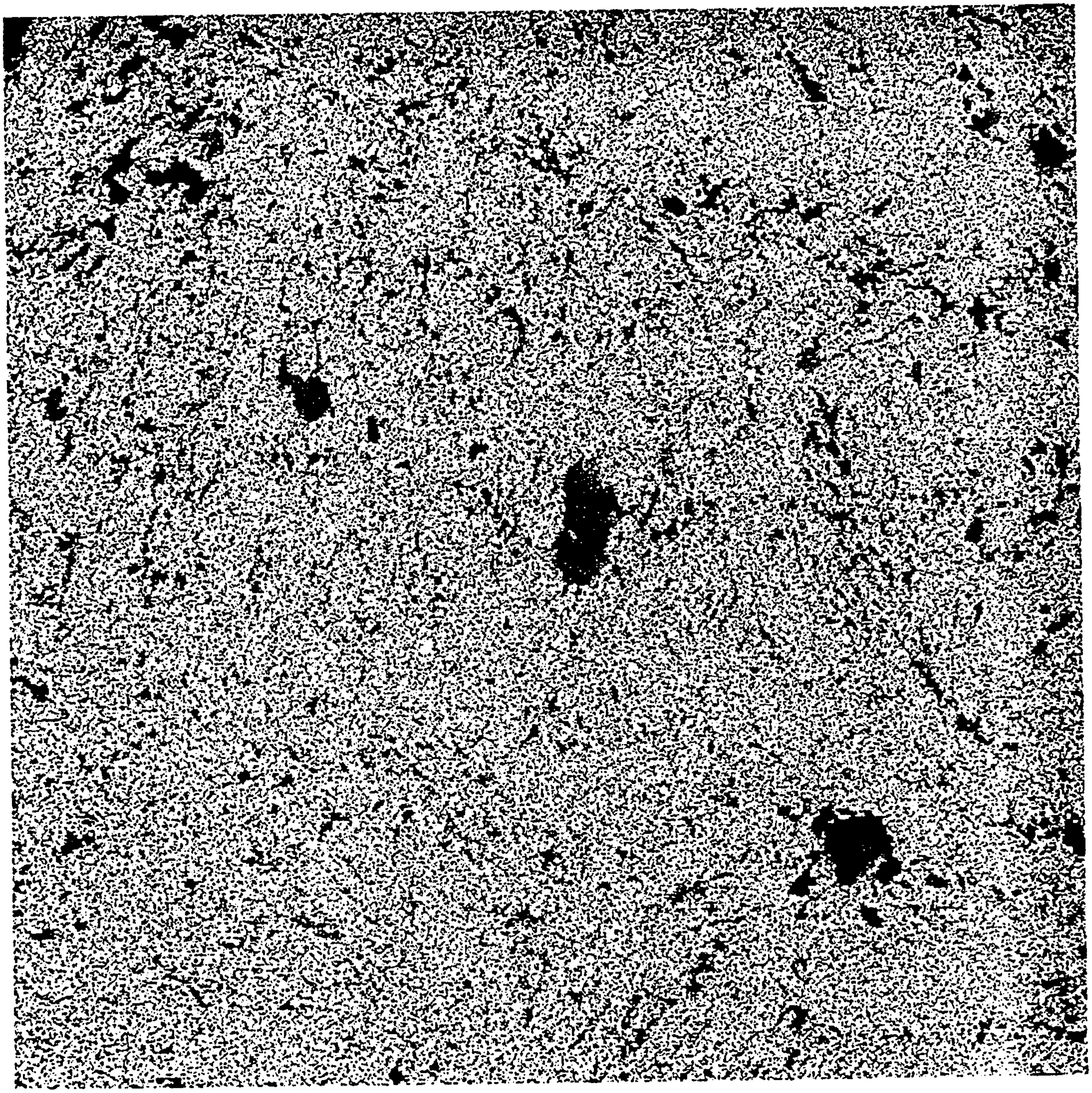


Figure E-7. Microprobe Photo D-10, Silicon

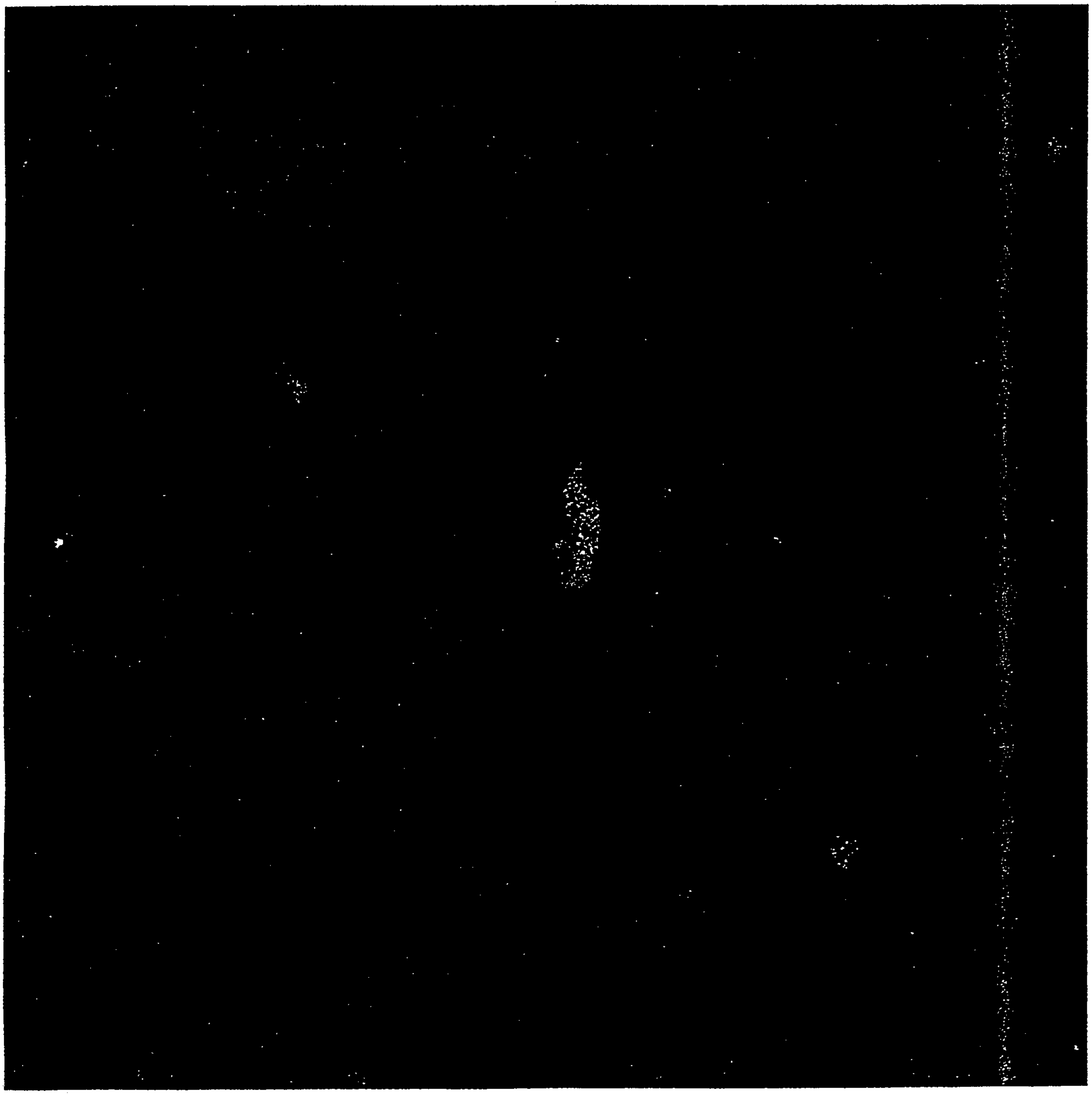


Figure E-8. Microprobe Photo D-10, Aluminum

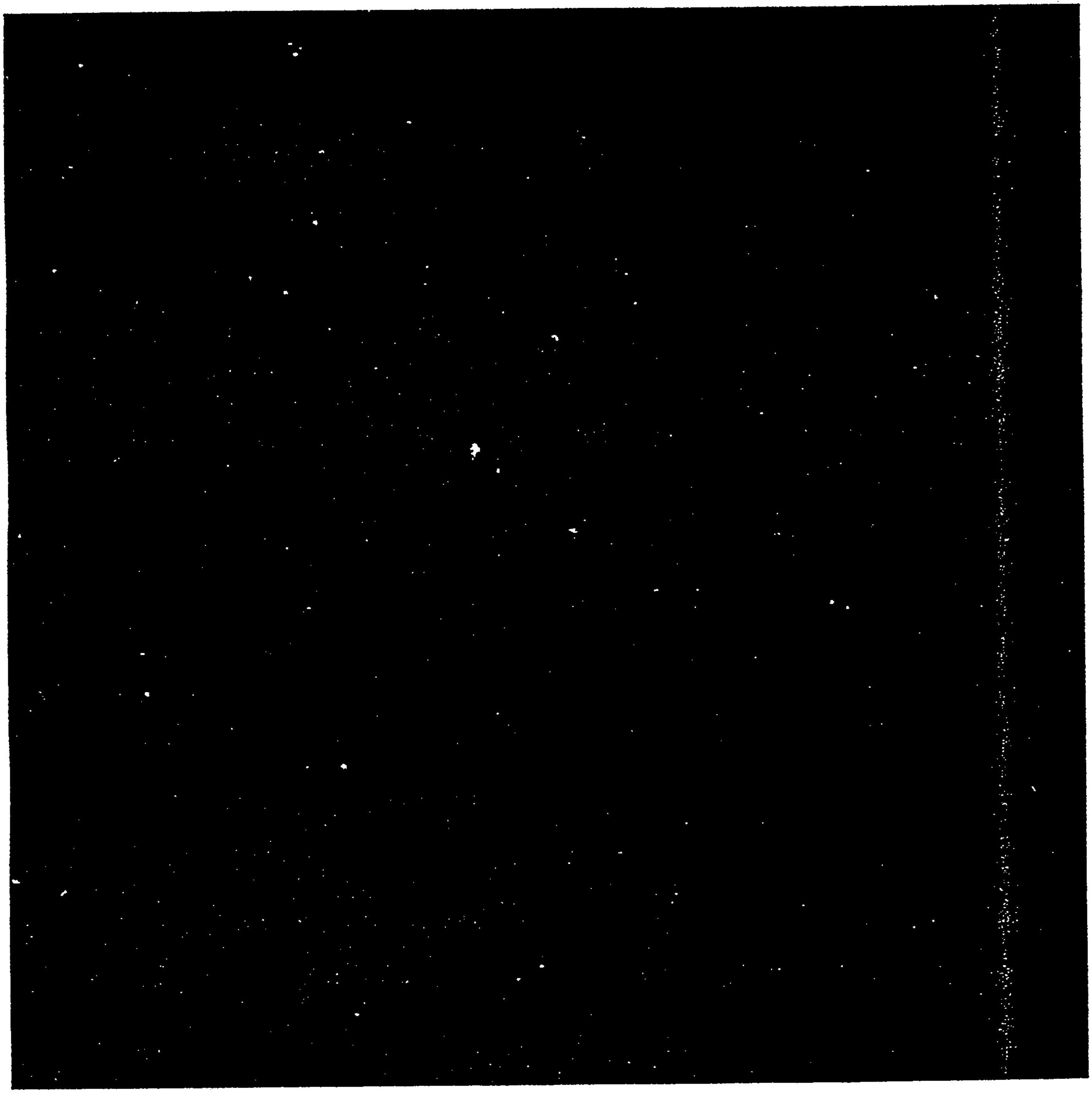


Figure E-9. Microprobe Photo D-13, Magnesium

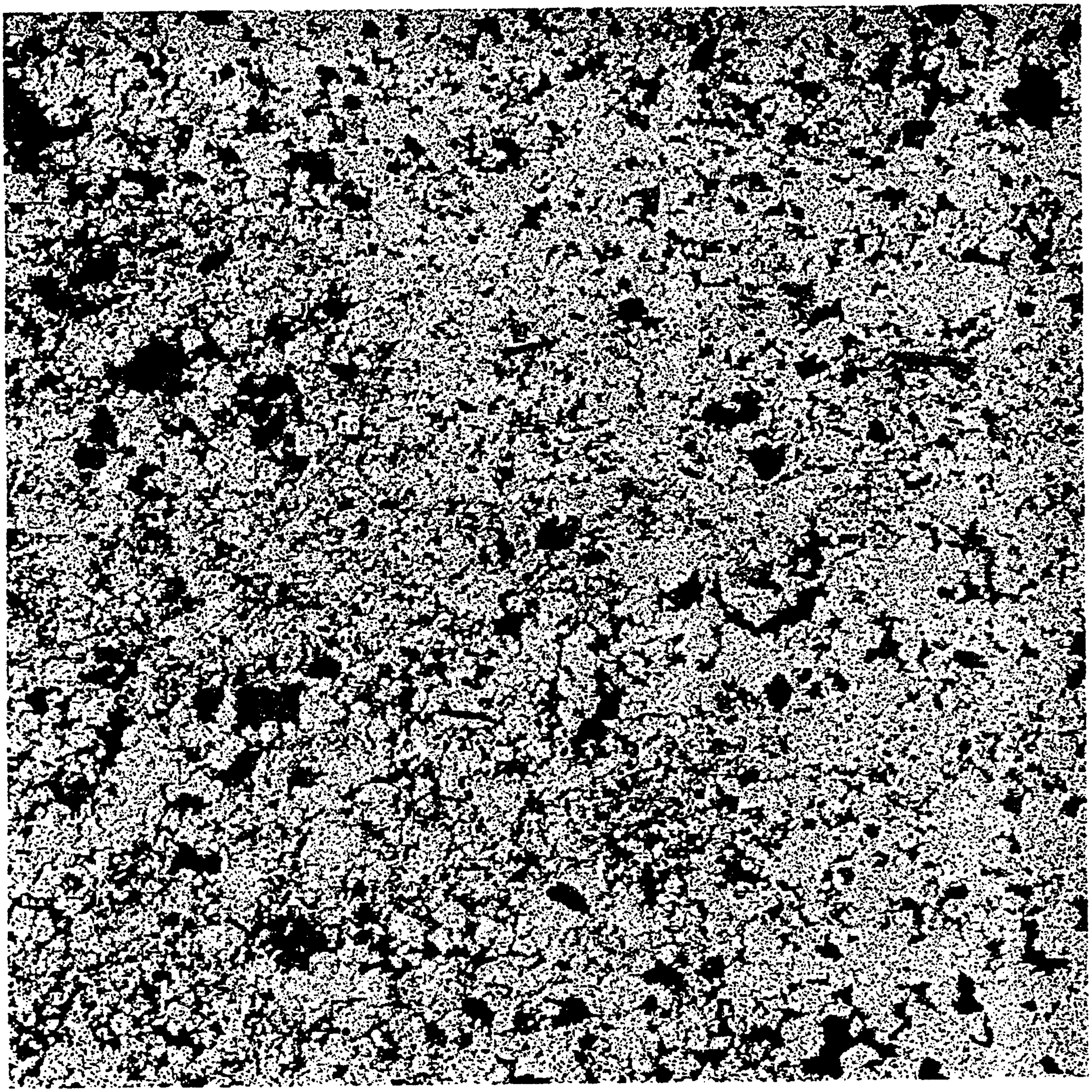


Figure E-10. Microprobe Photo D-13, Calcium

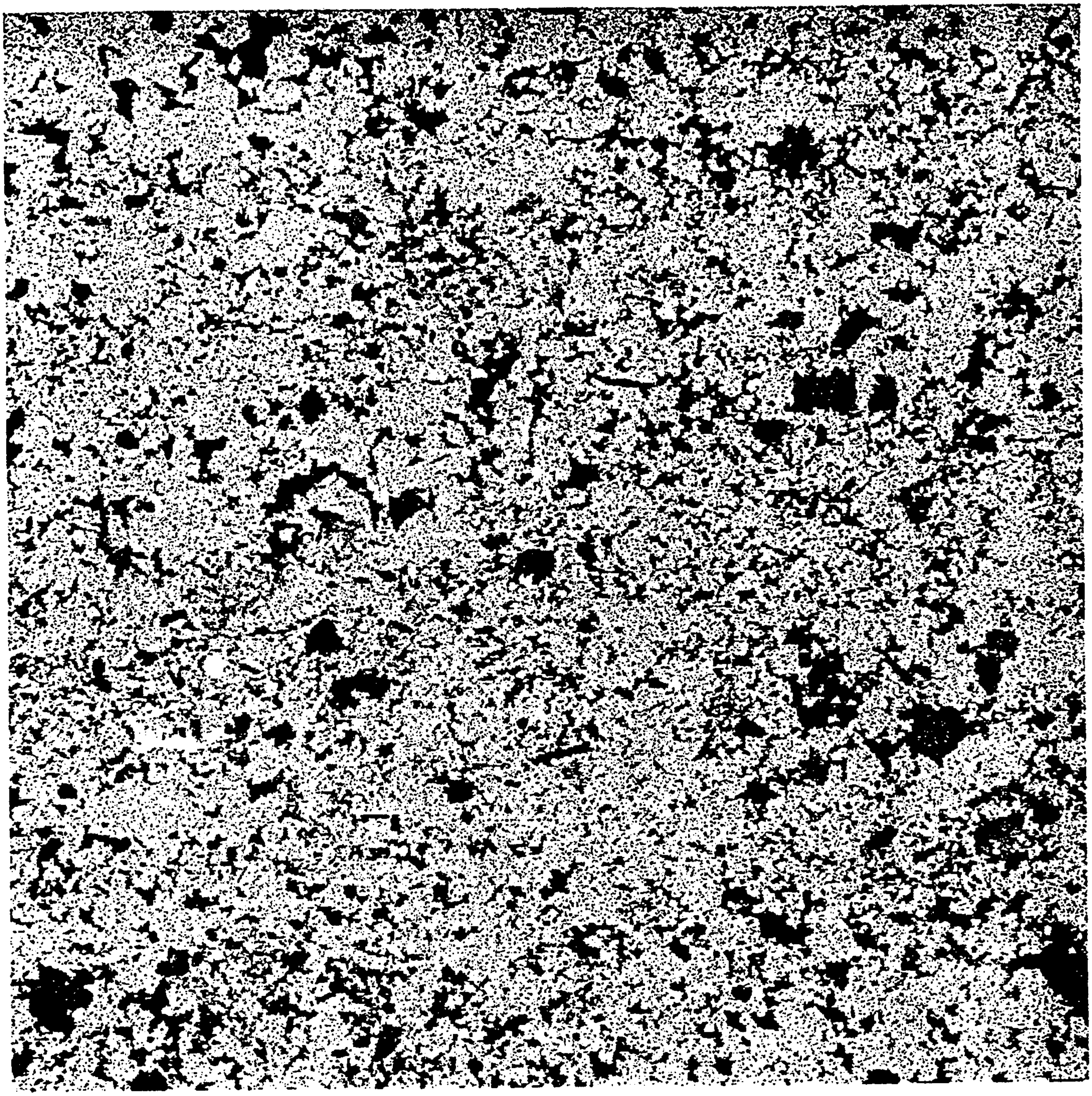


Figure E-11. Microprobe Photo D-13, Silicon

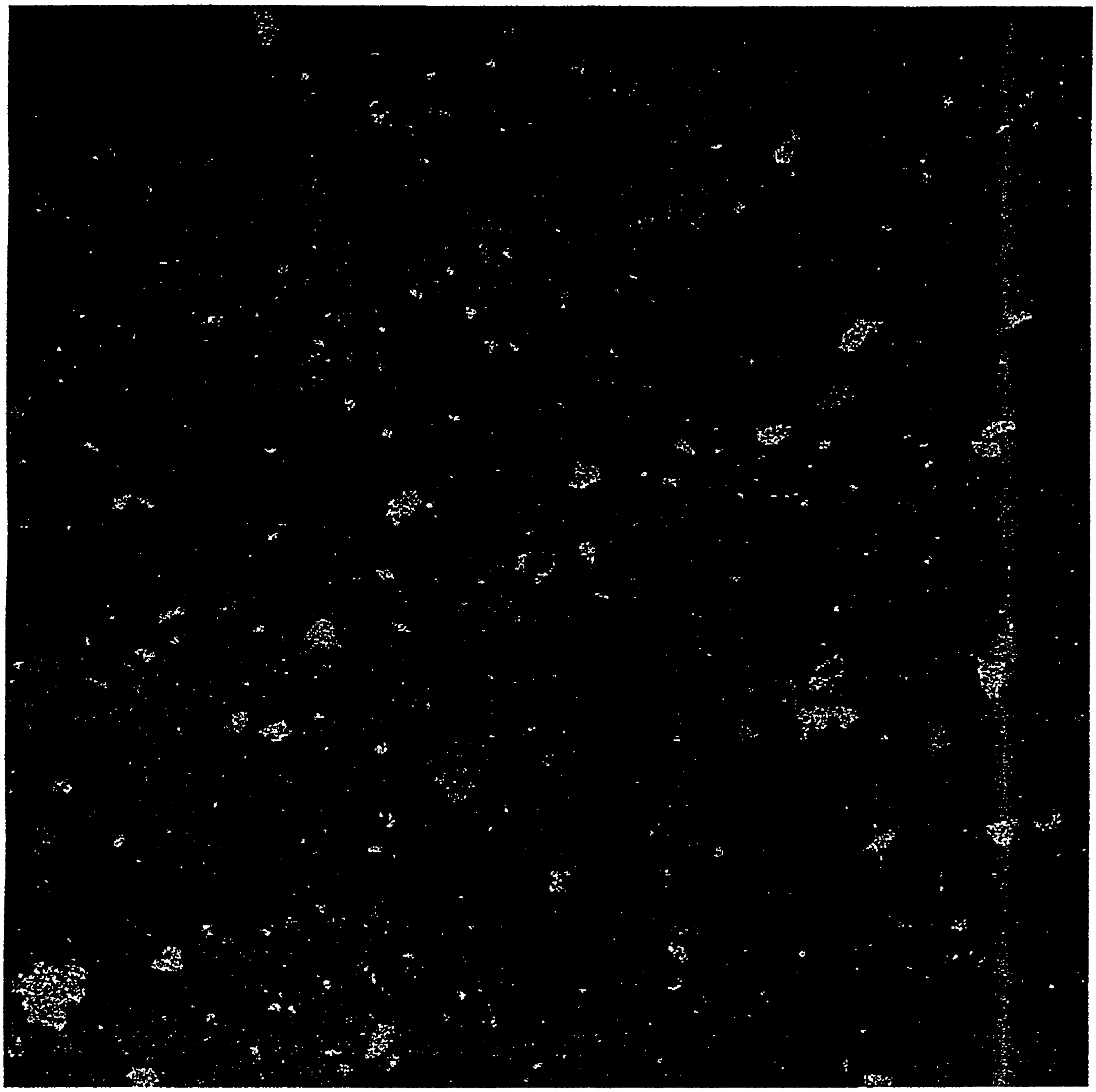


Figure E-12. Microprobe Photo D-13, Aluminum

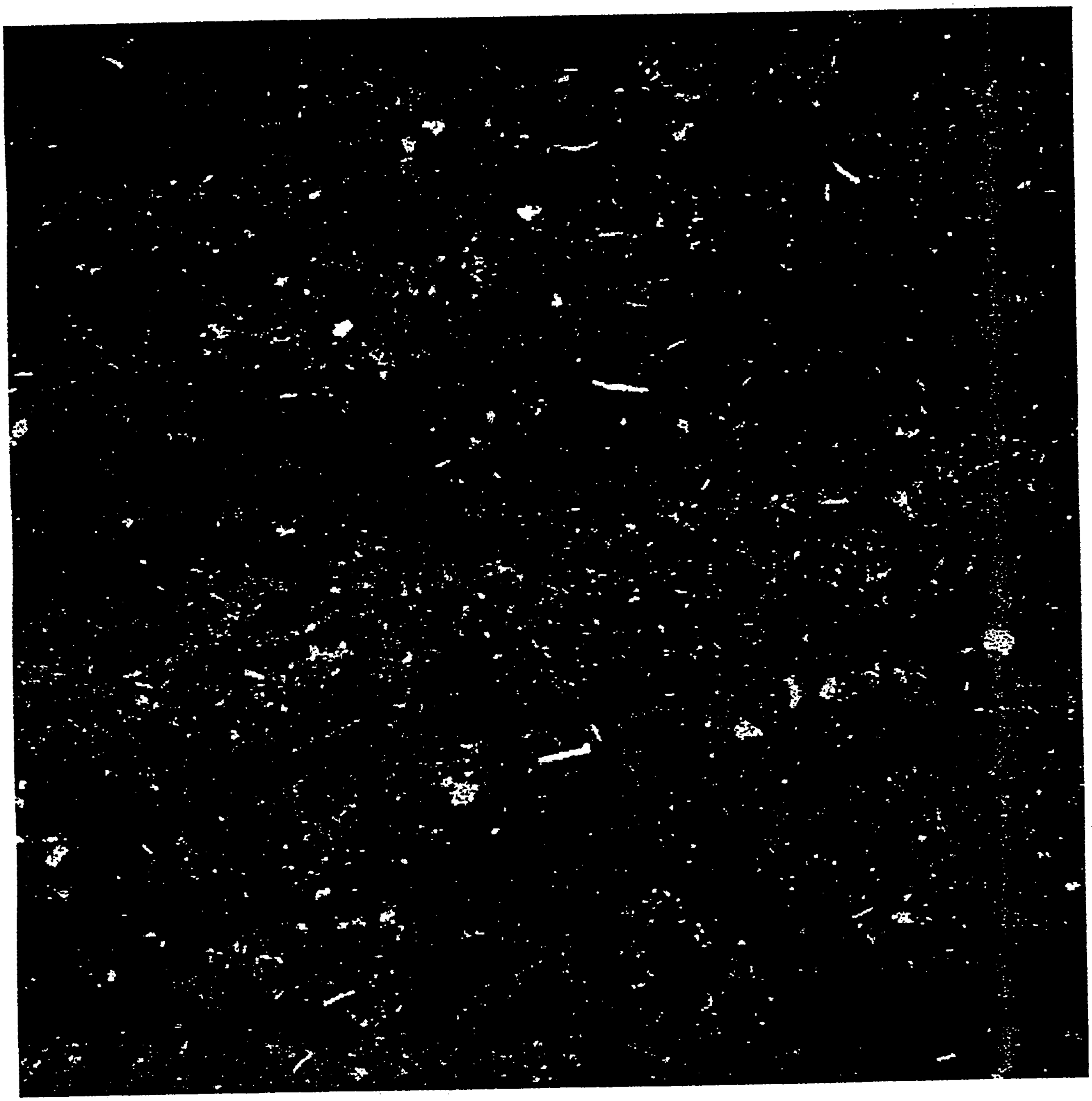


Figure E-13. Microprobe Photo D-19, Magnesium

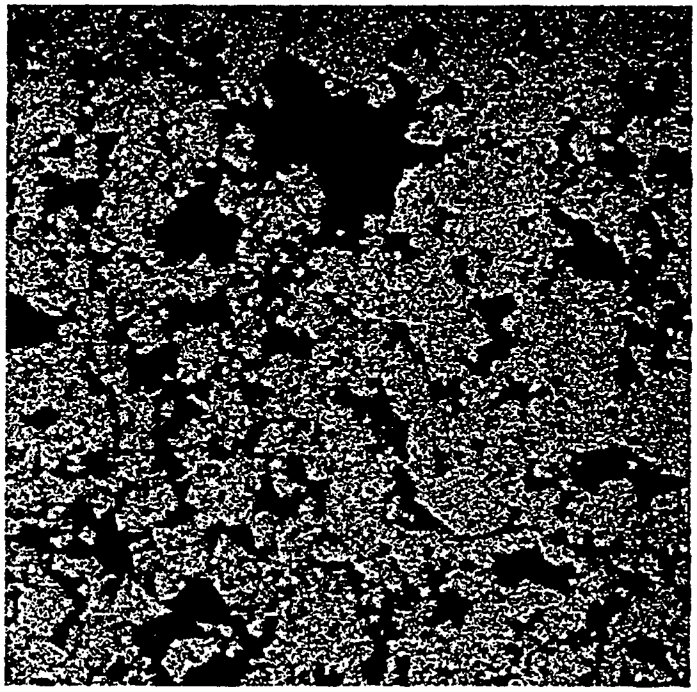


Figure E-14.   Microprobe Photo D-19, Calcium

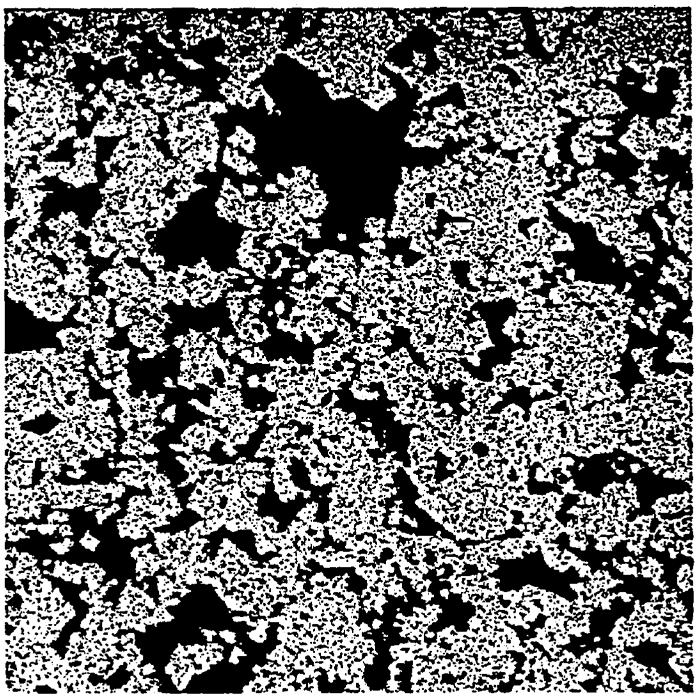


Figure E-15. Microprobe Photo D-19, Aluminum

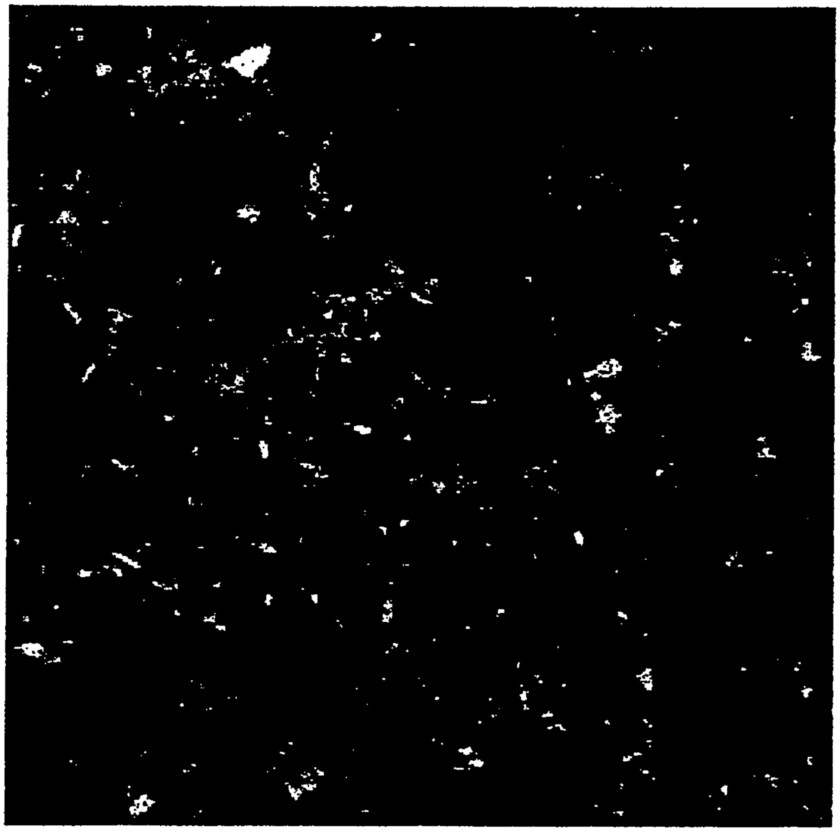
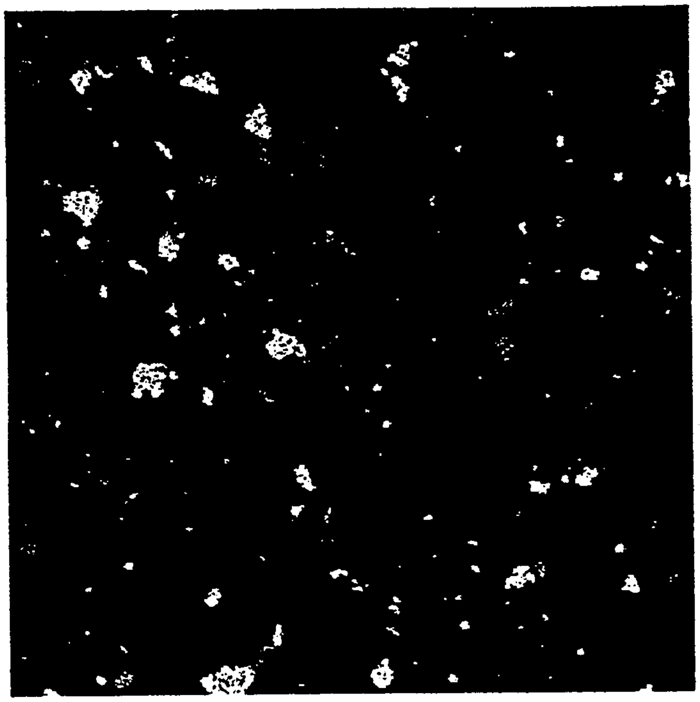


Figure E-16. Microprobe Photo D-19, Silicon



Appendix F

Photographs of Pavement Cores

Pavement Core Photographs

Photograph #	Description
1	I-94 (WB Core #1) Photograph shows wearing surface side of the pavement core slice. Somewhat rounded aggregate in upper left-hand corner (scale @ bottom of picture) is a fractured dolomite. Scale units given in millimeters for all photos.
2	I-80 (WB Core #1) Shattered appearance of dolomites shown here are common in other core slices from I-80 site.
3	I-80 (WB Core #1) Aggregate in the bottom center has horizontal fracture. Top portion of the piece is missing.
4&5	I-65 (SB Core #2) Large, fractured dolomites common on lower side of pavement core slice.
6	I-65 (SB Core #2) I-65 cores generally are smoother than I-94 with less fracturing of dolomite.
7	SR 67 (SB Core #5) Photo shows a fractured dolomite with weathered, worn slag on right and pitting on left. Pitting is likely due to natural porosity of the slag.
8	SR 67 (SB Core #5) Deep pitting with fracturing in the bituminous material. Less common occurrence in cores observed in this study. Appears that force normal to surface caused bituminous material to be displaced into void.
9	SR 67 (NB Core #1) Although not closely shown in photo, a groove is worn around aggregate piece at bottom of photograph near scale. Worn nature of bituminous matrix around perimeter of aggregates is common in cores for all roads studied.
10	SR 67 (NB Core #1) Photograph shows a well worn piece of slag with deep pitting, likely associated with natural porosity of the slag.
11	SR 67 (SB Core #1) Rare but interesting quartz pebble showing radial fracturing or possibly deep radial grooves. Photo shows grooving around aggregates more clearly than previously described photos.

**Note that WB = West Bound, NB = North Bound, and SB = South Bound.*

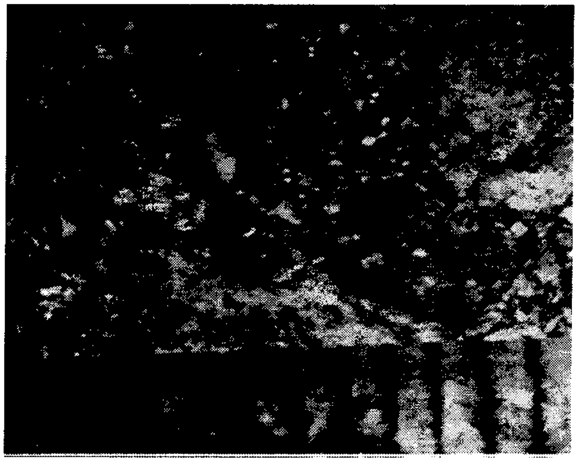


Photo 1

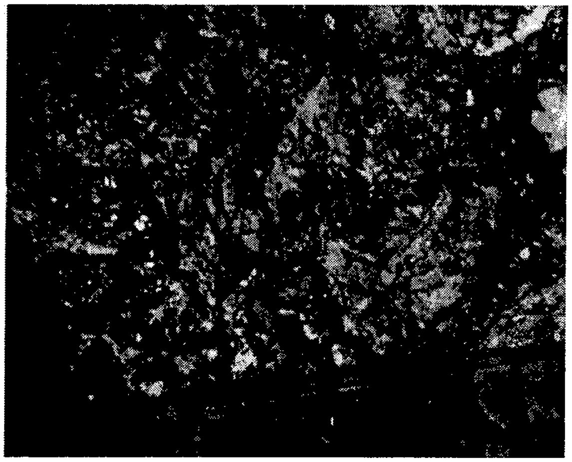


Photo 2

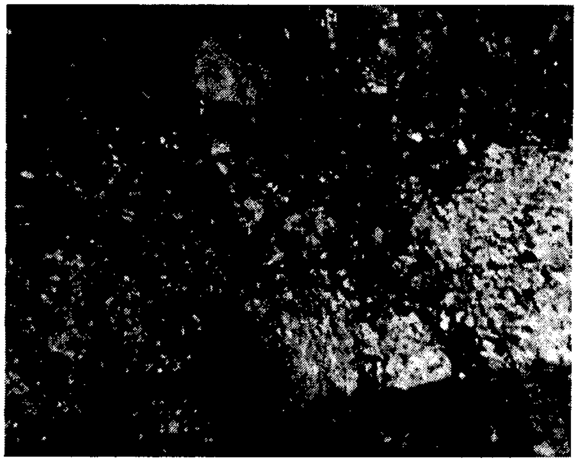


Photo 3

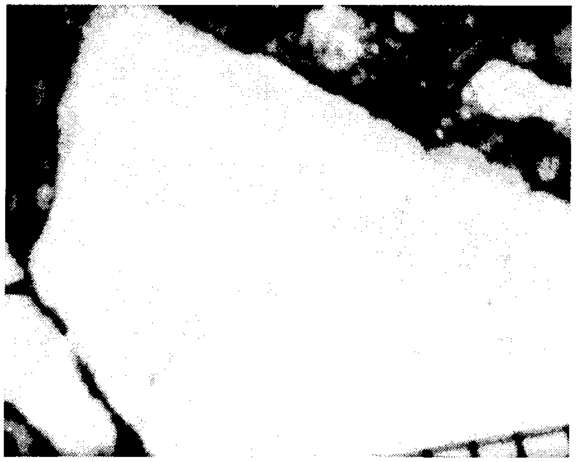


Photo 4

Photo 5

Photo 6

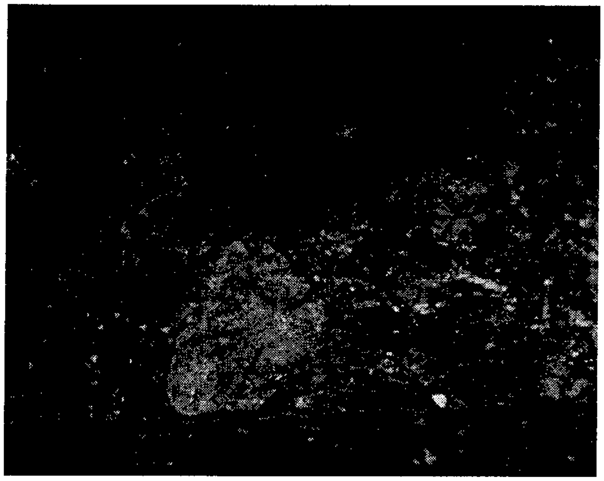


Photo 7

Photo 8

Photo 9

Photo 10

Photo 11