

PREPRINT

**EVALUATION OF
CARBONATE AGGREGATE
USING X-RAY ANALYSIS**

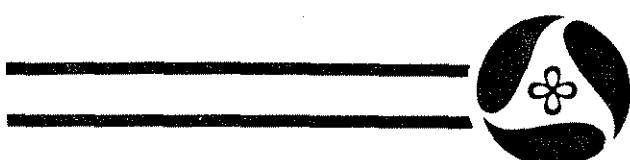
by

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Research Project HR-266**

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**Iowa Department
of Transportation**

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Using X-Ray Analysis

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DISCLAIMER

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ABSTRACT

Iowa has more than 13,000 miles of portland cement concrete (pcc) pavement. Some have performed well for over 50 years while others have been removed or overlaid due to the premature deterioration of joints and cracks. Some of the premature deterioration is classical D-cracking which is attributed to a critically saturated aggregate pore system (freeze/thaw damage). However, some of the premature deterioration is related to adverse chemical reactivity involving carbonate coarse aggregate.

The objective of this report is to demonstrate the value of a chemical analysis of carbonate aggregate using X-ray equipment to identify good or poor quality.

At least 1.5% dolomite is necessary in a carbonate aggregate to produce a discernible dolomite peak. The shift of the maximum-intensity X-ray diffraction dolomite d-spacing (XRD d-s) can be used to predict poor performance of a carbonate aggregate in portland cement concrete (pcc). A limestone aggregate with a low percent of strontium (less than 0.013) and phosphorus (less than 0.010) would be expected to give good performance in pcc pavement. Poor performance in pcc pavement is expected from limestone aggregates with higher percentages (above 0.050) of strontium.

INTRODUCTION

Over 13,000 miles of Iowa's 35,000 miles of paved highways are portland cement concrete. Many of the 22,000 miles of asphalt concrete pavement were originally pcc pavement that have been overlaid. The extent of the maintenance-free life of these pcc pavements is very dependent on the quality of coarse aggregate used. Some pcc pavements have performed well for over 50 years while others have failed prematurely. The premature failures are predominantly deterioration of joints and cracks (Figure 1). This deterioration is generally referred to as D-cracking. In a recent synthesis (1), D-cracking is defined as "a form of pcc deterioration associated primarily with the use of coarse aggregates in the concrete that disintegrate when they become saturated and are subjected to repetitive cycles of freezing and thawing".

Iowa research in the 1960's identified many crushed stone aggregate sources that would, in a saturated condition, cause rapid deterioration of pcc due to freezing and thawing. Initial testing and identification was based on ASTM C666, Method B. Research beginning in 1978 established a relationship between the freeze/thaw failure of carbonate aggregates and their pore systems (2, 3).

Within the last ten years, poor performance of many miles of pcc pavement has been determined to be related to the use of carbonate aggregates with open, large pore systems not prone

to critical saturation and subsequent freeze-thaw disintegration. These aggregates cause adverse chemical reactivity resulting in pavement deterioration visibly similar to D-cracking. The amount or rate of deterioration is directly related to the amount of deicing salt used on the pavement (4). This finding then suggests that there are at least two types of rapid joint deterioration. They are the classical D-cracking failure as defined in the recent synthesis (1) and another type that can be related to the trace elements in the carbonate aggregate. The trace elements contribute to chemical reactions which alter and weaken the crystalline structure of the carbonate aggregate and the cement paste.

High quality aggregates are essential to durable concrete. Iowa is using the Mercury Porosimeter and the Iowa Pore Index Test to identify carbonate aggregates with pore systems that are susceptible to rapid freeze/thaw deterioration. Previous Iowa research (5) has shown that aggregates that exhibit a predominance of pore sizes in the 0.04 to 0.2 micron diameter range are susceptible to critical saturation and subsequent freeze/thaw deterioration. Generally, aggregates that do not contain a predominance of these pore sizes are not prone to freeze/thaw (D-cracking) deterioration. Similar deterioration of some pcc pavements made using coarse aggregates with pore sizes larger than 0.2 micron diameter prompted the research on chemical reaction associated premature failure.

OBJECTIVE

The objective of this report is to demonstrate the value of chemical analyses using x-ray equipment to identify good or poor carbonate aggregate quality.

GEOLOGIC IDENTIFICATION OF IOWA CARBONATES

Beginning in the 1940's, Iowa geologists began developing geologic sections for quarries producing crushed carbonates for use in Iowa. The geologic sections describe the beds in all the ledges that are or at one time were approved for use in pcc pavement. These sections show the correlation of all concrete ledges with the geologic column for Iowa. Final acceptance of coarse aggregate for pcc concrete is based on quality testing of the aggregate produced.

Pavement history records which include the quarry ledges by bed used to produce the coarse aggregate have been maintained since 1940. Iowa geologists continually inspect pavements and record when visible deterioration of joints due to the aggregate is identified. A service record (SR) of years to visible deterioration is thereby developed for all the ledges of stone used in Iowa pcc pavement.

Approximately 75 percent of the coarse aggregate used in Iowa pcc pavement is crushed carbonate. The other 25 percent is river gravel containing 20 to 70 percent carbonate particles.

Geologically, the Paleozoic Strata of Iowa are part of the Forest City Basin which extends into Missouri, Kansas and Nebraska (6). A cross-sectional view of the stratigraphic system is shown in Figure 2. With the exception of a small amount of sandstone and a very small area of gypsum the quarried bedrock is carbonate. The Pennsylvanian System generally yields poor quality stone. The Mississippian, Devonian and Ordovician Systems contain units that yield carbonate aggregates of poor to good quality. The best aggregates are obtained from the Silurian System.

X-RAY EQUIPMENT

The x-ray equipment used for elemental analysis of the carbonate aggregate is located at Iowa State University (7). The ISU facilities include two scanning electron microscopes (SEM), a sequential x-ray spectrometer, and an x-ray diffractometer. Since 1980, a software package providing automated image analysis has been added, thereby expanding the capabilities of the SEM.

Electron microscopes scan across a specimen using a highly focused beam of electrons. When the beam strikes the surface of the sample, x-rays and secondary electrons are emitted. A positively charged detector attracts the negatively charged electrons and convert them into an electric current. An image of the sample is then displayed on a cathode ray tube. The newly obtained JEOL JSM-840A scanning electron microscope al-

lows for easier operation and observations with higher resolution of ultramicro surface structures than the JEOL JSM-U3, which was purchased in 1971. Both SEM's are equipped with an energy dispersive x-ray analyzer, which can identify elements present in the sample by the x-rays they emit when the electron beam strikes the sample.

The SRS 200 automated sequential x-ray spectrometer analyzes elements by fluorescence. The spectrometer excites the samples using x-rays, and by measuring the x-rays emitted from the sample, elemental concentrations can be determined. The SRS-200 is capable of analyzing ten samples at a time for up to thirty elements.

Using the Siemen D500 x-ray diffractometer, analysis of compounds is performed through the determination of spacings of crystallographic planes by the diffraction of x-rays. The Siemens D500/SRS200 system uses two microcomputers for its analytical operations. The data can also be transmitted to the Iowa State University main frame computer for further evaluation.

SAMPLE SIZE AND PREPARATION

The new JEOL JSM-840A SEM will accommodate samples up to 4 inches in diameter and 1/2 inch thick. For initial evaluation, a broken piece with a relatively flat plane can be used without further preparation. A quick elemental analysis can be conducted to determine if further analysis is warranted. This also gives direction as to what elements should be selected for final analysis. Photographs are taken at relatively low voltage. Sometimes good quality photographs can be taken without a special coating, but normally a gold, carbon or silver coating is applied to prevent an electrical charge from occurring, and interfering with the photograph. For final chemical analysis, the area of interest must be flat and polished. Rapid sawing, with a diamond blade, had been used to obtain a smooth flat face. This is no longer used as the procedure heats igneous and other hard particles and causes damage to the surface. Slow sawing with a lapidary diamond blade does not overheat the hard particles.

For x-ray fluorescence (XRF), the material to be analyzed is crushed for two minutes in a Spex Shatterbox (ring grinder) to produce material passing the number 300 sieve. If a mechanical mixture is being prepared, care should be taken to try to obtain uniform particle size. Silica is a problem when crushed with the softer carbonate material. Grinding of a mixture of harder and softer particles produces non-uniform particle sizes which do not allow for precise quantitative

analysis. An extended period of grinding alleviates this problem. The sample holder is a one inch diameter plastic cylinder. A plastic ring is used to attach a sheet of 6 micron thick mylar to the bottom making a cup. A 1/4 inch layer of loose crushed dolomite or limestone is poured into the cup. The top of the sample is covered with a sheet of 6 micron thick mylar and fastened with another plastic ring.

The sample for x-ray diffraction (XRD) is crushed to the same size as for x-ray fluorescence. When the determination of a very exact d-spacing or a minor shift in a d-spacing is desired, approximately ten percent crushed zirconia is mixed with the carbonate sample as a standard reference. Enough powdered sample material is placed in a one inch diameter, 1/16 inch deep plastic cup to overfill the cup. A strike off is used to remove excess material.

COMPOSITION OF AGGREGATE

There are times when the composition of an aggregate is an important factor in ascertaining its quality. Laboratory testing of the igneous fraction of Iowa river gravels always yields good performance in pcc. In some areas of Iowa, the carbonate fraction (up to 70%) of a river gravel produces premature failure in pcc pavement. In the past, it took an experienced petrographer to manually separate the various rock types.

With X-ray diffraction, there is a much quicker, more accurate and less labor intensive method of conducting a petrographic analysis. The material to be analyzed is first crushed to pass the number 4 sieve size. This material is then split to yield a uniform representative sample. This representative sample is then ground in the Spex Shatterbox as previously explained. A software package has been obtained by Iowa State University that will graphically display the percentage of each of the different rock types. Petrographic analysis of river sands is now being conducted. A sample of the computer output is shown in figure 3.

A program to systematically determine the elemental analysis of Iowa carbonates began five years ago. During that time, percentages of various elements have been determined on over 1700 samples. Some elements (Na and Cl) have shown no relationship to aggregate performance and are no longer included. The Iowa DOT has recognized the benefits of using x-ray analysis of carbonate aggregate as an important factor in determining quality of coarse aggregate for use in pcc pavement. Some carbonates from other states, generally exhibiting poor performance, have been included in the x-ray analysis program. The X-ray analyses of this limited number (57 samples) do not conflict with the findings for Iowa carbonates.

Because the research being conducted is on dolomites $\text{CaMg(CO}_3)_2$ and limestones CaCO_3 many of the elements are almost entirely contained as one compound. For instance, the calcium is almost entirely combined as CaCO_3 or limestone. Almost all of the magnesium would be in the form of $\text{CaMg(CO}_3)_2$ or dolomite. All of the x-ray chemical analyses for this research has been elemental by XRF with an assumption as to the compounds. The percentages of calcium and magnesium provide information to determine the ratio of limestone to dolomite. XRD peak intensities can also provide this ratio information. Current research with XRD verifies the validity of the limestone/dolomite relationship determined using XRF elemental analysis.

Analysis by XRF yields the percentages of elements present in a sample. Prior to X-ray analysis the percent of strontium found in a sample was usually expressed as strontium oxide. The authors do not believe that these elements are contained in the carbonates as oxides and, therefore, will not be expressed in that manner. All percentages expressed in this report will be in regard to elemental strontium and elemental phosphorus. The conversion factor that may be used to determine how much strontium oxide this would represent is 1.1826. The conversion factor from elemental phosphorus to phosphorus oxide is 2.2913.

Currently the Iowa DOT uses a 16 cycle water-alcohol freeze/thaw test (FTA) with a specified loss of not more than six percent of the total sample weight for acceptance of coarse aggregate for pcc. The percent loss is directly related to the amount of clay in the carbonate. Most of the elemental aluminum and titanium are contained in clays and, therefore, they correlate with the results of the water-alcohol freeze/thaw test (Figures 4 and 5). Two factors that hinder this correlation are the presence of authigenic feldspar containing aluminum and titanium and the use of different gradations of aggregate in the freeze/thaw test. When the same gradation is used for the freeze/thaw test and the aggregate is from the same quarry (Festina) a high correlation ($r=0.93$) is obtained (Figure 6). The correlation coefficient of elements (for 86 samples) with each other and with water-alcohol freeze/thaw is given in Table 2. Aluminum ($r=0.65$) and titanium ($r=0.62$) show the greatest correlation with the FTA.

The elemental analysis of a few carbonates selected to give a range of limestone and dolomite aggregates from good to poor performance is given in Table 1.

The SEM is used to examine limestones, dolomites and pcc. A deteriorated dolomite aggregate in pcc exhibited a dark rim (Figure 7). An initial assumption was that the rim would be high in iron. A SEM spot analysis showed no iron, but a high

sulfur, calcium, and silicon content. An investigation of the dark rim is continuing. Speculation is that a chemical reaction has occurred at the boundary of the dolomite particle which adversely affects the bond.

EVALUATION OF DOLOMitic AGGREGATE

X-ray diffraction has become the principal Iowa method of identifying dolomitic aggregate that will result in poor performance in pcc pavement. In a stone consisting of a mixture of limestone and dolomite, at least 1.5% dolomite is needed to yield a discernable dolomite peak.

If a discernable dolomite peak of an Iowa carbonate aggregate can be identified, poor performance in pcc pavement can be predicted by the maximum-intensity x-ray diffraction dolomite d-spacing (XRD d-s) (4). A high maximum-intensity dolomite/ankerite d-spacing (greater than 2.899) would identify a carbonate with poor performance in pcc pavement (Figure 8). The pore system of the aggregate significantly affects the rate of adverse chemical reactivity. At this time, the relationship of the pore size effect on chemical reactivity has not been fully determined. This relationship is not intended to show that 1.5 percent poor quality dolomite can cause rapid deterioration of pcc pavement. Speculation is that the detrimental element, compound or characteristic is also present in the limestone fraction of the carbonate particle.

X-ray analyses of over 120 samples have shown very low amounts (less than 0.025%) of strontium in dolomite aggregates.

EVALUATION OF LIMESTONE AGGREGATE

X-ray analyses have yielded data that will generally predict good or bad performance of limestone (CaCO_3) aggregate in pcc pavement. A good correlation for the full range has not been identified. There is a very small d-spacing shift of the maximum-intensity limestone (CaCO_3) peak as compared to the large d-spacing shift for dolomites. Zirconia was added to the carbonate sample to improve accuracy in the analysis of peak shifts. Research to date has not shown a good correlation of this shift with limestone (calcite) performance records.

Strontium, an element not found in significant amounts in dolomite aggregates tested by the Iowa DOT, relates to performance of limestone aggregate in pcc pavement. Phosphorus is another element that exhibits a relationship to the performance of limestone aggregate in pcc pavement. If a limestone aggregate is low in percent strontium (below 0.013) in the limestone fraction of the carbonate particle (Figure 9) and low in percent phosphorus (below 0.010) in the limestone fraction (Figure 10) good performance in pcc pavement is expected. Poor performance is expected if the percent strontium in the limestone fraction of the aggregate exceeds 0.050.

Iowa limestones from the Silurian System (percent strontium below 0.013) exhibit the best performance while Pennsylvanian limestones (percent strontium generally above 0.040) exhibit the poorest performance. Within the Pennsylvanian system, the Stanzel Quarry with the poorest performance has the highest percent strontium (0.055) and the Weeping Water, NE Quarry with the best performance has the lowest percent strontium (0.041). The SR in years to visible deterioration of pcc pavement shows a correlation coefficient of $r=-0.38$ with strontium (for 367 samples), $r=-0.22$ with phosphorus (for 376 samples) and $r=-0.40$ with strontium plus phosphorus (for 367 samples).

CONCLUSIONS

This research on x-ray analysis of carbonate aggregates supports the following conclusions:

1. X-ray analysis of Iowa carbonate aggregates is an effective method of predicting their performance in pcc pavement.
2. For Iowa carbonate aggregates with more than 1.5 percent dolomite, the maximum-intensity x-ray diffraction dolomite d-spacing can be used to predict poor performance in pcc concrete.

3. Iowa limestone aggregates with large, open pore systems with percent strontium below 0.013 and percent phosphorus below 0.010 exhibit good performance in pcc pavement.
4. Iowa limestone aggregates with percent strontium above 0.050 will exhibit poor performance when used in pcc pavement.

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TABLE TITLES

1. XRF Elemental Analyses of a Range of Carbonates
2. A Summary of Correlation Coefficients for Elements in
Carbonates and the Water-Alcohol Freeze/Thaw

TABLE 1

XRF ELEMENTAL ANALYSES
OF A RANGE OF CARBONATES

Quarry	SR	XRD d-S	PERCENTAGES								
			Sr	Mg	Ca	Al	Fe	S	Ti	Si	P
Maryville	40	2.889	0.010	12.66	21.78	0.13	0.16	0.02	0.01	0.44	0.00
Aurora	40	2.887	0.011	11.72	22.51	0.17	0.19	0.02	0.01	1.04	0.00
Pape	30	None	0.011	0.22	38.32	0.28	0.12	0.04	0.01	1.22	0.00
Daley	30	2.887	0.008	12.59	21.36	0.22	0.58	0.07	----	0.63	----
Gil City	30	None	0.016	0.23	38.33	0.30	0.21	0.13	0.01	1.05	0.00
Kendall-ville	25	2.889	0.015	6.41	28.72	0.37	0.55	0.38	0.02	1.69	0.02
Skyline	25	2.891	0.024	3.42	30.84	0.58	0.43	0.19	0.03	3.87	0.03
Weeping Water	25	None	0.041	0.23	39.11	0.08	0.18	0.08	0.00	0.44	0.01
LeGrand	20	2.902	0.014	8.02	27.84	0.12	0.83	0.17	0.01	0.38	0.03
Smith	15	2.901	----	10.89	23.83	0.15	0.49	0.17	0.01	0.62	0.01
Logan	15	2.902	0.054	0.76	35.33	0.53	0.54	0.19	0.03	3.13	0.03
Garrison	15	2.904	0.021	10.67	23.53	0.16	1.06	0.65	0.01	0.71	0.01
Glory Rapid	10	2.903	0.016	9.86	19.40	1.48	1.05	0.43	----	5.47	----
Kingston, ONT	10	2.908	0.038	5.21	24.94	1.98	1.04	0.23	0.11	5.96	0.01
Ullin, IL	10	2.890	0.259	1.15	35.83	0.18	0.17	0.37	0.01	2.34	0.05
VA	?	2.903	0.033	1.34	36.36	0.35	0.39	0.19	0.02	1.34	0.01
Tyrone, KY	?	2.904	0.032	2.76	32.09	0.86	0.50	0.16	0.04	3.20	0.13
Jefferson	5	2.906	0.068	0.46	37.55	0.20	0.29	0.04	0.01	1.66	0.02
Stanzel	5	2.907	0.055	0.43	38.30	0.14	0.47	0.06	0.01	0.79	0.01
Ada, OK	?	2.908	0.017	0.18	38.16	0.22	0.28	0.10	0.01	0.74	0.54
C. Hill, TN	?	None	0.055	0.41	38.64	0.16	0.15	0.14	0.01	0.53	0.05

TABLE 2
 A SUMMARY OF CORRELATION COEFFICIENTS (*r*)
 FOR ELEMENTS IN CARBONATES
 AND THE WATER-ALCOHOL FREEZE/THAW

	Sr	Mg	Fe	S	Ti	Mn	Si	Ca	K	P	Al
Mg	-0.30	1.00									
Fe	0.27	-0.05	1.00								
S	0.01	0.00	0.09	1.00							
Ti	0.34	-0.12	0.63	0.08	1.00						
Mn	0.10	0.05	0.32	0.04	0.16	1.00					
Si	0.21	-0.19	0.48	0.00	0.55	0.17	1.00				
Ca	0.07	-0.56	-0.41	-0.16	-0.36	-0.16	-0.54	1.00			
K	0.21	-0.11	0.33	-0.00	0.43	0.06	0.52	-0.44	1.00		
P	0.43	-0.24	0.44	0.00	0.58	0.11	0.34	-0.11	0.32	1.00	
Al	0.41	-0.21	0.59	0.03	0.73	0.21	0.80	-0.47	0.68	0.50	1.00
FTA	-0.08	-0.07	0.30	0.23	0.62	-0.10	0.60	-0.10	0.61	0.35	0.65

FIGURE CAPTIONS

1. A Close-up of Premature Deterioration at an Intersection of the Transverse and Longitudinal Joints
2. A Stratigraphic Cross-sectional View of Iowa Geologic Systems
3. XRD Petrographic Analysis of River Sand
4. Alcohol Freeze/Thaw vs Percent Aluminum
5. Alcohol Freeze/Thaw vs Percent Titanium
6. Alcohol Freeze/Thaw vs Percent Aluminum for the Festina Quarry
7. Portland Cement Concrete Containing a Deteriorated Dolomite Aggregate With a Dark Rim
8. Graph of d-spacing of Maximum-Intensity Dolomite Peak vs Years to Visible Deterioration of the Concrete
9. The Relationship of Percent Strontium in the Carbonate Aggregate to Service Record When Used in PCC Pavement
10. The Relationship of Percent Phosphorus in the Carbonate Aggregate to Service Record When Used in PCC Pavement

**FIGURE 3 XRD PETROGRAPHIC ANALYSIS OF RIVER SAND
CONN PIT**

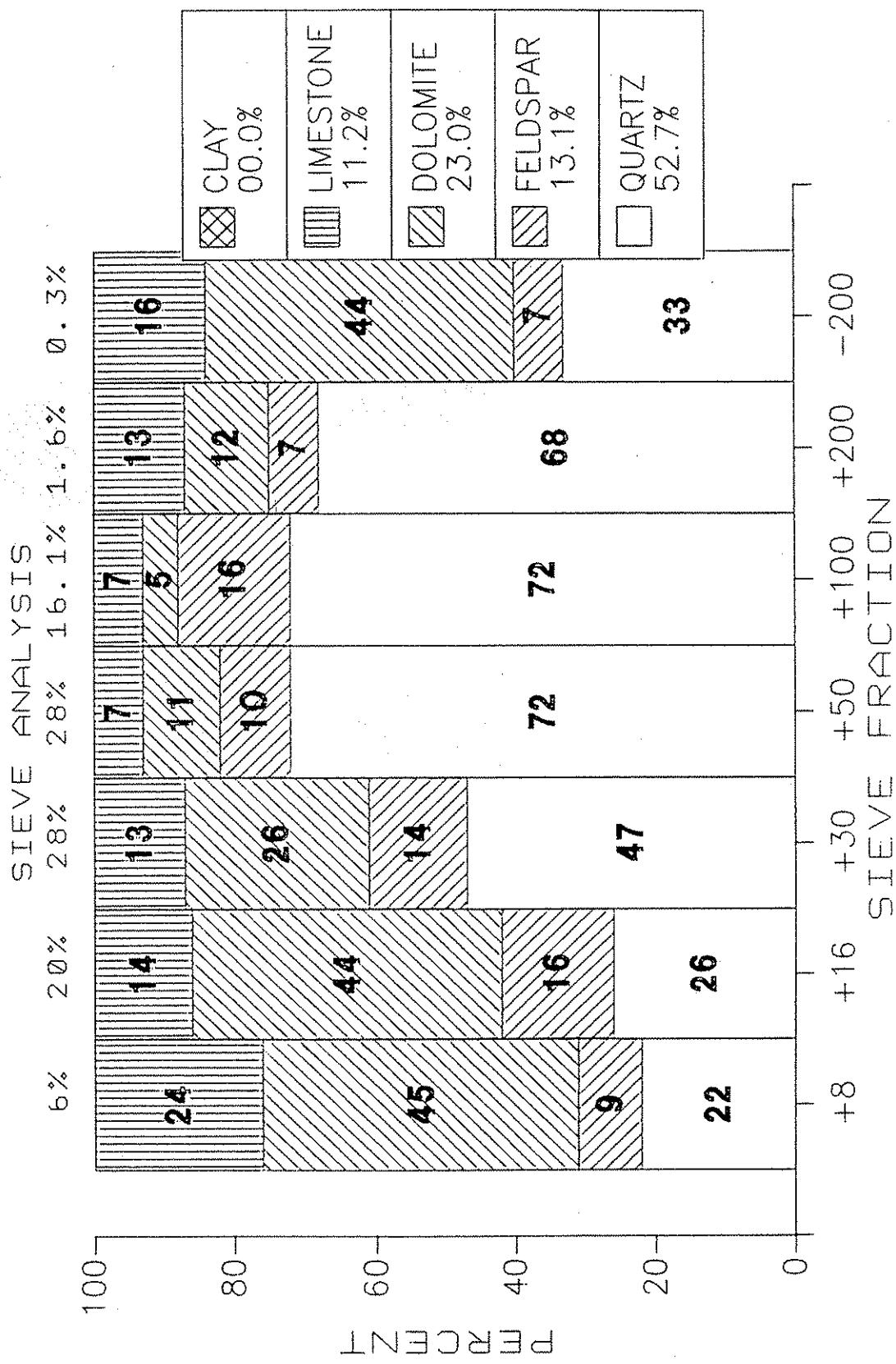


FIGURE 4

ALCOHOL FREEZE/THAW VS. PERCENT ALUMINUM
ALL LABORATORY FTA SAMPLES DURING JAN & FEB 1985

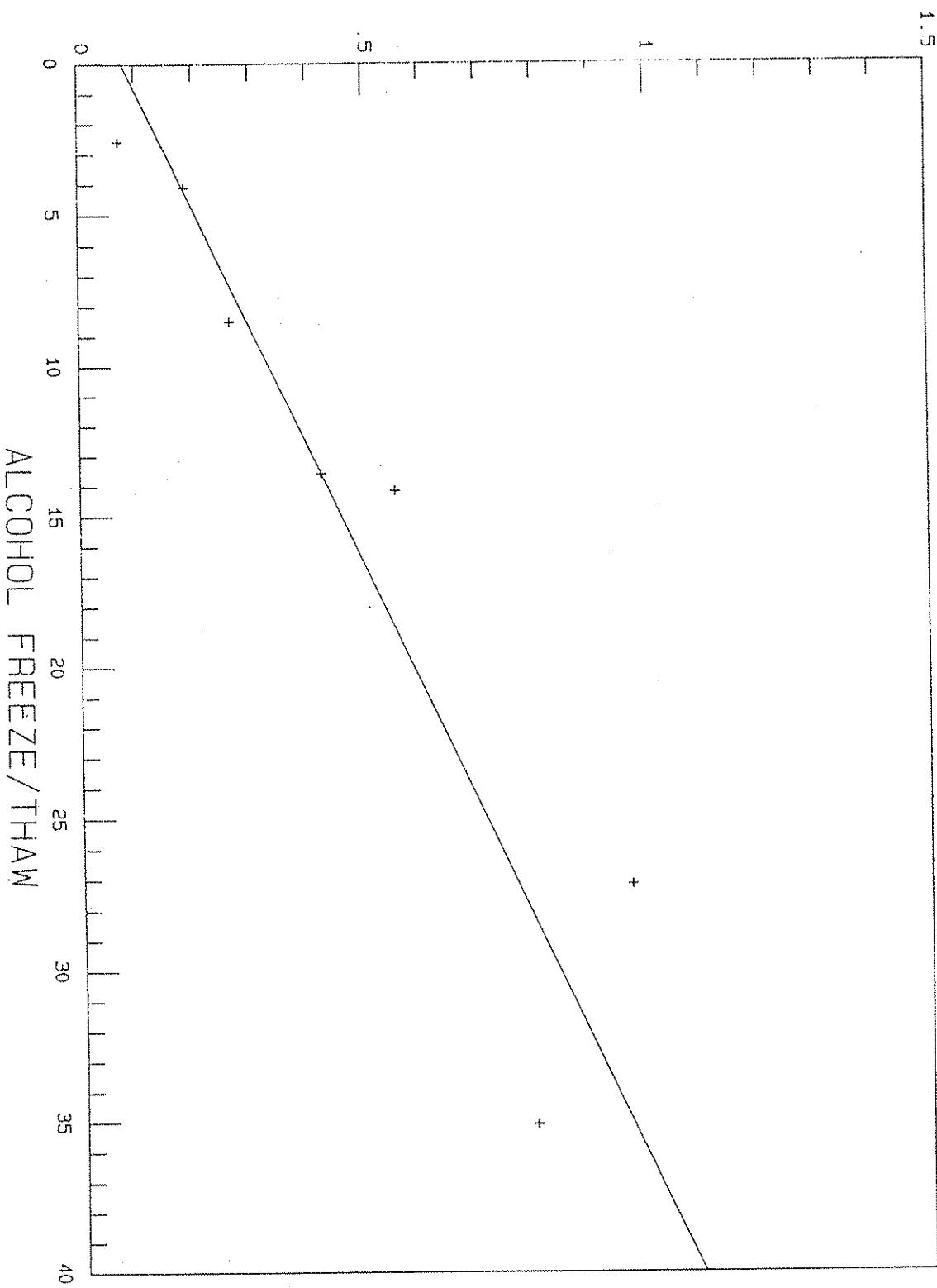


FIGURE 5
ALCOHOL FREEZE/THAW VS. PERCENT TITANIUM
ALL LABORATORY FTA SAMPLES DURING JAN & FEB 1985

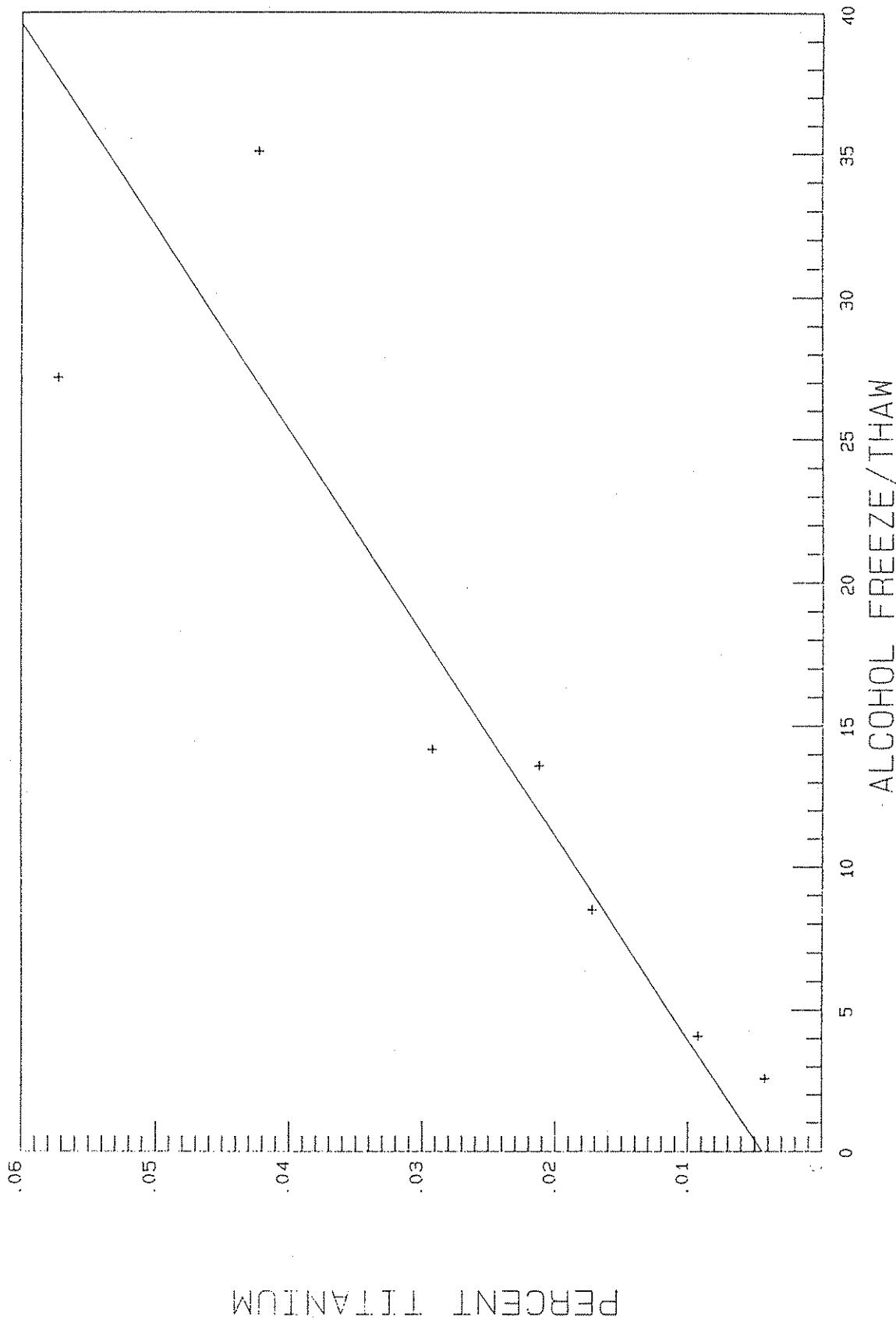
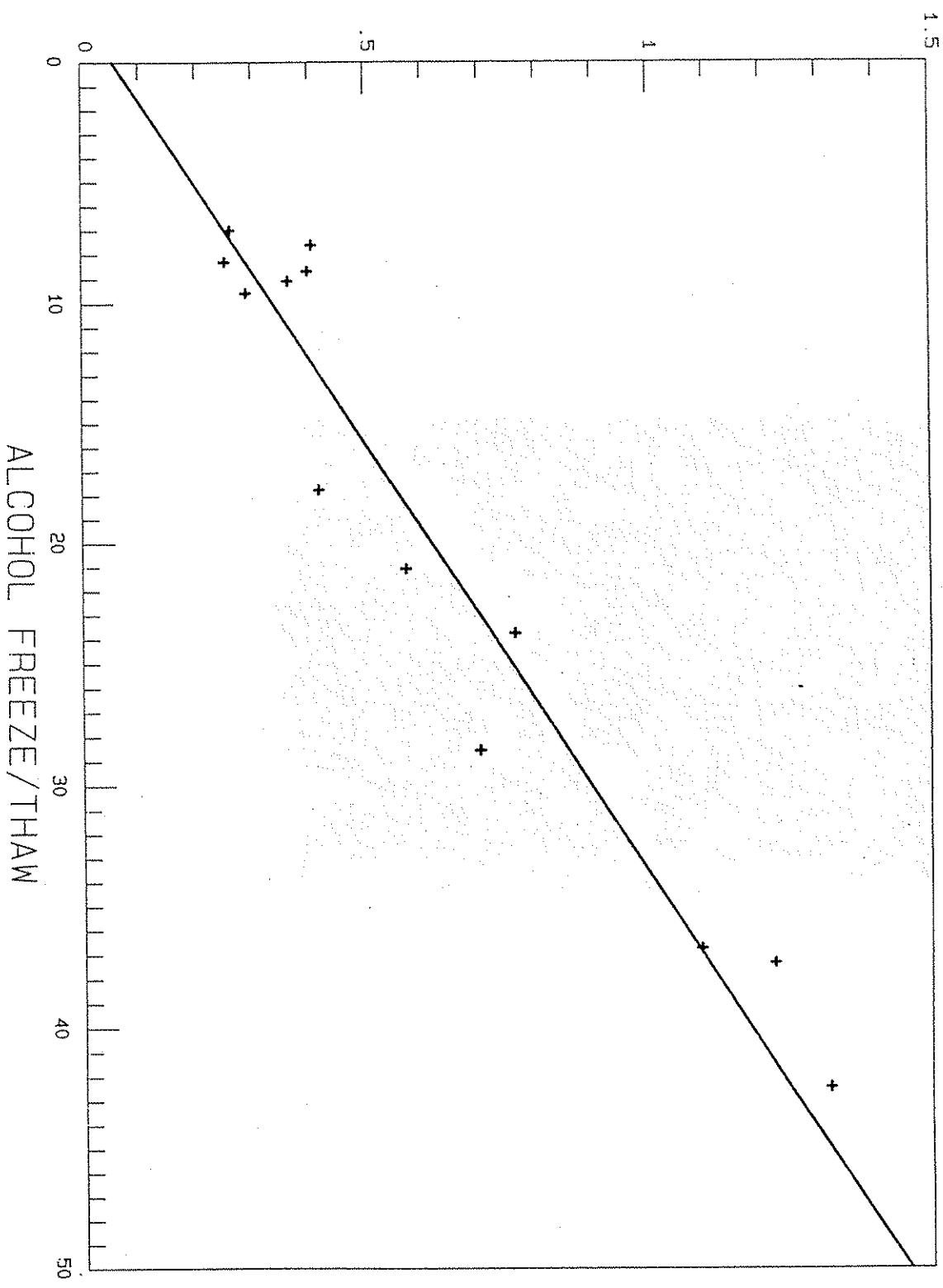


FIGURE 6

ALCOHOL FREEZE/THAW VS. PERCENT ALUMINUM
FESTINA QUARRY BEDS 1B THROUGH 3C



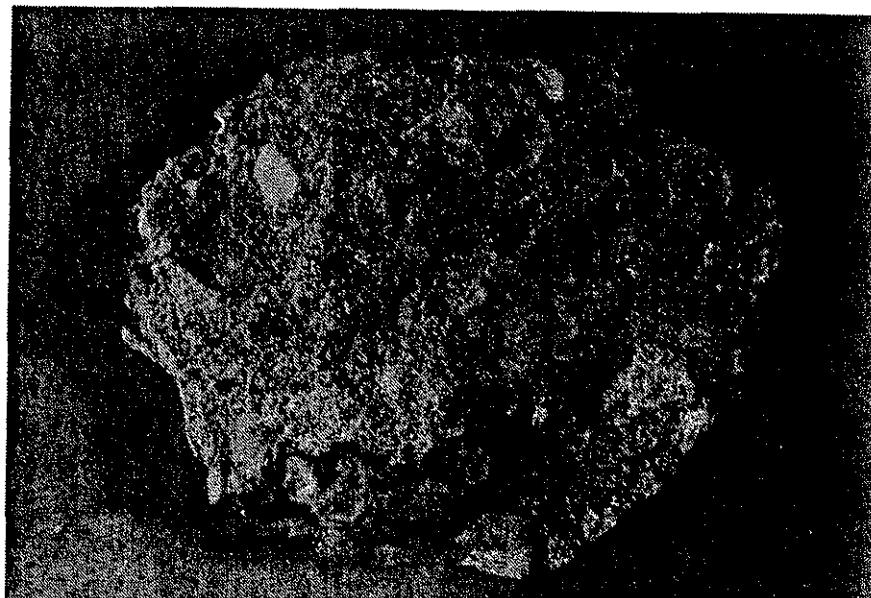


Figure 7 Portland Cement Concrete Containing a Deteriorated
Dolomite Aggregate With a Dark Rim

FIGURE 8

d SPACING OF MAXIMUM INTENSITY DOLOMITE PEAK VS.
YEARS TO VISIBLE DETERIORATION OF PCC PAVEMENT

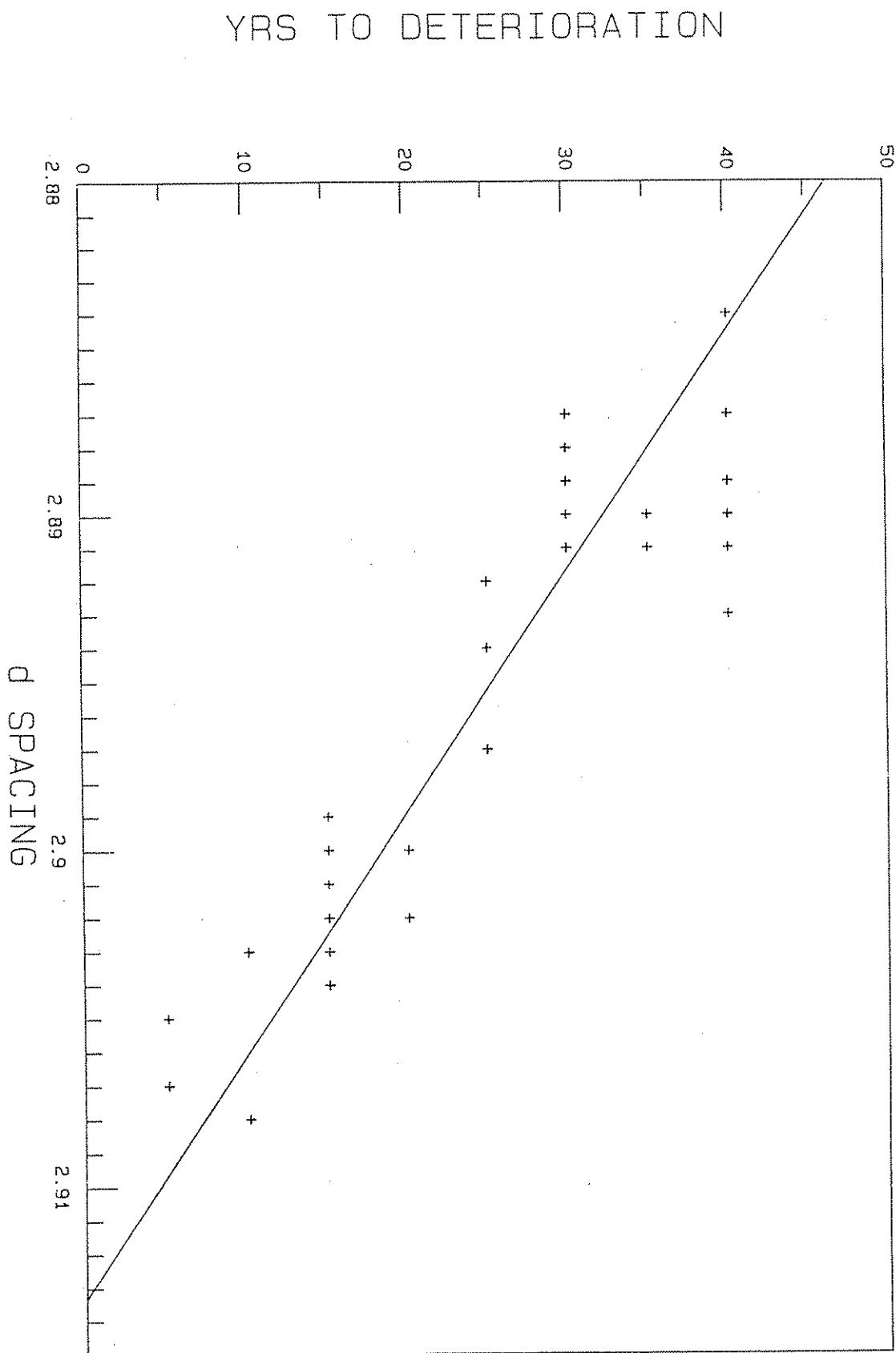


FIGURE 9
RELATIONSHIP OF PERCENT STRONTIUM IN THE COARSE AGGREGATE
TO SERVICE RECORD WHEN USED IN PCC PAVEMENT

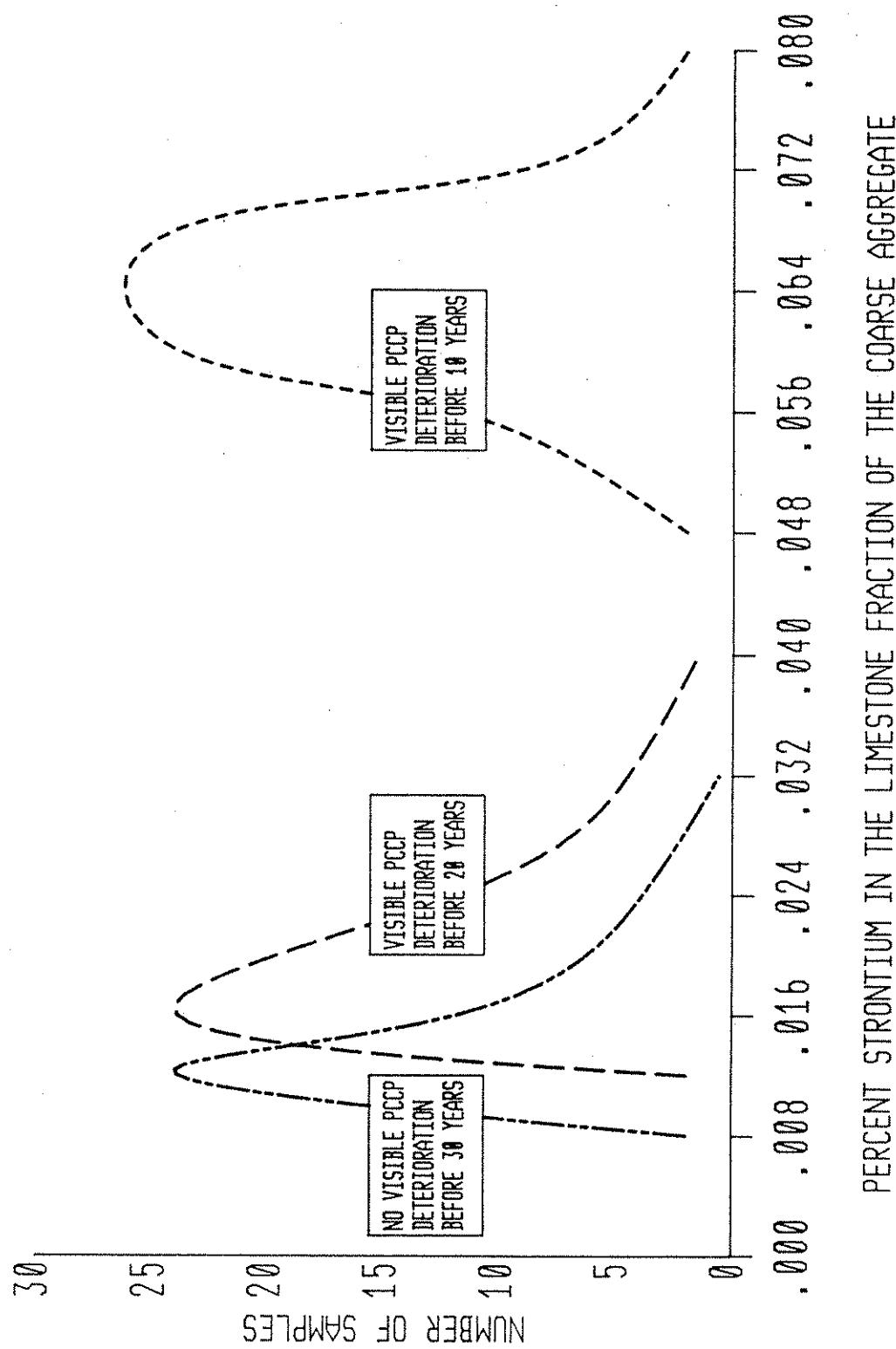
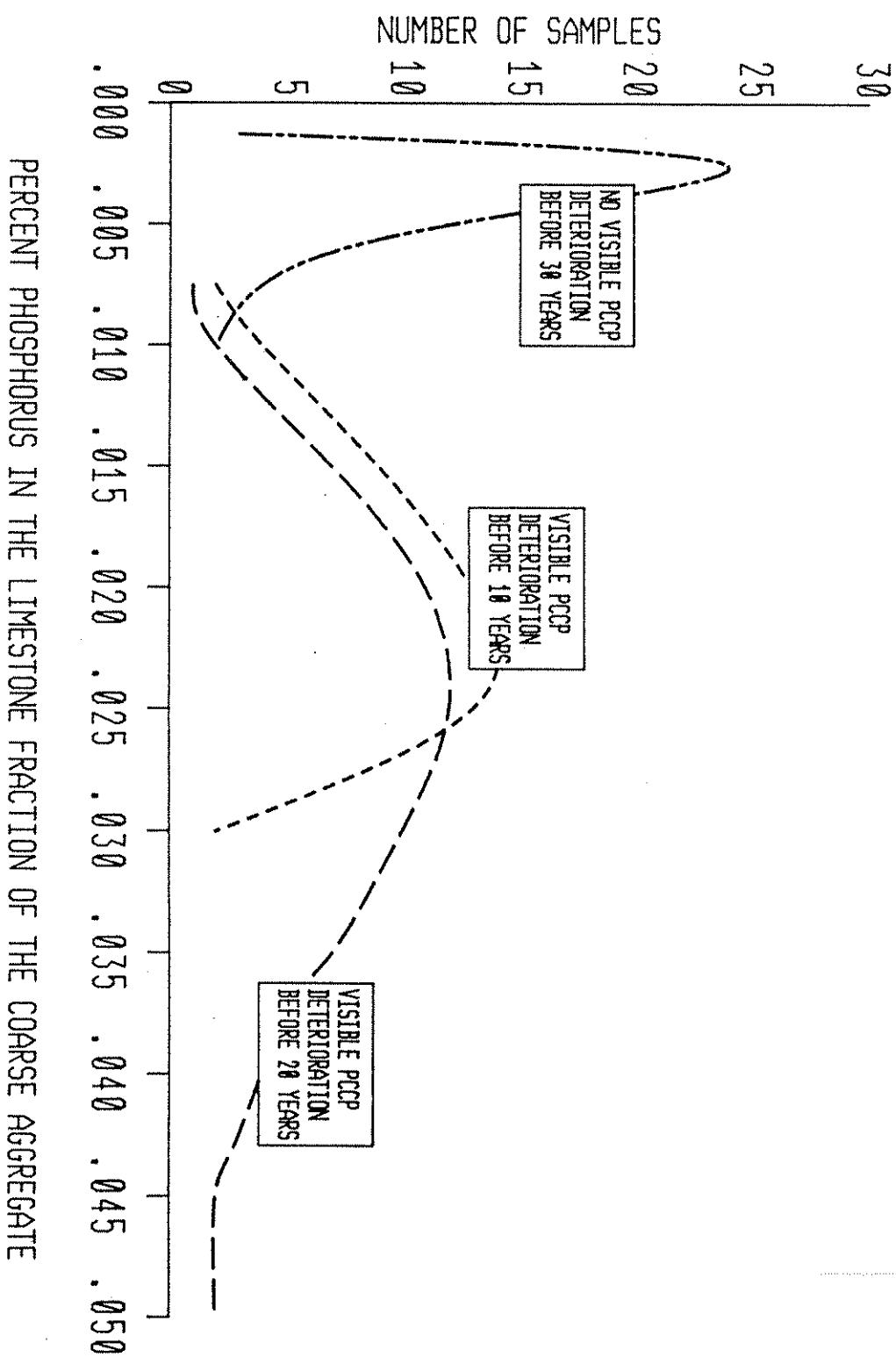


FIGURE 10
RELATIONSHIP OF PERCENT PHOSPHORUS IN THE COARSE AGGREGATE
TO SERVICE RECORD WHEN USED IN PCC PAVEMENT



PERCENT PHOSPHORUS IN THE LIMESTONE FRACTION OF THE COARSE AGGREGATE