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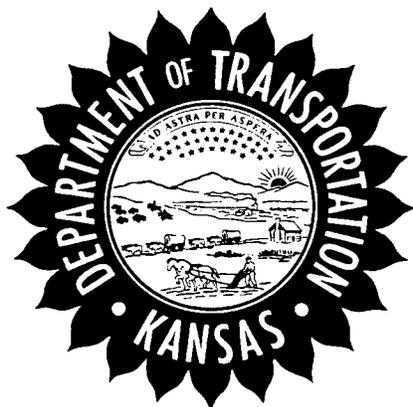


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Final Report

**TEXTURAL AND MINERALOGICAL CHARACTERIZATION OF
KANSAS LIMESTONE AGGREGATES IN RELATION TO
PHYSICAL TEST RESULTS**

Barbara J. Smith and Ralph G. Pollock



November 1997

KANSAS DEPARTMENT OF TRANSPORTATION

**Division of Operations
Bureau of Materials and Research**

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Ralph G. Pollock**

Kansas Department of Transportation

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Correlation of expansion and durability factor results with percent contamination in the beams cut from pavement having 30% crushed limestone in the mix are shown. Compare this pavement mix with that in Figures 21 and 22.

Summary of test results for expansion and durability on laboratory beams made with both mixes found in the paving projects is shown with 0-40% contamination levels. Aggregates used were from the same sources as the paving projects.

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Abstract

Suitable limestone aggregate for transportation construction use is becoming less available in Kansas. The more accessible and better aggregates of known sources are being used. Characterization of suitable and unsuitable aggregates by petrographic descriptions and tests has not been done in the state. This study includes characterizing quarry ledge samples by mineralogy and texture and sorting these characteristics for suitability for construction uses. To do this, quarry samples collected in the early 1980s were cut, polished and treated with a standard acid stain to differentiate the carbonate minerals probably present. Then the surface was treated with acetate to make a permanent, more easily handled record of the mineralogy and texture of each polished and treated surface. The information from these acetate records form a database that also includes the information from the physical testing on the ledge samples. The database includes 550 records from 243 ledges, some ledges providing two or more acetate records called peels for description. The database supplies the means to sort characteristics for statistical testing. The statistical test chosen was Chi Square (χ^2) with Cramer's V test used to norm the results of the values. Contingency tables were built using categories of color of stained peels, texture and passing or failing classifications for concrete construction.

Results of the statistical analyses indicate that there are relationships between the characteristics chosen and passing or failing. The results are discussed by characteristic. An application concerning verification of aggregate sources is included and discussed as it illustrates the usefulness of the petrographic information. Recommendations are given to further define relationships to mineralogy and texture, to verify these relationships as predictors of passing or failing with new ledge samples, and to summarize the results from these endeavors in a preliminary field tool for use in prospecting for resources and verifying production stockpile consistency with source ledge test results. Tables of relationship based on peel data are included.

Introduction and the Need for Study

Suitable limestone aggregate for transportation construction use is becoming less available in Kansas. The more accessible and better aggregates of known sources are being used or quarries closed. Characterization of suitable and unsuitable aggregates by petrographic descriptions and tests has not been done in the state. This study includes characterizing quarry ledge samples by mineralogy and texture and sorting these characteristics for suitability for construction uses. To do this, quarry samples collected in the early 1980s were cut, polished and treated with a standard acid stain to differentiate the carbonate minerals probably present. Then the surface was treated with acetate to make a permanent, more easily handled record of the mineralogy and texture of each polished and treated surface. The information from these acetate records, called "peels", form a database that also includes the information from the physical testing on the ledge samples. The database includes 550 records from 243 ledges, some ledges providing two or more acetate records for description. The database supplies the means to sort characteristics for statistical testing. The resulting information of geologic characterization associated with qualified aggregates for construction will help in searching for new sources of limestone aggregate and possibly in using existing sources more effectively.

Study Construction

The limestone quarry ledge samples for this study were selected from routine ledge samples submitted for physical testing at the Materials and Research Center during the years 1981 through 1984. Very few from 1981 and 1984 were included. Samples

from Oklahoma were excluded. The majority came from those submitted in 1982 and 1983. At the time of selection of samples for the study, an attempt was made to select handsized samples that exhibited the range of quickly recognizable characteristics of the particular ledge sample submitted. No attempt was made to select for characteristics in proportion to the total sample.

The physical tests for limestone include durability factor, expansion, and soundness (modified freeze and thaw). These test procedures are included in the appendix. In order to be used for concrete pavement construction, the limestone aggregate must pass all three tests. After passing the test requirements these aggregates are called Class I aggregates. Those that fail any test are Class 0 aggregates. Recently a neural network-reliability based approach has been demonstrated predicting the expansion and soundness test results and using specific gravity, acid insoluble residue and absorption to determine durability factor (Najjar, Basheev, 1997). This will be discussed later.

The selected ledge samples were given a geology research identifying number and individually marked with the number before sawing. Each sawed sample was polished on a lap using fine grit before the acid etching and staining process. Treatment with acetone and acetate resulted in an acetate record, called a peel, holding the staining and texture of each treated piece of the sample. This procedure is given in the appendix. After labeling with the proper identifying number the peels were mounted in laboratory books for easy access and study.

Table 1 describes the mineralogy differentiated by the staining procedure. To provide information suitable for a database each peel was described by Munsell color notation and by percent of each color noted in the peel. To describe a color, three

Table 1 Etching and Staining Characteristics of Carbonate Minerals*

Mineral	Effect of etching	Stain color with Alizarin Red S	Stain color with potassium ferricyanide	Combined result
Calcite (non-ferroan)	Considerable (relief reduced)	Pink to red-brown	None	Pink to red-brown
Calcite (ferroan)	Considerable (relief reduced)	Pink to red-brown	Pale to deep blue depending on iron content	
Dolomite (non-ferroan)	Negligible (relief maintained)	None	None	Colorless
Dolomite (ferroan)	Negligible (relief maintained)	None	Very pale blue	Very pale blue (appears turquoise or greenish in thin section)

Note: The staining procedure used for this study produces results summarized in the last column.

Adapted from: Atlas of Sedimentary Rocks Under the Microscope, A.E. Adams, W.S. MacKenzie, and C. Guilford, 1984, John Wiley and Sons, pp104.

variables are used: hue, value, and chroma. Hue indicates the color in relation to red, green, yellow, blue or purple designated by one or two letters. Those used in this study included R, RP, P, PB, B, and YR representing red, red purple, purple blue, blue and yellow red. Colorless sections of the sample were coded as CL for clear. Value is notated in terms of lightness with the smaller numbers closer to black and the larger numbers closer to white. Chroma notation in the description indicates the strength of the hue as it departs from the neutral gray of the same lightness as the color to be notated. Value and chroma are separated by a /. As examples, Kansas University "blue" is close to 5PB 3.5/8 and Kansas State University "purple" is close to 5P 4/10. The 5PB and 5P give the blue and purple hues respectively. The 3.5 and 4 indicate that these are on the darker end of the value scale. The /8 and /10 indicate the blue or purple are strongly present or highly saturated. Not a part of the Munsell notation, but included for each color present in the peel was the percent of each color in the total peel. Up to five color descriptions with percents were allowed for each peel.

Texture for each peel is coded by number. The descriptions for the code numbers are given in Table 2. The determinations were made by eye with a 10X lens from the peels. Samples that exhibited zones of differing textures were given two code numbers in separate fields to correspond to the two dominant textures present.

The database program used in this study is Microsoft Access®, a part of Microsoft Office®. Queries of the database provided the information needed to construct r x c contingency tables for statistical analysis. The statistics were run in the spreadsheet program Microsoft Excel®, another part of Microsoft Office®. For this study only parts of the database were accessed heavily, the geologic descriptions of peels and the

Table 2 Carbonate Texture Code and Description

Code	% Fine grained carbonate (mud)	% fossils and/or coarser grained carbonate
1	100-99	0-1
2	98-90	2-10
3	89-50	11-50
4	less than 50	more than 50
5	about equal	about equal coarse grained cement/fossils
6	less than 30	fossils unsorted
7	less than 30	fossils sorted well
8	less than 30	fossils rounded

a) One to two codes can be used to correspond to the mud or or fossils or coarser grained carbonate constituents identified.

b) Identification is aided by 10x lens.

classifications, either 0 or 1, “failed or passed”. Since the geologic descriptions and classifications are categorical, χ^2 tests were used for statistical analysis followed by Cramer’s V tests to render the χ^2 results comparable. These will be discussed more fully in the following section.

Description of the Statistical Analysis Process

The discussion that follows is explanation and illustration of the steps in the analysis of the data; it is not the results of the analysis. Discussion of results is in the following section. Summation of the results is included in the summary. The contingency tables organize the frequency data used for analysis in this study. Classifications of the descriptive data are made into groups that are exhaustive and mutually exclusive, then counted giving frequency, and recorded as a table of the groups. A classification is exhaustive when it provides categories to accommodate all members. It is mutually exclusive when each member is correctly allocated to one and only one category. Categories used were failed or passed, then cross classified with color, or texture plus color. Color in these classifications may be hue, value, chroma or value/chroma. The members in each category are counted and placed in a contingency table of frequencies.

The working hypothesis for the study is that there are differences in mineralogy and texture between the two groups of limestone determined to be passing or failing by physical tests. The null hypothesis is that there will be no relationship of mineralogy as determined by stain color in Munsell notation or texture as coded in the two groups of limestone classed as 0 or 1 by physical test results. The alternate hypothesis is that there

will be relationships present between mineralogy and class and/or texture and class. The test statistic chosen is χ^2 with the dependent variable the classification 0 or 1 and the independent variables as "color" or "texture" or "color with texture". Conventionally the row headings are reserved for the dependent variable and the column headings for the independent variables. These data are shown in reverse of the convention for ease of working with the computer programs available. This makes no difference in calculation or outcomes.

The null hypothesis is accepted, or we fail to reject it, if the calculated value of χ^2 is less than or equal to the tabulated value from χ^2 distribution tables for the relevant degrees of freedom. This acceptance means that for all combinations of the categories presented the probability that an item falls into a particular category combination is equal to the product of the respective category probabilities. This requires determining the expected counts for each category combination and comparing these to the observed counts in the same category combination. This is shown mathematically by the formula

$$\chi^2 = \sum \left[\frac{(O-E)^2}{E} \right]$$

with O for observed counts and E for expected counts. See the publications included in the references for more detailed descriptions.

When the observed cell counts do not agree with the expected cell counts then the null hypothesis is suspect and should be rejected at the .05 level of significance when the tabulated value exceeds the calculated χ^2 value. Once the null hypothesis is rejected it is

known that the variables tested are related or dependent. Almost every calculation showed some degree of relationship.

Once a relationship is shown, Cramer's V test allows comparison of the strength of the relationship to other relationships. This test takes into account the sample size, n, and size of the table by using t, defined as the smaller of the two values found by subtracting one from the number of rows or columns in the table. This is a normed test which means V will fall in the interval between 0 (no strength of relationship) to 1 (complete relatedness of the variables). The formula for Cramer's V test is:

$$V = \sqrt{\frac{\chi^2}{nt}}$$

The strength of relationship as determined by the Cramer's V calculation will be used in the following discussion. Further explanation and discussion of the methods employed can be found in Ott, 1993; Ott, Rexroat, Larson and Mendenhall, 1991; and Everitt, 1977.

Three variables, classification, mineralogy, and texture are considered here. Classification is either passed or failed as determined by physical tests. Mineralogy as evidenced by color notation from the stain peel will be considered first followed by texture and mineralogy divided into the respective texture codes. The process of obtaining the descriptive data of mineralogy and texture for this study included making acetate peels that recorded the staining and texture of the prepared ledge samples, determining up to five colors with percent of the total for each peel, and determining the one or two textures present in each peel. This information was then made accessible from a relational database by querying for specific information and combining results of successive queries when that was required.

From the data of peel descriptions another set of data was derived. Most of the peels had one hue that dominated. The dominant hue might be in one or more color descriptions for that peel. These individual descriptions were added together to obtain the total area of the peel of that hue. One field in the derived database contained the percent of each peel that was in one hue, when one hue totaled 50% or more of the peel. Another field recorded the color code for the dominant hue. The other hues present in the peel were recorded in other fields as associated hues. While there is one dominant color and up to four possible associated colors, the dominant color, divided into percent of area ranges, was paired with each recorded associated color and counted. Then χ^2 and V were calculated. Figure 1 shows the dominant color PB by pass/fail and percent of peel area paired successively with the colors P, RP, and CL with the respective values of χ^2 and V. This example demonstrates the advantages for using Cramer's V for comparisons over χ^2 alone. The variations in χ^2 are larger as the sample size n increases. The sample size varies from 19 to 39. Values of V show easily that when PB is the dominant color and RP is associated with it, there is a strength of dependence of passing or failing that is twice that of PB associated with P.

Mineralogy is evidenced by the color (see Table 1). The dominant colors are given with χ^2 , n and V in Figure 2. While P is the most numerous dominant color, it has the lowest significance among the other dominant colors. PB and RP have a much higher strength of relationship. The fewest in colors are those of YR, CL, R, and B which have been combined in Figure 2 to provide nominally more strength of relationship than P. "No Top" refers to those peels having three or more sub equal color hues such that no one

Associate Classification*	P		RP		CL	
	0	1	0	1	0	1
PB by % Area						
50-59	2	5	3	3	2	2
60-69	5	6	0	2	1	2
70-79	7	3	4	0	4	0
80-89	2	2	4	1	5	2
90-100	5	2	2	0	6	2
X²	4.06		8.45		4.43	
n	39.00		19.00		26.00	
t	1.00		1.00		1.00	
V	.32		0.67		0.41	

*0 is Fail or Class 0, 1 is Pass or Class I.

Figure 1 Dominant color PB when associated with P, RP or CL has stronger relationship to passing or failing when paired with RP as when paired with P.

Dominant Color	Chi-sq	n	V
P	4.77	229	.14
PB	12.25	79	.39
RP	6.89	48	.38
R+YR+CL+B	1.12	33	.18
No Top*	1.58	51	.18

*No dominant color hue present.

Figure 2 Comparison using Cramer's V of dominant colors indicates PB and RP are twice as likely to determine passing or failing as the other groups.

hue dominates. Placing the totals for each dominant color by pass or fail categories into the contingency table shown in Figure 3 gives a very high strength of relationship indicated by the V value of .98. Also notice that P and PB are the only colors that fail more than pass when totaled.

The dominant colors paired with associates are given with V in Figure 4. Using only the dominant color and its pairs gives the most strength to the occurrence of PB associated with RP. There is a group of similar strengths comprised of PB with CL, YR with RP, RP with P, R with any color, RP with R, P with R or RP. The next lowest group is RP with YR+CL+B and last is P with CL. This information is shown rearranged in Figure 5. By the separation of the dominant colors into groups by their associates there is now more information available than by the use of dominant color alone.

Since mineralogy alone does not describe the aggregate, the effect of texture needs to be accorded analysis also. Each hue as listed in the peel description has been sorted by texture code or pair of code numbers. In Figure 6 the hues are listed by pass or fail for texture 1. Note that for texture 1, P and PB fail more often than pass. Overall the table has more failing than passing. Cramer's V is .51, indicating a moderate dependence between class and hue for the texture. The pairs of textures containing texture 1 are different (Figure 7). Several hues are combined to accommodate restrictions in the calculations. Everitt (1977) points out that conservative rule requires each expected outcome cell to be one or greater. He also states that in the majority of cases valid results are obtained if the expected values are above 0.5 in the smallest cell. Combining data decreases the degree of freedom available in the table which lowers the χ^2 value and

Dominant Color	Classification*		
	0	1	
P	118	111	$X^2=516.34$ $n=541$ $t=1$ $V=.98$
PB	48	31	
RP	44	104	
R	4	11	
YR	1	6	
CL	2	3	
B	2	4	
No Top*	16	36	

*0=fail or Class 0, I is pass or Class I

* No Top=No dominant hue present.

Figure 3 Classification of peels by dominant colors and Class 0 or I including those having no dominant hue indicates high relationship between class and hue.

Dominant	Associate	Chi-sq	n	V
P	PB	6.81	168	.20
P	RP	3.10	24	.36
P	R	4.64	37	.35
P	CL	.89	107	.09
P	*	11.20	350	.18
PB	P	4.06	39	.32
PB	RP	8.45	19	.67
PB	CL	4.43	26	.41
PB	**	5.33	101	.23
RP	R	5.95	44	.37
RP	YR+CL+B	1.14	61	.14
RP	P	6.52	40	.40
RP	PB	.99	41	.16
YR	RP	9.98	51	.44
R	P+PB+RP+CL	3.25	22	.38
CL+B+YR	***	1.78	16	.33

*All pairs of P and associates including YR+B combined.

**All pairs of PB and associates including R+YR combined.

***Combining of all colors associated except the pair YR-RP.

Figure 4 Comparison of dominant peel colors with associates indicates PB-RP is strongest and P-CL is weakest using values of V.

Dominant	Associate	V
PB	RP	.67
YR	RP	.44
PB	CL	.41
RP	P	.40
R	P+PB+RP+CL	.38
RP	R	.37
P	RP	.36
P	R	.35
CL+B+YR	***	.33
PB	P	.32
PB	**	.23
P	PB	.20
P	*	.18
RP	PB	.16
RP	YR+CL+B	.14
P	CL	.09

- * All pairs of P and associates including YR+B combined.
- ** All pairs of PB and associates including R+YR combined.
- ***Combining of all colors associated except the pair YR-RP.

Figure 5
Rearrangement of V scores from Figure 4
from the highest to lowest values eases comparison.

Texture 1 by Hue	Class	
	0	1
P	9	4
PB	4	2
R	3	0
B	1	0
YR	0	2
RP	7	12

Figure 6 Sorting by the texture code 1 and then by the hue produces frequencies in each color by classification of passing or failing.

Class	Tx(1-2)		Tx(1-3)		Tx(1-5)	
	0	1	0	1	0	1
Hue						
P	2	2	6	15	0	0
PB	0	1	2	2	0	0
RP	1	8	1	11	0	1
R	0	1	0	1	0	0
YR	0	1	0	0	0	0
B	1	0	0	0	0	0
CL	0	0	0	1	0	0

Texture Pairs (1-2) + (1-3) + (1-5)

Hue	Class	
	0	1
P	8	17
PB	2	3
RP	2	20
R	0	2
YR	0	1
B	1	0
CL	0	1

Texture Pairs Table

Hue	Class	
	0	1
P	8	17
PB	2	3
RP	2	20
No Top	2	5
R+YR+B+CL	1	4

Figure7. The upper table contains data as it was found in the database. The middle table contains the three texture pairs combined. The lower table shows addition of data from some hues to make the contingency table for the χ^2 calculation.

consequently the Cramer's V value. Care must be taken not to lose more information than necessary. For this study R, YR, B, and CL are frequently combined if it is required. The textures of peels with no dominant colors have been added where appropriate. Figure 7 is an example of the progress of the kinds of combining done in this study. Figure 8 presents all the textures exhibited by hue P. Compared with the information in Figures 2, 3, and 4, it is seen that using texture adds information to P that otherwise was not apparent. We note that certain textures or pairs result in more passing or failing while others are nearly evenly represented in passing or failing.

For those contingency tables shown to have significant χ^2 values causing the rejection of the null hypothesis, further analysis can be done by partitioning the original table into 2x2 tables. This procedure is described in Appendix D. For each significant table there are as many of 2x2 partitioned tables within it as the number of degrees of freedom of the originating table. By comparison of these 2x2 tables the source of the significance of the χ^2 value can be found. In Figure 9 the textures for hue RP are presented. The combining of data and 2x2 table partitions are presented in Figure 10.

More information is contained in the color description of each peel. Each Munsell description contains not only hue or color but also value (lightness versus darkness) and chroma (saturation of the color) in the peel portion described. The percent of area of the peel portion is an added component to the description. These variables are placed in contingency tables by hue and texture and percent of total peel. Examples are seen in Figure 11. Geologic literature attributes these as indicators of several factors such as crystallite size, quantity of iron present and crystal chemistry of the minerals. Since these

P Texture	Classification	
	0	1
1	9	4
1-2 *	2	2
2	9	10
2-3	19	21
2-5	0	3
3	55	52
3-4	10	4
3-5	3	7
3-1	6	5
4	0	4
4-5	3	0
5	10	0
5-6	0	2
6+7+8 **	2	0
	n=243	

* Pairs of textures indicated with hyphenation.

** Combination of data for textures 6, 7 and 8.

Figure 8. Textures of hue P sorted by passing or failing show the complexity of combinations and variety of results in this hue.

Textures for RP by Class

RP Texture	Class 0	Class I
Tx1	7	2
2-1*	1	8
Tx2	4	20
Tx3	15	26
3-1	1	11
4-5	2	0
Tx5	3	2
5-6	0	1
Tx6	1	0
Tx7+Tx8	0	3
		n=117

*2-1 denotes the pair having textures 2 and 1.

*Tx7+Tx8 denotes the combining of information for the textures 7 and 8.

Figure 9 Data for hue RP sorted by textures and passing or failing contains several zero and small values. Most peels exhibit textures in the codes 1,2,3, or in combinations of their pairs.

RP Texture	Class	0	1
Tx1		7	12
1-2 + 1-5		1	9
1-3		1	11
Tx 2		4	20
2-3		12	16
Tx 3		15	26
3-4 + 3-5		1	4
Group A*		1	15
Group B*		6	2

*A is Tx4 + 4-5 + Tx 5

**B is Tx 6 + 5 - 6 + Tx 7 + Tx 8

n = 163
= 25.8
V = .40

a	$\frac{7}{41} \mid \frac{12}{83}$	$\chi^2=.11$ V=.03	b	$\frac{8}{40} \mid \frac{21}{74}$	$\chi^2=.58$ V=.06	c	$\frac{9}{39} \mid \frac{32}{63}$	$\chi^2=3.48$ V=.15	d	$\frac{13}{35} \mid \frac{52}{63}$	$\chi^2=4.64$ V=.17
e	$\frac{25}{23} \mid \frac{78}{47}$	$\chi^2=1.53$ V=.10	f	$\frac{40}{8} \mid \frac{104}{21}$	$\chi^2=0$ V=0	g	$\frac{41}{7} \mid \frac{119}{17}$	$\chi^2=.14$ V=.03	h	$\frac{42}{6} \mid \frac{134}{2}$	$\chi^2=10.38$ V=.25

Figure 10 The combination of some data results in a table with fewer small value occurrences. The table above results in eight 2x2 partitions for analysis. These partitions are shown with χ^2 and V values.

(a)

Value	%	Class 0			Class 1		
		0-20	21-60	61-100	0-20	21-60	61-100
9+8+7/*		5	3	6	12	1	2
6/*		12	0	2	1	3	2
5+4/*		2	1	3	1	3	1

$\chi^2 = 19.10$ $df = 5 \times 2 = 10$
 $n = 60$
 $t = 2$
 $V = .40$

(b)

Chroma	%	Class 0		Class 1	
		0-20	21-100	0-20	21-100
*1-4		8	9	7	6
*6		5	6	7	6
*8-10		6	0	0	0

$\chi^2 = 15.20$ $df = (3-1)(4-1) = 6$
 $n = 60$
 $t = 2$
 $V = .36$

Figure 11 When a single hue is sorted for value (lightness) as in 11a or chroma (saturation) as in 11b more information can be obtained about the distribution of characteristics.

indicators can overlap or be absent altogether depending on the source ledge rock, each variable of value and chroma will be taken separately in each hue. Data was counted for the percent of peel area ranges of 0-20, 21-40, 41-60, and 61-100 for both Class 0 and Class I. Often occurrences did not appear in each range and ranges were combined as seen in Figure 11.

Discussion of Results

For the discussion that follows information was obtained from two sets of data. The information about dominant colors and their associates and textures was derived from compiling descriptions of each peel in the original database. If an individual peel had three color descriptions for hue P and the three added together to total more than 50% of the peel area, then P was designated the dominant color with the total area as calculated. The entries from this derived data refer to single peels and not the segmented descriptions of the peel. When value and chroma information is assessed, the data as it appears in the original database is used. If a peel had five colors described and three were in the hue P, the three would differ in value and/or chroma and would contribute individually in the sorting and summations. This information was not sorted for texture pairs but for any incidence of the desired texture code.

The dominant color purple blue, PB, does not exhibit a significant relationship when the associated colors are purple, P, or red, R. When the associated colors are red purple, RP, or clear, CL, then the physical test results are twice as likely to be failing. With only a few samples with associated yellow red, YR, the tendency is for the physical test results to be passing. When the dominant color PB is sorted by percent of combined total peel area and texture, for the textures 1, 3, 5, and pairs 3-2, 3-4, 3-5, the total is 65

peels. These tend to be twice as likely to fail. The remaining textures and pairs are 4, 2, 2-1, 3-1, and 5-2 which total 17 peels that pass only slightly.

When individual peel color descriptions containing PB are sorted by value (darkness or lightness) or chroma (saturation of the color) as well as texture, new information is available. For texture 1 a value of middle gray (6/*) is the only value that gives significant information. It is twice as likely to fail as pass (14 entries versus 6 out of 34 failing to 26 passing). Sorting by chroma indicates that only the highly saturated or least saturated give any information; their tendency is to fail. Using the percent of peel area, the groups 1-40% and 41-100% are equal in the failing portions, but the 41-100% group is half as numerous in the passing portion. Overall, this is confirmed when looking at the texture 1 of dominant PB; those peels with texture 1 or its pairs with other textures are twice as likely to fail.

Sorting entries for those with textures 2 or 3 yields very little relational significant information (total of 128 entries in texture 2 and 372 in texture 3) when sorting only by value or chroma. All categories are very close in numbers for passing versus failing. This is also the situation in dominant color sorting for texture 2. With texture 3, dominant PB favors failing twice as much as passing.

For texture 4, PB fails two times more for either the value or chroma sortings or for dominant color and texture groupings (45 and 12 entries respectively). There are no other significant differences in the categories. The situation is similar for texture 5 information (47 entries) except for dominant color PB that has six peels, five of which fail physical tests.

Dominant color red purple RP when sorted for its associated colors yields much information about relationship. With purple P, purple blue PB, yellow red YR, and clear CL, there are totals of 40, 41, 3 and 61 peels respectively, each color associate having over two times the rate of passing as failing. When associated with red R there are 37 passing to 7 failing in the 44 peels for over 5 times passing as failing. There is only one peel with the associate blue B and it is passing.

Sorting the dominant color RP by texture 1 and its pairs gives a total of 41 peels, 9 failing and 32 passing. Of these 41, texture 1 accounts for 7 failing and 12 passing. Of the remaining peels 2 fail, one each in pairs 1-2 and 1-3 with 8 and 11 passing respectively. One peel exhibiting texture pair 1-5 passes. These three pairs average 10 times the rate of passing over failing. Peels exhibiting texture 2 or its pairs 2-3 or 2-1 (2-1 is the same as 1-2 above) number 58, with 14 failing and 44 passing. Texture 2 shows 5 times more passing than failing and pair 2-3 gives 2 times more passing. Sorting by texture 3 and its pairs gives a total of 64 peels passing and 31 failing. Except for pair 3-1 discussed earlier, texture groups in this sort have similar relationships as the average. Sorting for textures 4, 5, 6, 7 and 8 yields 19 peels total, 7 failing, some texture groups having only one peel exhibited. These small values give little meaningful information. Overall, dominant color RP sorted by textures shows greater tendency to pass compared to fail but there is also great variation depending on the texture exhibited.

Using the data for incidences of RP in peel descriptions with textures gives additional information when sorting on chroma and value. Texture 1 included 114 entries, 89 passing and 25 failing. When sorting by value each category from light (9+8/*), medium (7/*) to dark (6-3/*) has about the same rate of passing over failing of 3

to 4 times. Passing is 5 times more likely when the chroma is highly saturated (*8-10) and around 3 times in medium (*6) and low (*2-4) saturations. Notation (9+8/*) refers to values of 9 and 8 with any chroma and (*6) refers to any value with the chroma notation 6. Looking at occurrences of value and chroma together, the light values have the same rate of passing in both high and low saturation. For medium value, high saturation occurs 5 times more in passing than failing and only about 2.5 times more when the saturation rate is low. In dark gray values the highly saturated entries are 6 times more likely to pass and the low saturation entries are only slightly more likely to pass over fail.

The RP texture 2 information shows 107 passing out of 151 entries for an overall average ratio of passing over failing of two to three. In addition, the number of entries over 41% in area have about four times more likelihood of passing over failing. The total number in the category of area over 41% is 53, a larger portion of total entries than is usual in the peel area descriptions. Using value criteria, the light or dark entries have 3 to 4 times more chance of passing; the medium values of (6/*) or (7/*) have only a slight favor or two times the rate of passing. In the chroma sorting, the information shows that about 3/4 of the entries have low saturation with 2.5 to 3 times more likelihood to pass than fail. Medium saturation is slightly favored to pass, but highly saturated entries pass 2.5 times more than fail. The value with chroma sorting shows this complex pattern with 76 entries in the light and dark values of any saturation passing with about 3.5 times the rate of failing. The middle values show 75 entries with passing twice the rate of failing.

Over half the total of 262 entries in RP texture 3 have areas over 21%. Of that group, passing is twice as likely as failing. In the 1-20% area category, the entries are

equal in passing and failing. When the area of the peel is over 60%, the peel has a 3 times better chance of passing. If the area falls between 21 and 60%, it drops to 2 times the chances of passing over failing. About half of each of the passing or failing categories is in the light value range so it shares the same probabilities of passing or failing as the texture 3 overall average which is slightly favoring passing. Chroma sortings indicate that light saturation is numerous in the entries with incidences in each other category reducing as the saturation increases. Still the average for the whole describes the tendencies for each saturation range and not much more information is gained. When value and chroma are sorted together, the frequencies of occurrence indicate that except for the lightest value with the lowest saturation which has a rate of passing over failing of 3 times, there is a tendency to fail when the saturation is low. Moderate and high saturations both have twice the rate of passing over failing.

Very little relational information is contained in the RP texture 4 for sorting except that 19 of its 23 entries are light values with low saturations and nearly evenly split between passing and failing. There are 39 entries in the texture 5 sorting almost equally split between passing and failing, but there is not a pronounced incidence of light over dark values as in texture 4. The chroma saturations show even less favoring of any range of saturations. With 15 entries total for textures 6, 7, or 8 there are two failing. Not much more information is available.

The dominant color P shows very little information for the associated colors of RP, clear CL, B or PB. The exceptions are associates R and YR. There is nearly two times the chance of passing over failing when dominant P is associated with R, five times

the chance of passing when associated with YR. These two associates account for only 50 of the 400 peels.

When P texture 1 is sorted for value and chroma the chance of passing is twice that of failing in 94 peels. If the description of value and chroma are both medium then the chance of passing is three times greater than failing. All other categories favor failing even if only slightly. This is shown more when dominant P is sorted for texture 1 and its pairs. There is slightly more chance of passing over failing in texture 1. Taken together texture 1, pairs 1-3 and 1-2 total 38 peels, with 23 passing. When P texture 2 is sorted for value and chroma there are 68 failing and 97 passing and all categories show about the same proportions. For the dominant P texture 2 sorts, those passing and failing are about evenly split only slightly favoring passing. Even in P texture 3, the information is of little significance whether sorting for value or chroma, or those together or for dominant color. There is only slight favor of passing or failing in any sort or category.

Sorting for P textures 4 or 5 gives more information. Texture 4 peel descriptions are more likely to fail 2 to 1 and texture 5 will fail 3 to 2. These sorts total over 100 entries. When value and chroma of texture 4 are sorted together then light values with low saturation are more likely than the average to fail as well as medium value with medium saturation and dark values with high saturation. There is not any clear trend in texture 5.

The colors B and CL are not represented as dominant peel colors in large enough numbers to gain much information. The color B occurs in 24 peel descriptions. It is favored to pass by over 2 to 1. In texture 1 all three entries pass, while in texture 5 both entries fail. Textures 2 and 3 comprise the remaining entries with 5 failing to 14 passing.

The colors discussed to this point are those associated with the carbonate minerals calcite or dolomite with varying amounts of iron included in the structures. The color R describes calcite with no iron. Only 55 color descriptions contain R. Passing to failing is a ratio of 3 to 2. Sorting for textures 1 and 2 indicates that these ratios of passing to failing are 2 to 1. Textures 3 and 5 have ratios of 1 to 1. Using the dominant color R to sort for associated colors, we find that the ratio of 2 passing to 1 failing is similar overall but again the details differ. Associated colors P and RP have 4 times more passing than failing peels, PB is 1 to 1 and clear is associated very weakly with failing. Interestingly, R usually passes by over 2 to 1 when it is an associated color for the other dominant colors.

The color YR or yellow red is not considered to represent carbonate minerals as the colors B, P, PB, RP and R do. This color probably indicates other minerals having more iron content. However, it occurs in enough of the descriptions that some study was indicated. As a dominant color YR is mostly associated with RP with 30 of 51 peels failing. Texture sorting of dominant YR data shows YR passing. Using the peel descriptions (not dominant YR data but all incidents of YR) and sorting by textures gives some relationship information. All texture 1 entries pass, texture 3 entries pass 3 times as often as fail and textures 2 and 5 have about equal chances of passing or failing. As a whole nearly 3 times more entries with YR pass as fail.

To summarize, each color hue sorted by itself shows at least a small amount of relationship to passing or failing since most have calculated χ^2 values exceeding tabulated values. By dividing the color hue descriptions into value or chroma categories there is

usually a useable increase in information at least in portions of the table. Adding texture divisions greatly increases the information on relationship in most cases. When dominant color with associated colors for each peel is studied, there are similar increases in information as texture is included in the components being analyzed.

The categories of Class I and Class 0 are determined by several tests, each having a passing or failing result. Since Class I and Class 0 were not divided for analysis by individual test results of passing or failing or by their scores, there is a source of information available for further refinement and definition of relationships. That this refinement is probable is predicted upon the results of the divisions of color and texture that added information upon analysis that has been demonstrated in this study. Also an early application of the staining procedure for forensic use reinforces some of the findings of this study. This study had just begun with no data available. A description of the application follows.

The use of the staining technique had begun by Research Geology personnel when there was a question of aggregate source in a producing quarry. The making of peels was in progress for this study so peels were also made of the ledge samples from the quarry. There were three production ledges and originally three non-production ledges. Stained peels results indicated that purple colors were obtained from the production ledges with the exception of thin veins that stained blue in some zones. From the non-production ledges there was much more variety. One ledge stained with some purple areas similar to the production ledges; the remaining zones stained shades of green, blue green and blue. Another non-production ledge stained very consistently blue. The third ledge and the one visually much different from the others stained poorly to a bronze color or the treated

portion failed to retain the stain. The washing step removed the majority of staining and yielded a darker yellow color than was apparent before the staining process in the sample.

Since the portion of non-production contamination had to be determined in the crushed aggregate, some of the ledge rock was crushed and sieved, then stained by immersion and washed as if in preparation for making a peel but then dried and the colors noted. Each ledge sample in the crushed and stained state yielded darker, more intense colors than the corresponding ledge sample peels. For the ledges yielding purple stained peels, the corresponding aggregates stained similarly to the "KSU purple" color given as an example earlier. The ledges yielding blue or blue green stained peels produced blue in the stained aggregates similar to the example "KU blue" in color and intensity. It was noted however, that no production sample yielded blue stain as in the non-production samples. In the production ledges samples there were some thin blue veins surrounded by purple stain in the crushed aggregate. This gave a characteristic, spidery pattern that was not found in the non-production samples. This was easy to distinguish. Since some of the non-production ledges also stained purple as did the production ledges, any count of stained particles would give a conservative estimate of contamination in favor of the quarry. Staining the crushed aggregate of a stockpile became a quick way to check stockpile contamination.

Once contamination was shown in the stockpiles the next task was to find the level of contamination in the pavements since considerable production had already been incorporated into the placed concrete. Random, unbiased core sampling to determine pavement thickness served as a source of cores to help in this determination. Tests indicated that concrete cores could be stained and rinsed just as the aggregate had been

treated. Whole cores could be immersed in staining solution, rinsed, airdried and the resultant colored aggregates tallied on each core. Locations were known for each core and these corresponded to placement dates. Time sequencing of contamination could be found. As litigation was a possibility, the request for more in-depth identification of the aggregate by the petrographic description of the natural aggregate was made. Stained cores were cut longitudinally yielding exposed aggregate that could be examined with no interference from the staining. Figure 12 and 13 indicate the results of contamination found in the cores from two paving projects.

Examination of the ledge samples by X-ray diffraction was also done to further detail the differences between the production and non-production portions of the quarry. Since the stain colors implied differences in mineralogy and chemistry of the carbonate minerals calcite and dolomite, these differences should be apparent in the diffraction peaks of the rocks made of the differing minerals. A Philips 1840 X-ray diffractometer was utilized for this part of the study. The samples were selected from the previously submitted ledges and stockpiles and also from samples hand selected by quarry operators and KDOT geologists. The samples were powdered, mounted into holders and radiated by copper k alpha X-rays and the results recorded from 2° to $70^{\circ} 2\theta$. Typical peaks from the production ledges were identified as calcite and quartz. Calcite has a strong peak at $29.44^{\circ} 2\theta$ or d-value of 3.03. All the production samples showed this peak and many others corresponding to calcite and also quartz, a silicate. Examples of patterns characteristic of calcite and quartz are shown in figures 14,15 and 16 from this project. Those samples from the production ledges having blue veins in stained samples or pink

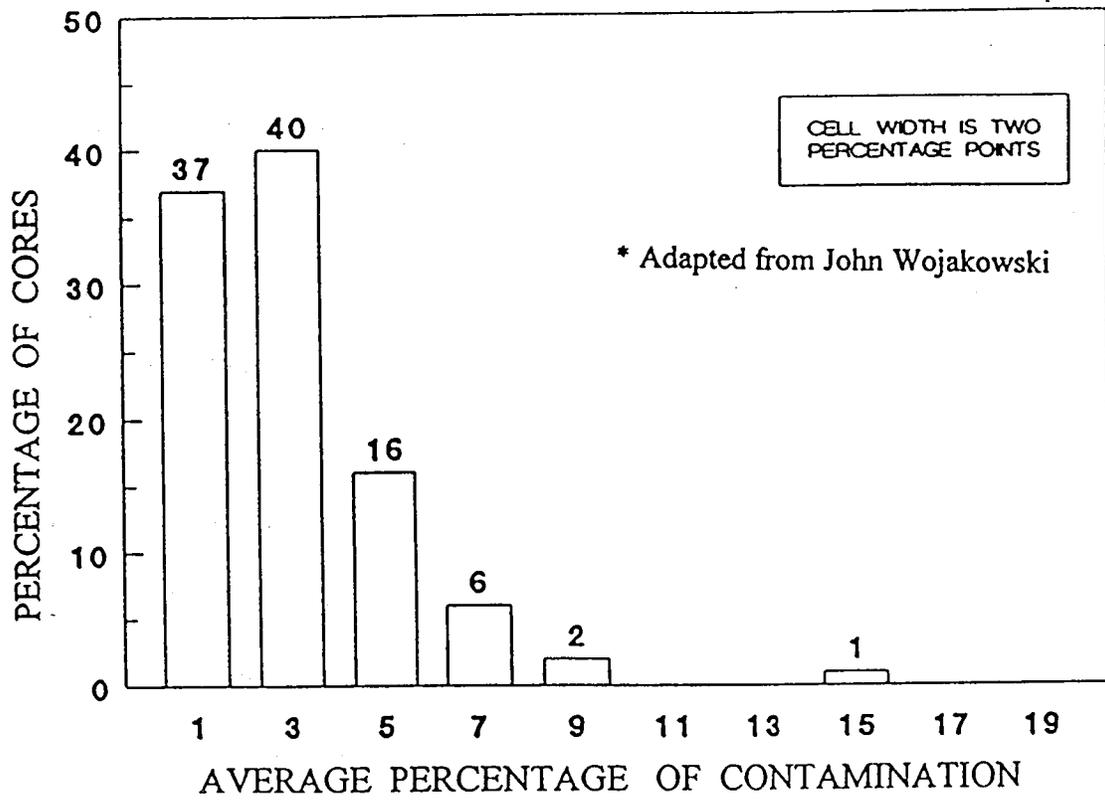


Figure 12 One project receiving crushed aggregate from the suspect quarry had a concrete mix with 30% limestone crushed aggregate. The resulting cores yielded these proportions of contamination in the limestone aggregate. Contamination was identified by staining the concrete cores as described in the text.

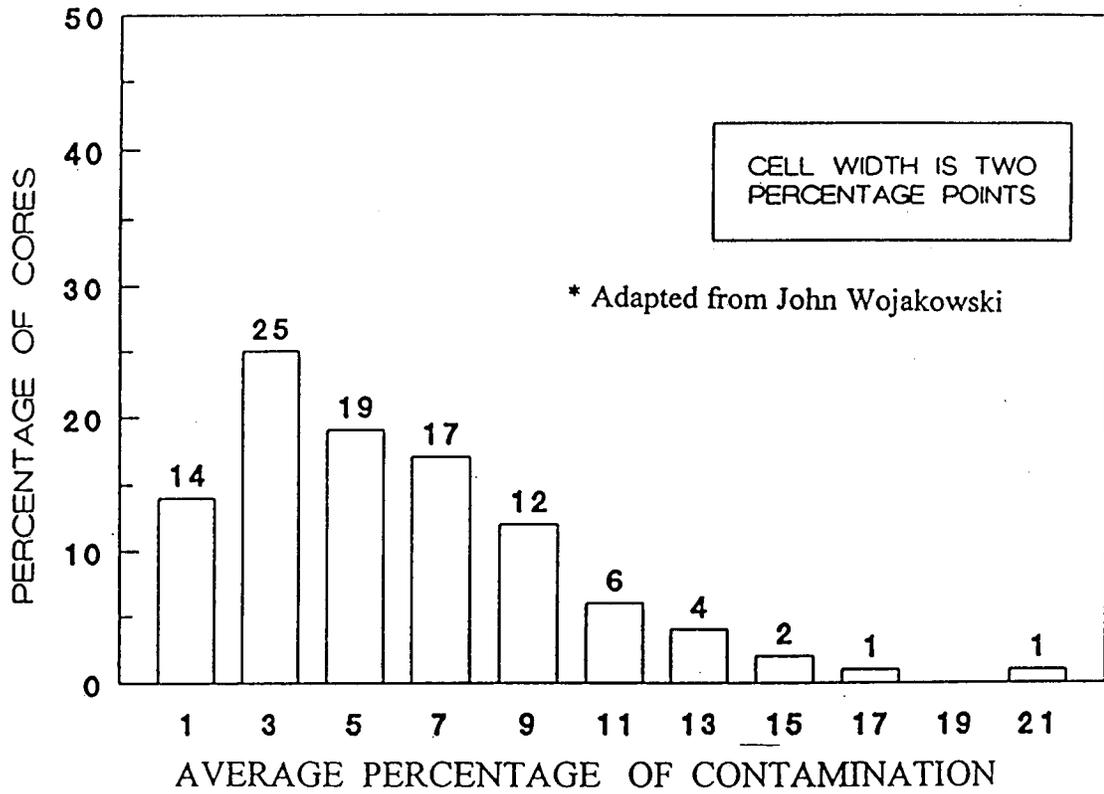


Figure 13 A different project using the same quarry used 50% crushed limestone in the mix. Contamination was identified by staining the concrete cores.

sample: 202 sf=5, tc=.2

5-nov-93 16:1

PHILIPS Analytical

PC-APD, Diffraction Software

data file name: 202.RD 5-nov-93 15:55
diffractometer: PW1840
x-ray tube: Cu LFF 40kV 50mA
wavelength(a1,a2): 1.54060 1.54438
full scale(*1000): 5
time constant: .2
receiving slit: 0.05
range 2theta: 10.000 - 70.000
step width, time: 0.020 0.40
peaks file name: 202.D1 5-nov-93 16:19
range d-value: 1.3430 - 8.8382
range tip width: 0.00 - 2.00
minimum significance: 0.75
threshold(cps): 250 counts: 100
conversion to: AUTO

peak no.	d-value (Ang)	angle (deg)	width (deg)	peak (cts)	backg (cts)	I/Imax (%)	significance
1	3.8554	23.050	0.100	169	49	8.89	1.72
2	3.0341	29.415	0.160	1901	53	100.00	18.24
3	2.4944	35.975	0.100	342	61	18.00	2.18
4	2.2840	39.420	0.160	524	62	27.59	8.45
5	2.0943	43.160	0.100	484	66	25.46	3.21
6	1.9273	47.115	0.100	166	69	8.75	1.00
7	1.9120	47.515	0.100	449	69	23.64	2.23
8	1.8748	48.520	0.120	506	69	26.63	3.46
9	1.6245	56.610	0.200	102	74	5.37	1.90
10	1.6039	57.405	0.140	250	76	13.13	2.52
11	1.5245	60.700	0.160	166	79	8.75	2.18
12	1.4396	64.700	0.120	199	79	10.46	1.73

Figure 14 This typical sample from the producing ledges of the quarry gives the peaks characteristic of calcite.

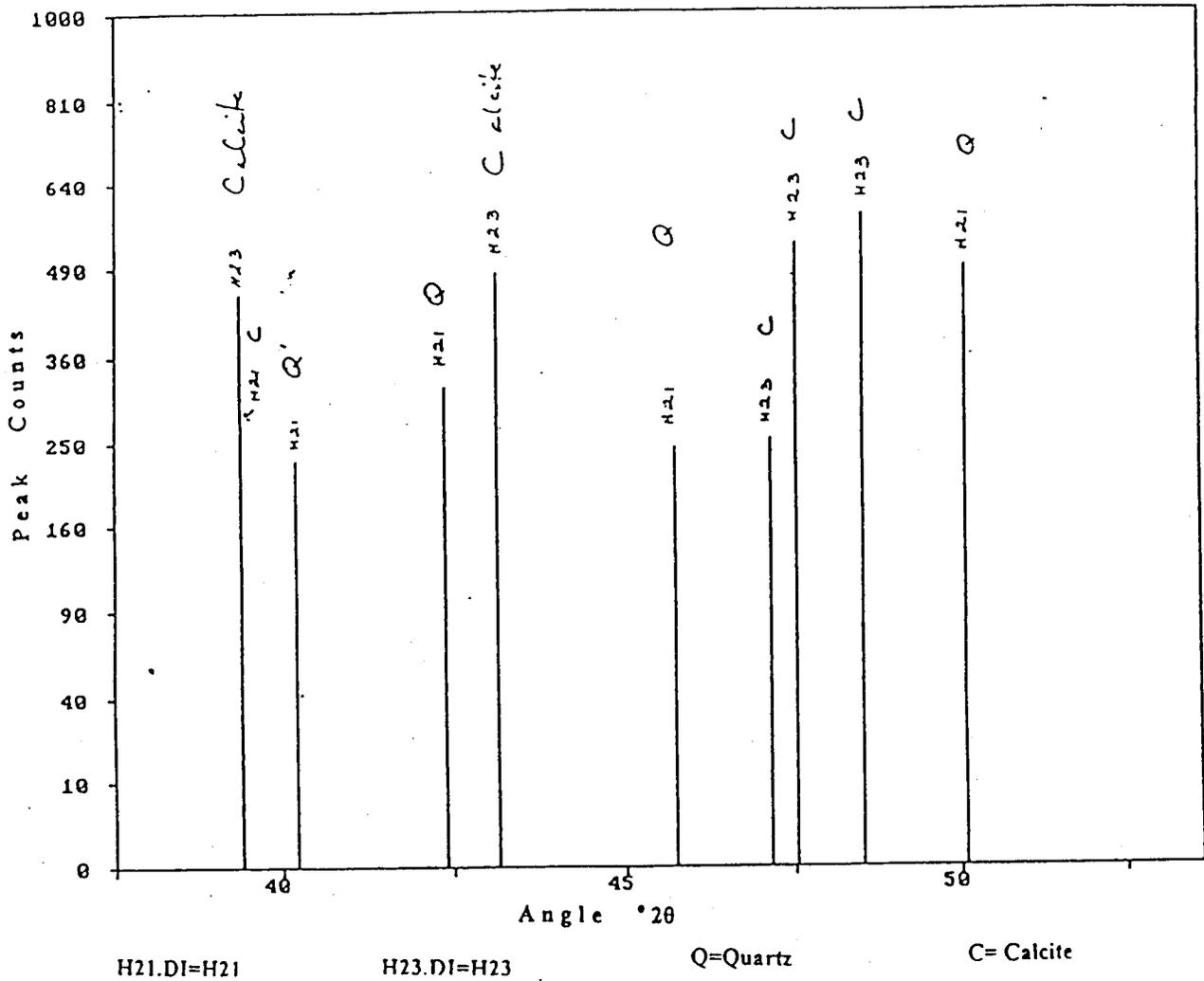


Figure 16 Use of software for X-ray pattern analysis can aid in identifying minerals in samples. This is a small part of the peak patterns shown in Figure 15.

minerals in hand samples also exhibited one or more peaks characteristic of an iron-bearing dolomite named ankerite. The strong peak for ankerite occurs at $30.7^\circ 2\theta$ or d-value of 2.90. When non-production samples were tested, many more peaks characteristic of ankerite were detected along with characteristic peaks for calcite. Figures 17 through 20 give examples from X-ray data. One exception was the non-production ledge that was yellow in color and did not stain well. Most of the non-production samples also had peaks for calcite and quartz. These results indicated that other chemistry tests on the ledge and concrete samples might be able to detect contamination presence. Since there was more high iron bearing dolomite in the non-production samples than the production ledges, differences might be detectable for iron and associated elements.

A Thermo Jarrell Ash ICAP-61E Spectrometer (ICAP) was used for chemical element analysis. This is a plasma emission spectrometer programmed to read all the chosen elements in a single sample run. The emission source of ultraviolet radiation is an inductively coupled argon plasma. Several sample sets were run against several standards prepared in the same manner as the samples. Acid digestion and fusion with lithium borate were preparation methods tested. Results of the testing varied with the manner of sample selection and preparation. During this time another laboratory used X-ray fluorescence to test concrete beams from the project. Our testing of the same beams by the fusion method on ICAP produced comparable results to their chemical element pattern results. It was decided that the ICAP method did not give quick enough results to justify the trouble involved in sample preparation and testing for information obtained.

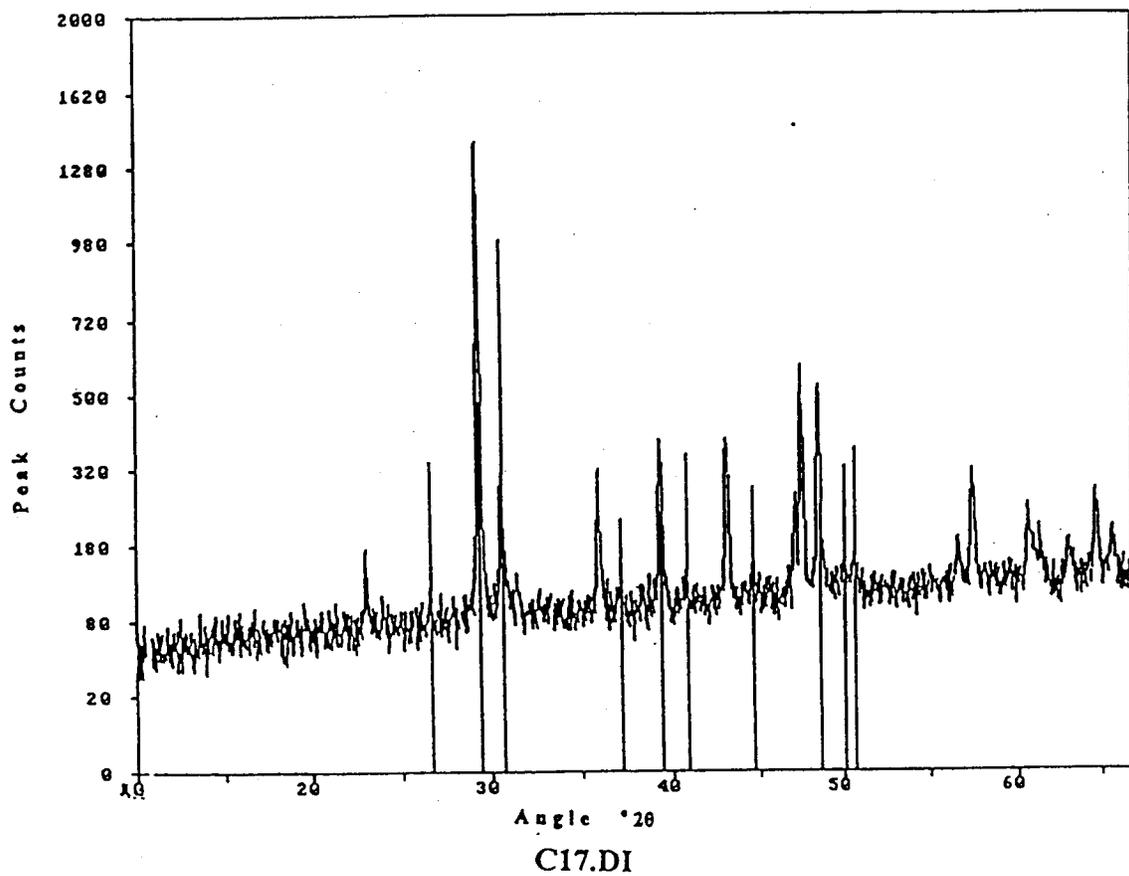


Figure 17 Raw data for a stock pile sample produces the jagged lines. The DI pattern is the result from one non production sample. Superposition of the two files can aid in identifying minerals in the samples. This stockpile sample contains ankerite similar to that in sample C17.DI

sample: C17

27-oct-93 16:42

PHILIPS Analytical

PC-APD, Diffraction Software

data file name: C17 27-oct-93 13:09
diffractometer: PW1840
x-ray tube: Cu LFF 40kV 50mA
wavelength(a1,a2): 1.54060 1.54438
full scale(*1000): 5
time constant: .2
receiving slit: 0.05
range 2theta: 10.000 - 70.000
step width, time: 0.020 0.40
peaks file name: C17.DI 27-oct-93 16:42
range d-value: 1.3430 - 8.8382
range tip width: 0.00 - 2.00
minimum significance: 0.75
threshold(cps): 250 counts: 100
conversion to: AUTO

peak no.	d-value (Ang)	angle (deg)	width (deg)	peak (cts)	backg (cts)	I/Imax (X)	significance
1	3.3435	26.640	0.140	246	88	27.76	3.36
2	3.0330	29.425	0.120	388	94	43.70	3.16
3	2.9104	30.695	0.180	888	104	100.00	9.46
4	2.4166	37.175	0.200	108	117	12.18	1.17
5	2.2812	39.470	0.200	114	121	12.89	1.42
6	2.2063	40.870	0.160	228	123	25.68	2.18
7	2.0266	44.680	0.280	149	132	16.76	3.24
8	1.8720	48.595	0.240	110	139	12.41	1.61
9	1.8201	50.075	0.160	182	142	20.52	0.79
10	1.8008	50.650	0.120	222	144	25.00	0.99

Figure 18 Sample C17 peaks show a mixture of the mineral ankerite. This sample reduced to a DI pattern is shown in Figure 17. Differences in the proportions of the minerals in the mixtures making the sample account for many of the differences in presence or absence of peaks in comparing two samples.

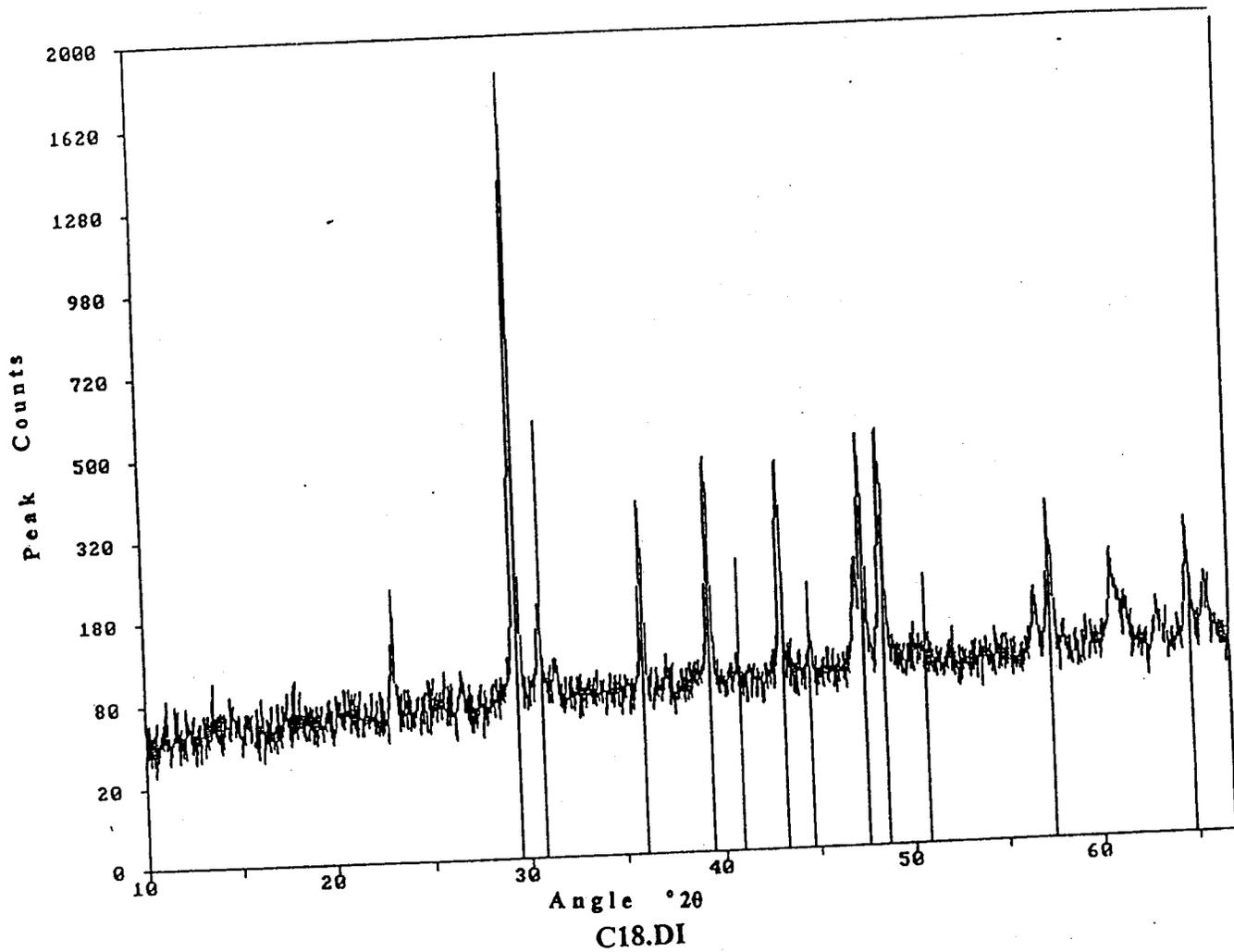


Figure 19 Raw data from a different stock pile sample and a different DI file of a non production sample again show identifying characteristics of the mineral ankerite.

match score list sample: C18 3-dec-93 10:33

PHILIPS Analytical PC-APD. Diffraction Software

```

data file name: C18                      27-oct-93 13:30
diffractometer: PW1840
x-ray tube: Cu LFF 40kV 50mA
wavelength(Å): 1.54060 1.54438
full scale(*1000): 5
time constant: .2
receiving slit: 0.05
range 2theta: 10.000 - 70.000
step width, time: 0.020 0.40
peaks file name: C18.D1                      3-dec-93 10:28
range d-value: 1.3430 - 8.8382
range tip width: 0.00 - 2.00
minimum significance: 0.76
threshold(cps): 250                      counts: 100
conversion to: AUTO
reference directory: \POW\
number of patterns: 134                      group: 1 2 3 4
  
```

score	reisc.	IX	disp	DI-file	grp	name	formula
5.07	0.32	48	24	240027	3	CALCITE	CaCO3
4.21	0.22	33	37	50586	3	CALCITE	CaCO3
1.81	0.10	28	-77	120088	3	ANKERITE	Ca(Hg,Fe)(CO3)2
1.80	0.09	28	-77	340517	3	DOLONITE, ferroan	Ca(Hg0.67Fe0.33)(CO3)
1.80	0.09	11	-52	330282	3	ANKERITE	Ca(Fe,Hg)(CO3)2
1.43	0.06	11	-185	360426	3	DOLONITE	CaHg(CO3)2
0.58	0.03	6	-294	110078	3	DOLONITE	CaHg(CO3)2
0.47	0.08	3	-294	210540	3	SPINEL, Fe	Hg(Al,Fe)2O4
0.18	0.00	2	3	361461	3	ANGLESITE	PbSO4
0.09	0.01	1	-294	211152	3	SPINEL	HgAl2O4
0.09	0.01	1	-88	70239	3	BRUCITE	Hg(OH)2
0.09	0.01	1	-294	251376	3	MAGNETITE	(Fe,Hg)(Al,Cr,Fe,Ti)
0.00	0.00	2	-294	160613	1	VERMICULITE	Hg _x (Hg,Fe) ₃ (Si,Al) ₄ O
0.00	0.00	1	-294	20045	3	BIOTITE-1M	K(Fe,Hg)3AlSi3O10(OH)
0.00	0.00	1	-294	20714	3	CALCITE, manganoan	(Ca,Mn)CO3
0.00	0.00	1	-294	50453	3	ARAGONITE	CaCO3
0.00	0.00	1	-294	50628	3	HALITE	NaCl
0.00	0.00	1	-294	60263	3	MUSCOVITE-2M1	KAl2(Si3Al)O10(OH,F)
0.00	0.00	1	-294	60710	4	PYRITE	FeS2
0.00	0.00	1	-294	70025	3	MUSCOVITE-1M	KAl2Si3AlO10(OH)2

Figure 20 Peak matching by a software program produced this list of possible minerals to account for the peaks found in the nonproduction sample. Analysis by comparing the listed patterns with the peaks in the sample indicated this sample contained ankerite and calcite.

The quick identification of levels of contamination by staining allowed the cutting of beams from the pavements for laboratory testing of expansion and durability. The beam sites were selected to reflect the range of contamination levels found in the cores. The beams obtained gave the results summarized in Figures 21,22 and 23 for the two paving projects.

Late in the forensic process we discovered that two sources of aggregates found in the paving cores but unidentified from the original three Class I and three Class O ledges actually came from zones below the production ledges and were constituting the floor of the quarry. Previous to this identification these aggregates were categorized as "unknown". Once identification was made it was verified that these also were unacceptable for Class I and should be regarded as part of the contamination. One zone stained dark blue and one stained light blue. Natural color and mineralogy were much different from the six original ledges.

Test beams were made using a range of contamination levels from 0 to 40%. The laboratory results confirmed that the tendencies for the contaminated field beams to perform poorly in testing were not the artifacts of sawing and retrieval from the pavement and transferring to the laboratory to be tested. This data is summarized in Figure 24. The contentions with the quarry supplying limestone aggregate for these pavements were settled out of court in favor of KDOT.

The quarry forensic application is interesting for several reasons. It was done when this study was beginning with no data available for relationship to Class I or Class 0 aggregate. The quarry peel colors P and PB fall into the categories of passing and failing respectively, which follows the general relationship findings of this study. The

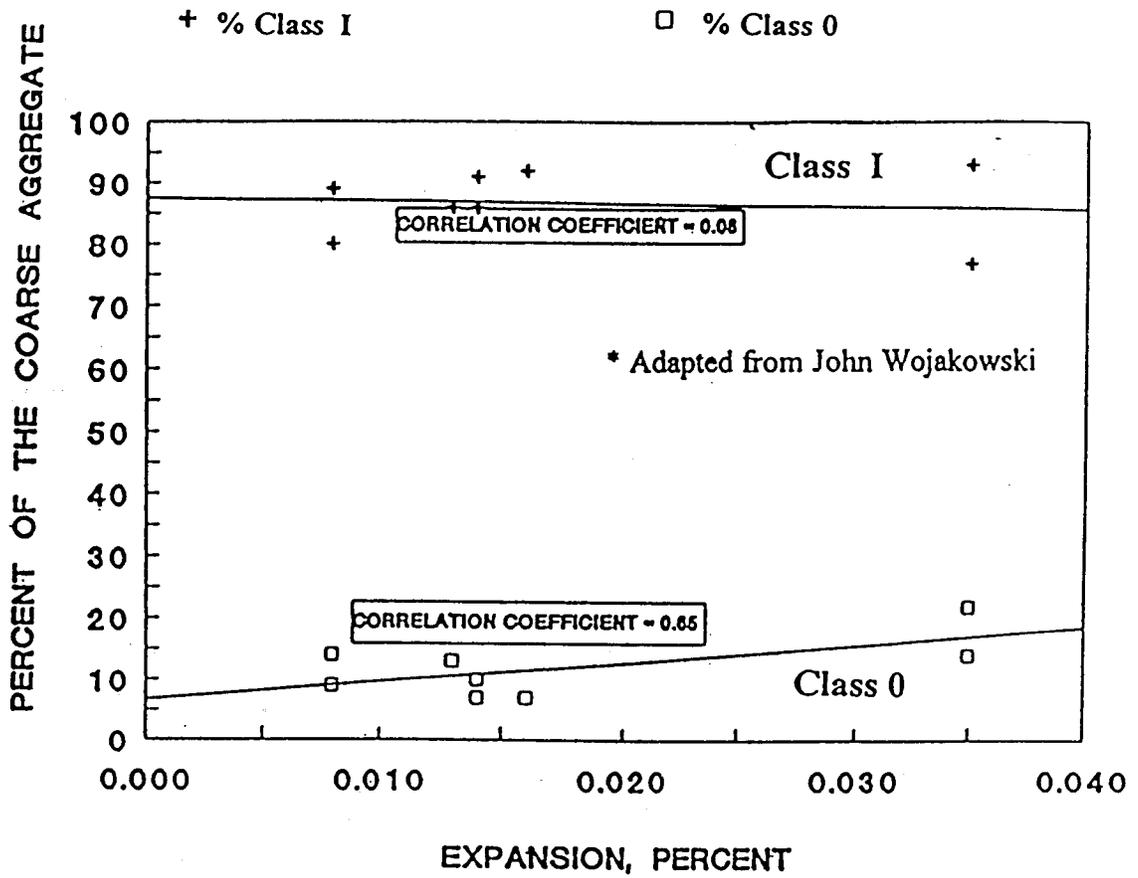


Figure 21 Correlation of aggregate contamination found in pavement with 50% crushed limestone aggregate in the mix with expansion test results is shown by two lines, one based on the percent of Class I aggregate and one on the percent of known contamination. These were beams cut from the pavement and tested in the laboratory.

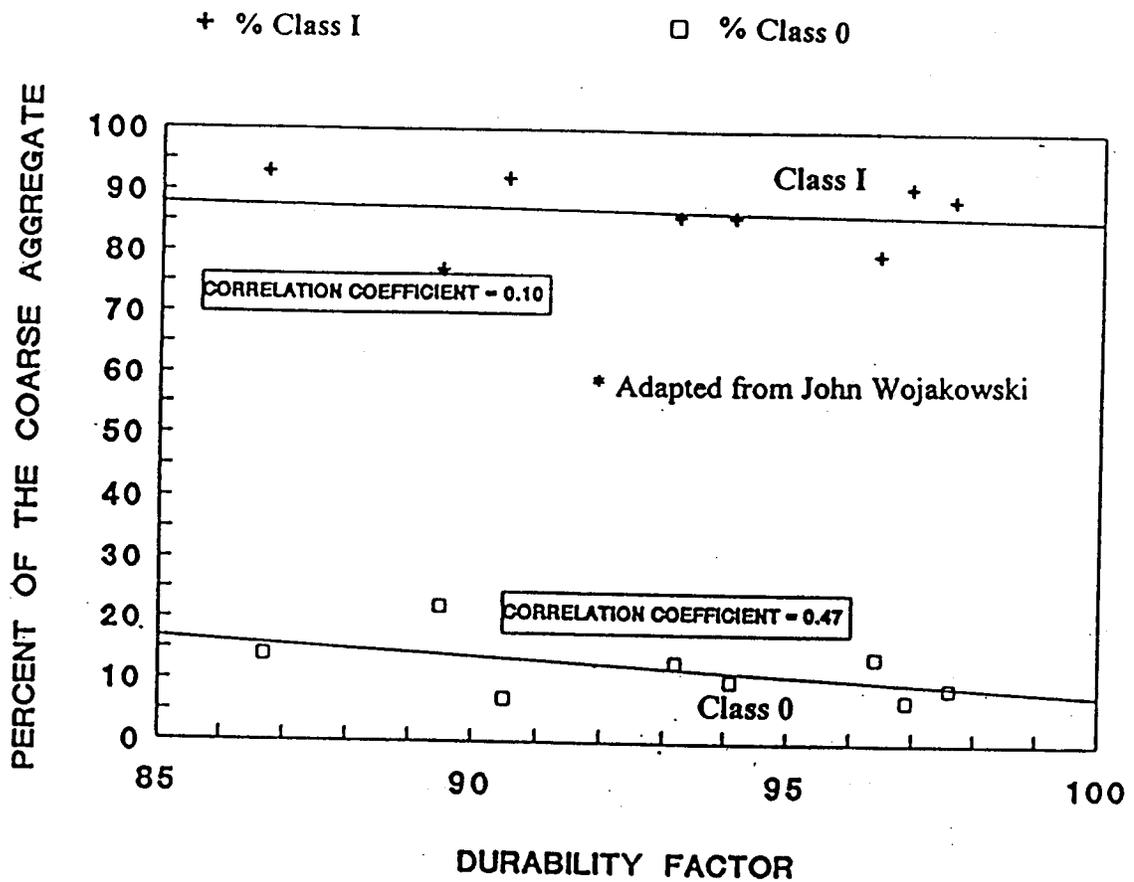


Figure 22 Durability test results correlated to contamination in beams cut from the pavement made with 50% crushed limestone are shown as in Figure 21. Note that in Figures 21 and 22 higher correlations are obtained when using the values of contamination than the values of acceptance aggregate. Aggregate noted as "unknown" was not included in amounts of contamination until late in the investigation.

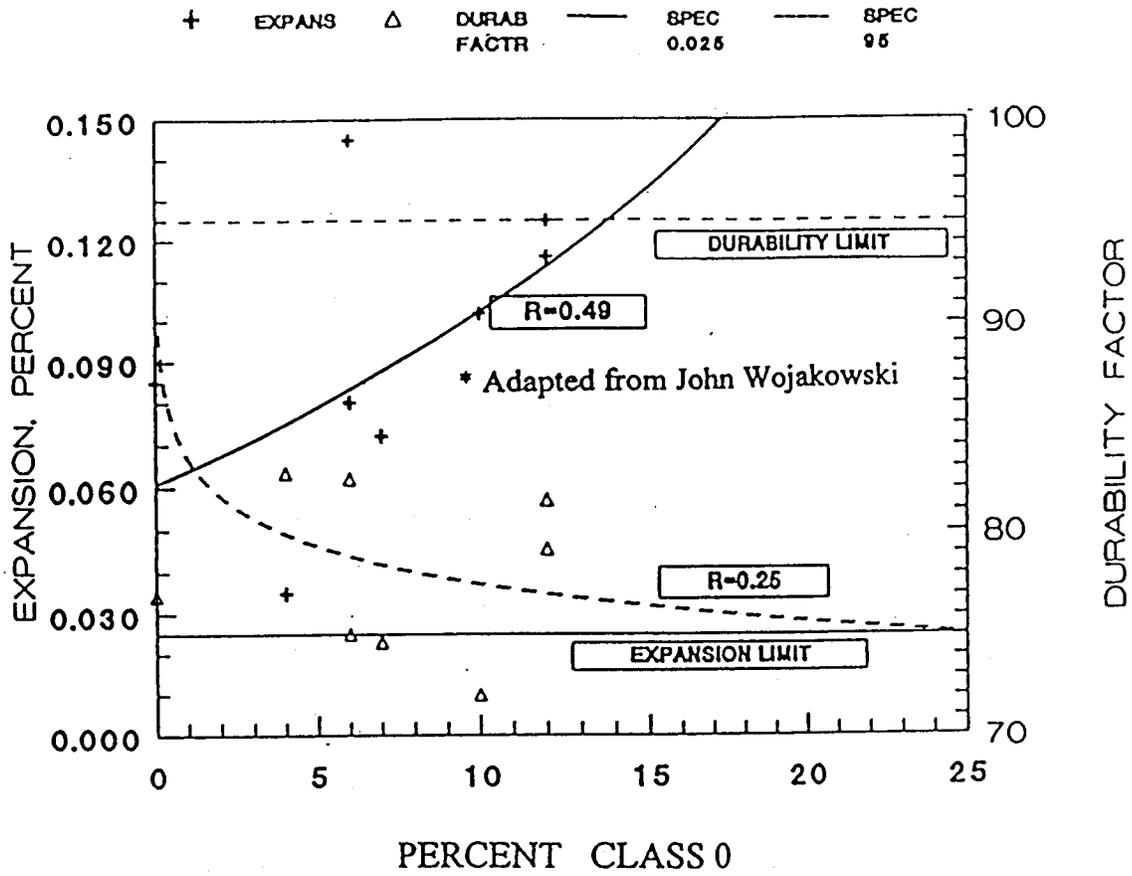


Figure 23 Correlation of expansion and durability factor results with percent contamination in the beams cut from pavement having 30% crushed limestone in the mix are shown. Compare this pavement mix with that in Figures 21 and 22.

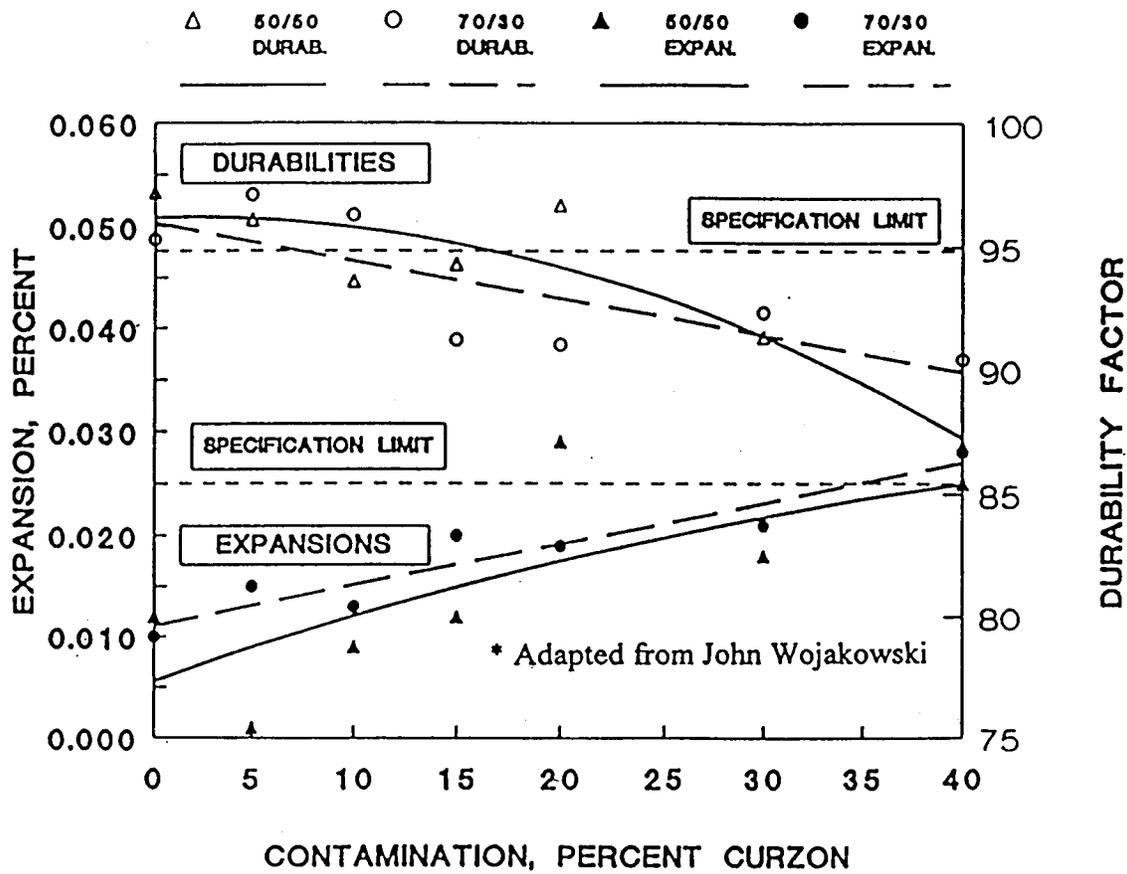


Figure 24 Summary of test results for expansion and durability on laboratory beams made with both mixes found in the paving projects are shown with 0-40% contamination levels. Aggregates used were from the same sources as the paving projects.

quarry ledge that was “yellow” in natural and stained color (YR in color notation), while failing physical tests was the best performing of the non-production aggregates. As a statistical category the color YR performed better than the blue hues. There was no texture categorization in the quarry project for closer comparison to the statistical analyses.

The quarry project forced finding ways to determine quickly whether a stockpile sample had unacceptable components and for this project the staining procedure worked. Staining is usually used as a research tool but handled carefully can be used in the field as a screening or prospecting tool. What needs to be determined is the relationship of stained peel colors from polished samples to colors resulting from staining crushed aggregate or freshly broken quarry ledges. Also the experience with crushed aggregate provided a means to determine aggregate identification in cored concrete pavement. The tools available in the research laboratory, notably X-ray diffraction (XRD) and ICAP spectrometry, while valuable for identifying individual samples, were not the best choices for processing the large volumes of work and providing the type of information needed in the quarry project application. XRD and ICAP were valuable by validating the staining color differences that proved useful.

Since the mineral ankerite is not mentioned in the general literature with the intense blue stain results found in this quarry, more research to better define this relationship may be useful. The carbonate minerals constituting limestone are difficult to differentiate since they are very similar. Kansas limestones thought to be potential aggregates for concrete, when studied in the past with XRD to determine ratios of dolomite to calcite, have failed to show any relationships to passing or failing physical

test requirements. The iron content of the different carbonate minerals and its patterns of occurrence rather than just the mineral types themselves may be determinative of performance in concrete. If this can be shown then prospecting or inspecting quarry ledges might be easier and quicker.

Natural aggregate color was also used effectively as part of the petrographic determination of ledge sources in the quarry application. Natural color descriptions are included in the study database, but have not been accessed for statistical analysis. Are there relationships between natural colors and stain colors or physical test scores? This should be investigated.

Summary

Two tables summarize the information from the statistical analysis and discussion. Table 3 gives the ratio of passing to failing peels in the dominant color column. The notation "with" indicates that the color mentioned after "with" is an associated color. Associated colors, single textures and texture pairs have been placed in the table in the appropriate dominant color row and the most fitting category between passing and failing. Tx refers to a single texture and hyphenated numbers in parenthesis indicate a texture pair in the same peel. Table 4 presents information in a similar format. Since this table uses the entire color description of each peel and each texture code, but not texture pair information, the category contents reflect the differences in information. In both tables the categories from "Passes" to "Fails" are based on both the ratios of Passing to Failing counts and the significance of the scores. For this reason the categories are indicators of tendencies and not defined by percentages. By utilizing the numerical test scores of individual physical tests, relationships may be defined more clearly in the

Table 3 Summary Table from Dominant Colors

Pass/Fail in Dominant Color Only	Passes	Passes more than fails	50/50	Fails more than Passes	Fails
P 1/1	(6-5) Tx4 (2-5) with YR	(1-3) (3-5) with R	Tx1 with CL with RP Tx3 (2-3) Tx2 (1-2)	(3-4) with PB	(4-5) Tx5 Tx6 Tx7 Tx8
PB 2/3	-----	-----	(2-5) (2-1) Tx2 (3-1) (3-2) Tx4 with P with R	Tx1 Tx3 (3-4) with RP with CL	Tx5
RP 2/1	(2-1) (3-1) (5-6) Tx7 Tx8	(2-3) Tx1 Tx2 with P with PB with CI with R	Tx5	(4-5) Tx6 Tx3	-----
Minor Colors YR, R, CI, or B 3/1	When these colors are dominant, then sample passes except when YR is associated with RP it fails more than it passes				
No Dominant Color* 2/1	No preferences shown by texture group				

*3 or more colors present each less than 50% of the total area

Table 4 Summary Table of Color, Texture, Value, Chroma and Areal Distribution

Color	Passes	Passes more than fails	50/50	Fails more than Passes	Fails
PB	-----	-----	Tx1 light or dark values Tx2 Tx3	Tx1 medium values Tx3 when area > 40% Tx4 Tx5	Tx4, dark value Tx5, dark value or highly saturated
RP	Tx 1, high saturation Tx6 Tx7 Tx8	Tx1, medium and low saturation Tx2 better for areas > 40% Tx3, increases for areas > 60% or light value and less saturated	Tx1 medium value with low saturation Tx2, medium value Tx4 Tx5	-----	-----
P	Tx 1 medium value and saturation	Tx1 Tx2	Tx3	Tx5 Tx4, increases when light value with low saturation and medium value with high or medium saturation	-----
B	Tx1	Tx2 Tx3	-----	-----	Tx5
YR	Tx1	Tx3	Tx2 Tx5	-----	-----

future. With the ability to differentiate a near failing score from a very definite failure and likewise the borderline passing score from an excellent score on a physical test, there may be more obviousness in statistical results. Different statistical approaches can further future analysis now that relationships have been shown in this study.

This study has shown that there are useful relationships between the indicators used and the designation of aggregates of Class I and Class 0. The color of a treated sample can be broken into three components: hue, value, and chroma. The texture and color characteristics together can often indicate acceptable or non-acceptable aggregate for concrete construction. The use of computerized sorting of the characteristics makes combinations easily accessible and analyzable. As more components are included in the analysis of color or texture or color with texture, the relationships become more defined. More information than was used in this analysis is easily accessible in the database. Since Class I is defined by the results of several tests and each of these is numerically measured and recorded in the database, these test results could be used with the geologic characteristics to further define the relationships that are shown to exist. Further definition of relationships can be desirable for several reasons: streamlining test procedures, improving relationships with resource managers/owners, improving prospecting for and utilization of natural aggregate sources, and anticipating the need to computerize characteristics. These are discussed further.

With definition of which geologic descriptors relate to failure of specific physical tests, the least costly testing procedure can be requested for each aggregate. Once the aggregate fails a test no further testing is required. Testing is thorough for verifying good aggregates. This would also improve relationships with limestone resource owners since

they could be informed of failure sooner, or conversely know that the longer time to get classification results was indicative of better chances of having a qualified limestone resource. Since this research project began, some streamlining has already taken place that was independent of this project.

The geologist in the field prospecting limestone ledges for suitability would have guidelines that would indicate probable acceptable limestone for sampling and testing. The field geologist would also know what factors would decrease the acceptability of the limestone and could recommend particular testing sequencing to be done to eliminate suspect sources before more costly testing was begun. This knowledge would also aid in verifying the stockpile quality consistency once qualifying limestone sources begin production.

Research of the current literature (Frohnsdorff, Clifton, 1997) and visits with other agencies verify that computerized database searches and analyses of test results and materials characteristics to aid in performance based standards and specifications are looming. KDOT recently printed a report (Najjar, Basheer, 1997) modeling durability factor and expansion upon easily tested properties of aggregates, absorption, specific gravity, and acid insoluble residue. Inclusion of geologic characteristics could improve prediction for many aggregates. Florida has been finding petrographic tests to be more useful and faster than soundness tests for predicting carbonate aggregate soundness (Eades, 1997, personal communication). A history of efforts in petrography of carbonate aggregates for use in concrete is summarized in Oyen, et al., (1998). KDOT currently has equipment and trained personnel to produce the data suitable for inclusion in database form for faster, reliable aggregate characterization.

Recommendations

For anticipating the future the following items are recommended:

- 1) Use the existing limestone characteristics database to further define relationships between various physical test results and specific geologic characteristics, probably using different statistical testing more appropriate for the new application.
- 2) Verify relationships defined in item 1 above with characterization of new samples and performing the proper statistical analysis to predict results, which could in turn, improve overall predictions.
- 3) Using known relationships and peel data, perform staining tests on aggregates and tabulate resulting colors for inclusion in the database.
- 4) Summarize the results of items 1 and 3 above for use as a field tool in prospecting quarry sites, inspecting quarries, and verifying production stockpile consistency. When new information from item 2 is available, incorporate it into the field tools.
- 5) Add a color image analysis system to the research tools currently owned so that color characteristics could be objectively and quickly determined by non-geologically trained personnel, reducing personal variations in determining characteristics of hue, value, and chroma as well as measuring areas of colors in a peel.
- 6) Use K-TRAN research to clarify and verify relationships that appear in statistical analyses and other research.

Implementation

With summarization of the statistical data of this study into the table using dominant staining colors, there is the beginning of a field tool. The Appendix of the report includes the stain recipe, the descriptions of the stain colors resulting from carbonate mineralogy in thin-section and the summary table of dominant staining colors found in this study. Together these constitute a preliminary field tool. The geologist performing staining tests and recording the results also records the natural colors of the tested limestones. This information recorded in the database can make future statistical analysis more powerful and determinative for prospecting quarries and verifying stockpiles in quality control and assurance. This can begin immediately requiring no addition of personnel and a small amount of training. Field geologist and crews sample quarries on request as usual and add other quarries for sampling to develop a diverse geographic representation of quarries as well as mineralogy and texture representation. An extra ledge sample designated for research is collected for testing in the Materials and Research Center by research personnel. Training for field personnel and the purchase of chemicals and simple implements to perform the staining procedure are all that are needed. Research personnel provide lists of quarries for the additional sampling to be scheduled. Data from the sampling made available in a database for statistical testing would be collected for a year then analyzed. The results can be incorporated into a refined field-prospecting tool. Appendix F provides more descriptive detail of this implementation.

The database already has more information that has not been tapped. The initial analysis resulting in promising relationships should be followed with different sampling

of the database and statistical testing to better define the relationships. KDOT has available the statistical tools and the geologic research personnel with other tools such as SEM (scanning electron microscope) and XRD to clarify and verify results. Using the information to predict the performance of new ledges brought in for physical tests would also add to verification of results. Currently the ledges coming in for testing have been sampled and these samples are available for use.

One current K-TRAN project is already producing indications that texture is one of the primary reasons for lateral variations in single geological units that pass physical test requirements in one quarry and fail tests in a neighboring quarry. Quarry owners in the areas under the K-TRAN research scrutiny are positive towards the interest taken in finding causes of lateral variation of test results and have requested the investigators to explain the research results to local governing agencies. The investigators met with the Overland Park Department of Public Works to discuss the concerns of both the quarry owners and the agency about limestone variability (Goldstein, personal communications, 1998). The same K-TRAN project is producing petrographic indicators that parallel those of this study and other indicators that have the potential to be tested with the database from this study. This co-operation between KDOT, the university geologic departments, the local quarry owners /managers and local governing agencies should be continued and supported.

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Appendix

Contents

Part A: Making a Stained Peel

Part B: Stain Color and Mineralogy

Part C: Testing and Classifying Limestone Aggregate

Part D: Preparation of 2x2 Partitions Using a General Example

Part E: Summary of Statistical Analysis and Recommendations for
Implementation in the Field

Part F: Implementation Plan

Appendix Part A
Making a Stained Peel

The procedure detailed below is adapted from Dickson (1965). It has been found generally satisfactory. Two stains are required-Alizarin Red S and potassium ferricyanide.

1. Clean the polished surface of the rocks. Ensure that no dirt or grease adheres to the surface.
2. Prepare two staining solutions.

Solution A: Alizarin Red S-concentration of 0.2g/100 ml of 1.5% hydrochloric acid (15 ml pure acid made up to 1 litre with water.

Solution B: Potassium ferricyanide-concentration of 2g/100 ml of 1.5% hydrochloric acid.
3. Mix solutions A and B in the proportion 3 parts by volume of A to parts of B.
4. Immerse the polished face in the mixture of solutions 30-45 seconds, agitating gently for at least part of the time to remove gas bubbles from the surface, being careful not to touch the edges or the bottom of the stain container.
5. Wash the stained section in running water for a few seconds.
6. Allow to dry.
7. Level the prepared surface face up.
8. Flood with acetone then quickly and smoothly roll a film of acetate over the surface to cover. Leave in place until dry.
9. Gently peel off the acetate from the rock surface. The acetate retains an impression of the etched surface along with the dye that adhered to the surface.

Adapted from: Atlas of Sedimentary Rocks Under the Microscope, A.E. Adams, W.S. MacKenzie, and C. Guilford, 1984, John Wiley and Sons, pp104.

Appendix, Part B Stain Color and Mineralogy

The optical properties of calcite and dolomite are similar and therefore they can be difficult to distinguish optically. Simple chemical staining techniques are often employed by carbonate sedimentologists to distinguish calcite from dolomite and to distinguish ferroan from non-ferroan minerals.

The dye Alizarin Red S is used to differentiate calcite and dolomite, whereas potassium ferricyanide is used to differentiate ferroan and non-ferroan minerals. The dyes are dissolved in a weak acid solution. This also helps to distinguish dolomite from calcite, as dolomite does not react with cold dilute acid whereas calcite does, producing a contrast in relief between the two minerals.

The intensity of the stain color is partly related to the intensity of the etching with acid. Fine-grained crystal fabrics with many crystal boundaries etch more rapidly and thus show deeper stain colors than coarse crystal fabrics with few crystal boundaries.

Etching and Staining Characteristics of Carbonate Minerals*

Mineral	Effect of etching	Stain color with Alizarin Red S	Stain color with potassium ferricyanide	Combined result
Calcite (non-ferroan)	Considerable (relief reduced)	Pink to red-brown	None	Pink to red-brown
Calcite (ferroan)	Considerable (relief reduced)	Pink to red-brown	Pale to deep blue depending on iron content	
Dolomite (non-ferroan)	Negligible (relief maintained)	None	None	Colorless
Dolomite (ferroan)	Negligible (relief maintained)	None	Very pale blue	Very pale blue (appears turquoise or greenish in thin section)

Note: The staining procedure used for this study produces results summarized in the last column.

Adapted from: Atlas of Sedimentary Rocks Under the Microscope, A.E. Adams, W.S. MacKenzie, and C. Guilford, 1984, John Wiley and Sons, pp104.

NOTES

Appendix Part C

SUBSECTION 1102

AGGREGATES FOR CONCRETE

1102.01 DESCRIPTION.

This specification covers the requirements for coarse aggregate, mixed aggregate, fine aggregate and lightweight aggregate for use in concrete for all types of construction. Also covered are the requirements for mortar sand for use in grout or mortar.

1102.02 REQUIREMENTS.

(a) Coarse Aggregate.

Coarse aggregates shall conform to the requirements hereinafter specified.

(1) Quality.

(1.1) Coarse Aggregate for Concrete other than Pavement

Soundness, minimum..... 0.90
Wear, maximum..... 50%

* Soundness will be waived if the aggregate meets all requirements for Durability Class I aggregate.

(1.2) Coarse Aggregate for Concrete Pavement

(1.2.1) Siliceous Gravel, Chat or Calcite Cemented Sandstone

Soundness, minimum..... 0.90
Wear, maximum..... 50%

(1.2.2) Crushed Limestone or Dolomite

Wear, maximum..... 50%

Additionally this aggregate shall meet the requirements shown in Table A below for Durability Class I or VI.

Table A

Durability Class	Durability Factor (min.)	Expansion (%) (max.)	Soundness (Modified F&T) (min.)	PVF (max.)	Acid Insol. Residue (%) (max.)
I	95	0.025	0.85	35	3.5
VI			0.95		

Durability Class VI aggregates shall be used only with the approval of the Chief of Materials and Research. A listing of durability classed aggregates will be maintained at the Materials and Research Center.



DURABILITY FACTOR in accordance with ASTM C666 Procedure B Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing¹

This standard is issued under the fixed designation C 666; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of the resistance of concrete specimens to rapidly repeated cycles of freezing and thawing in the laboratory by two different procedures: Procedure A, Rapid Freezing and Thawing in Water, and Procedure B, Rapid Freezing in Air and Thawing in Water. Both procedures are intended for use in determining the effects of variations in the properties of concrete on the resistance of the concrete to the freezing-and-thawing cycles specified in the particular procedure. Neither procedure is intended to provide a quantitative measure of the length of service that may be expected from a specific type of concrete.

1.2 The values stated in inch-pound units are to be regarded as the standard.

1.3 All material in this test method not specifically designated as belonging to Procedure A or Procedure B applies to either procedure.

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- C 157 Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete²
- C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory²
- C 215 Test Method for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens²
- C 233 Test Method for Testing Air-Entraining Admixtures for Concrete²
- C 295 Guide for Petrographic Examination of Aggregates for Concrete²
- C 341 Test Method for Length Change of Drilled or Sawed Specimens of Hydraulic-Cement Mortar and Concrete²
- C 490 Practice for Use of Apparatus for Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete²

C 494 Specification for Chemical Admixtures for Concrete²

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials²

C 823 Practice for Examination and Sampling of Hardened Concrete in Constructions²

3. Significance and Use

3.1 As noted in the scope, the two procedures described in this test method are intended to determine the effects of variations in both properties and conditioning of concrete in the resistance to freezing and thawing cycles specified in the particular procedure. Specific applications include specified use in Specification C 494, Test Method C 233, and ranking of coarse aggregates as to their effect on concrete freeze-thaw durability, especially where soundness of the aggregate is questionable.

3.2 It is assumed that the procedures will have no significantly damaging effects on frost-resistant concrete which may be defined as (1) any concrete not critically saturated with water (that is, not sufficiently saturated to be damaged by freezing) and (2) concrete made with frost-resistant aggregates and having an adequate air-void system that has achieved appropriate maturity and thus will prevent critical saturation by water under common conditions.

3.3 If as a result of performance tests as described in this test method concrete is found to be relatively unaffected, it can be assumed that it was either not critically saturated, or was made with "sound" aggregates, a proper air-void system, and allowed to mature properly.

3.4 No relationship has been established between the resistance to cycles of freezing and thawing of specimens cut from hardened concrete and specimens prepared in the laboratory.

4. Apparatus

4.1 Freezing-and-Thawing Apparatus:

4.1.1 The freezing-and-thawing apparatus shall consist of a suitable chamber or chambers in which the specimens may be subjected to the specified freezing-and-thawing cycle, together with the necessary refrigerating and heating equipment and controls to produce continuously, and automatically, reproducible cycles within the specified temperature requirements. In the event that the equipment does not operate automatically, provision shall be made for either its continuous manual operation on a 24-h a day basis or for the storage of all specimens in a frozen condition when the equipment is not in operation.

4.1.2 The apparatus shall be so arranged that, except for

¹ This test method is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.67 on Resistance of Concrete to Its Environment.

Current edition approved Sept. 15, 1992. Published November 1992. Originally published as C 666 - 71. Last previous edition C 666 - 90.

² Annual Book of ASTM Standards, Vol 04.02

necessary supports, each specimen is: (1) for Procedure A, completely surrounded by not less than $\frac{1}{32}$ in. (1 mm) nor more than $\frac{1}{8}$ in. (3 mm) of water at all times while it is being subjected to freezing-and-thawing cycles, or (2) for Procedure B, completely surrounded by air during the freezing phase of the cycle and by water during the thawing phase. Rigid containers, which have the potential to damage specimens, are not permitted. Length change specimens in vertical containers shall be supported in a manner to avoid damage to the gage studs.

NOTE 1—Experience has indicated that ice or water pressure, during freezing tests, particularly in equipment that uses air rather than a liquid as the heat transfer medium, can cause excessive damage to rigid metal containers, and possibly to the specimens therein. Results of tests during which bulging or other distortion of containers occurs should be interpreted with caution.

4.1.3 The temperature of the heat-exchanging medium shall be uniform within 6°F (3.3°C) throughout the specimen cabinet when measured at any given time, at any point on the surface of any specimen container for Procedure A or on the surface of any specimen for Procedure B, except during the transition between freezing and thawing and *vice versa*.

4.1.3.1 Support each specimen at the bottom of its container in such a way that the temperature of the heat-exchanging medium will not be transmitted directly through the bottom of the container to the full area of the bottom of the specimen, thereby subjecting it to conditions substantially different from the remainder of the specimen.

NOTE 2—A flat spiral of $\frac{1}{8}$ -in. (3-mm) wire placed in the bottom of the container has been found adequate for supporting specimens.

4.1.4 For Procedure B, it is not contemplated that the specimens will be kept in containers. The supports on which the specimens rest shall be such that they are not in contact with the full area of the supported side or end of the specimen, thereby subjecting this area to conditions substantially different from those imposed on the remainder of the specimen.

NOTE 3—The use of relatively open gratings, metal rods, or the edges of metal angles has been found adequate for supporting specimens, provided the heat-exchanging medium can circulate in the direction of the long axis of the rods or angles.

4.2 *Temperature-Measuring Equipment*, consisting of thermometers, resistance thermometers, or thermocouples, capable of measuring the temperature at various points within the specimen chamber and at the centers of control specimens to within 2°F (1.1°C).

4.3 *Dynamic Testing Apparatus*, conforming to the requirements of Test Method C 215.

4.4 *Optional Length Change Test Length Change Comparator*, conforming to the requirements of Specification C 490. When specimens longer than the nominal $11\frac{1}{4}$ in. (286 mm) length provided for in Specification C 490 are used for freeze-thaw tests, use an appropriate length reference bar, which otherwise meets the Specification C 490 requirements. Dial gage micrometers for use on these longer length change comparators shall meet the gradation interval and accuracy requirements for Specification C 490 for either the inch or millimetre calibration requirements. Prior to the start of measurements on any specimens, fix the comparator at an appropriate length to accommodate all of the speci-

mens to be monitored for length change.

4.5 *Scales*, with a capacity approximately 50 % greater than the weight of the specimens and accurate to at least 0.01 lb (4.5 g) within the range of ± 10 % of the specimen weight will be satisfactory.

4.6 *Tempering Tank*, with suitable provisions for maintaining the temperature of the test specimens in water, such that when removed from the tank and tested for fundamental transverse frequency and length change, the specimens will be maintained within -2°F and $+4^{\circ}\text{F}$ (-1.1°C and $+2.2^{\circ}\text{C}$) of the target thaw temperature for specimens in the actual freezing-and-thawing cycle and equipment being used. The use of the specimen chamber in the freezing-and-thawing apparatus by stopping the apparatus at the end of the thawing cycle and holding the specimens in it shall be considered as meeting this requirement, provided the specimens are tested for fundamental transverse frequency within the above temperature range. It is required that the same target specimen thaw temperature be used throughout the testing of an individual specimen since a change in specimen temperature at the time of length measurement can affect the length of the specimen significantly.

5. Freezing-and-Thawing Cycle

5.1 Base conformity with the requirements for the freezing-and-thawing cycle on temperature measurements of control specimens of similar concrete to the specimens under test in which suitable temperature-measuring devices have been imbedded. Change the position of these control specimens frequently in such a way as to indicate the extremes of temperature variation at different locations in the specimen cabinet.

5.2 The nominal freezing-and-thawing cycle for both procedures of this test method shall consist of alternately lowering the temperature of the specimens from 40 to 0°F (4.4 to -17.8°C) and raising it from 0 to 40°F (-17.8 to 4.4°C) in not less than 2 nor more than 5 h. For Procedure A, not less than 25 % of the time shall be used for thawing, and for Procedure B, not less than 20 % of the time shall be used for thawing (Note 4). At the end of the cooling period the temperature at the centers of the specimens shall be $0 \pm 3^{\circ}\text{F}$ ($-17.8 \pm 1.7^{\circ}\text{C}$), and at the end of the heating period the temperature shall be $40 \pm 3^{\circ}\text{F}$ ($4.4 \pm 1.7^{\circ}\text{C}$), with no specimen at any time reaching a temperature lower than -3°F (-19.4°C) nor higher than 43°F (6.1°C). The time required for the temperature at the center of any single specimen to be reduced from 37 to 3°F (2.8 to -16.1°C) shall be not less than one half of the length of the cooling period, and the time required for the temperature at the center of any single specimen to be raised from 3 to 37°F (-16.1 to 2.8°C) shall be not less than one half of the length of the heating period. For specimens to be compared with each other, the time required to change the temperature at the centers of any specimens from 35 to 10°F (1.7 to -12.2°C) shall not differ by more than one sixth of the length of the cooling period from the time required for any specimen and the time required to change the temperature at the centers of any specimens from 10 to 35°F (-12.2 to 1.7°C) shall not differ by more than one third of the length of the heating period from the time required for any specimen.

NOTE 4—In most cases, uniform temperature and time conditions can be controlled most conveniently by maintaining a capacity load of specimens in the equipment at all times. In the event that a capacity load of test specimens is not available, dummy specimens can be used to fill empty spaces. This procedure also assists greatly in maintaining uniform fluid level conditions in the specimen and solution tanks.

The testing of concrete specimens composed of widely varying materials or with widely varying thermal properties, in the same equipment at the same time, may not permit adherence to the time-temperature requirements for all specimens. It is advisable that such specimens be tested at different times and that appropriate adjustments be made to the equipment.

5.3 The difference between the temperature at the center of a specimen and the temperature at its surface shall at no time exceed 50°F (27.8°C).

5.4 The period of transition between the freezing-and-thawing phases of the cycle shall not exceed 10 min, except when specimens are being tested in accordance with 8.2.

6. Sampling

6.1 Constituent materials for concrete specimens made in the laboratory shall be sampled using applicable standard methods.

6.2 Samples cut from hardened concrete are to be obtained in accordance with Practice C 823.

7. Test Specimens

7.1 The specimens for use in this test method shall be prisms or cylinders made and cured in accordance with the applicable requirements of Practice C 192 and Specification C 490.

7.2 Specimens used shall not be less than 3 in. (76 mm) nor more than 5 in. (127 mm) in width, depth, or diameter, and not less than 11 in. (279 mm) nor more than 16 in. (406 mm) in length.

7.3 Test specimens may also be cores or prisms cut from hardened concrete. If so, the specimens should not be allowed to dry to a moisture condition below that of the structure from which taken. This may be accomplished by wrapping in plastic or by other suitable means. The specimens so obtained shall be furnished with gage studs in accordance with Test Method C 341.

7.4 For this test the specimens shall be stored in saturated lime water from the time of their removal from the molds until the time freezing-and-thawing tests are started. All specimens to be compared with each other initially shall be of the same nominal dimensions.

8. Procedure

8.1 Immediately after the specified curing period (Note 5), bring the specimen to a temperature within -2°F and $+4^{\circ}\text{F}$ (-1.1°C and $+2.2^{\circ}\text{C}$) of the target thaw temperature that will be used in the freeze-thaw cycle and test for fundamental transverse frequency, weigh, determine the average length and cross section dimensions of the concrete specimen within the tolerance required in Test Method C 215, and determine the initial length comparator reading (optional) for the specimen with the length change comparator. Protect the specimens against loss of moisture between the time of removal from curing and the start of the freezing-and-thawing cycles.

NOTE 5—Unless some other age is specified, the specimens should be removed from curing and freezing-and-thawing tests started when the specimens are 14 days old.

8.2 Start freezing-and-thawing tests by placing the specimens in the thawing water at the beginning of the thawing phase of the cycle. Remove the specimens from the apparatus, in a thawed condition, at intervals not exceeding 36 cycles of exposure to the freezing-and-thawing cycles, test for fundamental transverse frequency and measure length change (optional) with the specimens within the temperature range specified for the tempering tank in 4.6, weigh each specimen, and return them to the apparatus. To ensure that the specimens are completely thawed and at the specified temperature place them in the tempering tank or hold them at the end of the thaw cycle in the freezing-and-thawing apparatus for a sufficient time for this condition to be attained throughout each specimen to be tested. Protect the specimens against loss of moisture while out of the apparatus and turn them end-for-end when returned. For Procedure A, rinse out the container and add clean water. Return the specimens either to random positions in the apparatus or to positions according to some predetermined rotation scheme that will ensure that each specimen that continues under test for any length of time is subjected to conditions in all parts of the freezing apparatus. Continue each specimen in the test until it has been subjected to 300 cycles or until its relative dynamic modulus of elasticity reaches 60 % of the initial modulus, whichever occurs first, unless other limits are specified (Note 6). For the optional length change test, 0.10 % expansion may be used as the end of test. Whenever a specimen is removed because of failure, replace it for the remainder of the test by a dummy specimen. Each time the specimen is tested for fundamental frequency (Note 7) and length change, make a note of its visual appearance and make special comment on any defects that develop (Note 8). When it is anticipated that specimens may deteriorate rapidly, they should be tested for fundamental transverse frequency and length change (optional) at intervals not exceeding 10 cycles when initially subjected to freezing and thawing.

NOTE 6—It is not recommended that specimens be continued in the test after their relative dynamic modulus of elasticity has fallen below 50 %.

NOTE 7—It is recommended that the fundamental longitudinal frequency be determined initially and as a check whenever a question exists concerning the accuracy of determination of fundamental transverse frequency, and that the fundamental torsional frequency be determined initially and periodically as a check on the value of Poisson's ratio.

NOTE 8—In some applications, such as airfield pavements and other slabs, popouts may be defects that are a concern. A popout is characterized by the breaking away of a small portion of the concrete surface due to internal pressure, thereby leaving a shallow and typically conical spall in the surface of the concrete through the aggregate particle. Popouts may be observed as defects in the test specimens. Where popouts are a concern, the number and general description should be reported as a special comment. The aggregates causing the popout may be identified by petrographic examination as in Practice C 295.

8.3 When the sequence of freezing-and-thawing cycles must be interrupted store the specimens in a frozen condition.

NOTE 9—If, due to equipment breakdown or for other reasons, it becomes necessary to interrupt the cycles for a protracted period, store

the specimens in a frozen condition in such a way as to prevent loss of moisture. For Procedure A, maintain the specimens in the containers and surround them by ice, if possible. If it is not possible to store the specimens in their containers, wrap and seal them, in as wet a condition as possible, in moisture-proof material to prevent dehydration and store in a refrigerator or cold room maintained at $0 \pm 3^\circ\text{F}$ ($-17.8 \pm 1.7^\circ\text{C}$). Follow the latter procedure when Procedure B is being used. In general, for specimens to remain in a thawed condition for more than two cycles is undesirable, but a longer period may be permissible if this occurs only once or twice during a complete test.

9. Calculation

9.1 *Relative Dynamic Modulus of Elasticity*—Calculate the numerical values of relative dynamic modulus of elasticity as follows:

$$P_c = (n_1^2/n^2) \times 100$$

where:

- P_c = relative dynamic modulus of elasticity, after c cycles of freezing and thawing, percent,
- n = fundamental transverse frequency at 0 cycles of freezing and thawing, and
- n_1 = fundamental transverse frequency after c cycles of freezing and thawing.

NOTE 10—This calculation of relative dynamic modulus of elasticity is based on the assumption that the weight and dimensions of the specimen remain constant throughout the test. This assumption is not true in many cases due to disintegration of the specimen. However, if the test is to be used to make comparisons between the relative dynamic moduli of different specimens or of different concrete formulations, P_c as defined is adequate for the purpose.

9.2 *Durability Factor*—Calculate the durability factor as follows:

$$DF = PN/M$$

where:

- DF = durability factor of the test specimen,
- P = relative dynamic modulus of elasticity at N cycles, %,
- N = number of cycles at which P reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less, and
- M = specified number of cycles at which the exposure is to be terminated.

9.3 *Length Change in Percent (optional)*—Calculate the length change as follows:

$$L_c = \frac{(l_2 - l_1)}{L_e} \times 100$$

where:

- L_c = length change of the test specimen after C cycles of freezing and thawing, %,
- l_1 = length comparator reading at 0 cycles,
- l_2 = length comparator reading after C cycles, and
- L_e = the effective gage length between the innermost ends of the gage studs as shown in the mold diagram in Specification C 490.

10. Report

10.1 Report the following data such as are pertinent to the variables or combination of variables studied in the tests:

10.2 *Properties of Concrete Mixture:*

10.2.1 Type and proportions of cement, fine aggregate, and coarse aggregate, including maximum size and grading (or designated grading indices), and ratio of net water content to cement,

10.2.2 Kind and proportion of any addition or admixture used,

10.2.3 Air content of fresh concrete,

10.2.4 Unit weight of fresh concrete,

10.2.5 Consistency of fresh concrete,

10.2.6 Air content of the hardened concrete, when available,

10.2.7 Indicate if the test specimens are cut from hardened concrete, and if so, state the size, shape, orientation of the specimens in the structure, and any other pertinent information available, and

10.2.8 Curing period.

10.3 *Mixing, Molding, and Curing Procedures*—Report any departures from the standard procedures for mixing, molding, and curing as prescribed in Section 7.

10.4 *Procedure*—Report which of the two procedures was used.

10.5 *Characteristics of Test Specimens:*

10.5.1 Dimensions of specimens at 0 cycles of freezing and thawing,

10.5.2 Weight of specimens at 0 cycles of freezing and thawing,

10.5.3 Nominal gage length between embedded ends of gage studs, and

10.5.4 Any defects in each specimen present at 0 cycles of freezing and thawing.

10.6 *Results:*

10.6.1 Values for the durability factor of each specimen, calculated to the nearest whole number, and for the average durability factor for each group of similar specimens, also calculated to the nearest whole number, and the specified values for minimum relative dynamic modulus and maximum number of cycles (Note 10),

10.6.2 Values for the percent length change of each specimen and for the average percent length change for each group of similar specimens (Note 11),

10.6.3 Values of weight loss or gain for each specimen and average values for each group of similar specimens, and

10.6.4 Any defects in each specimen which develop during testing, and the number of cycles at which such defects were noted.

NOTE 11—It is recommended that the results of the test on each specimen, and the average of the results on each group of similar specimens, be plotted as curves showing the value of relative modulus of elasticity or percent length change against time expressed as the number of cycles of freezing and thawing.

11. Precision

11.1 *Within-Laboratory Precision (Single Beams)*—Criteria for judging the acceptability of durability factor results obtained by the two procedures in the same laboratory on concrete specimens made from the same batch of concrete or from two batches made with the same materials are given in Table 1. Precision data for length change (optional) are not available at this time.

NOTE 12—The between-batch precision of durability factors has been found to be the same as the within-batch precision. Thus the limits

TABLE 1 Within-Laboratory Durability Factor Precision for Single Beams

NOTE—The values given in Columns 2 and 4 are the standard deviations that have been found to be appropriate for Procedures A and B, respectively, for tests for which the average durability factor is in the corresponding range given in Column 1. The values given in Columns 3 and 5 are the corresponding limits that should not be exceeded by the difference between the results of two single test beams.

Range of Average Durability Factor	Procedure A		Procedure B	
	Standard Deviation ^A	Acceptable Range of Two Results ^A	Standard Deviation ^A	Acceptable Range of Two Results ^A
0 to 5	0.8	2.2	1.1	3.0
5 to 10	1.5	4.4	4.0	11.4
10 to 20	5.9	16.7	8.1	22.9
20 to 30	8.4	23.6	10.5	29.8
30 to 50	12.7	35.9	15.4	43.5
50 to 70	15.3	43.2	20.1	56.9
70 to 80	11.6	32.7	17.1	48.3
80 to 90	5.7	16.0	8.8	24.9
90 to 95	2.1	6.0	3.9	11.0
Over 95	1.1	3.1	2.0	5.7

^A These numbers represent the (1S) and (D2S) limits as described in Practice C 670.

given in this precision statement apply to specimens from different batches made with the same materials and mix design and having the same air content as well as to specimens from the same batch.

NOTE 13—The precision of this method for both procedures has been found to depend primarily on the average durability factor and not on the maximum *N* or minimum *P* specified for terminating the test nor

on the size of the beams within limits. The data on which these precision statements are based cover maximum *N*'s from 100 to 300 cycles, and minimum *P*'s from 50 to 70 percent of *E_c*. The indexes of precision are thus valid at least over these ranges.

11.1.1 The different specimen sizes represented by the data include the following: 3 by 3 by 16-in.; 3 by 3 by 16¼-in.; 3 by 4 by 16-in.; 3½ by 4½ by 16-in.; 3 by 3 by 11-in.; and 3½ by 4 by 16-in.; and 4 by 3 by 16-in. The first dimension given represents the direction in which the specimens were vibrated in the test for fundamental transverse frequency. The most commonly used size was 3 by 4 by 16-in.

11.2 *Within-Laboratory Precision (Averages of Two or More Beams)*—Specifications sometimes call for comparisons between averages of two or more beams. Tables 2 and 3 give appropriate standard deviations and acceptable ranges for the two procedures for two averages of the number of test beams shown.

11.3 *Multilaboratory Precision*—No data are available for evaluation of multilaboratory precision. It is believed that a multilaboratory statement of precision is not appropriate because of the limited possibility that two or more laboratories will be performing freezing-and-thawing tests on the same concretes.

12. Keywords

12.1 accelerated testing; concrete-weathering tests; conditioning; freezing and thawing; resistance-frost

TABLE 2 Within-Laboratory Durability Factor Precision for Averages of Two or More Beams—Procedure A

Range of Average Durability Factor	Number of Beams Averaged									
	2		3		4		5		6	
	Standard Deviation ^A	Acceptable Range ^A								
0 to 5	0.6	1.6	0.5	1.3	0.4	1.1	0.4	1.0	0.3	0.9
5 to 10	1.1	3.1	0.9	2.5	0.8	2.2	0.7	2.0	0.6	1.8
10 to 20	4.2	11.8	3.4	9.7	3.0	8.4	2.7	7.5	2.4	6.8
20 to 30	5.9	16.7	4.8	13.7	4.2	11.8	3.7	10.6	3.4	9.7
30 to 50	9.0	25.4	7.4	20.8	6.4	18.0	5.7	16.1	5.2	14.7
50 to 70	10.8	30.6	8.8	25.0	7.6	21.6	6.8	19.3	6.2	17.6
70 to 80	8.2	23.1	6.7	18.9	5.8	16.4	5.2	14.6	4.7	13.4
80 to 90	4.0	11.3	3.3	9.2	2.8	8.0	2.5	7.2	2.3	6.5
90 to 95	1.5	4.2	1.2	3.5	1.1	3.0	0.9	2.7	0.9	2.4
Above 95	0.8	2.2	0.6	1.8	0.5	1.5	0.5	1.4	0.4	1.3

^A These numbers represent the (1S) and (D2S) limits as described in Practice C 670.

TABLE 3 Within-Laboratory Durability Factor Precision for Averages of Two or More Beams—Procedure B

Range of Average Durability Factor	Number of Beams Averaged									
	2		3		4		5		6	
	Standard Deviation ^A	Acceptable Range ^A								
0 to 5	0.8	2.1	0.6	1.8	0.5	1.5	0.5	1.4	0.4	1.2
5 to 10	2.9	8.1	2.3	6.6	2.0	5.7	1.8	5.1	1.7	4.7
10 to 20	5.7	16.2	4.7	13.2	4.1	11.5	3.6	10.3	3.3	7.4
20 to 30	7.4	21.0	6.1	17.2	5.3	14.9	4.7	13.3	4.3	12.2
30 to 50	10.9	30.8	8.9	25.1	7.7	21.8	6.9	19.5	6.3	17.8
50 to 70	14.2	40.2	11.6	32.9	10.1	28.5	9.0	25.5	8.2	23.2
70 to 80	12.1	34.2	9.9	27.9	8.5	24.2	7.6	11.6	7.0	19.7
80 to 90	6.2	17.6	5.0	14.4	4.4	12.5	3.9	11.1	3.6	10.2
90 to 95	2.8	7.8	2.3	6.4	2.0	5.5	1.7	4.9	1.6	4.5
Above 95	1.4	4.1	1.2	3.3	1.0	2.9	0.9	2.6	0.8	2.3

^A These numbers represent the (1S) and (D2S) limits as described in Practice C 670.

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EXPANSION in accordance with subarticle 1117(t)

(t) WETTING AND DRYING TEST OF TOTAL MIXED AGGREGATE CONCRETE.

(1) Description. This test shall be used to determine the acceptability of total mixed aggregate to be used in concrete for the construction of pavements, bases, bridges, flumes, slope drains, sidewalks, riprap, curb and gutter, building floors, ramps to buildings, parking lots, wash checks and ditch lining.

(2) Apparatus:

(2.1) Molds for casting 3" × 4" × 16" beams.

(2.2) Container suitable for mixing the concrete.

(2.3) Trowel.

(2.4) Balance sensitive to one gram.

(2.5) Slump cone.

(2.6) Mechanical convection oven capable of maintaining a temperature of 128° F. to 130° F.

(2.7) Water bath capable of maintaining a temperature between 60° F. and 80° F.

(2.8) Device for measuring length change.

(2.9) Testing machine for modulus of rupture determination.

(3) Preparation of specimens. Specimens for test shall be prepared using materials and procedures as follows:

(3.1) Cement. Cement shall be of the type and brand designated by the Engineer of Tests.

(3.2) Aggregate. The gradation of the aggregate being tested shall be within the middle 1/3 of the limits specified for MA-1, except for the 3/4" sieve. It shall be further prepared by screening it through the 3/4" sieve and all material retained on the 3/4" sieve shall be crushed to pass the 3/4" sieve and incorporated into the mix.

(3.3) Concrete Mix. The concrete mix, using the prepared aggregate and the designated cement, shall contain 0.51 pounds of water per pound of cement and shall have a slump between 2" and 3".

(3.4) Molding Procedure. Cast six 3" × 4" × 16" beams as follows:

(3.4.1) Placing. Place the concrete in the molds with a scoop of appropriate size. Select each scoopful in a manner to approximate a representative sample of the mix. Move the scoop around the edge of the mold as the concrete is discharged in order to ensure a symmetrical distribution and to minimize segregation of coarse aggregate. Further distribute the concrete by use of the tamping rod prior to the start of consolidation. Do not add nonrepresentative samples of concrete to an underfilled mold.

(3.4.2) Method of Consolidation. Consolidation shall be by rodding with a 5/8" diameter steel rod having a hemispherical tip of the same diameter as the rod.

(3.4.3) Consolidation. Place the concrete in the mold in two layers of approximately equal volume. Rod each layer 32 times with the rounded end of the rod. Rod the bottom layer throughout its depth, distributing the strokes uniformly over the cross

section of the mold. For the upper layer, allow the rod to penetrate about $\frac{1}{2}$ inch into the bottom layer. After each layer is rodded, spade the concrete around the edges of the mold with a trowel or spatula. The molds containing the concrete shall then be tapped lightly on the table top to close any remaining voids. Finish the surface with a wood float using the minimum amount of manipulation necessary to produce a plane surface that is essentially level with the top edge of the mold.

(3.5) Curing. Cure the beams seven days in a moist room maintained at $73.4^{\circ} \pm 3^{\circ}$ F. and at not less than 95% relative humidity, then 21 days in air at a temperature between 68° and 81.5° F and 50% plus relative humidity and then submerged in water maintained between 60° and 80° F. for two days. The beams to be tested in flexure at 60 days shall then be cured in the moist room for an additional 30 days.

(4) Test Procedure.

(4.1) Measurements. The beams shall be measured for length at the following ages: 30, 60, 120, 180, 240, 300 and 365 days. At each age the beams shall be submerged in water maintained between 60° and 80° F for not less than $15\frac{3}{4}$ hours prior to measurement.

(4.2) Initial Flexure Test. Sixty days after casting, three of the beams shall be tested for modulus of rupture. The test shall be conducted with the $3" \times 16"$ faces perpendicular to the applied load, with the load applied at the center of a 14" span.

(4.3) Wetting and Drying Test. Beginning 30 days after casting, the remaining three beams shall be subjected to the following wetting and drying test procedure:

(4.3.1) Place the beams in the oven and maintained at a temperature between 128° and 130° F. for eight hours.

(4.3.2) Remove the beams from the oven and submerge them in the 60° to 80° F. water bath for $15\frac{3}{4}$ hours. Procedures (4.3.1) and (4.3.2) shall constitute one cycle and shall be completed in 24 hours.

(4.3.3) The cycle shall be repeated each consecutive day throughout the 365 day period except for weekends and holidays when the beams shall remain in the water bath.

(4.4) Calculation of Expansion. The length change, expressed as percent expansion, shall be calculated and recorded at each of the ages stated under (4.1) using the length measured at 30 days as the base.

(4.5) Final Flexure Test. The beams shall be tested for modulus of rupture, upon completion of the 365 day test period, in accordance with the procedure given under (4.2).

(5) Requirements for Acceptability of the Aggregate.

(5.1) Modulus of Rupture. Each of the two groups of beams tested in flexure at 60 days and 365 days shall have an average modulus of rupture of not less than 550 psi.

(5.2) Expansion.

(5.2.1) At 180 days. The increase in length shall not exceed 0.050%.

(5.2.2) At 365 days. The increase in length shall not exceed 0.070%.

(v) MODIFIED SOUNDNESS TEST FOR AGGREGATES.

(1) Description. This test, known as the "Modified Freeze and Thaw" test, shall be used to determine the soundness characteristics of Durability Classed Aggregate (Class O, I and VI).

(2) Apparatus.

(2.1) Balance sensitive to one gram.

(2.2) Set of standard square mesh sieves $\frac{3}{4}$ ", $\frac{1}{2}$ " and $\frac{3}{8}$ ".

(2.3) Freezing equipment capable of maintaining a temperature between 0° and minus 20° F.

(2.4) Water bath capable of maintaining a temperature of 70° F to 80° F.

(2.5) Oven capable of maintaining a temperature of $230^{\circ} \pm 9^{\circ}$ F.

(3) Sample Preparation.

(3.1) Preliminary Preparation. Preliminary preparation shall include removal of all material retained on the $\frac{3}{4}$ " mesh sieve and that passing the $\frac{3}{8}$ " mesh sieve, and the removal of all mud, clay lumps or sticks. The material shall not be washed. Shale, shale-like material, coal, asphalt coated pieces, rotten stone, soft or friable particles and other foreign material shall not be removed prior to testing. The material shall then be oven dried to a constant mass at a temperature of $230^{\circ} \pm 9^{\circ}$ F.

(3.2) Final Preparation. Final preparation shall consist of screening the oven dried material over $\frac{3}{4}$ ", $\frac{1}{2}$ " and $\frac{3}{8}$ " mesh sieves to meet the following grading.

<u>Individual Sieves</u>	<u>Cumulative Weight Retained (gms)</u>
$\frac{3}{4}$ "	0
$\frac{1}{2}$ "	2,500
$\frac{3}{8}$ "	5,000

(4) Test Procedure:

After sieving, the material shall be placed in an open top container, covered with a No. 16 mesh sieve cloth and submerged in tap water maintained at a temperature from 70° to 80° F for a period of 24 ± 4 hours. The sample shall then be tested in accordance with subarticle 1117(s)(4.2) and (4.3). One freezing period and one thawing period shall be considered one cycle. After the sample has been subjected to 25 cycles of freezing and thawing it shall be washed over a No. 12 sieve and oven dried to a constant mass at a temperature of $230^{\circ} \pm 9^{\circ}$ F. The sample shall then be screened over a $\frac{1}{2}$ " and $\frac{3}{8}$ " mesh sieve.

(5) Computations:

Computations shall be in accordance with subarticle 1117(s)(5).

(5) Computations.

(5.1) The cumulative percentage of material retained on each sieve ($\frac{3}{4}$ ", $\frac{3}{8}$ ", No. 4 and No. 8) before testing shall be computed and recorded.

(5.2) The cumulative percentage of material retained on each sieve at the end of the test shall be computed and recorded.

(5.3) The sum of the cumulative percentages of aggregate retained on the sieves after 25 cycles of freezing and thawing shall be divided by the sum of the cumulative percentages of aggregates retained on the same screens before testing. The value obtained shall be known as the freeze-thaw loss-ratio.

(y) DETERMINATION OF TOTAL ACID INSOLUBLE RESIDUE.

(1) Description. This method of test covers the procedure for determining the total acid insoluble residue of crushed limestone or dolomite.

(2) PART I.

(2.1) Apparatus.

(2.1.1) Glass or plastic wide-mouth gallon jar.

(2.1.2) Concentrated hydrochloric acid.

(2.1.3) Stirring rod.

(2.1.4) Sample splitter.

(2.1.5) Balance, 800 gm capacity (minimum), 0.1 gm. readability.

(2.2) Sample Preparation

(2.2.1) Dry the sample in an oven at approximately 230° F for 24 hours.

(2.2.2) Split the sample to obtain approximately 200 gm.

(2.2.3) Crush the 200 gm. sample to pass a No. 4 sieve.

(2.2.4) Weigh the sample to 0.1 gm. and record as sample weight "B".

(2.3) Test Procedure.

(2.3.1) Place the sample in a wide-mouth gallon jar and add sufficient distilled water to cover the sample.

(2.3.2) Place the jar under an exhaust hood and add approximately 25 ml of concentrated hydrochloric acid. Stir until reaction stops. If reaction is violent, direct a stream of distilled water along the walls of the jar to subdue the reaction.

(2.3.3) Continue adding increments of 25 ml of hydrochloric acid until the reaction stops.

(2.3.4) Add an additional 20 ml of concentrated hydrochloric acid and allow to stand overnight to assure complete removal of all carbonates.

(3) PART II.

(3.1) Apparatus.

(3.1.1) Buchner Funnel.

(3.1.2) Whatman No. 42 filter paper.

(3.1.3) Vacuum filtering flask.

(3.1.4) Rubber stopper.

(3.1.5) Oven.

(3.2) Preparation.

(3.2.1) Assemble vacuum filtering apparatus—Buchner funnel, filtering flask and vacuum line.

(3.2.2) Place Whatman No. 42 filter paper in the Buchner funnel and open the vacuum line slightly. Pour distilled water over the filter paper and smooth the filter paper with the stirring rod to seal it to the bottom of the funnel. Check for leaks around the edge of the filter paper.

(3.3) Procedure.

(3.3.1) Transfer the sample residue and solution to the Buchner funnel for filtration.

(3.3.2) Wash the residue with distilled water to remove all free chloride.

(3.3.3) Remove residue from the Buchner funnel, place in an evaporating dish and dry in oven for 24 hours at approximately 230° F.

(3.3.4) Weigh the residue to the nearest 0.1 gm and record as sample weight "A".

(3.3.5) Compute percent acid insoluble residue.

$\%AI = (100) (\text{Sample Weight A}) / (\text{Sample Weight B})$

Appendix Part D

Construction of 2 x 2 Tables

For a contingency table of size four rows and four columns, 2 x 2 partitions result in nine tables. Each 2 x 2 partition table is the result of moving the quadrant boundaries. The resultant tables are used for χ^2 and V calculations. In general, the 2 x 2 partition quadrant a is the sum of all numbers in that partition. Quadrant b is the sum of all the numbers in its partition and so on for c and d. The progression of changes is shown schematically in the nine tables below.

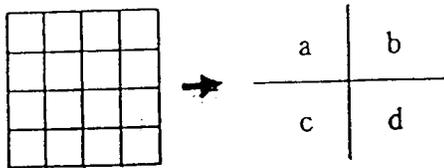


Table 5

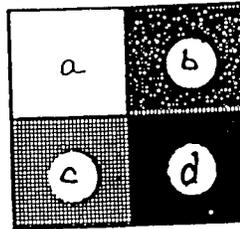


Table 1

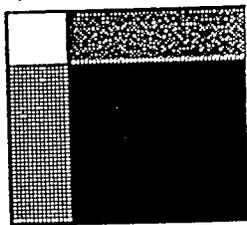


Table 6

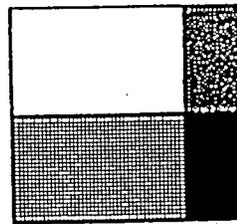


Table 2

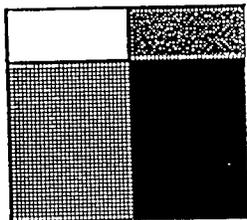


Table 7

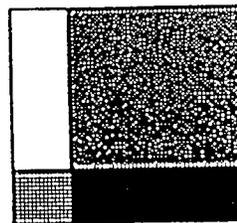


Table 3

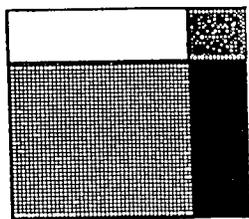


Table 8

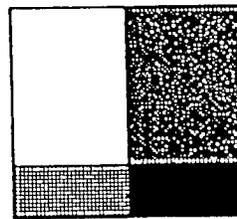


Table 4

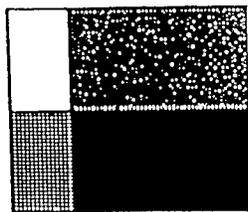
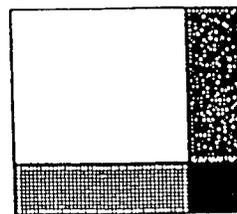


Table 9



Appendix Part E

Summary of Statistical Analysis and Recommendations for Implementation in the Field.

The table, Summary table from Dominant Colors, is based on the data obtained from stained peels using the Munsell color categories for description. When a peel had 50% or more of its area in the color category regardless of chroma or value variations, that color was considered dominant. Future study of chroma and value variations within the dominant color may change the relationships in the table. Since non-dominant colors were only considered singly with the dominant color, nothing can be implied when more than one color is found with the dominant color and these associated colors are shown to have opposing tendencies in the table.

If staining is attempted in the field of unpolished samples the surface to be stained should be newly broken. Obtaining peels will be nearly impossible and not expected. Care should be taken to duplicate the laboratory procedure bearing in mind that the results will not be the same for various reasons. Future laboratory and statistical study may simplify the ordeals of comparisons by clarifying the field results as they relate to the laboratory descriptions. Color identification can be more difficult since the broken surfaces will generally yield more intense or darker colors than those obtained from the peels. Field staining should be attempted with at least two square inches and the stained area fully rinsed and air-dried before determining the color categories and areas. Attention should also be given to assessing whether layers are evident in the field sample.

Segregation of mineralogy or textural characteristics into layers when present might influence the color outcome or overall assessment of passing or failing potential. Information concerning total textural classification may be difficult in unpolished samples. A 10X magnifier was used in the lab and again a newly broken surface of substantial area should be used for classifying.

Future study may add information comparing the natural color of samples to the other characteristics and potentials for passing or failing. For this reason full Munsell descriptions of natural colors may prove useful if recording the field staining colors. The Munsell Soil Color Charts are adequate to give full descriptions of natural rock colors usually found in this study.

Appendix Part E

Summary Table from Dominant Colors

Pass/Fail in Dominant Color Only	Passes	Passes more than fails	50/50	Fails more than Passes	Fails
P 1/1	(6-5) Tx4 (2-5) with YR	(1-3) (3-5) with R	Tx1 with CL with RP Tx3 (2-3) Tx2 (1-2)	(3-4) with PB	(4-5) Tx5 Tx6 Tx7 Tx8
PB 2/3	-----	-----	(2-5) (2-1) Tx2 (3-1) (3-2) Tx4 with P with R	Tx1 Tx3 (3-4) with RP with CL	Tx5
RP 2/1	(2-1) (3-1) (5-6) Tx7 Tx8	(2-3) Tx1 Tx2 with P with PB with CI with R	Tx5	(4-5) Tx6 Tx3	-----
Minor Colors YR, R, CI, or B 3/1	When these colors are dominant, then sample passes except when YR is associated with RP it fails more than it passes				
No Dominant Color* 2/1	No preferences shown by texture group				

*3 or more colors present each less than 50% of the total area

Implementation Plan

Using the preliminary field tool of FHWA KS-97/4, quarry sampling can lead to refinement of the field tool. Sampling should be representative of textural and mineralogical categories found to show relationship to passing or failing aggregate tests as well as represent the geographical extent of Kansas quarries.

The quarries to be sampled are those on a list agreed upon by the Research Geologist and the Field Geologist. Approval for the additional sampling needs to be in place. The list should provide enough optional choices to accommodate problems of closed quarries or those unable to accommodate the additional activity of the geology field crew and still provide the diversity needed.

Field samples come to the Materials and Research Center at which time research personnel prepare the samples for data collecting, store the data in a suitable database and perform statistical tests for refinement of physical test outcomes. The scheduling for these tasks is shown in the included table.

The following equipment and supplies will be utilized:

Sample bags and sampling equipment

Chemicals for staining

Acetone and acetate sheets for preparation of peels

Field and laboratory books for records and peels storage

Normal laboratory glassware

Rock saw with nonaqueous lubricant and sufficient blades

Polishing lapidary wheels and polishing grits

Color image analysis package if available

Computer and appropriate software for selected statistics

Sample storage space and storage containers

Implementation Plan

Task: Personnel

