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POTENTIALLY REACTIVE CARBONATE ROCKS

Progress Report No. 5

An Evaluation of Several Methods for Detecting Alkali-Carbonate Reaction

by

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(The opinions, findings, and conclusions expressed in this report are those of the authors and not necessarily those of the sponsoring agencies.)

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SUMMARY

Despite an extensive international research effort on alkali-carbonate reactions spanning almost twenty years, there is still no agreement on the most satisfactory testing methods for use in the control and acceptance of aggregates from commercial sources. A major portion of the research effort in Virginia has been directed toward this problem.

Four test methods were evaluated using a suite of 22 rocks exhibiting a wide range of potential expansion and mineralogical compositions. The purposes of the study were:

- (1) To evaluate the ability of the methods to predict detrimental expansions of concrete caused by alkali-carbonate reactions, and
- (2) to evaluate the capability of the methods for characterizing the reactive potential of commercial aggregate sources.

The aggregates were evaluated in concretes made with three cements having different alkali contents. Testing methods included the measurement of the expansion of concrete moist cured for 5 years, resistance of concrete to freezing and thawing, expansion of mortar bars, and length changes of rock prisms stored in a NaOH solution. Detailed petrographic examinations were made on both the concrete and rocks.

The results from the several testing methods were compared with each other, with the results from testing of quarries conducted by the Virginia Department of Highways and those from tests by other agencies.

Based upon the results of this study the following conclusions are drawn:

- (1) The expansion after 5 years of continuous moist storage of the concretes made with high alkali cements and containing the various aggregates ranged from 0.01% to 0.27%. Expansions of the apparently nonreactive aggregates are generally higher than values that have been reported in the literature. But an expansion value of 0.05% appears to separate the aggregates that evidence detrimental expansion in other test methods from those that do not.
- (2) The expansion of the rocks during testing in accordance with ASTM C 586 in general reflected their behavior in concrete. While the method is essentially a "screening method", when used in conjunction with other procedures, such as petrographic examination, it can be used for providing a rapid quantitative evaluation of potential for expansion of the aggregate in concrete.

- (3) Correlation of the prism expansions at various ages with 5 year expansions of concrete made with high alkali cement resulted in correlation coefficients between 0.87 and 0.95. Slightly higher correlations were found for 4- and 8-week prism expansions than for those at either 1 week or later ages. This appears to be related to the role of restraint in concrete and the time at which expansion begins.
- (4) For concrete made with the medium alkali cement, the correlation between 5-year expansions of concrete and expansions or prisms at various ages, improved with increasing age of prism. The correlation coefficients were less than those for the concretes with high alkali cement. Very low correlation was obtained for the low alkali cements, which is indicative of limited expansive reaction.
- (5) The correlation between concrete expansions and prism expansions reduces as fewer prisms are used for the average. There appears to be a slight decline in predictive power where less than 5 prisms are used; but considering the fact that the variation of expansions within a given lithology may be as large or larger than those between lithologies, the use of only 1 or 2 prisms per lithology provides an adequate indicator of the potential behavior of the rock in concrete and permits the sampling of more lithologies.
- (6) The value of 0.2% at 16 weeks in C 586, which has been used in Virginia to indicate potentially detrimentally reactive rocks, appears reasonable. Considering the variability of the method and the shape of the expansion vs. the curve, the value of 0.2% at 16 weeks is not too different from the value of 0.1% at 84 days suggested for use in C 33. Either value seems reasonable, but the more tolerant (.2% at 16 weeks) appears sufficient.
- (7) The results from mortar bar tests (ASTM C 227) are indicative of expansion in **concrete**. The expansions are of the same order as those in concrete. Values necessary to separate detrimentally expansive from innocuous aggregates are about one-half of those used when the method is applied to alkali-silica reaction. In view of the low expansions and high variability, C 227 appears to be of limited value in testing for alkali-carbonate reaction.
- (8) The characteristic reactive lithology described in ASTM C 294, whenever and wherever it occurs in a dolomitic limestone, will undergo reaction or dedolomitization in an alkaline environment. Whether the reaction will ultimately lead to expansion of a rock prism, concrete beam, or mortar bar, insofar as the rock is concerned, depends upon the perfection of the type lithology, including such factors as the calcite/ dolomite ratio and the grain size, and on the amount and distribution of the reactive texture within the rock. The factors governing expansion in concrete are related to the properties of the particular concrete in question; e.g. water cement ratio, total water soluble alkalies, relative amounts of paste and aggregates, etc.

- (9) In sampling of ledge rock, as in quarries, for alkali-carbonate reactive material should take cognizance of the evidence that the characteristic reactive texture and composition are only subtly and indirectly related to the primary factors of sedimentation. The texture is a secondary or replacement fabric which may or may not conform faithfully and uniformly to a given lithologic or stratigraphic unit as defined macroscopically in the field. While this lithology is not completely random in distribution, its presence or absence cannot be predicted with certainty in a given sequence of dolomitic limestones. Sampling for the reactive lithology and delineation of it should therefore be guided by petrographic considerations, using as many thin or polished sections as necessary to select samples for further tests.
- (10) Because of the variability that may exist, any thin or polished section that shows even a minor area of characteristic reactive texture should be classified as reactive and the field sampling unit which it has been taken to represent should be considered suspect.
- (11) While a total specification and acceptance program will vary with local conditions, and include all testing consistent with time and funds, the petrographic examination, while qualitative, appears to be the most satisfactory method since it is cheap, fast, and sufficiently predictive of the behavior of the aggregates in concrete. If one needs a quantitative measurement from a direct test, the fabrication and testing of concrete beams is the surest method, but the prism test will give a rapid and satisfactory prediction of the concrete expansion at an early age. The choice between the petrographic examination and prism test depends upon the equipment and/or personnel available to the evaluating agency.

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INTRODUCTION

In the December 1956 issue of the ASTM Bulletin, Swenson described what in the title of the article was designated "A Cement-Aggregate Reaction Undetected by ASTM Tests" (Swenson 1956). Since this initial attention, several extensive research projects, including that in Virginia, have been reported (HRB 1964) which have established that detrimental reactions can occur between cement alkalies and certain otherwise sound carbonate aggregates. Although several types of reactions have been identified, most attention correctly has focused upon the reactions which result in detrimental expansion with attendant cracking and deterioration. Despite the extensive research effort, however, ASTM Committee C-9 on Concrete and Concrete Aggregates, acting upon the recommendation of its Subcommittee III-e on Aggregates, in 1970 found itself unable to place quantitative limits in its standard specification for aggregates because of lack of agreement as to a test method and on specific limits on expansion using available test methods. Instead, Committee C-9 has recently placed in its aggregate specification a statement calling attention to the possible occurrence of the reaction but offering no quantitative guidance as to its control. Thus, for those faced with the need to assure the quality of aggregates for concrete, the situation is not greatly changed from that existing at the time of Swenson's original paper on the subject.

The first indication that some carbonate aggregates being quarried in Virginia were alkali reactive came from measurements obtained in 1957 from a bridge that was instrumented to measure volume change. Abnormal expansions were recorded from both the bridge deck and special test beams fabricated for comparison. Subsequently laboratory and field investigations led to the initiation of the present research project, which was designed to locate and study potentially reactive carbonate rocks in Virginia (Newlon 1961). The initial investigations were reported in 1962 (Newlon and Sherwood) and the

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project was then organized into several identifiable parts. Several remedial measures designed to reduce the expansion of reactive combinations were studied (Newlon and Sherwood 1963). The locations of potentially reactive aggregates were identified from a statewide survey of commercial sources (Sherwood and Newlon 1964); and the levels of alkalies in cement supplied in Virginia were evaluated (Newlon and Sherwood 1964). Throughout the study attempts have been made to elucidate the mechanism of the reaction (Sherwood and Newlon - 1964; Walker - 1967; and Hilton - 1968).

An important part of the project has been the evaluation of available methods of testing aggregates and concrete to permit prediction of expansion and thus reduce the detrimental effects which result when the reaction is unanticipated. This report covers the part of the project directed toward evaluating various of the available test methods.

PURPOSE AND SCOPE

Four test methods were evaluated using a suite of rocks exhibiting a wide range of potential expansion and mineralogical compositions. The purposes of this study were:

- (1) To evaluate the ability of the methods to predict detrimental expansions of concrete caused by alkali-carbonate reactions, and
- (2) to evaluate the capability of the methods for characterizing the reactive potential of commercial aggregate sources.

In the study, the expansion of concrete specimens made in conformance with one of the methods, ASTM C 157, "Length Change of Cement Mortar and Concrete", and stored continuously moist was taken as the indicator of performance, the prediction of which was the goal of the 3 remaining test procedures, some of which provide results of an earlier age than does Method C 157 and/or eliminate the need to fabricate concrete specimens.

VARIABLES

Test Methods

As reflected in the literature, at least 5 methods have been utilized for testing the potential reactivity of carbonate aggregates. These include:

- (1) Expansion of concrete beams containing the aggregate when tested in essential conformity with ASTM C 157, "Length Change of Cement Mortar and Concrete".
- (2) Expansion of mortar bars as described in ASTM C 227, "Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)".

- (3) Expansion of rock specimens (prisms or cylinders) stored in alkali solution using procedures of ASTM C 586, "Potential Alkali Reactivity of Cement-Aggregate Combinations (Rock Cylinder Method), developed as a screening test for the alkalicarbonate reaction.
- (4) Petrographic examinations as outlined in ASTM C 295, "Petrographic Examination of Aggregates for Concrete".
- (5) Expansion of powders in the Powder Cell Test (Gillott 1963), which has not yet been standardized.

All of these methods except the Powder Cell Test were evaluated in this project.

Materials

Aggregates

Twenty-two aggregates were used. These were limestones selected on the basis of performance or compositional characteristics obtained during earlier studies (Sherwood and Newlon 1964). One of the aggregates, designated "1-8"*, definitely has been shown to be responsible for premature deterioration of concrete in highway structures in Virginia (Ozol and Newlon 1971).

For each of the 22 aggregates, a 300-lb. sample was obtained by selecting from a quarry face, pieces of rock with lithologic characteristics comparable to those for the same sample designation in the previously reported statewide survey of commercial quarries (Sherwood and Newlon 1964). From the 300-lb. sample 6 pieces were randomly selected. From each of these a single rock prism was made for storage in 1N NaOH solution and periodic measurements of length change in accordance with the method subsequently standardized as ASTM C 586. Also a thin section corresponding to each rock prism was made for petrographic examination. The remainder of the sample was crushed and sized to permit the fabrication of concrete beams and mortar bars.

The variability of the rock properties within what was intended to be a homogeneous sample makes characterization of the rocks difficult. Average values for important properties are given in Table 1. The values obtained for each of the 6 pieces are given in Appendix A. Of the 22 samples, all but 2 contained some dolomite although in 2 cases dolomite comprised less than 10% of the carbonate fraction. Of the 22 samples, 7 were predominantly dolomite, 12 were predominantly calcite, and 2 had approximately equal amounts of the 2 minerals. Many had high insoluble residues. The material comprising this residue was predominantly illitic clay plus quartz. The absorptions were all below

^{*}Throughout the Virginia studies a two-number designation has been used to identify sources. The first number indicates a specific quarry and the second a specific lithology. Thus "1~8" indicates a sample from Quarry 1 and Lithology 8.

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0.4% with the exception of aggregate 13-1, which had an absorption of 1.3%. All of the aggregates would be judged physically sound on the basis of conventional acceptance tests.

Table 1

Rock	Ratio: Calcite Dolomite	Insoluble Residue, %	Absorption, $\%$
1-8	37/63	28	0.3
1-X	$\frac{68}{31}$	17	0.2
5-1	81/18	22	0.3
6-2	0/100	13	0.2
7-5	14/86	8	0.2
11-X	$\frac{-1}{76}/25$	7	0.1
12-1	87/13	. 8	0.1
12-9	58/42	21	0.4
13-1	1/99	33	1.3
15-9	$\frac{2}{78}/22$	4	0.4
21-2	97/3	$\hat{\overline{6}}$	0.1
23-9	0/100	16	0.1
24-7	0/100	10	0.2
27-4	45/55	39	0.3
27-6	25/75	26	0.2
29-4	55/45	25	0.1
29-5	70/30	13	0.1
34-7	80/20	10	0.2
35-5	79/21	11	0.1
36-X	93/7	6	0.1
48-4	100/0	20	0.4
48-5	100/0	8	0.3

Average Characteristics of Test Aggregates

Cements

Three cements, intended to provide a range in alkali contents, were used in the project. Throughout the report these cements will be designated as "high", "medium", and "low", reflecting their relative alkali contents. The chemical composition of the cements is shown in Table 2. The cements were Type II, although the calculated C_3A content of the "high" cement was 8.2%.

Subsequent to the initiation of this project, McCoy and Eshenour (1968) emphasized the importance of the water soluble alkalies. The total and water soluble alkali contents for the 3 cements are shown in Table 3.

Table	2
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Chemical Analyses of Cements (Values in Percent)

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Constituent	Cement Identified According to Anticipa Alkali Level		
	Low	Medium	High
\mathbf{s}_{2}	23.88	22.12	21.76
Al ₂ O ₃	3.78	4.40	4.68
Fe ₂ O ₃	3.28	2.80	2.48
CaO	63.25	64.60	63.46
MgO	1.38	1.64	2.51
so ₃	2.29	2.33	2.67
Ig. loss	1.16	0.76	1.52
Na ₂ O	0.08	0.20	0.32
к ₂ о	0.53	0.72	0.96
Na ₂ O (Equiv.)	0.43	0.67	0.95
с ₃ s	39.4	54.7	50.4
$c_2 s$	38.8	22.4	24.4
C ₃ A	4.5	6.9	8.2
C ₄ AF	10.0	8.5	11.7
Type Cement	11	п	Ш

Table 3

Cement	N	a ₂ 0	K	2 ⁰	Na	2 ^O (equiv.)
	Total	Water Soluble	Total	Water Soluble	Total	Water Soluble
''High''	0.32	0.05	0.96	0.52	0.95	0.39
"Medium"	0.20	0.11	0.72	0.61	0.67	0.51
"Low"	0.08	0.01	0.53	0.14	0.43	0.10

Alkali Content of Cements (Values in Percent)

It can be seen that the cement designated "medium" actually has a higher proportion of water soluble alkalies than that designated "high". Throughout this report the cements are identified on the basis of the total rather than water soluble alkalies, using the designations "high", "medium", and "low". As will be seen subsequently this ranking reflects the relative expansions of concrete containing the 3 cements.

TESTING PROGRAM

A single concrete mixture was made using each of the aggregates with each of the 3 cements. The coarse aggregates were artificially graded as shown in Table 4. The supply of aggregate was not sufficient to permit extensive adjustments by trial mixtures. For several of the aggregates, the supply was insufficient to permit mixing with all 3 cements. Sixty-one batches of concrete were prepared with the mixture characteristics shown in Table 5.

Table	4
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Sieve Sizes	Amount Retained, %
-1 + 3/4	15
-3/4 + 3/8	35
-3/8 + 4	35
-4 + 100	15

Table 5

	Average, $\overline{\mathbf{X}}$	Standard Deviation, V
Cement Content, lb./ey	592	7.5
$W_{\ell}C$, by wt.	0.49	. 004
Slump, in.	2.3	0.75
Air Content. %	5,5	0.80
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Important Characteristics of the 61 Concrete Batches Used in the Project

From each concrete batch, 3 beams 4" x 4" x 12" were made for measurements of expansion in accordance with ASTM C 157, with the beams stored continuously in the moist room. Expansion was measured periodically on each of the 3 beams for 18 months. After 18 months, the single beam showing the greatest expansion for each combination was removed from test and subjected to a detailed petrographic examination. The beam showing the lowest expansion was tested for flexural and indirect tensile strengths. $E_{X^{so}}$ pansion measurements have continued on the remaining beam from each combination, which at age 18 months was that nearest the average of the set of 3.

Three beams, $3'' \ge 4'' \ge 16''$ were also made from each batch for evaluating resistance to freezing and thawing in accordance with ASTM C 291. This procedure was intended to assess the physical durability of the aggregates. The freezing and thawing tests were begun after 14 days of moist curing followed by 7 days of drying in laboratory air. The freezing and thawing tests were continued for 300 cycles,

From the fine material developed during crushing, 2 sets of mortar bars, of 2 bars each, were prepared for each cement-aggregate combination and tested in conformance with ASTM C 227. The testing program is summarized in the schematic diagram shown in Figure 1.



Figure 1. Schematic outline of testing program.

RESULTS

This project involved the preparation of 61 batches of concrete and the fabrication and testing of 366 concrete specimens, 132 rock prisms, and 264 mortar bars. Even so, the number of specimens representing a given cement-aggregate combination are too limited for a sophisticated statistical treatment of the concrete and mortar bar data. The number of prisms representing a given aggregate are sufficient to warrant statistical analyses. These analyses are important because they draw attention to the inherent variability present on a microscale for rocks which on a macroscale appear homogeneous. For the concrete data the usual parameters of average and standard deviation will be presented simply to provide indications of the general levels of variability among the various cement-aggregate combinations.

Length Changes

Concrete

The average length changes of the concretes containing the various cementaggregate combinations after 1 year of continuous moist storage are shown in Figures 2-4, along with the corresponding standard deviations from the length changes of the 3 beams from a single batch.

In Figure 2 the data are arranged in order of increasing expansion for the concretes made with high alkali cement. The data for the concretes made with medium and low alkali cement are given in Figures 3 and 4. In these figures and in many that follow in the report, the data for the various aggregates are presented in the same order as those in Figure 2 to facilitate comparisons among the results from the several test methods and with those for the concretes containing the high alkali cement.

It is apparent from the data in Figure 2 that the expansions at 1 year for the cement-aggregate combinations containing the high alkali cement represent almost a continuum of values from 0.01 to 0.13%. The aggregates at the right end of the diagrams; i.e., those that are most reactive, show a definite relationship between expansion and alkali content. For concretes containing the less reactive aggregates and those combinations containing the low alkali cement the expansion is more or less random. The average length change for all concretes containing high alkali cement was 0.037%; medium, 0.026%; and 0.0079%.

As indicated earlier, after 18 months, length change measurements were continued only on the specimen nearest the average of the 3. The length changes after 5 years of continuous moist storage for the single specimen that was nearest the average at 18 months are given in Figure 5. The arrangement is again by aggregate source in order of increasing expansion, based upon 1-year expansions of the concrete made with high alkali cement (Figure 2).

Concretes containing aggregates 1–8 and 12–9 show significantly higher expansions at both 1 and 5 years than those containing the remaining aggregates. With the exception of a few obvious anomalies, the expansions are related to the level of cement alkalies and show similar behavior at both ages. In several cases at both ages the expansion values show a greater divergence than would be expected solely from the differences in alkali contents. The limited amount of aggregate available permitted the preparation of only a single batch so that exact accord would not be expected. The largest anomaly is in the behavior of concrete containing aggregate 13–1, in which that made with the low cement showed very high expansion.

The 5-year expansions of concretes containing the high cement range from 0.01% to 0.27% and present a progression similar to that for the 1-year data,

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Figure 2. Length change at 1 year of concrete beams made with high alkali cement.

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Figure 5. Length change at 5 years of concrete beams made with high alkali cement.

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The wide range of expansions raises the question as to what is "normal" expansion of concrete subjected to continuous moist storage. Despite the extensive literature on volume changes of concrete, there are comparatively few published results for concrete specimens kept continuously moist. The emphasis in most of the published research has been on drying shrinkage or autogenous volume changes, both of which under normal circumstances are more realistic conditions for simulating practical problems than is continuous moist storage. Washa (1966) states, "the ultimate amount of expansion (for moist storage) is usually less than 0.025 percent." L'Hermite (1960) indicates that concrete cured moist for 1,000 days will swell about 0.01%. Gonnerman, Lerch, and Whiteside (1953), in reporting comprehensive studies of expansion characteristics of cement pastes and mortars, include a few data from measurements on concrete. The average 5-year expansions from these tests approximate 0.02%.

No noncarbonate reference aggregates were included in this study, but some of the limestone aggregates are undoubtedly, by all other criteria, nonexpansive. Thus, the expansions of the concretes with nonreactive aggregate are greater than can be reconciled with previously published values. It was reported in an earlier paper that expansions of reference concretes containing a granite coarse aggregate were 0.030% (Newlon and Sherwood 1964) after moist storage for 1 year. Comparable expansions have been observed for "reference concretes" in unpublished studies being conducted at the Waterways Experiment Station (Buck 1970). Since, with the exception of concretes containing aggregates 1-8 and 12-9, there is no clearly defined separation among the expansions, the value of 0.05% expansion after 5 years continuous moist storage was taken as a point of reference for discussion purposes. While this value is somewhat arbitrary, the rationale for its selection is as follows:

- (1) It is consistent with the addition of a tolerance to the value measured by Newlon and Sherwood (1964) for concrete containing a granite coarse aggregate.
- (2) It has been used by Smith (1964) as a value for judging reactivity although he also used a time of 84 days.
- (3) It is consistent with the values reported by Swenson and Gillott (1964) and Hadley (1964) for cement-aggregate combinations giving expansions definitely lower than those combinations known to have detrimental performance records.
- (4) It reflects the uncertainty as to the relationship of laboratory expansions to field performance by doubling the values generally considered as normal. If controls were placed on the use of aggregates exhibiting potential for expansion, there would be some justification for establishing a "tolerant" rather than a "restrictive" value, which could subsequently be refined with further experience.

Rock Prisms

As noted earlier, a $1/4" \ge 1/4" \ge 1-1/4"$ prism was made from each of 6 rocks randomly selected from the 300-lb. sample that had been selected to represent a given

lithology. It is important to reemphasize that the 300-lb. sample was selected from the ledge under the supervision of 2 of the authors using for comparison a hand sample of the same lithology obtained from the previous statewide gathering (Sherwood and Newlon 1964). This sample meets as closely as is humanly possible a uniform lithology as described in ASTM Methods D 75 and C 295.

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The 6 prisms were stored in 1N NaOH and tested in accordance with procedures conforming to the subsequently adopted Method C 586. The average expansions for 4 samples (12-9, 1-8, 15-9, and 13-1) representing typical behavior by rocks with different expansion characteristics that will be the basis for subsequent discussion are shown in Figure 6. In addition to the unrestrained expansions measured using C 586, expansions measured for specimens of 12-9 and 1-8 under restraint using equipment developed by Hilton (1968) are also shown. As seen in Figure 6, samples 12-9 and 1-8 without restraint expanded approximately 1.2 and 5.3% respectively. Under restraint, intended to simulate that provided in concrete by the mortar matrix, the expansions of both 12-9 and 1-8 were about 0.3%, despite the large difference in unrestrained expansions. Hilton demonstrated that this was a logical consequence of textural differences between the rocks. It is also understandable that because of the restraint offered by the mortar matrix some plateau of expansion would be reached beyond which the unrestrained expansion of aggregate would not be manifested in concrete. This point will be significant in subsequent discussion.



Figure 6. Length change of typical rock prisms.

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The prism expansion of sample 15-9 is typical of those samples which showed slight initial shrukage with insignificant subsequent expansion. Sample 13-1 showed initial shrukage and subsequent expansion, which began between 4 and 8 weeks and ultimately reached 0.5%. This behavior is typical of rocks characterized as "delayed expanders" and studied extensively by Dolar-Mantuani (1964). Sample 13-1 is the only lithology from any of the Virginia studies that has exhibited delayed expansion of this magnitude and it was included in this evaluation for that reason.

The distribution of average expansions and standard deviations of the rock prisms after 1 week, 16 weeks, and 1 year are shown in Figures 7-9. The expansions are arranged from left to right in the same order as the 1-year expansions of the corresponding concretes shown earlier in Figures 2-4.

As is apparent in Figures 7-9, after 1 week only 1-8 and 12-9 showed significant expansion. It is also obvious that the expansions of the 6 prisms from the given lithology were highly variable at all ages as evidenced by the fact that in about one-half of the cases the standard deviation is larger than, or of the same magnitude as, the average. Despite this variability, there is for both the 16-week and 1-year data a general increase of prism expansions, which reflects a relationship with the concrete expansions at 16 weeks and 1 year than does aggregate 1-8, the concrete expansions shown in Figure 5 are about the same.



Figure 7. Length change of 6 prisms from each sample (age - 1 week).





Figure 8. Length change of 6 prisms from each sample (age -16 weeks).



Figure 9. Length change of 6 prisms from each sample (age -1 year).

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This is consistent with the findings of Hilton (1968) discussed in connection with Figure 6. The delayed expansion of aggregate 13-1 prisms was not reflected in the expansion of concrete. Concrete containing aggregate 13-1 showed the lowest expansion of any combination, except for the apparent anomalous behavior with the low alkali cement. The failure of aggregate 13-1 to expand in concrete may also be explainable from considerations of the time of the onset of expansion and the degree of restraint offered by the matrix. It is not difficult to visualize that for rapid expanders like 1-8 and 12-9, the expansion would begin almost immediately, when the surrounding paste would offer comparatively little restriant (i.e., it would have a low modulus of elasticity), and the aggregate expansion would be reflected in the concrete expansion. When a rock begins to expand after several months, the matrix has developed a much higher modulus of elasticity and thus more restraint to the expansion.

For the prism expansions, there are only 4 significant anomalies in the ranking of expansion as compared with that of the concrete. These are aggregates 23-9, 35-5, 5-1, and 29-5. The latter two show substantial expansions although less might be anticipated from the concrete expansions. These differences are explained by differences in the characteristics of the rocks in the prisms, and these supposedly similar rocks in concrete. This lack of correspondence is discussed in detail later in the section on petrographic examination.

Correlation Between Expansion of Prisms and Concrete

The rock prism test was developed as a rapid screening method. Its primary attractions are simplicity and early indication of potential reactivity. Criticisms of the procedure focus upon the variability of the results obtained from samples that purportedly represent the same rock and the uncertain agreement between the expansion of unrestrained rock specimens and those when the aggregate is placed in concrete.

The average expansions at 1 year of concretes containing the high alkali cement are plotted in Figure 10 as a function of the average expansions of the 6 prisms at an age of 8 weeks. The ranges of prism expansions are also indicated. Despite the variability of the prism expansions, there is a general prediction of concrete expansion by the expansion of rock prisms. This relationship is typical of that obtained at other ages.

Correlation and regression analyses were made to compare concrete and rock prism expansions using the results for all of the various combinations of ages at which measurements were made from 1 week through 5 years. The following types of relationships between concrete expansion (Y) and rock prism expansion (x) were considered.

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$$Y = Ax + B$$
 $Y = Ax^2 + Bx + C$ $Y = A1nx + B$ $Y = Ax^3 + Bx^2 + C$ $Y = Ax^{1/2} + B$ $Y = Ax^3 + Bx^2 + Cx + D$



Figure 10. Relationship between length change at 1 year of concrete containing high alkali cement and length change of prisms at 8 weeks. Average and range of prism length change are shown for each sample.

Preliminary analyses suggested that the correlations involving the logarithmic and square root functions were inappropriate because the negative values of x required use of the quantity (x + 1), which was both cumbersome and of little significance. The shape of polynomials where the order of x was 3 or greater had no physical significance. Thus the correlations centered on relationships of the form:

$$Y = Ax + B$$
, and $Y = Ax^2 + Bx + C$

. .

The results from aggregates 13-1 and 12-9 were not used in these correlations. For reasons that were discussed earlier, it was reasoned that both of these rocks presented somewhat unique behavior that was not typical of the behavior of the remaining 20 rocks. It is speculated that the relationship between the expansion of concrete and rock prisms with expansions above some limiting value, say 1%, would be different from that for less expansive rocks. It is likely that had more examples of such highly expansive rocks been included in the study sample, a meaningful relationship could have been

developed. Sample 13-1 was a delayed expander whose expansion was not reflected in the concrete. While intensive studies have been made of this type of rock by Dolar-Mantuani (1964), these have been limited to studies of rock without parallel studies of concrete. The single sample in this project obviously does not permit generalization, but it suggests that the delayed expansive tendency in rocks is not manifested in conerete, at least up to 5 years of exposure.

The correlation coefficients, r, and standard errors, $S_{\rm YX}$, are shown in Table 6 for the 3 cement-aggregate combinations. Prism expansions at 1 week, 4 weeks, 8 weeks, 16 weeks, and 1 year were correlated with concrete expansions at 6 months, 1 year, $1\frac{1}{2}$ years, and 5 years.

As would be expected, the correlation coefficients for the concrete made with low alkali cement are very poor. This confirms the fact that the amount of expansive reaction is low and only to a very limited extent related to the reactivity of the aggregate as manifested in the expansion of the rock prisms.

The correlation coefficients for the medium and high cements are both higher than those for the low cement. The correlation with high alkali cement concrete is better than that with the medium. The correlation coefficients generally improved with length of exposure of the concrete. In the case of the rock prisms, it appears that after 4-8 weeks the improvement in the correlation did not improve significantly. In several cases, particularly with the high alkali cement, it became slightly poorer. A typical plot of the relationship between concrete and prism expansion is shown in Figure 11.

As seen in Table 6, the use of the polynomial improved the correlation, but only slightly. The correlation coefficients between the prism expansions at any age and the expansion of concretes containing the high alkali cement are between 0.858 and 0.933. Those for the medium cement concretes are somewhat lower.

The correlation between the prism expansions and those from corresponding concretes is sufficient to warrant the use of the prism test as a rapid and comparatively simple indicator of reactivity. For the data developed in these tests, there is little improvement to be gained from running the prism tests beyond 4 weeks. Axon (Missouri 1967), from his studies, concluded that the prism test period could be reduced below 8 weeks based upon correlations with expansions at later ages although such shortening would increase the number of delayed expanders that would go undetected. It should be reemphasized that the highly expansive aggregate 12-9, which also expanded in concrete, and the delayed expanding aggregate 13-1, which did not, were excluded from this correlation.

Despite the excellent correlation between the average values of prism and concrete expansions, the variability of the expansions of the 6 prisms taken from a macroscopically homogeneous lithology is bothersome. Similar behavior in other studies has been the basis for opposition to the use of Method C 586 for specification purposes. The method as adopted by ASTM in 1966 does not furnish guidelines as to the number of samples necessary to characterize a given source, but treats rather the accomplishment of expansion measurement for a given piece of rock.

										τ Γ	Ax +]	B (Str	aight I	(jine)										
			Lc	ow Alk	tali Cei	ment					Ŵ	edium	Alkali	Ceme	t				High	Alkali	Cemei	f		
	24 w.	ks.	52 wl	ks.	78 n	rks.	5 yr	s.	24	wks.	52	wks.	78	wks.	5 yı	s.	24 w	ks.	52 wk	s.	78 wks		5 yrs	
	r	s xy	អ	$^{\rm s}_{\rm xy}$	น	$\mathbf{x}_{\mathbf{x}\mathbf{y}}^{\mathbf{S}}$	ង	s xy	ม	ω _x	r A	°× v×	ч Л	s _{xy}	' r	$_{\rm xy}^{\rm s}$	អ	$^{\rm s}_{\rm xy}$	ħ	s xy	ы	sxy xy	น	s
1 week	. 268	.02	.663	.01	. 577	.01	.472	.01	. 602	. 02	. 666	. 01	. 587	.01	. 659	.02	.877	.01	.863	.01	. 856	. 01	898	.02
4 weeks	.115	.02	.611	.01	.572	.01	.474	01	.724	.01	801	. 01	.728	.01	.791	.02	. 866	.01	.911	.01	. 900	. 10.	945	.02
8 weeks	.519	• 01	.643	.01	.615	.01	.513	.01	.721	.01	. 817	.01	. 758	.01	.820	.02	. 836	.01	. 897	.01	. 891	. 01	933	.02
16 weeks	.514	.01	. 655	.01	.641	.01	.490	.01	.718	.01	.815	.01	.774	.01	.843	.01	. 798	.01	.872	.01	.862	. 10	903	.02
52 weeks	.485	.01	.652	.01	.674	.01	.454	.01	.708	.01	.810	. 01	. 773	.01	.870	.01	. 750	.02	.825	.01	.821	. 10.	868	. 03
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								Υ	$= Ax^2$	+ Bx	с +	(Secol	nd Deg	ree Po	lynom	ial)								
1 week	.357	.02	. 696	.01	.602	.01	.486	.01	.656	.02	. 696	.01	.600	.01	.674	.02	, 933	.01	.914	.01	. 896	. 10	938	02
4 weeks	.134	.02	.622	.01	.615	10.	.496	.01	.726	.01	177 .	.01	.771	.01	.824	.02	.919	.01	.927	-01	. 60£	. 10	955	02
8 weeks	.521	.01	.648	.01	.662	.01	.520	.01	.723	.01	.819	.01	. 775	.01	. 839	.02	.904	.01	.930	.01	911	. 10	955	02
16 weeks	.515	.01	.656	.01	• 687	.01	.506	.01	.726	.01	.815	.01	.781	.01	. 859	.01	. 883	.01	.920	.01	268	. 01	940 .	02
52 weeks	.489	.01	.653	.01	. 705	.01	.473	.01	.738	.01	.815	.01	.773	.01	.874	.01	.858	.02	.906	. 01	878	. 10	926	02
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Table 6

Correlation Coefficients, r, and Standard Errors, S_{xy}, for Rock Prisms vs. Concrete Beams V - And N Concrete Deams

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Figure 11. Concrete vs. prism length change.

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Because the 6 pieces of rock were randomly drawn from a single lithology, some estimate of the influence of sample size can be obtained by treating the average expansions indicated from 6, 5, 4, 3, 2, and 1 prisms respectively. The correlation coefficients and standard errors so obtained based upon comparison of prism expansions at several ages with those for concretes made with high alkali cement after 5 years are shown in Table 7.

Table 7

Influence of Sample Size on Results of Simple Regression Analysis Rock Prisms vs. Concrete Beams with High Alkali Cements (age 5 years) (Aggregates 12-9 and 13-1 Removed)

	1 w	k.	4 wk	S.	8 wł	s.	16 w	XS.	52 w	ks.
Ave. of	r	s _{xy}	r	\mathbf{s}_{xy}	r	s _{xy}	r	s_{xy}	r	s _{xy}
6	.898	.02	. 945	.02	.932	.02	.903	.02	.867	.03
5	.896	.02	.924	.02	.893	.02	.873	.03	.831	.03
4	.873	. 03	。865	. 03	.787	.03	.804	.03	。734	.04
3	.854	.03	.871	.03	.869	.03	.817	.03	.736	.04
2	.813	.03	. 803	. 03	.800	.03	.747	.04	.652	.04
1	.829	.03	.857	. 03	.856	.03	.819	.03	.587	.04

Straight Line (Y = Ax + B)

2nd Degree Polynomial $(Y - Ax^2 + Bx + C)$

6	. 938	.02	.955	.02	. 954	。02	.940	.02	.925	.02
5	.942	.02	.947	.02	. 926	.02	. 908	.02	.872	. 03
4	.940	.02	.915	.02	.793	.03	.812	. 03	. 736	. 04
3	. 935	.02	.932	.02	.918	.02	.854	.03	.741	.04
2	.924	.02	.910	.02	。865	.03	.767	.03	.655	. 04
1.	. 909	.02	, 935	. 02	. 932	.02	.878	.03	.625	.04
							ļ			

As would be expected, the correlation coefficient in general decreases as fewer prism results are used to compute the average for use in the correlation. The decrease is not, however, very great. Even if only 1 prism is used, the correlation coefficient remains above 0.80. It can be seen, however, that the standard error of estimate increases when 4 rather than 5 samples are used. Thus, less confidence could be placed in the conclusions drawn from using fewer prisms. Although the applicability of this relationship can be considered to hold only for the samples included in this study, the results do give some idea as to the improvement of the relationships with increased sample size. The results also provide a certain degree of comfort that 1 or 2 samples provide an adequate correlation and, therefore, an adequate indication for making preliminary judgments of potential reactivity.

The fact that prism expansions at early ages correlate better with concrete expansions than prism expansion at later ages is also reflected in these comparisons.

Mortar Bar Results

The expansions of mortar bars made and tested in conformance with ASTM C 227 are shown in Figure 12. The arrangement from left to right is that of the 1 year concrete expansions shown earlier in Figure 2. For the mortar bars made with high alkali cement, there is a general pattern of agreement with the concrete data from Figure 2 and the prism data from Figure 7 reflecting an increasing expansion with the degree of rock reactivity. There are 3 anomalies in the results wherein the expansion of the mortar bars was less than would be expected based upon the expansion of the concrete (1-X, 12-1, and 29-5). Two of these samples, 12-1 and 29-5, also showed lower expansions in the prism test than were reflected in the expansions of concrete. The other samples that showed lower prism expansion than the corresponding concrete expansion (23-9, 35-5, and 5-1) showed higher than average mortar bar expansion. All of the mortar bars made with the medium cement showed substantially lower expansions than were observed with either the high or low cements. The "average" expansion for the 22 aggregates and the cement characteristics are summarized in Table 8, along with the alkali contents.

One possible explanation for the results might be that the low expansion with the medium cement was related to the possibility that the alkalies were so readily soluble that a rapid reaction took place - a reaction that would not be reflected in the measurements of the hardened mortar.

Table 8

-	of Three Differ	ent Alkali Co	ontents
Cement	Total All H ₂ O Sol.	kalies Total	Mortar Bar Expansion, C 227, 6 months, %
high	0.39	0.95	。037
medium	0.51	0.67	.004
low	0.10	0.43	. 016

Expansions of Mortar Bars Made with Cements

As reported in a detailed analysis of the mortar bar results (Hilton 1964), the expansions were highly variable. As stated in Method C 227, the repeatability is considered satisfactory if the expansion of any specimen does not differ by more than 0.003





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percentage points from the average value of the expansion of all specimens molded from the same batch or mortar. Where the average expansion exceeds 0.02%, the repeatability is acceptable if the expansion of any specimen does not differ from the average by more than 15%. For this study, the repeatability is based on the deviation from the average of 2 specimens since only 2 were made from each batch. Table 9 shows the number of batches with an acceptable repeatability and the total number of batches produced.

Table 9

Designated Cement Alkali Group	Total Number of Batches	Total Batches with Acceptable Repeatability	Percent Acceptable Repeatability
Low	44	41	93
Medium	44	24	55
High	44	33	75
Totals	132	98	74 (Avg.)

Summary of Batch Repeatability for Mortar Bars

During the progress of this project, Subcommittee II-b of ASTM Committee C-9 undertook a cooperative testing program to determine the ability of Method C 227 to differentiate among the carbonate aggregates of different reactivities. Five of the aggregates from the Virginia study were included in this testing program. These results are discussed later in the report.

Comparison Between Expansion of Mortar Bars and Concrete

A comparison of the expansion of concrete beams and mortar bars at an age of 6 months is shown in Figure 13. The significant point of this plot is that the concrete expansion is of the same order as that of the mortar bars; i.e., no acceleration of expansion is gained from processing the aggregate for use in mortar bars. The mortar bar test was developed for use with the alkali-silica reaction in which reducing the size of the aggregate accelerates the reaction and concommitantly the expansion. For the alkali-carbonate reaction it has been conclusively demonstrated that the expansion increases with increasing aggregate size (Swenson and Gillott 1960), so that little is to be gained by using the mortar bar test for judging the alkali reactivity of carbonate aggregates.

The variability of the mortar bar method also reduces the usefulness of the procedure.

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Figure 13. Mortar bar vs. concrete expansion (6 months).

Resistance to Freezing and Thawing

When this project was initiated, there were, and to some degree there still are, questions as to the extent to which carbonate aggregates that are susceptible to reaction with cement alkalies would be otherwise satisfactory as concrete aggregates. Early work with aggregate 1-8 had shown that it met conventional soundness requirements. To evaluate the quality of the other aggregates, from each batch of concrete 3 beams $3" \times 4" \times 6"$ were fabricated and tested for resistance to freezing and thawing in accordance with ASTM Method C 291 (rapid freezing in air and thawing in water). The beams were moist cured for 14 days, stored for 7 days in laboratory air at approximately 73° F and 50% RH and placed under test at an age of 21 days.

The results are shown in Figure 14, where the durability factors for the various cement-aggregate combinations are presented, again using the order of increasing expansion at 1 year of concretes containing the high alkali cement. Using the usual criterion that a durability factor of less than 60% suggests questionable performance, it is seen that only aggregate 1-8 was below this value with both the high and low cements. Observations made on these beams suggest that this low result might be due to contamination with a slightly weathered variety of the typical aggregate 1-8.





In the field situations where concrete containing aggregate 1–8 has been exposed to freezing and thawing as well as deicing salts, it has sometimes been difficult to separate the effects of expansive reaction from those of freezing and thawing, but in at least 1 case there seems to be little doubt that the balance was tipped to poor performance by the reaction (Ozol and Newlon 1971). There is certainly no consistent relationship among expansion, alkali content, and the durability factor, and except for aggregate 1–8 and possibly 13–1 these rocks would all be classed as acceptable. As noted earlier, aggregate 13–1 is untypical of most Virginia aggregates, particularly with respect to its high absorption of 1.3%.

Strength

As explained earlier, the volume change specimen showing the lowest expansion after 18 months was broken in flexure using one-third point loading in accordance with ASTM Method C 78. The 2 end portions were then sawed to provide blocks 4" x 4" x 3", and these were tested for splitting tensile strength using procedures as outlined in ASTM C 496. The specimens were approximately 2 years old at the time of testing. During the period between removal from moist storage and testing, the specimens were stored in laboratory air. All specimens were subjected to approximately the same conditions so that any differences are comparatively small when compared with the age at the time of test. The strength results were highly variable and no consistent relationships were found between expansion and tensile or flexural strengths.

Petrographic Examination*

Concrete

Each of the concrete beams available was cut with a diamond saw and a slab with a finely ground finish was prepared for petrographic examination. The following features were examined: (1) Cracks within the aggregate, (2) cracks within the aggregate which extend into the paste, (3) separations at the boundary between the aggregate and the paste, (4) cracks in the paste apparently unrelated to cracks within the aggregate, and (5) rims (internal) around the edge of the aggregate. Each feature was graded as to severity and abundance by the following schedule:

- NO --- feature not observed
- R --- feature rare
- NC feature more frequent than rare but not common
 - C --- feature of common occurrence
 - A --- feature abundant
- VA --- feature very abundant

^{*}Note: The petrographic examinations of these concretes were conducted by Mrs. H. N. Walker, Highway Materials Research Analyst, with the Research Council.

A numerical value was assigned to each grade, and these values summed for the first four features of each slab (all except rims). This sum was then used to place the slabs in order of descending amount of deterioration. Whenever the sums for 2 or more specimens were equal, the exact order was determined by further examination of the slab and by considering those cracks which were in the paste more deleterious than either the cracks entirely within the aggregate or boundary separations. A tabulation of the results of this examination is given in Table 10 in which the slabs from the beams are listed in order of descending deterioration; i.e., number 1 is the most deteriorated slab while number 56 is least effected.

Of the 66 possible cement-aggregate combinations available, 4 were not mixed due to a lack of aggregate. No beams were available for petrographic examination from any of the 3 mixtures containing aggregate 1-X, the mixtures of aggregate 48-5 with high alkali cement, aggregate 24-7 with high cement, or aggregate 27-4 with medium cement. Thus, 56 beams were studied.

Detailed correlations of the features observed in the petrographic examination with other properties are probably not warranted but several relationships are evident. Of the 14 aggregates that had expansions as shown in Figure 5 greater than 0.05% after 5 years, 13 are represented in 20 of the 28 beams representing the most deteriorated half of the specimens. Only aggregate 35-5 is absent. It is also evident that concretes containing the high alkali cement were generally much more affected by cracking, separation, etc. than those with low alkali cements. The differences between the most reactive aggregates (1-8 and 12-9) when used with the 3 different cements are of particular interest and are compared in Table 11.

The defects are obviously related to cement-aggregate interactions. The complexity of these interactions is vividly demonstrated by the behavior of aggregate 12-9. As shown in Table 11 cracks in the paste and boundary separations became less numerous with lower alkali cements. Cracks in the aggregate showed a reverse trend. These were associated with rims that developed in the aggregate. Typical sections from each of the 3 concretes containing aggregate 12-9 are shown in Figures 15-17. As indicated in Figures 15-17, the expansions of the 3 beams were proportional to the alkali contents of the cement, as was the frequency of paste cracking. The tendency to form rims within the aggregate however showed an opposite tendency. Pronounced rims are seen in Figure 17 for the low alkali cement. The aggregate particles also exhibited cracking that is confined to the rim area. Despite this cracking there is no evident separation between either the outer rim and the inner aggregate or between the rim and the paste. The mechanism of the formation of these cracks is not clear. Their presence implies shortening of the rim as compared with the interior. Such relative movement would be expected to generate some boundary separations but none are evident. It is conceivable that some autogenous healing has taken place, but this is not observable. When etched in 3N HCL, the rim areas are very slightly "negative"; i.e., they appear slightly more soluble than does the interior of the rock particle. Ideas relating to the formation of rims have been summarized by Hadley (1964), and the susceptibility of rocks in Virginia to such rim formation has been previously presented (Sherwood and Newlon 1964).

At this time, no significance is placed on the conditions shown in Figures 15-17 beyond the fact that they emphasize the vast differences in the behavior of this aggregate when mixed with different cements and reemphasize the dynamic processes which occur at the paste-aggregate interface.

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Occurrence of Defects as Observed During Petrographic Examination

Rank	Aggregate	Cement Alkali Content	Cracks in Aggregate	Cracks in Aggregate Extending into Paste	Boundary Separations	Cracks in Paste	Rims
1	11 - X	High	NC-C	NC-C	NC	VA	NO D
2	29-5	High	NC	c	NC	VA A-WA	NO-R
3	5-1	High	NC	NC	NC	$\Delta = V\Delta$	NO
4	12-9	High	NC	C	NC	A	NO VA
5	1-8	High	NC	NC	NC	A-VA	NC
6	29-4	High	NC-C	C	R	Δ	NO
7	7-5	High	NC-C	С	NC	Ĉ	NO
8	21-2	High	С	NC	NC	č	NO
9	11 - X	Medium	С	NC-C	NC	č	R
10	27-4	High	R-NC	NC	R	VA	NO
11	21-2	Low	VA	R-NC	NC	NC	NO
12	23-9	High	NC-C	С	R	NC	C C
13	34-7	High	NC	NC	R	A	ŇO
14	11-X	Low	VA	R-NC	R	R-NC	B-NC
15	27-6	High	NC	NC-C	R	C-A	R
16	12-1	High	NC	NC	NC	C	NO
17	36-X	High	NC	С	NO	R	NO
18	12-1	Low	С	R	R	NC	NC
19	1-8	Medium	NC	R	R	C	NO
20	15-9	Low	NC	NC	R	NC	NO
21	27-6	Medium	С	R	NC	R	NO
22	34-7	Low	NC	R	R-NC	NC-C	NO
23	29-5	Medium	NC-C	R	NC	R	NC
24	6-2	Medium	R-NC	R	R	Ċ	NC
25	12-9	Medium	NC	NO	NC	NC	VA
26	15-9	High	R	NC	R	NC	NO
27	29-4	Low	NC-R	NC-R	NC	NC	NO
28	48-4	High	NC	NC	R	R	NO
29	27-4	Low	NC	R	R	NC	NO
30	5-1	Medium	NC	R	NO-R	NC-C	NO
31	34-7	Medium	NC	R	R	NC	NO
32	29-5	Low	С	NO	R	R	NC
33	6-2	High	R	R	R	NC	NO
34	15-9	Medium	NC	R	R	R	NO
30	13-1	High	NC	R	R	R	VA
30	7-5	Low	C	NO	\mathbf{R}	R	NO
31 90	7-0	Medium	NC	NO	NC	R	NO
30	0-1 05 5	Low	NC	R	R	R	NO
39	30-0	Medium	R	NO-R	R	NC	NO
40	24-1	Medium	NC	NO-R	R	R	R
42	48-5	Low	NC	NO	R	R-NC	NO
43	40-0 95_5	Medium	ĸ	NO	NC	R	NO-R
44	00-0 19-0	nign Lorr	ĸ	K	R	R	NO
45	12-3 36-Y	LOW	A	NO	NO	NO	А
46	20-A 23-0	Medium	NC	NO	R	R	С
47	20-0 97-6	Low	NC	NO	NC	NO	R
48	29-4	Modium	NC	NO	R	R	NC-C
49	13-1	Low	NC C	NO	R	NO-R	NO-R
50	13-1	Madium	NU-U P	NO	NO-R	NO-	VA
51	48-5	Low	n D	NU	R	R	VA
52		Low	к D	NO	R	R	NO
59	27-1 26_V	LOW	к NO	NO	R	R	NC
54	30-A 1-9	LOW	NC D NG	NO	R	NO	NO
55	6-9	LOW	R-NC	NO	R	NO	NC
56	22-0	LOW	NU NO D	NO	R	NO-R	R
	20-3	LOW	NO-K	NO	R	NO	R

Table 11

Condition of Concrete Containing Most Reactive Aggregates (1-8 and 12-9)

Rank in Table 10	Aggregate	Cement Alkali Content	Cracks in Aggregate	Cracks in Aggregate Extending into Paste	Boundary Separations	Cracks in Paste	Rims
4	12-9	High	NC	с	NC	Α	V۵
5	1-8	High	NC	NC	NC	A-VA	NC
19	1-8	Medium	NC	R	R	с	NO
25	12-9	Medium	NC	NO	NC	NC	VA
44	12-9	Low	А	NO	NO	NO	4
54	1-8	Low	R-NC	NO	R	NO	NC



Figure 15. Aggregate 12-9 with high alkali cement. Expansion of concrete after 1 year equals 0.096%. Primary features are cracks in aggregates, cracks in aggregates extending into the mortar and very minor rims on aggregates. (3x)



Figure 16. Aggregate 12-9 with medium alkali cement. Expansion of concrete after 1 year equals 0.086%. Primary features are slight boundary separation, cracks extending into the mortar and some rims on aggregates. (3x)



Figure 17. Aggregate 12-9 with low alkali cement. Expansion of concrete after 1 year equals 0.025%. Primary features are lack of cracking in mortar and pronounced rims on aggregates and cracks within rim areas. (7x)

The characteristics observed in the petrographic examinations of the concretes confirm in practically all cases the expansive behavior of the various cement aggregate combinations.

Rocks

Consideration of previous work on the petrography of the rocks involved in the alkali-carbonate reaction tempts one to attempt to verify the ideas that:

- (1) All limestones which possess the characteristic texture and composition as described in ASTM C 294 and shown in Figure 18 will react or dedolomitize in an alkaline environment; and
- (2) all limestones which dedolomitize in an alkaline environment possess the characteristic texture and composition.

The words "dedolomitize" or "react" are used particularly rather than the word "expand", since recognition must be given to both the propensity for a rock to react chemically and, depending on considerations related to texture and structure, to manifest that reaction in expansion if conditions are favorable for that.





The foregoing syllogistic ideas might then be modified to include these additional ones.

All rocks that possess the characteristic texture and composition may expand in an alkaline environment or in concrete. All rocks which expand in an alkaline environment or in concrete do possess the characteristic texture and composition. Or, alternatively, no rock that does dedolomitize or expand in alkaline solution or in concrete does not possess the characteristic texture and composition.

Reliable and unequivocal answers to these propositions, if they are posed in the form of questions, will measurably aid the correlation of methods for detecting the reaction.

As noted earlier, thin sections were made from each of the 6 randomly selected rock samples from which a prism was made. The section was taken adjacent to the prism location.

In order to test the propositions outlined above, an attempt was made to organize the test samples according to the scheme illustrated in Figure 19.

Ideally there should be the mutually exclusive organization depicted in the chart, where all those samples that have the characteristic texture do expand in rock prism and in concrete and all those samples that do not have the characteristic texture and composition do not.

	ROCK PRISM EXPANDS	ROCK PRISM DOES NOT EXPAND	MORTAR-BAR & /OR BEAM EXPANDS	MORTAR-BAR & /OR BEAM DOES NOT EXPAND
CHARACTERISTIC	12-9		12-9	
POTENTIALLY	1-8		1-8	
REACTIVE	29-4		29-4	
TEXTURE	22.4	1 < 1	22-6	2
			<u>ب</u> بر الم	
	ĮĮ:		ĨŠ	
		11-X -		
		6-2		6-2
NOT		7-5		7-5
POTENTIALLY		25.4		29
REACTIVE		7-5		·
TEXTURE	12-1 -			
		23-9-		
		5-1 🔫	≻ 5-1	
	13-1 -			

Figure 19. Schematic representation of relationship between petrographic characteristics of rocks and expansion of rocks, mortar bars, and concrete beams.

The initial grouping of the samples is shown in Figure 19. Most, but not all, of the samples fall in the anticipated categories. For simplicity, not all of the samples in the study are shown, but rather illustrations of proper arrangement and several types of deviations which are typical of the entire suite of samples are presented.

It was now necessary to examine critically the deviations from the anticipated categories, mindful of the facts that the rock expansion represented the average of 6 separate prisms, that each of the 6 prisms had a corresponding thin section, and that the concrete beam and mortar bar aggregates were composites and that thus their behavior in concrete would represent the composite behavior of all of the samples from which the prisms and thin sections were made. Immediately, the possibility of sample variability could be examined as a chief reason to account for inconsistent behavior.

If close microscopic inspection of the anomalous or irrational samples, with regard to outright variability or systematic lithologic gradation did not explain the anomalous behavior, then other operational variables in the system would need to be examined.

Based on examinations of all of the thin sections in the entire suite of samples studied, there are no other operational variabilities affecting the relationship between the characteristic lithology and the propensity of that lithology to react (and of virtually all those samples that react to expand) in an alkaline solution or in concrete. The characteristic lithology will expand if possible, and those rocks that expand in alkaline solution or in concrete have the characteristic lithology.

Some of the reasons for the initially untidy organization in Figure 19 can be illustrated.

With regard to the sample designated 5-1, initial observation of thin sections connected with the selection of a portion for the prism test indicated no characteristic reactive texture, as shown in Figure 20, and the prisms did not expand. However, the beams and the mortar bars expanded .08% and .05% respectively. Thin sections of the beams that expanded showed the characteristic reactive texture in the coarse aggregate as illustrated in Figure 21. The same material was not in the beams and mortar bars as in the thin sections and prisms. The differences in materials was not due to a mixup of samples or other error or oversight. The anomalous behavior due to gross sample variability of the apparently homogeneous lithology present in Quarry 5 will be discussed in the next section of the report.

Another sample, 11-X, the original thin sections of which did indicate the presence of the characteristic texture, did not expand in rock prisms or mortar bars but did expand in concrete beams. Reexamination of the thin sections indicated an intrinsically nonhomogeneous lithology, with, in many cases, veins of sharply defined reactive type material in an innocuous type matrix (see Figures 22 and 23). The variable behavior in this case can be attributed to interlithologic variability rather than to gross sample variability.

Similarly, sample 12-1 was not classed as petrographically potentially reactive but its rock prism did expand .3%, and its beam .07% percent, and the mortar bar only .02%. Most of the 12-1 thin sections are composed of areas of uniform microspar with a few isolated dolomite rhombs as seen in Figure 24, but there are areas of well developed characteristic reactive textures, such as shown in Figure 25. It must be presumed that the concrete beams have a sufficient amount of the reactive phase to expand.

To conclude the discussion of anomalous samples, it is helpful to examine the behavior of the samples designated 23-9 and 13-1, neither of which was classed grossly as having the characteristic reactive texture, but the first of which (23-9) expanded in concrete beams and mortar bars but not in rock prisms and the second of which (13-1) showed delayed expansion in rock prisms but not in concrete beams or mortar bars. Both of these rocks are richer in dolomite than the typical reactive rock. By chemical analysis the dolomite constitutes over 90% of the carbonate fraction in both. Together they illustrate anomalous behavior once again due to a given lithology not being where it is presumed to have been, but also the influence of composition and texture on a particular kind of behavior as described below.



Figure 20. Texture of aggregate 5-1 representing material in prisms that did not expand. (125x)



Figure 21. Texture of aggregate 5-1 representing material in concrete beams that expanded. (125x)



Figure 22. A vein of reactive texture in aggregate 11-X enclosed by an innocuous texture. (45x)



Figure 23. A close-up of the area shown in Figure 22. (125x)



Figure 24. Usual texture of aggregate 12-1 showing areas of uniform microspar with a few isolated dolomite rhombs. (125x)



Figure 25. An area of well developed reactive texture in aggregate 12-1. (125x)

The rock (23-9) that did not expand in rock prisms is shown in Figure 26. It is a relatively coarsely crystalline subhedral mosaic of dolomite. The aggregate 23-9 that caused the concrete beam to expand .06% is shown in Figure 27. It is finer grained with more euhedral dolomite rhombs in a mixture of fine grained calcite and dolomite. There appears to be little interstitial clay. Numerous areas of the coarse grained mosaic phase intergrade imperceptibly with the other end member; i.e., the isolated rhomb phase. Figures 26 and 27 illustrate only those two end members.

The other rock, 13-1, which expanded in rock prisms but not in mortar bars or concrete beams, is shown in Figures 28 and 29 and is similar to the isolated rhomb phase of 23-9. It has, however, more quartz, silt, and sand and greater porosity and absorption. In this case the same lithology is present in the concrete beams as in the thin sections. This rock is the delayed expander mentioned earlier and its petrography corresponds extremely well to those delayed expanders described by Dolar-Mantuani (1964). It is presumed that the consistency in its behavior, i.e., why it expands in rock prism and not in concrete, is accounted for by the idea mentioned earlier — that when this rock approaches the threshold for expansion in concrete, mortar matrix is stronger and more able to resist expansion than it is in the case of the much more frequently encountered early expanding type of rock.

In retrospect, it appears that this highly dolomitic, more porous, more quartzose, and less argillaceous lithology is a variant of the characteristic reactive rock and should be considered as such.

It is suggested that a possible reason for the delayed expansion is an original deficiency of calcite in the rock. Hadley (1964) has written that one of the products of the dedolomitization reaction is calcium carbonate and has shown that the reaction is maximized and accelerated as the proportion of calcite and dolomite approaches 50/50. In these more highly dolomitic reactive rocks which are slow to expand but which are nevertheless reactive, perhaps the production of calcite (or calcium carbonate) in the early stage of the reaction supplies an amount which increasingly approaches the ideal proportion for ready reaction at a later time.

In summary of these illustrations, petrographic studies of the rocks show that all deviations from consistent behavior with respect to characteristic reactive texture, reactivity, and expansion have been resolved by examination of the samples in question, and that all the deviations relate to variability of some kind.

Variability of the kind exhibited by a nonhomogeneous or mixed lithology, as for instance within a given thin section or suite of adjacent thin sections, can account for the situation where mortar bars, beams, and rock prisms do not agree and can cause a misclassification of the rock as to whether or not the characteristic reactive texture is present. Samples 11-X and 12-1 are of this type. In order to counter the threat of this kind of situation one might use many thin sections concentrated from a relatively small area.



Figure 26. Texture of aggregate 23-9 representing material in prisms that did not expand. (125x)



Figure 27. Texture of aggregate 23-9 representing material in concrete that showed expansion. (125x)



Figure 28. Texture of aggregate 13-1 that showed delayed expansion in prisms but no expansion in mortar bars or concrete.



Figure 29. Close-up of texture of aggregate 13-1. (460x)

Results from Multiple Sampling of Virginia Quarries

Variability of the types illustrated in the petrographic examination is bothersome and obviously must be considered in any evaluation of test methods. Despite this variability, however, experience has demonstrated that quarries can be identified and subsequently confirmed as possessing or not possessing potentially reactive rocks with a degree of certainty sufficient for practical purpose.

During the summer of 1962, 42 Virginia quarries were visited and 226 samples were collected. Within each quarry samples were chosen to represent each clearly identifiable lithology. The least number of samples from a single quarry was 2, and the most was 12. The results of physical, chemical and expansion tests on these samples were presented in an earlier report (Sherwood and Newlon 1964). Subsequently 6 additional quarries were similarly sampled. Based upon the results from these two preliminary samplings, the Virginia Department of Highways in 1964 conducted in-depth surveys of 7 quarries which from the preliminary survey appeared to have potentially reactive material.

Independently but using generally similar criteria for sample selection, the Corps of Engineers in 1963 sampled 5 quarries which were in the original statewide survey, 2 of which had also been included in the detailed sampling by the Virginia Department of Highways. Consideration of the results obtained from these several independent sampling and testing programs provides some insight as to the ability to characterize production from commercial quarries. The quarries involved in these several samplings are shown in Table 12.

Comparing the Virginia statewide survey and that of the Corps of Engineers, both of which were aimed at preliminary screening of sources, it is interesting to observe that each quarry was characterized in each survey by approximately the same number of samples. Apparently the gross lithologies as viewed by the 2 sampling parties were identified in the same way.

For all samples from these 3 gatherings, length change measurements were made on rock prisms or cylinders. In Virginia the criterion used to identify aggregates showing expansions which might be viewed with caution was 0.2% at 16 weeks (112 days). The Corps of Engineers used 0.1% at 84 days (12 weeks). Experience has shown that these 2 criteria are not greatly different with regard to separating specific aggregates. Using these criteria, the portion of the samples from the 3 sample gatherings indicated as expansive are shown in Table 13. (1728

Table 12

Number of Samples Gathered in Multiple Sampling of Quarries

Quarry	Preliminary Statewide Survey Research Council, 1962	Detailed Study, Va. Dept. of Highways, 1964	Corps of Engineers Preliminary, 1963
1	18	72	_
5	2	69	-
11	· 26	47	·
12	5	124	7
13	8	anijen,	7
15	7	Callera	3
21	7		9
27	6	107	
29	5	124	
48	5	31	4

Table 13

Amounts of Potentially Reactive Material as Indicated from Various Samplings

	Virginia Statewide Survey		Virginia Detailed Survey		Corps of Engineers		
Quarry	No. of Samples	Potentially* Reactive, %	No. of Samples	Potentially* Reactive, %	No. of Samples	Potentially** Reactive, %	
1	18	44	72	21			
5	2	50	69	27			
11	6	20	47	9			
12	5	60	124	6	10	53	
13	8	12	(2 3)	فتجته	7	15	
14	7	0	C###2270	Corport	3	0	
15	10	10	-		9	30	
27	6	33	107	4	- 		
29	5	0	124	2		0000	
48	5	20	31	19	5	70	
*	> 0.2%	at 112 days					
**	> 0.1%	at 84 days					

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From the data in Table 13 the indications from the preliminary samplings of the Research Council and the Corps of Engineers show excellent agreement for 3 quarries (12, 13, and 14), fair agreement for one (15), and poor agreement for the other (48). It can also be seen that the more extensive samplings usually indicated the presence of less reactive material than did the preliminary samplings. The reason for this is not clear.

The distribution of rock prism expansions from the detailed surveys are shown in Figures 30 through 36. Also indicated on the figures are the measurements obtained from the earlier statewide surveys and the "average" expansion indicated from each survey. These averages and the corresponding standard deviations are given in Table 14. From these data it can be seen that the 3 quarries (11, 27, and 29) which, based on the detailed survey had comparatively little expansive material, were so characterized by both surveys.

Table 14

Average Expansions,	$\overline{\mathbf{X}}$, and Variability of Expansion from the	
Preliminary and	Detailed Samplings of Seven Quarries	
	(Values in Percent)	

Quarry		Statewide $\overline{\mathbf{x}}$	σ	Detailed $\overline{\mathbf{X}}$	9
1		+.223	0.275	+.087	0.244
5	¢.	+.192		212	0.370
11		+.069	0.148	+.024	0.120
12		+.527	0.728	+.148	0.640
27		+.045	0.124	+.031	0.077
29		002	0.002	005	0.070
48		+.147	0.264	+.293	0.815

Quarry 12, which had a comparatively small volume of reactive material showed a high average expansion because of the presence of several beds of very highly expansive rock. It is also of interest to note that while the averages for a quarry change with the 2 surveys, the variability remains comparatively the same.



Length Change, percent (age - 16 wks.)

Figure 30. Distribution of length changes for prisms from Quarry 1. The bars represent results from the detailed survey (1964) and the numbers in parentheses indicate the number of samples exhibiting the indicated expansion in the preliminary statewide survey (1962).



Length Change, percent (age - 16 wks.)

Figure 31. Distribution of length changes for prisms from Quarry 5 in the same form as shown in Figure 30.



(age - 16 wks.)



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Figure 33. Distribution of length changes for prisms from Quarry 12 in the same form as shown in Figure 30.





Figure 36. Distribution of length changes for prisms from Quarry 48 in the same form as shown in Figure 30.

The problems of sampling and characterizing commercial sources are emphasized by the situations shown in Figures 37, 38, and 39. In Figure 37 are shown 66 rock prisms taken from a continuous core from the face of Quarry 27. Three of these were detrimentally expansive. As is obvious from the photograph, there is no correlation between expansion and appearance. Figures 38 and 39 illustrate the occurrence of expansive material in close proximity to non-expansive material. The expansions at 16 weeks of rock prisms taken from the locations indicated in Figure 38 were as follows:

12-4CC	+0.450
12–5A	-0.059
12 - 5B	+0.159
12-5C	+0.007
12-5CC	-0.055
12-5D	+0.179
12-5E	-0.022

Similarly the prism expansions at 16 weeks for the samples shown in Figure 39 were:

12–3A	+2.96
12–3AA	+0.015
12-3B	-0.007
12-4A	+1.532

;





Figure 38. Face of Quarry 12 illustrating the close proximity of moderately expansive and non-expansive rocks.

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Figure 39. Another face of Quarry 12 illustrating the close proximity of highly expansive and non-expansive rocks.

In many Virginia quarries, the bedding is not so clearly defined as that in Figures 38 and 39. In quarry 1, for example, the identification of beds is greatly complicated by structural features as seen in Figure 40. Such situations emphasize the need to utilize geologists in characterizing and directing the sampling of quarries. Despite the obvious complexities, the general agreement among the several surveys discussed in this section is encouraging.

While agreement among the several surveys is not exact, there does appear to be sufficient concurrence among the results to give a degree of confidence in the ability of the rock cylinder method, properly applied as a screening test, to warn of the occurrence of potentially reactive carbonate rocks in commercial sources.

Relationship of Virginia Results to Other Studies

During the conduct of this project, close contact was maintained with other agencies conducting similar studies of alkali carbonate reactivity.

In some cases, samples were exchanged and comparative or relative tests conducted on materials which have been discussed in this report. Some of these results have been published and these along with other unpublished work offer some confirmations and some extensions of the studies conducted in Virginia.

Mortar Bar Studies

McCoy and Eshenour (1968), in their studies of the significance of alkali solubility on expansion of mortar bars tested in accordance with ASTM Method C 227, utilized aggregate 1-8. Their testing program also included a carbonate sand from South Dakota (Buck 1962) and a well-known siliceous sand from the Republican River in Nebraska that has been intensively studied as an example of alkali silica reactivity. Some results obtained by McCoy and Eshenour are presented in Table 15.

In order to evaluate the applicability of the mortar bar method (ASTM C 227) to the detection of alkali carbonate reactivity, Subcommittee II-b of ASTM Committee C-9 conducted cooperative tests using 5 aggregates from the Virginia research project and 2 supplied by the Waterways Experiment Station of the Corps of Engineers. Four laboratories participated, including the National Research Council of Canada (NRCC), the Portland Cement Association (PCA), the Waterways Experiment Station (WES), and the Virginia Highway Research Council (VHRC).

The 5 aggregates supplied from Virginia were 1-8, 12-9, 27-4, 29-5, and 36-X. The samples were obtained from the crushed material used in the mortar bar studies described earlier. WES furnished 2 samples that had been studied by Buck and Dolch (1966) and found to be reactive in the sense of forming rims. One sample, a fine grained, non-dolomitic limestone from Missouri, was designated KAN-4 G-6 (3). It was selected for examination from a 250-lb. ledge rock obtained in 1964. The second WES sample was from Iowa and was designated OM-16 (G-1) (2). It was likewise selected from a ledge-rock sample, crushed, washed, and sieved to appropriate sizes.

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Figure 40. Folding of beds in Quarry 1, which is typical of many Virginia quarries and adds to the difficulties of sampling and characterization of quarries.

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Table 15

Relationship of Mortar Bar Expansions to Alkali Contents as Determined by McCoy and Eshenour (1968)

	Cement	Na ₂ O Total	Equivalent % Water Soluble	1 month	Mortar Bar I (ASTM C 227 3 months	Expansion,), Percent 6 months	1 year
	/ Aggregate 1-8	}					
	A	0.83	0.48	0.026	0.040	0.054	0.068
	в	0.83	0.32	0.024	0.037	0.049	0.062
	С	0.84	0.14	0.018	0.028	0.034	0.048
	Watertown Sar	nd (South L	Dakota)				
Inou	А	0.83	0.48	0.013	0.022	0.028	0.036
sher	В	0.83	0.32	0.008	0.014	0.015	0.022
й р	С	0.84	0.14	0.002	0.005	0.005	0.010
y an	Aggregate 1-8	5					
ວິວ	С	0.84	0.14	0.018	0.028	0.034	0.04 8
f M	D	0.62	0.19	0.024	0.042	0.052	0.064
k o	E	0.45	0.19	0.020	0.034	0.042	0.056
Wor	Republican Ri	ver					
	С	0.84	0.14	0.060	0.156	0.212	0.306
	D	0.62	0.19	0.031	0.138	0.186	0.204
1	E	0.45	0.19	0.012	0.102	0.175	0.234

	Aggregate 1-	8		
k of RC	High	0.95	0.39	+0.075
/or] VHI	Medium	0.67	0.51	+0.026
5	Low	0.43	0.10	+0.030

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Sufficient cement to conduct the required tests was furnished the participating laboratories by the Research Council from the cement designated high alkali in this report and previously described in Table 2. Additionally, some of the participants used their local cements and/or aggregates for comparative purposes.

The results obtained by the 4 laboratories are summarized in Figure 41 and in Tables 16-20. The values obtained by each laboratory are given in Table 16. In Table 17 are shown the rankings of the several aggregates from the mortar bar tests as well as the Research Council's prism and concrete tests described earlier in this report. Using the criteria shown in the table, it can be seen that after 12 months the 3 definitely expansive aggregates (1-8, 12-9, and 27-4) were separated from the remainder by all laboratories. One laboratory also separated aggregate 36-X. After 6 months the same separations were made except that the results from 1 laboratory (WES) were low based upon the arbitrary criteria established. At 3 months 2 labs (PCA and VHRC) separated the 3 aggregates; 1 lab (NRCC) separated 2, and the results from the third lab were low, although the proper ranking within the lab was obtained. One lab (PCA) separated an additional aggregate (36-X).

Although the orders of ranking of the aggregates differ slightly among the labratories, there is a generally consistent pattern to the results, and the definitely expansive rocks are identified by most participants.

Analyses of the data are given in Tables 18 and 19 to show the repeatability of Method C 227. In this method "repeatability" is considered satisfactory if the difference in the value for percentage expansion of any given specimen in a group from the average value for percentage expansion of the group does not exceed 0.003%, except that if the average expansion exceeds 0.020%, the repeatability is considered satisfactory if the percentage expansion of each specimen molded from the same cement-aggregate combination is within 15% of the average.

When these criteria are applied to the 12-month data in Table 17, the repeatability requirements are met for all samples by 2 labs (PCA and NRCC). The other 2 labs (VHRC and WES) exceed the requirements in 2 cases. At 3 and 6 months 1 lab (PCA) meets the repeatability for all cases. The remaining labs exceed the limits in from 1 to 4 cases, although the limits are usually only slightly exceeded. The most difficulty is an early age and/or low expansion. The repeatability at 6 months is slightly better than at other ages.

The ranges of expansions from the 4 laboratories are given in Table 20, along with similar data from an earlier interlaboratory program using reactive siliceous rocks. The repeatability of C 227 appears to be about the same for both types of aggregate.

Although the repeatability of the test results within a laboratory was within tolerable limits, significant differences among laboratories exist as shown in Table 16. The low expansions recorded at early ages by 1 lab (WES) were corrected by adjustments made to obtain a higher humidity in the containers. A likely cause of the other variations is from loss of moisture from containers, probably during the 24-hour temperature conditioning prior to measuring. VHRC relied upon the water tightness of the commercial containers to maintain moisture whereas NRCC used tape, and the VHRC results are generally below average while those of the NRCC are above. The VHRC specimen containers were exposed in a water bath that maintained a very high humidity during storage, so the moisture loss was minimized during this portion of the testing.

Figure 41. Results from cooperative tests by four laboratories of C 227 mortar

bars containing carbonate aggregates.



rength Change, percent

16	
Table	

AGGREGATE				LABORA	VTORY			
	д 	CA	M	HRC	NR(0C**	M	ES
	Average	Maximum Deviation	Average	Maximum Deviation	Average	Maximum Deviation	Average	Maximum Deviation
		цГ	ee Months					
1- 8	+0•056	. 001	+0,046	• 6• 4	+0.026	• 006***	+0.018	. 003
12-9	+0.056	.001	+0.035	. (1)3	+0, 022	.003	+0.009	***900"
27-4	+0.049	.003	+0.040	.002	+0.032	.004	+0.016	***200 *
29-5	+0.023	.001	+0.015	.003	+0.001	• 004***	-0.002	***600*
36 - X	+0.028	.002	+0.014	.003	+0.021	• 005***	+0.006	• 000***
Kan 4	+0.014	.001	+0.014	• 004***			+0,006	.002
OM -1 6	+0,008	. 003	+0.018	.002			-0.006	• 006***
		Six	Months					
1-8	+0.072	.001	+0,069	• 006	+0,053	• 004	+0.040	. 003
12-9	+0.081	.002	+0.059	.004	+0,067	.005	+0.027	.004
27-4	+0.075	.001	+0,071	.007	+0.082	. 008	+0.039	.004
29-5	+0.035	.002	+0.027	.002	+0.024	***200°	+0.014	***900"
36 - X	+0.046	• 003	+0.026	•000***	+0.074	.004	+0.027	.001
Kan 4			+0.027	.001	+0.019	.003	+0.012	.001
OM-16	+0,016	• 003	+0.038	.002	+0.023	.001	+0,005	• 006***
		эмТ	lve Months					
1-8	+0,087	.001	+0,083	. 008	+0.074	. 003	+0*04	. 005
12-9	+0.111	.004	+0.074	. 006	+0.120	.004	+0.085	. 006
27-4	+0.100	.004	+0.091	.005	+0.139	.013	+0.089	. 006
29-5	+0.040	.001	+0.034	. 006	+0.050	.010	+0.040	.008
36-X	+0.055	.005	+0.030	***800.	+0.129	.003	+0,069	.005
Kan 4	+0.019	.001	+0.026	.001	+0.020	.001	+0.018	.001
OM-16	+0.022	.002	+0.042	***:00°	+0.026	.001	+0.016	• 000*

** Average of 3 bars from same batch *** Exceeds the repeatability limits of ASTM C 227-63T

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Table 17

Relative Ranking of Aggregates by C 227, Concrete, and Rock Prism Tests in Descending Order of Expansion Within Each Column

		12 mo.	27-4	12-9	1-8				36-X	29-5	Kan 4	0M-16			
	WES	6 mo.							1-8	27-4	(12-9	(36-X	29~5	Kan 4	0M-16
		3 mo.							1-8	27-4	12-9	(36-X	Kan 4	29-5	
		12 mo.	27-4	36-X	12-9	1-8			29-5	0M-16	Kan 4			·	
C 227	NRCC	6 mo.	27-4	36-X	12-9	1-8			295	0M-16	Kan 4				
		3 mo.	27-4	1-8					12-9	36-X	29-5				
	VHRC	12 mo.	27-4	1-8	12-9				0M-16	29-5	36X	Kan 4			-
		6 mo.	27-4	1-8	12-9				0M-16	(29-5	Kan 4	36X			
		3 mo.	1-8***	27-4	12-9				29~5	36-X	Kan 4				
		12 mo.	12-9	27-4	1-8				36-X	29-5	0M-16	Kan 4			
	PCA	6 mo.	12-9	27-4	1-8				36-X	29-5	0M-16				_
		3 mo.	(12-9***	1-8***	27-4***	36-X			29-5	Kan 4					
	*	12 mo.	1-8	12-9	27-4				29-5	36 -X					
	ONCRETH	6 mo.	1-8	12-9	27-4				29-5	36-X					
	Ŭ	3 mo.	1-8	12-9	29-5	27-4			36-X						
		12 mo.	12-9	1-8	27-4				29-5	36-X					
	PRISM**	6 mo.	12-9	1-8	27-4				295	36-X					
		3 mo.	12-9	۴ 1	27.4				29-5	36-X	Kan 4	0M-16			
	CATEGORY*		Expansive				and the second se	Non-	Expansive						

^{*} Using as criteria 3 mo. 0.025 0.2
 ⁶ mo. 0.050 0.4
 ¹² mo. 0.075 0.6
 ^{*} Tests of VHRC except rim forming. which were studied by

Concrete

Prism

Mortar Bars

Expansion, %

0.015 0.03 0.05

** Tests of VHRC except rim forming, which were studied by WES

*** Rejection on basis of ASTM C 33 = Same Value

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Table	

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AGGREGATE	CEMENT (Alkali Na _o O Equiv.)	LABORATORY	3 MONTHS	6 MONTHS	12 MONTHS
	1				
1-8	B (1.19)	NRCC	+0,036	+0, 063	+0*036
	Med. (.67)	VHRC	+0.015	+0, 026	+0.047
	Low. (•43)	VHRC	+0.021	+0.030	+0,037
2-9	B (1.19)	NRCC	+0.032	+0, 080	+0.145
	E (0.45)	NRCC	-0.013	+0.010	I
	Med. (.67)	VHRC	+0° 004	0	+0.032
	Low (•43)	VHRC	+0.006	+0.017	+0.022
7-4	B (1.19)	NRCC	+0•047	+0.110	+0.179
	Med. (.67)	VHRC	+0.011	+0.025	+0* 067
	Low (.43)	VHRC	+0.025	+0.028	+0.029
9-5	B (1.19)	NRCC	+0.020	+0,052	+0,087
	Med. (.67)	VHRC	-0.014	-0.012	+0.002
	Low (•43)	VHRC	+0,007	-0.002	+0.001
6-X	B (1.19)	NRCC	+0.028	+0,095	+0.151
	Med. (.67)	VHRC	-0-006	-0,003	+0.012
	Low (.43)	VHRC	+0.006	+0.013	+0.016
*0-	B (1.19)	NRCC	+0.027	+0,047	+0,060
	E (0.45)	NRCC	+0.012	+0.028	+0.038
tR**	B (1.19).	NRCC	+0,091	+0.123	ł
	B (0.45)	NRCC	-0.001	+0.021	ł
) M-1 6	Low (0.38)	WES	-0.016	-0, 003	+0,007
an 4	Low (0.38)	WES	-0,007	+0,002	+0.009

** Republican River Sand

* Composite of top 24 feet of Pittsburg Quarry at Kingston

Table 19

Statistical Analysis of Within Laboratory Mortar Bar Data on Reactivity of Carbonate Rocks

				Fynanci	on of		
				Tamden	<i>N N</i>		
	ł		3 Months			6 Months	
Aggregate	Laboratory	Average	Range	Coefficient of Variation	Average	Range	Coefficient of Variation
1-8	PCA VHRC NRCC WES	.056 .047 .026 .018	. 001 . 005 . 011	1 4 4 11 11	.072 .069 .053	. 001 . 009 . 006 . 006	0 0 Q I I
12-9	PCA VHRC NRCC WES	.058 .034 .022 .009	. 011 . 005 . 005 . 008	8 11 38	. 081 . 058 . 067	.004 .010 .008 .006	2 6 10
27-4	PCA VHRC NRCC WES	. 048 . 040 . 032 . 016	• 010 • 004 • 014	8 20 40	. 075 . 071 . 082 . 039	.001 .009 .013	1 5 11
29-5	PCA VHRC NRCC WES	.023 .014 .001	• 001 • 004 • 007 • 008	2 11 150	. 034 . 027 . 024 . 014	.002 .003 .012 .009	3 4 21 27
36-X	PCA VHRC NRCC WES	.028 .014 .021 .006	. 003 . 005 . 009 . 008	4 14 56	.046 .026 .074	.005 .011 .008 .001	4 18 5

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	uis Report (4 laboratories)	n at Indicated Ages	6 Months	0.044	0.054	0.043	0.020	0.048	
No. of the second	ock Studies Described in t	Ranges of Expansio	3 Months	.038	.049	.032	.022	.022	
	Results from Carbonate R	Aggregate		1-8	12-9	27-4	29-5	36-X	

(b) Results from Prior Studies by ASTM Committee C-1 for Silicate Rocks (10 laboratories)*

Age, weeks	16	16	16	œ	
Range of Expansion	.01	. 04	.02	. 03	
Cement	4	5	6	4	
Aggregate	ъ	5	ນ	4	

*Private communication: Paul Seligmann to W. C. Hansen, November 12. 1965. Based upon data from C-1 Cooperative Tests reported in <u>ASTM Bulletin</u>, October, 1946.

Interlaboratory Ranges of Mortar Bar Expansion Tests of Carbonate and Silicate Rocks

(a)

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Table 20

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As seen from Table 17, based upon 3-month data, PCA would have rejected 3 samples and VHRC 1 under existing criteria established for alkali-silica reactivity. Based upon 6 month data, no rejections would have resulted. It should be noted that expansions are related to alkali content and at 6 months rejection of 1 sample (27-4) would have resulted from use of NRCC Cement B (Na₂O equivalent -1.19%).

These results confirm the Research Council's findings, reported earlier, that use of ASTM C 227 for the detection of alkali-carbonate reactivity would require that limits be approximately one-half of those used for alkali-silica reaction. The following limits would have resulted in separation by ASTM C 227 of expansive from non-expansive aggregates:

Age, months	Expansion, $\%$
3	0.025
6	0.050
12	0.075

In addition to the data gathered on the test method, the data in Table 20 show that the test aggregates from Virginia generally show higher expansions than the Kingston reference aggregate but lower expansion than the siliceous Republican River reference aggregate in tests by NRCC. The fact that these Virginia aggregates were potentially more expansive than the Kingston rock has also been demonstrated by Hilton (1968). The poor field performance of the Kingston aggregate is well documented (Swenson and Gillott 1964). Poor field performance of concrete containing aggregate 1–8 has also been conclusively documented (Ozol and Newlon 1971) but the influence of the aggregate is not as spectacular as that observed with the Kingston aggregate. The apparent difference may be related to the fact that the alkali contents of the cements used in Virginia are substantially lower than those in the Kingston area.

Prism Studies

During the Corps of Engineers' evaluation of rocks from the quarries described in the discussion accompanying Tables 12 and 13, prisms of rock taken from Quarry 12 were sent to the Research Council for comparative measurements. The prisms were taken from vertically adjoining positions and perpendicular to the bedding. The details of sampling and complete data are included in a published report (Buck 1969). Comparative measurements are given in Table 21. Eleven of the 16 prisms indicated a very high degree of expansion. Two more showed excessive expansion. Only 3 showed acceptable behavior. The characterization of samples A and B was the same by both labs. While different results were obtained by each, both the sets of results for samples C and D would indicate the presence of aggregates demanding caution and would suggest further testing. Data from tests of rock from quarry 12, presented earlier in Tables 12-14, would substantiate these indications.

The lack of agreement for samples C and D was attributed by Buck to unexpected variation in the rock between 2 supposedly identical specimens. This situation has been extensively discussed previously.

Table 21

<u>Prism</u>	1 week	4 weeks	12 weeks	6 months	<u>1 year</u>
A-1 (WES)	-0.01	0.61	1.82	2.18	2.25
A-2 (WES)	0.22	+			
A-1 (VHRC)	0.01	0.69	+		
A-2 (VHRC)	0.00	0.72	3.16	3.77	3.88
B-1 (WES)	-0.01	-0.01	0.06	0.60	1.74
B-2 (WES)	-0.01	0.04	1.68	2.09	+
B-1 (VHRC)	0.00	0.10	1.64	2.96	3.33
B-2 (VHRC)	0.00	0.06	1.31	2.71	3.15
C-1 (WES)	-0.02	0.00	0,02	0.14	0.38
C-2 (WES)	-0.01	-0.03	-0.02	0.05	0.13
C-1 (VHRC)	-0.01	-0.04	0.02	0.78	1.27
C-2 (VHRC)	-0.02	-0.04	0.01	0.80	1.20
D-1 (WES)	-0.01	0.00	0.01	0.00	-0.02
D-2 (WES)	0.05	0.27	0.45	0.48	0.64
D-1 (VHRC)	0.03	0.10	0.18	0.26	2.55
D-2 (VHRC)	-0.01	-0.02	-0.03	-0.04	-0.05
and the second					

Length Change Data from Comparative Measurements of Expansion Made on Rock Prisms by the Corps of Engineers and the Research Council (Values in Percent)

+ Readings discontinued because specimen broke.

Similar results were obtained for samples measured by the Research Council and the National Crushed Stone Association on samples exchanged in 1967^{*}. The tests utilized 3 rock samples. The prisms were from the same sample, but not from adjacent locations, as was the case with the 27 prisms discussed in Table 21. The results are summarized in Table 22.

Again, since the rocks were all highly expansive, the differences are magnified and there is no way to judge the ability of the method to separate expansive from nonexpansive rocks. The results from NSCA are lower for the samples MO and 12-9-8. This may be because of the strong bedding apparent in these rocks. It has been clearly shown by many investigators that expansion is greatest in the direction perpendicular to bedding. Averaging the 3 results as specified in C 586 and as reflected in the NSCA data would mean that 2 of the 3 results would be lower than the maximum, which is that reflected by the VHRC data. By any criteria, however, both laboratories would have classified the rocks as expansive.

^{*}F. A. Renniger, March 14, 1969: Private communication.

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Table 22

Sample	4 w	veeks	16 weeks		
	VHRC*	NCSA**	VHRC*	NCSA**	
MO	5.73	0.75	7.43	1.17	
12-9B	1.80	0.47	6.06	2.07	
1-8	0.64	0.83	1.42	1.64	

Length Change Data	from Comparative Measurements Made on Rock Prisms
by the National	Crushed Stone Association and the Research Council
	(Values in Percent)

* Average of 3 parallel prisms cut perpendicular to bedding.

** Average of 3 mutually perpendicular prisms.

The ability of 2 laboratories to separate expansive from non-expansive rocks is indicated by the data in Table 23. During the early stages of this project, the Research Council exchanged samples with the Portland Cement Association, where the prism method was being studied by its developer, D. W. Hadley. The results of measurements made on rocks from 10 lithologies within Quarry 1 are given in Table 23. Each value is the average of 2 companion prisms from the same rock sample but not necessarily from the same piece.

Table 23

Length Change Data from Cooperative Measurements of Prisms by the Portland Cement Association and the Research Council (Values in Percent)

Sample	PCA	VHRC
1-3	+0.16	+0.22
1-4	+0.06	+0.08
1-5	+0.08	-0.04
1-6	+0.04	+0.02
1-7	-0.01	-0.02
1-8	+0.30	+0.54
1-9	+0.12	+0.08
1-10	-0.02	-0.02
1-11	-0.02	-0.01
1-12	+0.16	+0.07

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The agreement is seen to be quite good in that the high, moderate, and non-expanding rocks are consistently delineated by both laboratories.

The behavior of aggregates obtained from Virginia has been discussed by Gillett and Swenson (1970) but direct comparisons are not possible.

CONCLUSIONS

- The expansion after 5 years of continuous moist storage of the concretes made with high alkali cements and containing the various aggregates ranged from 0.01% to 0.27%. Expansions of the apparently nonreactive aggregates are generally higher than values that have been reported in the literature. But an expansion value of 0.05% appears to separate the aggregates that evidence detrimental expansion in other test methods from those that do not.
- 2. The expansion of the rocks during testing in accordance with ASTM C 586 in general reflected their behavior in concrete. While the method is essentially a "screening method", when used in conjunction with other procedures, such as petrographic examination, it can be used for providing a rapid quantitative evaluation of potential for expansion of the aggregate in concrete.
- 3. Correlation of the prism expansions at various ages with 5 year expansions of concrete made with high alkali cement resulted in correlation coefficients between 0.87 and 0.95. Slightly higher correlations were found for 4- and 8-week prism expansions than for those at either 1 week or later ages. This appears to be related to the role of restraint in concrete and the time at which expansion begins.
- 4. For concrete made with the medium alkali cement, the correlation between 5-year expansions of concrete and expansions or prisms at various ages, improved with increasing age of prism. The correlation coefficients were less than those for the concretes with high alkali cement. Very low correlation was obtained for the low alkali cements, which is indicative of limited expansive reaction.
- 5. The correlation between concrete expansions and prism expansions reduces as fewer prisms are used for the average. There appears to be a slight decline in predictive power where less than 5 prisms are used; but considering the fact that the variation of expansions within a given lithology may be as large or larger than those between lithologies, the use of only 1 or 2 prisms per lithology provides an adequate indicator of the potential behavior of the rock in concrete and permits the sampling of more lithologies.
- 6. The value of 0.2% at 16 weeks in C 586, which has been used in Virginia to indicate potentially detrimentally reactive rocks, appears reasonable. Considering the variability of the method and the shape of the expansion vs. the curve, the value of 0.2% at 16 weeks is not too different from the value of 0.1% at 84 days suggested for use in C 33. Either value seems reasonable, but the more tolerant (.2% at 16 weeks) appears sufficient.

- 7. The results from mortar bar tests (ASTM C 227) are indicative of expansion in concrete. The expansions are of the same order as those in concrete. Values necessary to separate detrimentally expansive from innocuous aggregates are about one-half of those used when the method is applied to alkali-silica reaction. In view of the low expansions and high variability, C 227 appears to be of limited value in testing for alkali-carbonate reaction.
- 8. The characteristic reactive lithology described in ASTM C 294, whenever and wherever it occurs in a dolomitic limestone, will undergo reaction or dedolomitization in an alkaline environment. Whether the reaction will ultimately lead to expansion of a rock prism, concrete beam, or mortar bar, insofar as the rock is concerned, depends upon the perfection of the type lithology, including such factors as the calcite/dolomite ratio and the grain size, and on the amount and distribution of the reactive texture within the rock. The factors governing expansion in concrete are related to the properties of the particular concrete in question; e.g. water cement ratio, total water soluble alkalies, relative amounts of paste and aggregates, etc.
- 9. In sampling of ledge rock, as in quarries, for alkali-carbonate reactive material should take cognizance of the evidence that the characteristic reactive texture and composition are only subtly and indirectly related to the primary factors of sedimentation. The texture is a secondary or replacement fabric which may or may not conform faithfully and uniformly to a given lithologic or stratigraphic unit as defined macroscopically in the field. While this lithology is not completely random in distribution, its presence or absence cannot be predicted with certainty in a given sequence of dolomitic limestones. Sampling for the reactive lithology and delineation of it should therefore be guided by petrographic considerations, using as many thin or polished sections as necessary to select samples for further tests.
- 10. Because of the variability that may exist, any thin or polished section that shows even a minor area of characteristic reactive texture should be classified as reactive and the field sampling unit which it has been taken to represent should be considered suspect.
- 11. While a total specification and acceptance program will vary with local conditions, and include all testing consistent with time and funds, the petrographic examination, while qualitative, appears to be the most satisfactory method since it is cheap, fast, and sufficiently predictive of the behavior of the aggregates in concrete. If one needs a quantitative measurement from a direct test, the fabrication and testing of concrete beams is the surest method, but the prism test will give a rapid and satisfactory prediction of the concrete expansion at an early age. The choice between the petrographic examination and prism test depends upon the equipment and/or personnel available to the evaluating agency.

ACKNOWLEDGEMENTS

Because of the length of time spanned by this research, it benefited from the contributions of more than the usual number of people to whom appreciation is expressed without naming them individually. Special mention, however, must be made of the work of Marvin H. Hilton, Highway Research Engineer, and Mrs. Holly Walker, Highway Research Analyst, for the portions of the project which each conducted as indicated in the report.

While not specifically a part of this research project, the data obtained during the sampling of several quarries by the Materials Division of the Virginia Department of Highways greatly increased the usefulness of the project. Appreciation is expressed to those who organized and conducted the quarry sampling program. Appreciation is also expressed to the National Crushed Stone Association, the National Research Council of Canada, the Portland Cement Association and the Waterways Experiment Station for their cooperation in exchanging specimens for comparative testing and permitting the use of their data. Individuals from these agencies were most helpful in discussions of the results at various times during the conduct of the work.

Without the continuing support of Clyde E. Giannini, Materials Technician who supervised the preparation of the concrete specimens and rock prisms as well as the thousands of measurements, this project would not have been possible. Celik Ozyildirim, Graduate Assistant, organized and programmed the analyses of the voluminous length change data.

The Council's clerical personnel and Report Section did their usual competent job in preparation, review and reproduction of this report.

The project began during the administration of the late Tilton E. Shelburne and has continued under the general direction of Jack H. Dillard.

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APPENDIX

Table	A-1
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Current Study	Cal./Dol.	Insoluble Residue	Absorption
1-8A	25/75	22	
В	30/70	25	
С	32/68	20	
D	40/60	28	
E	33/67	32	
F	32/68	35	
Average	32/68	27	
Statewide Survey	37/63	28	0.3
1-Xa	97/3	16	
В	73/27	26	
С	49/51	8	
D	41/59	8	
E	51/49	28	
F	98/2	17	
Average	68/31	17	
Statewide Survey	97/3 to 41/59	8-17	0.2
5 -1 A	80/20	20	
В	75/25	18	
С	72/28	23	
D	85/15	22	
E	80/20	21	
F	79/21	18	
Average	78/22	20	
Statewide Survey	81/18	22	0.3
6-2A	0/100	10.0	
В	0/100	10.6	
С	0/100	10.7	
D	0/100	17.6	
E	0/100	19.6	
F	0/100	8.1	
Average	0/100	13	
Statewide Survey	8/92	10	0.2

Compositional and Absorption Data for Rocks Used

1**58** Table À-1 (cont.)

Current Study	Cal./Dol.	Insoluble Residue	Absorption
7-5A B C D E F Average	$16/84 \\ 15/85 \\ 14/86 \\ 13/87 \\ 12/88 \\ 13/87 \\ 14/86$	6.8 17.3 7.1 6.0 5.6 6.9 8	
Statewide Survey	21/79	15	0.2
11-xA B C D E F Average	86/16 67/33 66/34 82/18 77/23 75/25 76/25	$ \begin{array}{r} 6.1\\ 3.2\\ 6.2\\ 11.2\\ 7.9\\ 8.4\\ 7 \end{array} $	
Statewide Survey	57/43 to 100/0	5 to 11	0.1
12-1A B C E F Average	100/0 89/11 100/0 78/22 70/30 87/13	5.1 6.7 7.3 9.1 12.2 8	
Statewide Survey	56/36	8	0.1
12-9A B D F Average Statewide Survey	$ \begin{array}{r} 49/51 \\ 65/35 \\ 54/46 \\ 62/38 \\ 58/42 \\ 73/27 \\ \end{array} $	26.0 27.6 25.4 24.3 21 12	0.4
13-1A C D E F Average	0/100 0/100 0/100 0/100 9/91 1/99	$ 34.2 \\ 30.8 \\ 35.1 \\ 35.0 \\ 31.0 \\ 33 $	
Statewide Survey	9/91	26	1.3
15-9A B C D E F Average	81/19 80/20 77/23 92/8 77/23 63/37 78/22	$2.7 \\ 5.6 \\ 3.6 \\ 3.7 \\ 4.8 \\ 4.6 \\ 4$	
Statewide Survey	85/15	30	0.4

Table A-1 (cont.)

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Current Study	Cal./Dol.	Insoluble Residue	Absorption
21-2C	90/10 100/0	9.1	
Б F	100/0	2. I 6. 2	
Average	97/0	6	
Statewide Survey	69/31	18	0.3
23-9-B	0/100	15.4	
F	0/100	15.7	
Average	0/100	16	
Statewide Survey	0/100	15	0
24-7A	0/100	9.2	
В	0/100	8.2	
D	0/100	8.0	
E	0/100	8.4	
F	0/100	15.5	
Average	0/100	10	
Statewide Survey	0/100	3	0.2
27 - 4A	25/75	44.9	
В	32/68	44.1	
C	81/19	21.9	
D	25/75	44.5	
E	60/40	38.5	
Average	45/55	39	
Statewide Survey	95/5	10	0.3
27-6A	23/77	29.8	
D	30/70	28.9	
E	0/100	20.4	
F	48/52	26.5	
Average	25/75	26	
Statewide Survey	33/67	22	0.2
29-4B	38/62	32.2	
C	51/49	22.7	
D	57/43	23.7	
E	60/40	22.5	
F	70/30	19.4	
Average	55/45	25	
Statewide Survey	91/19	9	0.1
34-7A	83/17	28.3	
В	100/0	5.8	
C	100/0	5.6	
D	61/39	7.5	
E	79/21	5.8	
F	55/45	7.4	
Average	80/20	10	
Statewide Survey	47/53	20	0.2

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Table A-1 (cont.)* 1753

Current Study	Cal./Dol.	Insoluble Residue	Absorption
35-5B	79/21	11.4	
Statewide Survey	77/23	10	0.1
36-XA	90/10	5.0	
В	85/15	5.0	
С	100/0	6.6	
D	100/0	5.8	
E	85/15	8.2	
F	100/0	6.6	
Average	93/7	6	
Statewide Survey	85/15 to 91/9	8 to 10	0.1
48-4A	100/0	19.7	
В	100/0	19.6	
С	100/0	18.1	
D	100/0	21.4	
E	100/0	21.6	
F	100/0	20.3	
Average	100/0	20	
Statewide Survey	38/62	20	0.4
48-5C	100/0	11.0	
E	100/0	6.9	
F	100/0	6.2	
Average	100/0	8	
Statewide Survey	94/6	8	0.3