

# HIGHWAY RESEARCH RECORD

**Number 124**

Aggregates  
for  
Concrete

4 Reports

Subject Classification

32 Cement and Concrete

35 Mineral Aggregates

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## Foreword

The large volume of concrete placed annually for highway pavements and structures places a burden on the materials engineer to insure that suitable aggregates are employed to provide enduring construction. Finishability and placing qualities of the fresh concrete can normally be achieved by proper choice of concrete mixture proportions and grading of the contained aggregates. However, geologic history provided certain basic shortcomings of the aggregates such as presence of undesirable particle types causing disruptive volume changes. Special processing or beneficiation to alleviate such shortcomings may be expensive and require thorough justification before being undertaken.

The papers contained herein seek to provide deeper insight into some of the problems perplexing materials engineers in the selection of concrete aggregates relative to these less easily controlled features.

Buth, et al., report results of strength tests, drying shrinkage, and elastic modulus on a series of laboratory made concretes, the main variable being the amount and type of contaminant passing the No. 200 sieve. Concretes containing siliceous and limestone coarse aggregates were employed yielding not entirely concordant results between the two. Substitution of the "sand equivalent test" for the "Texas loss by decantation test", is favored since the former enables estimate of both the quantity and quality of the contaminant whereas the decantation test evaluates only the total quantity of fines.

Chamberlin studied properties of 25 natural sands and the characteristics of mortars made with these sands. Interrelationships were developed between mortar drying shrinkage, clay content of the sand, sand equivalent, elastic modulus of the mortar, and magnesium sulfate soundness loss. Despite a large range of magnesium sulfate soundness losses provided by the sands, freeze-thaw results in air-entrained mortar were uniformly satisfactory. In other relationships the sulfate test provided a useful index to overall quality. The author thus questions past interpretations of the sulfate test and speculates, despite its relative success, that a less troublesome procedure might be developed around elastic modulus determinations of mortar containing the test sand, compared with similar mortar containing Ottawa sand.

The DePuy paper represents another step in the continuing effort to sort out the many facets of the problem of detrimental concrete expansions displaying the alkali-silica or cement-aggregate reaction. Reactive aggregates were studied in the laboratory using both sealed moist storage techniques and the Conrow cycle. Removal of the alkali-silica reactive particles from the aggregates reduced expansions but significant expansions remained when using the Conrow cycle.

The Ingham and Dunikowska-Koniuszy paper represents an effort to classify chert to facilitate selecting aggregate for concrete and bituminous mixtures. Seven varieties of chert were established which could be recognized visually by color and texture. Chert nodules occurring in limestone ledges as well as discrete particles in gravels were catalogued relative to geographical distribution. Measurements such as specific gravity, porosity, and unconfined freezing and thawing, were made on the various chert types.

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# Correlation of Concrete Properties With Tests for Clay Content of Aggregate

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•THE PRESENCE of clays in the fine aggregate used to make concrete is detrimental to the structural properties of the concrete. The Texas Highway Department currently uses the results of sand equivalent and loss by decantation tests as a means of detecting such clay and controlling the quality of fine aggregate used in portland cement concrete. Article 421 of the Texas Highway Department specifications states:

The loss by decantation of fine aggregate, including mineral filler when used (Test Method Tex-406-A), shall not exceed 2.5 percent. As an alternate to this, the fine aggregate may be used if, when subjected to the Sand Equivalent test, (Test Method Tex-203-F), the sand equivalent is equal to or higher than 80.

These quality control tests were developed independently, and the relationship between the numerical results of each test was not known. Since the two tests form apparently independent bases for accepting or rejecting a material, the relationship between them is very important.

The sand equivalent test separates the finer clay particles from the coarser particles and compares them on a volume basis, which magnifies the volume of the clay in proportion to its affinity for water. (This affinity for water is referred to hereafter as the activity of the clay fraction.) This magnification of the clay volume is not accomplished by the loss by decantation test, and consequently the relationship between the two is nonlinear. The liquid limit (AASHO T80-60) of the clay fraction was chosen as the parameter to indicate activity.

Information is presented in this paper which will aid engineers in establishing limits for the quantity and activity of minus 200 mesh material allowed in concrete aggregate.

## NOMENCLATURE

SE = sand equivalent value;

LD = loss by decantation given as a percentage;

P = decimal fraction of minus 200 mesh material in samples of sand;

A = sand reading in inches in sand equivalent test;

K = ratio of clay reading minus sand reading and sand reading in sand equivalent test;

$C = \frac{K}{P}$ ;

LL = liquid limit of minus 200 mesh fraction; and

$K_1$  = adherence factor, the ratio in percent between fraction decanted in loss by decantation test and fraction of minus 200 mesh material actually present in aggregate.

# STUDY OF LOSS BY DECANTATION AND SAND EQUIVALENT TESTS

## Testing Program

The sand equivalent test was performed in accordance with the Texas Highway Department Test Method Tex-203-F, which is a modification of the California Test Method No. 217-C (AASHTO T176-56). The loss by decantation test procedure used was in accordance with the Texas Highway Department Test Method Tex-406-A.

The sand equivalent test was developed by Hveem (4) as a rapid means of quality control of fine aggregate for bases, subbases, bituminous mixtures and portland cement concrete. The procedure developed by Hveem did not require that the samples be oven-dried before testing, and consequently results could be produced within 40 min.

The sand equivalent test method uses a calcium chloride solution to separate the clay and sand fraction. A cylinder graduated in tenths of inches is filled to the 4-in. mark with the calcium chloride solution. An oven-dried sand sample is then poured into the cylinder. Air bubbles are removed and the sample is allowed to soak for 10 min. After the soaking period, the cylinder is held horizontally and shaken vigorously by throwing the contents from end to end. The cylinder should complete 90 cycles in approximately 30 sec with a 9-in. throw. Following this operation, an agitator tube is used to flush the fine clay-like material into suspension above the coarse sand particles. The graduated cylinder and its contents are allowed to stand for 20 min. The heights of the sand

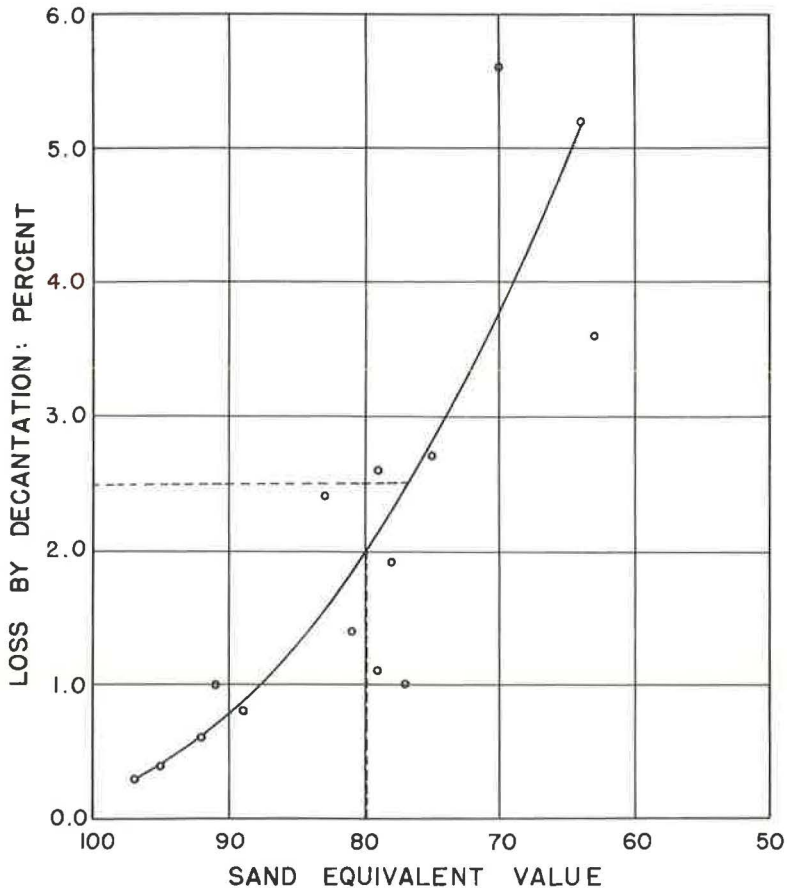


Figure 1. Relationship between loss by decantation and sand equivalent values for natural aggregate samples.

TABLE 1  
MEASURED AND CALCULATED DATA FOR  
AGGREGATE SAMPLES

Sample No.	Loss by Decantation	Measured Sand Equiv. Value	Liquid Limit	Calculated Sand Equiv. Value
1	1.4	81	29.7	92
2	2.6	79	33.1	86
3	5.2	64	36.2	73
4	3.6	63	30.1	82
5	1.9	78	27.8	90
6	2.7	75	30.5	86
7	5.6	70	36.2	72
8	2.4	83	25.8	89
9	1.0	77	33.6	94
10	0.6	92	24.2	97
11	0.8	89	— <sup>a</sup>	—
12	0.4	95	— <sup>a</sup>	—
13	0.3	97	— <sup>a</sup>	—
14	1.0	91	— <sup>a</sup>	—
15	1.1	79	— <sup>a</sup>	—
101	2.2	94	0.0	96
102	2.3	87	34.0	87
103	2.1	61	200	61
104	2.4	41	400	42
105	2.3	32	640	32
106	4.4	89	0.0	93
107	4.3	81	34.0	78
108	4.5	38	200	43
109	4.2	25	400	29
110	4.1	23	640	21

<sup>a</sup>There was not enough minus No. 200 mesh material in this sample for a liquid limit determination.

TABLE 2  
MANUFACTURED SAMPLES

Sample No.	Description
101	Washed sand with 2½ percent silica flour
102	Washed sand with 2½ percent natural clay (LL = 34%)
103	Washed sand with 2½ percent silica-montmorillonite (LL = 200%)
104	Washed sand with 2½ percent silica-montmorillonite (LL = 400%)
105	Washed sand with 2½ percent montmorillonite (LL = 640%)
106 to 110	Same as 101 through 105 but with 5 percent contaminant

and of the clay are then read and the sand equivalent value is calculated by the following formula:

$$SE = \frac{\text{sand reading}}{\text{clay reading}} \times 100$$

The loss by decantation test can be used for coarse as well as fine aggregates. The sample, no dryer than saturated surface dry (SSD), is placed in the pycnometer, and the pycnometer filled with water. After

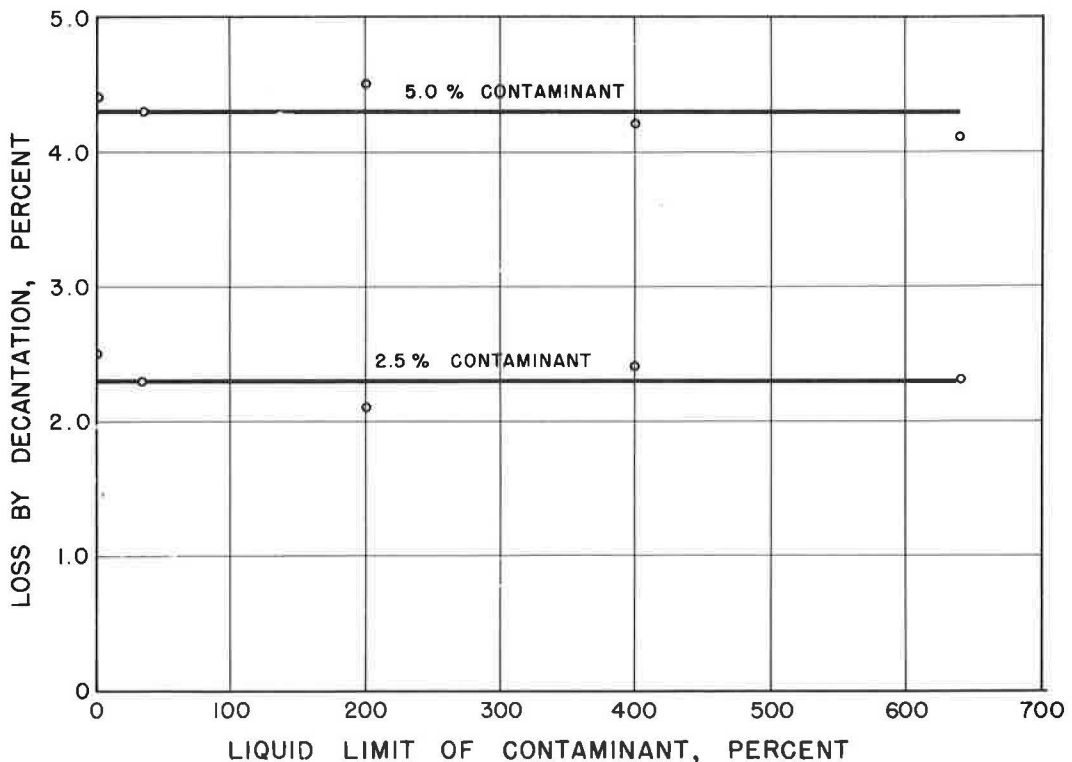


Figure 2. Relationship between loss by decantation and liquid limit of contaminant.

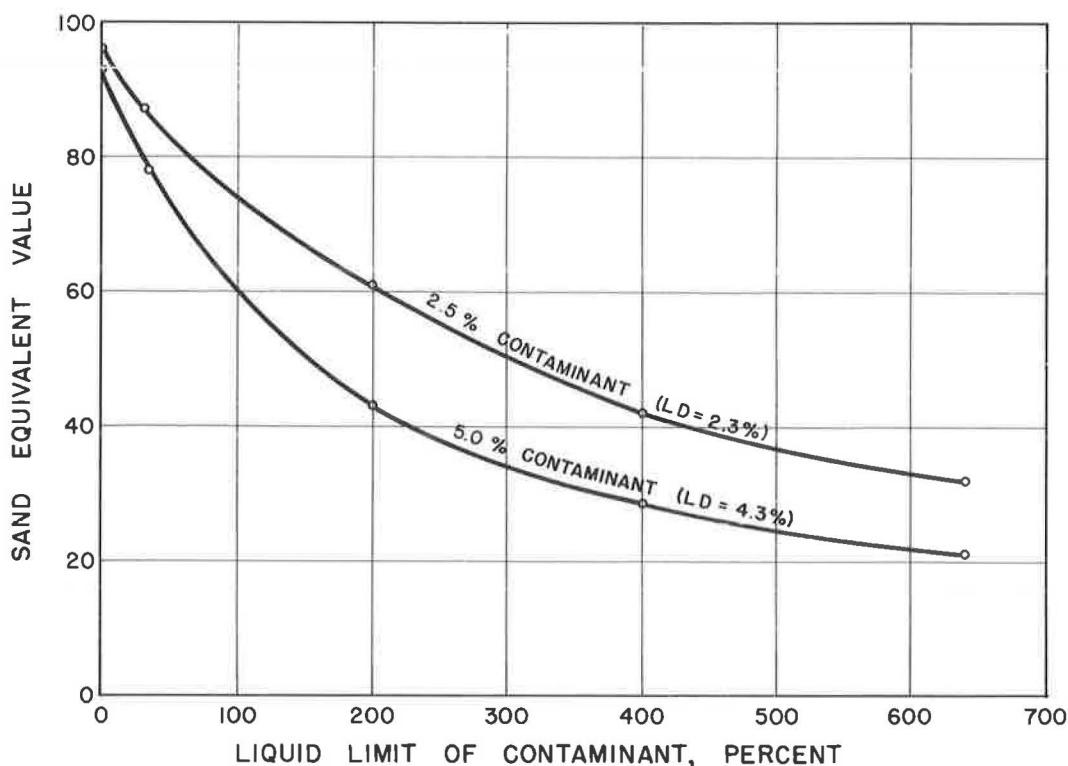


Figure 3. Relationship between sand equivalent value and liquid limit of contaminant.

weighing, the sample is agitated by rolling the pycnometer and then allowed to stand for a 15-sec settling period. The water containing the fine material is decanted and the washing process is repeated until the water remains clear after a 15-sec settling period. The pycnometer is again filled with water and weighed. The percent loss by decantation is calculated as follows:

$$\text{Percent loss} = \frac{Z_1 - Z_2}{Z_1 - Y} \times 100$$

where

$Z_1$  = weight of pycnometer containing sample and water before washing;

$Z_2$  = weight of pycnometer containing sample and water after washing and decanting;  
and

$Y$  = weight of pycnometer filled with water at approximately same temperature at which  $Z_1$  and  $Z_2$  were determined.

Examination of the loss by decantation and sand equivalent tests shows that the loss by decantation results reflect only the amount of clay-size materials in the aggregate, whereas the sand equivalent results give an indication of amount and activity of the clay-size fraction. A linking parameter was needed to draw a correlation between the two tests, and the liquid limit of the clay-size fraction was chosen.

Fifteen samples of concrete sand from various locations in Texas were obtained. Each of these samples was thoroughly mixed and split with a sample splitter to obtain test specimens for three loss by decantation tests and three sand equivalent tests. The loss by decantation and sand equivalent values for samples 1 through 15 are plotted in Figure 1 and given in Table 1.



To investigate the effect of liquid limit on the results of the two tests, 10 samples were manufactured and tested (Table 2). A high quality concrete sand was washed in the laboratory with a detergent to remove all minus 200 mesh material. The contaminants used were (a) pure silica flour with a liquid limit of zero, (b) a natural clay with a liquid limit of 34 percent, (c) a silica-montmorillonite mixture with a liquid limit of 200 percent, (d) a silica-montmorillonite mixture with a liquid limit of 400 percent, and (e) pure montmorillonite with a liquid limit of 640 percent.

The washed sand was air dried and divided with a sample splitter to obtain sand for 30 sand equivalent test specimens and 30 loss by decantation test specimens. Three test specimens, each containing the specified amount of minus 200 mesh material, were then made for each of the 10 samples. After each test specimen was thoroughly mixed, water was added and the specimen was mixed again, then dried in an oven at 105 C. Sand equivalent and loss by decantation tests were then run on these test specimens; the results are presented in Figures 2 and 3 and in Table 1.

## Results and Discussion

Figure 1 shows the data obtained from samples 1 through 15. Although these points form a definite curve, the irregularity of some points definitely indicates that some variable or combination of variables affects the two test values in different ways. A curve is drawn through the data points and the allowable limit for acceptable fine aggregate (by Texas Highway Department specifications) is indicated. Some materials would be acceptable on the basis of the loss by decantation value but rejected on the basis of the sand equivalent value.

Figures 2 and 3 show the effect of the liquid limit of the contaminant for two different percentages of contaminant on the results of the two tests. The sand equivalent values in Figure 3 verify those reported by Clough and Martinez (2). The variation in liquid limit of the contaminant has little or no effect on loss by decantation results, but has a pronounced effect on the sand equivalent value.

The relationship between loss by decantation and sand equivalent test values can be derived in the following manner. If the symbols in Figure 4 are used in the definition of the sand equivalent value, it can be written:

$$SE = \frac{100A}{A + KA} \quad (1a)$$

or

$$SE = \frac{100}{1 + K} \quad (1b)$$

For a given material, the factor K can be written as another factor, C times P, where P is the decimal fraction of the contaminant in the sample. Eq. 1b then becomes

$$SE = \frac{100}{1 + CP} \quad (2)$$

This equation can be written

$$C = \frac{100 - SE}{SE(P)} \quad (3)$$

If the values of C are plotted against values of the liquid limit (using the data in Table 3) and the data points fitted with a curve by the least squares method using  $C = A_1 (LL) + A_2$  as a model, the resulting equation is

$$C = 0.1318 (LL) + 1.79 \quad (4)$$

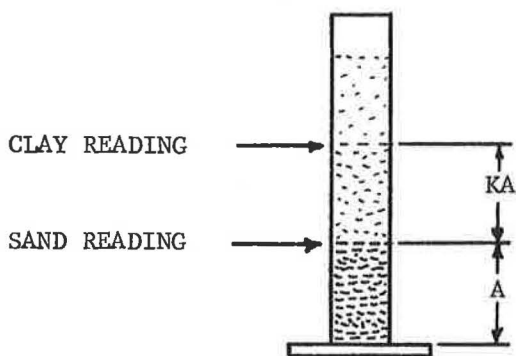


Figure 4. Clay and sand reading in sand equivalent test.

TABLE 3  
VALUES OF C USED TO DETERMINE  
 $C = F(LL)$

Sample No.	Measured Sand Equiv. Value	$C = \frac{100 - SE}{SE(P)}$
101	94	2.55
102	87	5.98
103	61	25.57
104	41	57.56
105	32	85.00

The relationship between sand equivalent (SE) and loss by decantation (LD) can be found by substituting the expression for C from Eq. 4 and the expression for P from Eq. 5 into Eq. 2.

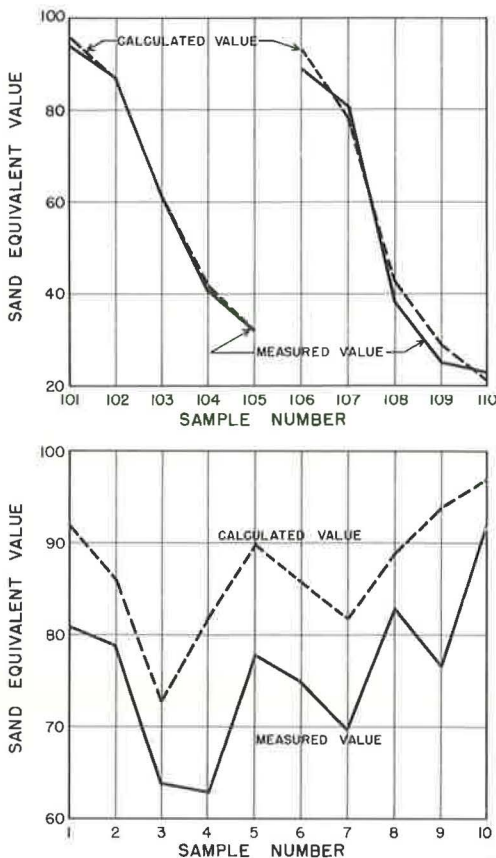


Figure 5. Correlation between calculated and measured sand equivalent values.

Figure 2 shows that the loss by decantation value varies insignificantly with liquid limit, and can be considered constant for all practical purposes. The average value of the loss by decantation was 2.3 for 2.5 percent minus 200 mesh material. If these values are used in

$$LD = K_1 P \quad (5)$$

the value of  $K_1$  is 92.

Eq. 5 can now be written

$$P = \frac{LD}{92.0} = 0.01087 LD \quad (6)$$

$$SE = \frac{100}{1 + \frac{LD}{K_1}(0.1318 LL + 1.79)}$$

This relationship ( $K_1 = 92$ ) has been used to calculate sand equivalent values for samples 106 through 110 and 1 through 10 (Table 1, Fig. 5).

There is a good correlation between the test values, and the calculated values are consistently higher than the test values for samples 1 through 10 (Fig. 5). The difference between the two sets of data for the latter samples can possibly be explained by considering the difference between the manufactured (106 through 110) and naturally occurring samples (1 through 10).

In naturally occurring aggregates there may be a definite adherence of the clay particles to the sand grains. The washing action in the loss by decantation test is not extremely vigorous, and a smaller percentage of the minus 200 mesh material is removed compared to the quantity removed by the vigorous washing action of the sand equivalent test. This causes the sand equivalent value to be lower for the naturally occurring samples than is predicted by the relations derived from the manufactured samples. The results on natural clay samples yield values of the adherence factor ( $K_1$ ) ranging from 21 to 86.

The equation relating sand equivalent loss by decantation and liquid limit illustrates one of the primary differences be-

tween the two tests; the sand equivalent test indicates activity in addition to amount, but the loss by decantation test does not. The sand equivalent test is also superior in that it requires less expensive equipment, can be performed in the field, and the results can be obtained in about 40 min.

## EFFECTS OF CLAY IN AGGREGATE ON PROPERTIES OF CONCRETE

### Testing Program

**Concrete Batches.**—Fourteen batches were cast in this phase of the program, using a high quality siliceous aggregate (batches S-1 through S-9) and a high quality crushed limestone aggregate (batches L-1 through L-5). All batches contained the same high quality natural sand. The fine and coarse aggregates were washed with a detergent to remove all minus 200 mesh material. Loss by decantation values for the washed aggregate were zero. Table 4 gives the physical properties of the aggregates.

A series of eight concrete batches was cast to determine the effects of different amounts of a natural clay contaminant on the physical properties of concrete. The batches are designated as S-1 through S-4 and L-1 through L-4 in Table 5. The nominal amounts of clay contaminant used were 0.0, 0.8, 1.6, and 2.4 percent of the total aggregate weight. The maximum amount of clay contaminant now allowed by Texas Highway Department specifications is 2.5 percent loss by decantation for the fine aggregate and 1.0 percent for the coarse aggregate. For typical batch designs this is 1.6 percent of the total coarse and fine aggregate weight.

A second series of five concrete batches was cast to determine the effect of the contaminant liquid limit on the physical properties of concrete. Each of these batches (S-5 through S-9) contained nominally 1.6 percent clay contaminant. The contaminant used was a mixture of silica flour and montmorillonite. The desired liquid limit could be obtained by varying the proportions of these two constituents. Batch S-5 contained pure silica flour and batch S-9 contained pure montmorillonite. These batches are also included in Table 5.

TABLE 4  
PHYSICAL PROPERTIES OF AGGREGATES

Property	Siliceous Coarse	Siliceous Fine	Crushed Limestone Coarse
Unit weight (lb/cu ft), dry loose	93.0	98.5	88.0
Specific gravity (SSD)	2.61	2.62	2.65
Absorption (% of dry wt)	1.24	0.81	1.44
Sieve analysis: cumula- tive percent retained on			
$\frac{3}{4}$ in.	0.0	—	0.0
$\frac{1}{2}$ in.	35.0	—	35.0
$\frac{3}{8}$ in.	60.0	—	60.0
No. 4	100.0	0.24	100.0
No. 8	—	10.10	—
No. 16	—	26.21	—
No. 30	—	41.21	—
No. 50	—	83.29	—
No. 100	—	98.62	—
No. 200	—	100.00	—

TABLE 5  
CONCRETE MIX DATA<sup>a</sup>

Batch Designation	Contaminant		Type I Cement		Aggregate		Total Water (lb)	Air Content (%)	Slump (in.)	Air Entrain. Admix (oz/cu yd)	Initial Unit Wt (pcf)
	Type	Amount (% tot. agg. by wt)	Sk	Lb	Coarse (lb)	Fine (lb)					
S-1 <sup>b</sup>	—	0.00	5.02	472	1,840	1,301	247	6.1	3 <sup>1</sup> / <sub>2</sub>	4.8	143.0
S-2	Natural clay	0.74	5.07	477	1,812	1,289	287	5.0	3 <sup>1</sup> / <sub>4</sub>	8.7	144.3
S-3	Natural clay	1.48	5.11	480	1,957	1,164	287	4.5	3	9.7	146.0
S-4	Natural clay	2.36	5.36	495	1,960	1,077	300	4.1	3	10.0	145.0
S-5 <sup>c</sup>	Silica flour	1.42	5.07	477	1,823	1,358	282	3.0	2 <sup>3</sup> / <sub>4</sub>	3.8	147.5
S-6	Sil-Mont LL = 35	1.48	5.05	475	1,814	1,277	273	4.9	2 <sup>3</sup> / <sub>4</sub>	5.8	144.0
S-7	Sil-Mont LL = 200	1.50	4.97	467	1,777	1,222	352	2.9	3 <sup>1</sup> / <sub>4</sub>	8.5	142.9
S-8	Sil-Mont LL = 400	1.57	5.11	480	1,842	1,101	386	3.0	3	9.7	142.9
S-9	Mont LL = 740	1.60	4.95	465	1,701	1,114	406	3.3	3	8.3	138.8
L-1 <sup>b</sup>	—	0.00	4.97	467	1,672	1,491	287	4.1	3	3.8	145.0
L-2	Natural clay	0.74	5.00	470	1,681	1,377	271	6.0	3 <sup>1</sup> / <sub>2</sub>	8.6	141.0
L-3	Natural clay	1.49	5.11	480	1,716	1,382	289	3.0	3	6.3	145.0
L-4	Natural clay	2.25	4.97	467	1,672	1,328	334	4.2	2 <sup>3</sup> / <sub>4</sub>	8.5	143.0
L-5	Limestone fines	1.48	5.05	475	1,698	1,405	296	3.1	3	4.8	145.5

<sup>a</sup>Quantities per cubic yard of concrete.

<sup>b</sup>Used as control batch for study of effects of percent contaminant.

<sup>c</sup>Used as control batch for study of effects of liquid limit of contaminant.

TABLE 6  
SPECIMEN TESTING SCHEDULE

Specimen No.	Dimensions	Curing	Test Type
1	3- × 4- × 16-in. prism	3 days moist	Specimens were subjected to ASTM freeze-thaw test C310-57T with dynamic modulus and weight determinations made periodically.
2	3- × 4- × 16-in. prism	3 days moist	
3	3- × 4- × 16-in. prism	3 days moist	
4	4- × 4- × 11-in. prism	3 days moist	Specimens were stored under atmospheric conditions of 52 percent RH and 72 F; shrinkage measured periodically.
5	4- × 4- × 11-in. prism	3 days moist	
7	3- × 4- × 16-in. prism	7 days moist	Weight, dynamic modulus, flexural and compressive strengths determined at 7 days of age.
8	3- × 4- × 16-in. prism	7 days moist	
9	3- × 4- × 16-in. prism	7 days moist	
10	3- × 4- × 16-in. prism	28 days moist	Weight and dynamic modulus determinations were made at 3, 7, 14, and 28 days of age; flexural and compressive strengths determined at 28 days of age.
11	3- × 4- × 16-in. prism	28 days moist	
12	3- × 4- × 16-in. prism	28 days moist	
13	3- × 4- × 16-in. prism	3 days moist	Specimens cycled between atmospheric conditions at 17 percent RH, 120 F and 100 percent RH, 72 F. Weight and dynamic modulus determinations were made periodically.
14	3- × 4- × 16-in. prism	3 days moist	
15	4- × 4- × 11-in. prism	3 days moist	Specimen subjected to same atmospheric conditions as specimens 13 and 14; shrinkage measured periodically.

Liquid limit determinations were made on the minus 200 mesh fractions of representative samples of Texas pit-run materials. On the basis of these results, a natural clay with a liquid limit of 34 percent was selected for contaminating six concrete batches.

All concretes were batched in a 2-cu ft vertical drum Lancaster mixer. The dry aggregate and contaminant were thoroughly mixed and then one-half of the mixing water was added. This was followed by the addition of the cement and about one-fourth of the estimated water containing the air-entraining admix. Water was then added until a slump of  $3 \pm \frac{1}{2}$  in. was obtained, after which air content and unit weight were determined.

Slump was determined in accordance with ASTM C143-39 and air content in accordance with ASTM C231-56T, except that vibration was used instead of the hand-rodding procedure. The testing schedule for these concretes is given in Table 6.

**Shrinkage.**—Shrinkage specimens were 4- by 4- by 11-in. prisms. Gage points were installed in the center of the end blocks which were free to move inward with the ends of the specimen. The gage points used were the same size as those used in ASTM C147-60T, Volume Change of Cement Mortar and Concrete, and provided a gage length of  $10.0 \pm 0.1$  in.

The comparator used to measure shrinkage was similar in design to that described in ASTM C157-60T, except that it could accommodate the 4- by 4-in. cross-section specimens. When changes in length were determined, the specimen was placed in the



TABLE 7  
PHYSICAL PROPERTIES OF CONCRETE<sup>a</sup>

Batch Design	Dynamic Modulus of Elasticity ( $10^{-6}$ lb/sq in.)		Modulus of Rupture (lb/sq in.) <sup>b</sup>		Compressive Strength (lb/sq in.) <sup>c</sup>	
	7 Day	28 Day	7 Day	28 Day	7 Day	28 Day
S-1	5.86	6.25	810	780	3,300	3,670
S-2	5.61	6.31	660	720	2,690	3,370
S-3	5.79	5.99	640	580	2,850	3,220
S-4	5.26	6.62	580	650	2,390	3,000
S-5	6.40	6.46	880	770	2,890	2,920
S-6	5.48	6.00	650	790	2,750	3,530
S-7	4.81	5.16	510	560	2,160	2,520
S-8	4.58	4.72	500	520	2,370	2,430
S-9	3.96	4.33	410	450	1,840	2,290
L-1	5.76	6.22	700	830	2,900	3,210
L-2	5.44	5.64	580	760	2,790	2,640
L-3	5.35	5.95	770	790	3,570	3,810
L-4	5.14	5.38	600	730	2,450	2,750
L-5	5.52	5.84	830	810	3,120	3,890

<sup>a</sup>All specimens were moist cured until time of testing.

<sup>b</sup>Center point loading 3- by 4- by 16-in. specimens.

<sup>c</sup>ASTM C116-49 modified cube.

comparator and allowed to rotate slowly. If any cyclic variation in the dial reading occurred as the specimen rotated, the lowest reading was recorded.

**Strength.**—Modulus of rupture was determined using 3- by 4- by 16-in. prismatic specimens under a midpoint loading condition. The specimens had a 14-in. span and were loaded with the 4-in. side in the vertical position. With the exception of the span length, this method of test conforms to ASTM C293-54T.

Compressive strength was determined using the two ends of the specimen remaining after the modulus of rupture test. The compressive strength test procedure conformed to ASTM C116-49.

**X-Ray Diffraction.**—Fifteen samples of clay from pit-run aggregates were analyzed using x-ray diffraction techniques. Cation exchange capacity and exchangeable cation tests were carried out to supplement the x-ray analysis.

### Results and Discussion

Non-dimensionalization of the ordinates of the graphs presented in this paper has been accomplished by expressing quantities as a percentage of control quantities. Control batches are indicated in Table 5 and absolute values of the various properties are given in Tables 4 and 7.

The effect of contaminant quantity on concrete strength is shown in Figures 6 through 9. A significant decrease in the modulus of rupture at 7 days is indicated as contaminant quantity increases. The same trend is evident in compressive strength at 28 days; the strength reduction is less for the concrete containing crushed limestone.

Data on relative shrinkage for concrete at 28 days and at 1 yr indicate that the presence of clay in the siliceous aggregate significantly increases concrete shrinkage at early ages, but has only a slight effect on shrinkage at 1 yr (Fig. 10). The effect of contaminant quantity on shrinkage of concrete containing crushed limestone appears quite different. This difference might be attributed to the angularity and texture of the crushed limestone. The clay provides effective lubrication for the more angular and

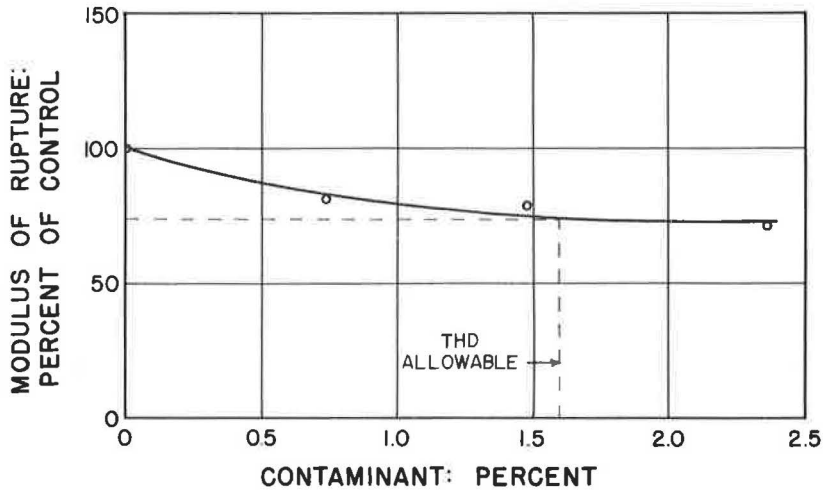


Figure 6. Relative 7-day modulus of rupture vs percent contaminant (natural clay LL = 34) for siliceous aggregate concrete.

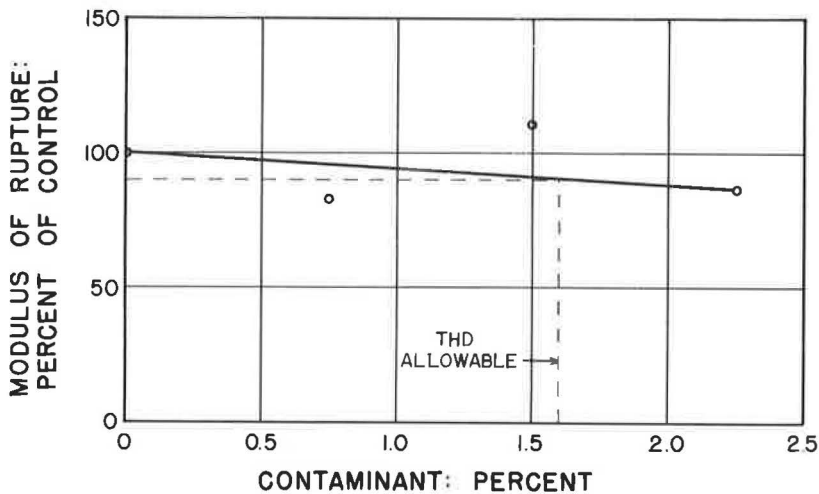


Figure 7. Relative 7-day modulus of rupture vs percent contaminant (natural clay LL = 34) for crushed limestone aggregate concrete.

rough textured limestone aggregate, which decreases the amount of water required for a given slump and tends to cancel the increased water requirement called for with the addition of clay. A point appears to be reached (at about 1.5% contaminant) where additional contaminant fails to provide additional lubrication, and the water requirement and consequently shrinkage begin to increase.

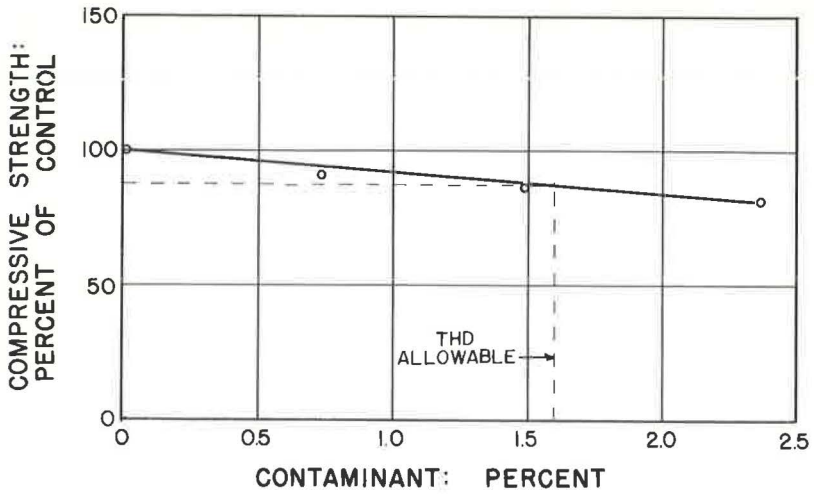


Figure 8. Relative 28-day compressive strength vs percent contaminant (natural clay LL = 34) for siliceous aggregate concrete.

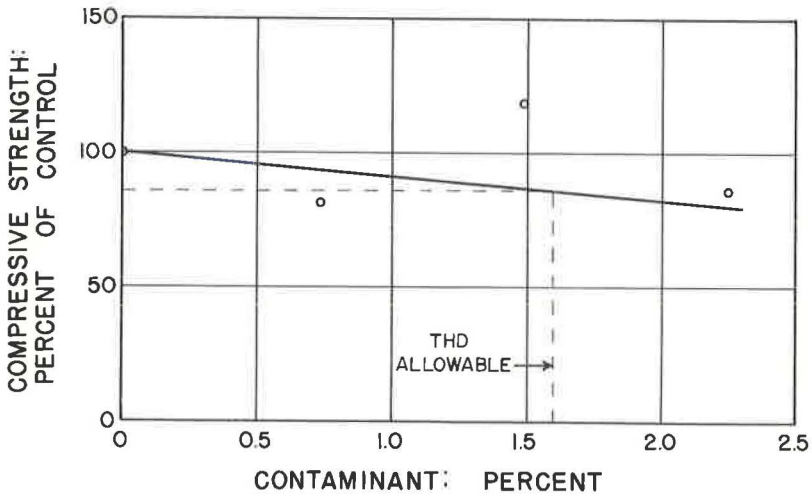


Figure 9. Relative 28-day compressive strength vs percent contaminant (natural clay LL = 34) for crushed limestone aggregate concrete.

The effects of contaminant liquid limit on various concrete properties are shown in Figures 11 through 13. A very pronounced decrease in both the modulus of rupture and the compressive strength is indicated with increasing liquid limit. In the case of the 7-day modulus of rupture, a large percentage of this decrease in strength is in the 0 to 40 percent liquid limit range. This strength reduction is of major concern because



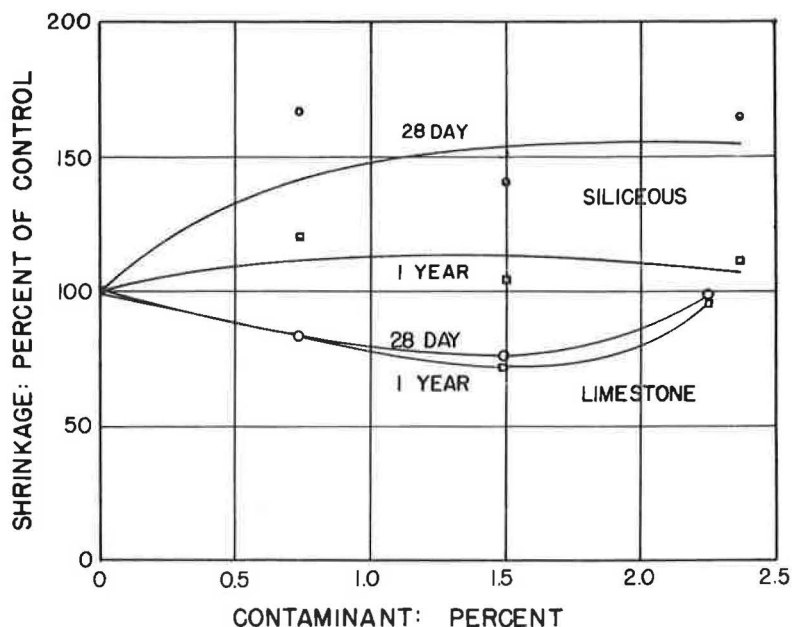


Figure 10. Relative shrinkage vs percent contaminant (natural clay LL = 34) for crushed limestone and siliceous aggregate concrete.

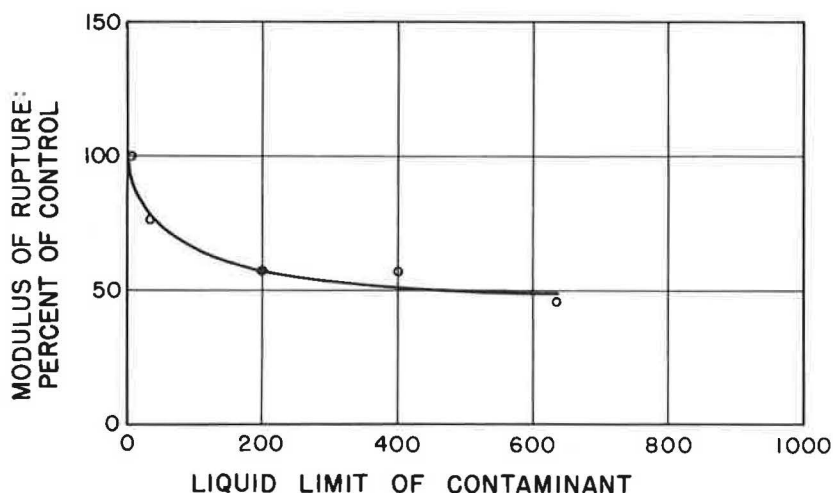


Figure 11. Relative 7-day modulus of rupture vs liquid limit (nominally 1.6% clay) for siliceous aggregate concrete.

the liquid limit of the clays naturally occurring in concrete aggregates in Texas is predominately within this range.

Relative shrinkage at ages of 28 days and 1 yr is shown in Figure 13. Here again, the influence of the contaminant liquid limit is quite pronounced.

The effects of contaminant quantity and liquid limit on the 7-day modulus of rupture and on the 28-day compressive strength are shown in Figures 14 and 15. Since limited

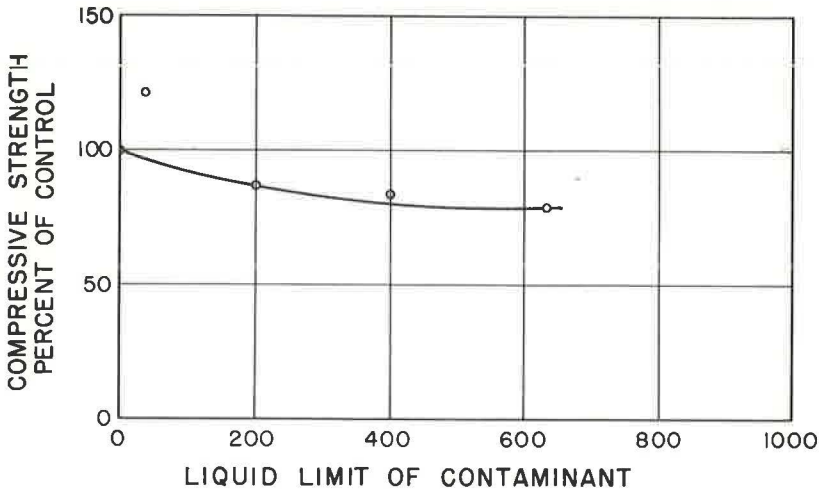


Figure 12. Relative 28-day compressive strength vs liquid limit (nominally 1.6% clay) for siliceous aggregate concrete.

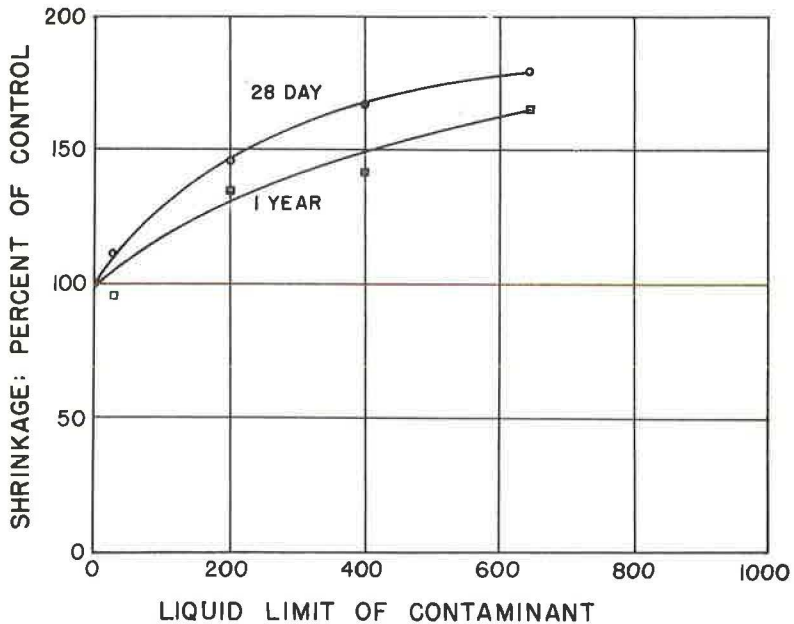


Figure 13. Relative shrinkage vs liquid limit of contaminant (nominally 1.6% clay) for siliceous aggregate.

data are available at this time, the dashed curves are speculative. These figures illustrate clearly that the effect of a contaminant depends on its activity as well as on the quantity present.

Figure 16 is a typical x-ray pattern obtained from clays found in natural concrete sand. The vertical scale is simply denoted intensity because this scale is an arbitrary, relative measure of the intensity of the refracted x-rays. The horizontal scale is twice the angle between the incident x-rays and the lattice planes of the clay. Most of the

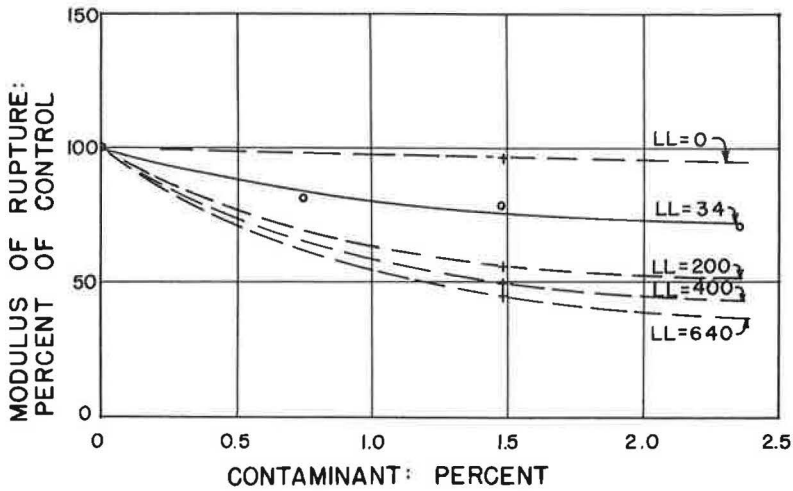


Figure 14. Relative 7-day modulus of rupture vs percent contaminant for different liquid limits, siliceous aggregate concrete.

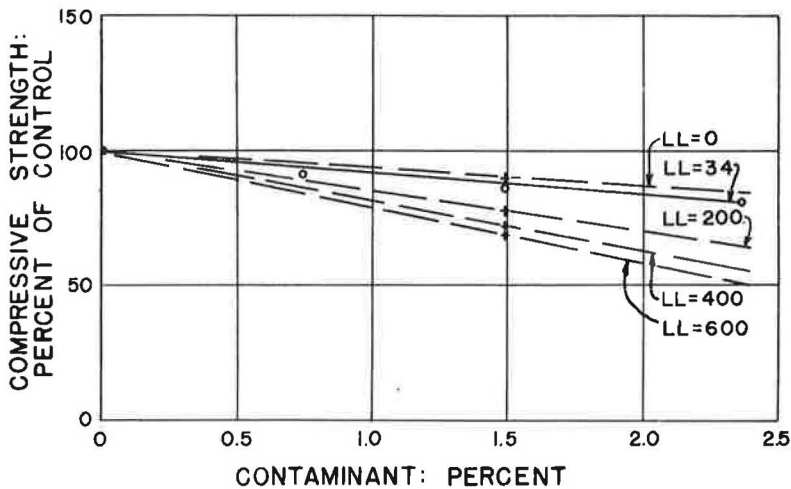


Figure 15. Relative 28-day compressive strength vs percent contaminant for different liquid limits, siliceous aggregate concrete.

clay minerals can be identified by the value of two theta at which the peak is found. Identification of some clay minerals is more difficult, however, and requires that test specimens be prepared using magnesium and water, magnesium and ethylene glycol, and potassium and water. In the diffraction patterns shown, the peaks of each clay mineral except montmorillonite occur at the same value of two theta for each of the three preparation solutions. This shift, or absence of the montmorillonite peak when the sample is prepared in different solutions, is an aid in identification. The width of

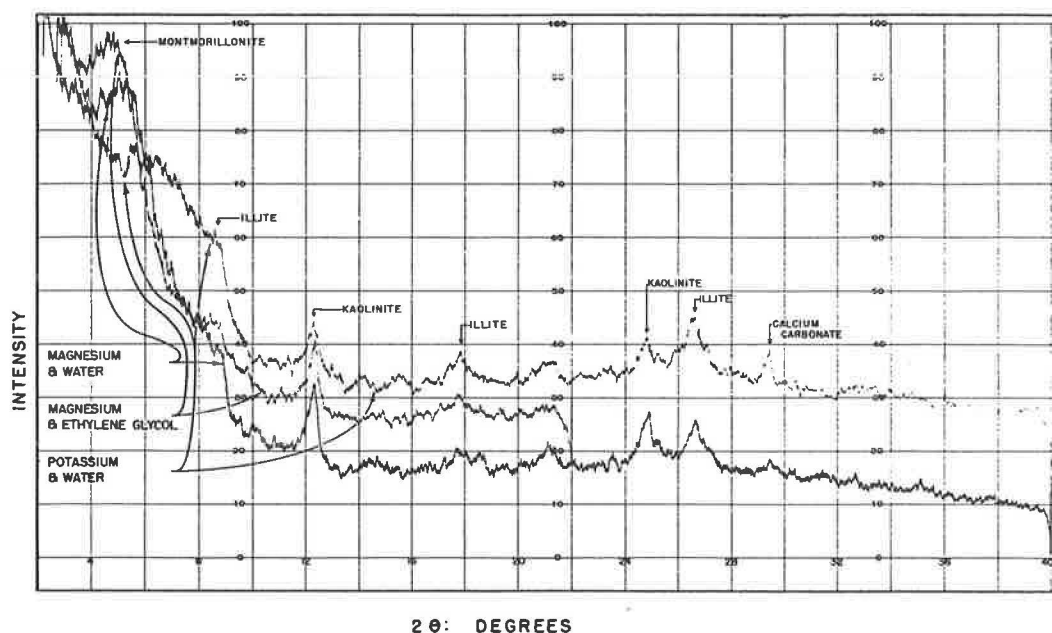


Figure 16. Typical x-ray diffraction pattern.

TABLE 8  
ANALYSIS OF CLAYS

Sample No.	Type of Clay and Estimated Amount <sup>a</sup>	Cation Exchange Cap. <sup>b</sup>	Exchangeable Cations <sup>b</sup>			
			Na	Ca	Mg	K
1	I2, M2, K2, Q3					
2	M1, I3, K3, Q3	17.3	0.24	Calc.	3.2	0.37
3	M1, K2, I3, Q3	18.6	0.11	Calc.	1.8	0.53
4	M1, I2, K2, Q3	7.5	0.10	Calc.	0.94	0.18
5	M1, I2, K2, Q3	11.0	6.2	Calc.	4.2	0.33
6	M1, K2, I2, Q3	13.5	0.44	Calc.	1.8	0.29
7	M1, I2, K2, Q3	32.3	0.23	Calc.	2.2	0.63
8	—	—	—	—	—	—
9	M1, K2, I3, Q3	10.2	4.9	Calc.	8.0	0.97
10	M1, K2, I3, Q3	9.6	0.58	Calc.	1.2	0.29
11	I2, K2, M2, Q3	14.3	0.3	7.9	1.7	0.55
12	M1, I3, K3, Q3	—	—	—	—	—
13	M2, I2, K2, Q3, F3	—	—	—	—	—
14	—	7.7	0.22	Calc.	1.4	0.20
15	—	—	—	—	—	—
16	M1, K2, I3, Q3	17.1	1.2	15.5	5.7	0.75
17	M1, I2, K2, C3	—	—	—	—	—
18	M1, K3, I3, Q3	8.3	0.39	Calc.	0.58	0.25

<sup>a</sup>Abbreviations used are M = montmorillonite, I = illite, K = kaolinite, Q = quartz, F = feldspar, C = calcium carbonate; numerical code: L = greater than 40 percent, 2 = 10 to 40 percent, 3 = less than 10 percent.

<sup>b</sup>Milliequivalents per 100 gm.

the montmorillonite peak indicates a poorly crystalline structure and decayed or decaying micaceous material.

Data obtained from x-ray analysis and related tests are given in Table 8. The amount of each clay mineral was estimated from the x-ray pattern and is not based on quantitative test results. Samples are predominantly montmorillonite. If several clay minerals have the same quantity code designation within a sample, they are arranged in order of descending magnitude. These data are of value in indicating the deleterious effect of the clay, as clay activity is dependent on the mineral composition.

### CONCLUSIONS

1. The strength of concrete is reduced as the quantity of contaminant in the aggregate is increased.
2. The strength of concrete is decreased as the liquid limit of the contaminant increases.
3. Shrinkage of the siliceous aggregate concrete is increased as the contaminant quantity increases.
4. Shrinkage of the siliceous aggregate concrete is increased as the liquid limit of the contaminant increases.
5. The dynamic modulus of elasticity of the concrete containing siliceous aggregate is decreased as the liquid limit of the contaminant increases.
6. Within the range of contaminant quantities tested, the dynamic modulus of elasticity of concrete does not change significantly as the quantity of contaminant increases.
7. Present Texas Highway Department specifications for concrete aggregate indirectly allow a 15 percent reduction in 28-day compressive strength and a 25 percent reduction in 7-day modulus of rupture values.
8. Some aggregates meet present Texas Highway Department specifications by the loss by decantation test while failing the requirements of the sand equivalent test.
9. A relationship exists between loss by decantation results, liquid limit of the minus 200 mesh fraction, and sand equivalent value.
10. Clay activity, as indicated by liquid limit, as well as the amount of the clay present in the aggregate, influence concrete strength. The sand equivalent test is an indicator of a combination of activity and amount of contaminant, whereas the loss by decantation test indicates only the amount. For this reason the sand equivalent test is a better indicator of the quality of fine aggregate for use in concrete. Loss by decantation results should be combined with liquid limit determinations to evaluate coarse aggregate.

### ACKNOWLEDGMENTS

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# Influence of Natural Sand Fine Aggregate on Some Properties of Hardened Concrete Mortar

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Most modern concrete specifications include requirements for quality of the fine aggregate portion to insure that it does not detract from the quality of the hardened concrete. The significance of many of the tests used to measure fine aggregate quality, however, is only poorly understood.

This paper reports a laboratory study of 25 New York natural sands and the effect that variations in their characteristics have on the freeze-thaw durability, drying shrinkage and water requirement of concrete mortar. The purpose of this study was to define significant aggregate properties and to suggest practical methods of measuring them.

It is concluded that except for varying amounts of surface damage in the form of small pits or "popouts," even extremely low quality fine aggregate, in itself, will cause no freeze-thaw deterioration of air-entrained concrete. The different fine aggregates, however, did cause variations in mortar shrinkage and water requirement. Modulus of elasticity was found to be the most significant aggregate property affecting shrinkage. Both aggregate elasticity and water requirement correlated empirically with results of the magnesium sulfate salt soundness test.

•FINE AGGREGATE is used in concrete principally to improve the properties of the plastic mix; that is, to provide workability, facilitate finishing, and promote uniformity by inhibiting segregation. The degree to which this is accomplished is determined largely by the grading, size, shape and surface texture of its component particles.

Most modern specifications include requirements for quality, as well, to insure that the sand selected for use does not detract from the properties desired in the hardened concrete. These requirements are usually based on mortar strength tests, sulfate soundness or freeze-thaw tests of unconfined aggregate, freeze-thaw tests of concrete containing the questionable aggregate, service records, or a combination of these. Unfortunately, the relationship between many of the tests available for judging quality and the properties imparted to concrete by the aggregate is poorly understood. The significance of these tests, therefore, is often difficult to interpret.

The increasing demand for aggregate of all types in the face of a decreasing supply resulting from depletion and zoning restrictions, has stimulated an increased interest in the influence of aggregate properties on the properties of concrete and in the search for simple tests to predict relative aggregate quality. The data reported here are part of the results of an investigation conducted in New York of natural sand concrete fine aggregate. This investigation was an attempt to contribute to an understanding of the influence of fine aggregate on the properties of hardened concrete, to define significantly related aggregate properties, and to suggest practical means of measuring them.

## HISTORY, SCOPE AND TEST PROCEDURES

### Historical Review

In 1929, Gonnerman (1) summarized the principal considerations in selecting fine aggregate for concrete as:

- ... (1) composition and structural characteristics of the particles, (2) cleanness of particles and freedom from injurious amounts of deleterious substances, and (3) size and grading of particles.

By and large, these same considerations are reflected in modern specifications (37). It was also recognized early that differences among fine aggregates cause variations in the properties of hardened concrete, principally, as they caused variations in the quantity of mixing water required to produce a given consistency (1). On the basis of these considerations, variations in the strength and durability of concrete and concrete mortar have frequently been explained by differences in their fine aggregate (2-9).

With the advent of air entrainment, however, it appeared that fine aggregate could be disregarded as a major contributing factor to concrete distress, at least from freezing and thawing (7, 8, 10). This view was supported by a growing understanding of the mechanism of frost damage in concrete, particularly of the effect of large numbers of small closely spaced air bubbles in the paste and of the significance of aggregate particle size (11). Accordingly, there have been only a few studies in recent years of the influence of fine aggregates on the properties of hardened concrete and mortar (12-15).

### Scope and Test Procedures

Twenty-five sands were sampled from the stockpiles of New York producers operating from deposits of natural sand and gravel. An effort was made to include the wide compositional variations represented in the State as well as the extremes in quality. Laboratory studies of corresponding mortars were conducted between April 1964 and May 1965 in which measurements of freeze-thaw durability, drying shrinkage and water requirement were made. The laboratory studies were supplemented by field surveys of air-entrained concrete conducted during the summer of 1963 and outdoor exposure tests begun during the winter of 1964-1965.

Each sand was sampled in sufficient quantity to meet the needs of the entire testing program, broken down into individual size fractions, washed, and recombined for each series of tests to a predetermined gradation with a fineness modulus of 2.85 and with 2 percent by weight of unwashed material passing the No. 200 screen. The 24-hr absorption, bulk specific gravity and 5-cycle magnesium sulfate soundness of each sand were determined by the appropriate ASTM procedures (38). Specific gravity distributions were determined by separation in liquid media prepared to different densities, and sand equivalents were measured by Test Method No. Calif. 217-E (July 1963) of the California Division of Highways. The proportion of various lithologic types in each of the experimental sands was determined by examining 100 grains each of the  $\frac{3}{8}$ -in.-No. 4, No. 4-No. 8, No. 8-No. 16, No. 16-No. 28, No. 28-No. 48, No. 48-No. 100 and No. 100-No. 200 sizes under the binocular microscope. The relative proportion of clay and silt size material in the minus No. 200 portion of each sand was determined by hydrometer analysis. The minerals comprising the clay-size portion (smaller than two microns) were determined by X-ray diffraction.

One hundred grains of the No. 14-No. 28 size of each sand were also examined for roundness and sphericity with a petrographic microscope and camera lucida. A qualitative roundness index was assigned to each sand which consisted of the average roundness value for the particles examined according to the following classification:

Classification	Roundness Value
Angular, all edges sharp	0
Subangular, < 50% of edges rounded	33
Subrounded, > 50% of edges rounded	66
Rounded, all edges rounded	100

The sphericity of each particle was computed from the lengths of its principal axes by the method of Krumbein (16) and the average value calculated for each sand. Specific surface areas were also estimated from the lengths of principal axes by the following formula:

$$\text{Specific surface area, cm}^2/\text{cm}^3 = 2/b (1/x + y + 1)$$

TABLE 1  
LITHOLOGIC AND SHAPE CHARACTERISTICS OF EXPERIMENTAL SANDS

Sand	Rock Types, in Order of Decreasing Amount <sup>a</sup>		Particle Shape <sup>b</sup>				Relative Specific Surface Area
	Major	Minor	B/A	C/B	Sphericity <sup>c</sup>	Roundness	
1	Q, G	M	0.80	0.70	0.75	33.7	8.18
2	Q	L, S	0.76	0.79	0.77	35.7	7.36
3	Q, G	—	0.74	0.73	0.73	44.3	7.27
4	S, Q	L, Sh, C	0.76	0.66	0.72	46.0	7.68
5	Q, L	S, Sh	0.76	0.71	0.73	41.3	7.45
6	Q	S, Sh	0.76	0.72	0.74	53.6	7.41
7	S, Q, L	C, Sh	0.78	0.68	0.74	55.7	7.67
8	Q, S	L, Sh	0.80	0.73	0.77	55.3	7.36
9	Q, S, Sh	C	0.78	0.69	0.74	45.3	7.57
10	S, Q, L	C, Sh	0.73	0.63	0.69	36.7	7.86
11	Q, L, S, Sh	C	0.76	0.67	0.72	38.4	7.66
12	S, L, Q	Sh, C	0.74	0.72	0.73	38.7	7.32
13	Q, S, Sh	—	0.76	0.74	0.74	43.6	7.28
14	S, Q, C	Sh	0.77	0.66	0.72	35.7	7.71
15	S, Q, Sh, C	L	0.77	0.71	0.74	47.0	7.47
16	S, Q, L	Sh	0.73	0.66	0.70	47.6	7.63
17	Sh, Q, S	—	0.75	0.66	0.71	43.6	7.72
18	S, Q	L, Sh	0.73	0.73	0.72	51.7	7.14
19	S, Q, Sh, L	—	0.74	0.64	0.70	49.3	7.81
20	S, Q	Sh, C, L	0.76	0.71	0.73	42.0	7.44
21	S, Sh, Q	L, C	0.76	0.69	0.73	47.3	7.52
22	S, Q	L, Sh	0.74	0.66	0.70	46.3	7.79
23	Sh, Q, S	—	0.75	0.65	0.71	44.6	7.74
24	Sh, S, Q	—	0.72	0.55	0.68	49.7	8.59
25	S, Sh, Q	L	0.73	0.62	0.78	33.1	8.16

<sup>a</sup>"Major" > 10 percent of total; "Minor" 2–10 percent of total; according to the following notation, Q = quartz and feldspar including quartzite, G = granite and gneiss, S = sandstone, L = limestone and dolomite, Sh = shale and siltstone, C = chert, M = mica.

<sup>b</sup>Average values for 100 grains examined in the No. 14–No. 28 size range.

<sup>c</sup>As defined by Krumbein (16) in which A, B and C are the major, intermediate and minor axes, respectively, of the enclosing rectangular prism.



TABLE 2  
ABSORPTION AND SPECIFIC GRAVITY OF EXPERIMENTAL SANDS

Sand	24-Hr Absorption (%) <sup>a</sup>	Bulk Specific Gravity (dry) <sup>a</sup>	Specific Gravity Distribution (% by wt lighter than)				
			2.00	2.20	2.40	2.60	2.80
1	0.78	2.62	0.0	0.1	0.5	8.3	97.6
2	0.40	2.63	0.0	0.0	0.3	33.8	97.5
3	0.66	2.66	0.0	0.0	0.1	18.1	88.3
4	1.56	2.57	0.0	0.1	0.8	13.3	95.8
5	1.05	2.63	0.0	0.1	0.2	9.4	96.5
6	0.99	2.59	0.2	1.1	3.5	16.8	97.4
7	1.50	2.58	0.0	0.0	0.9	11.0	98.7
8	1.39	2.59	0.0	0.2	2.0	22.7	96.8
9	2.04	2.53	0.1	0.2	4.4	52.5	98.6
10	1.86	2.58	0.0	0.1	0.4	11.4	96.8
11	1.65	2.62	0.0	0.0	0.2	7.8	76.6
12	1.41	2.60	0.0	0.0	0.4	6.1	90.7
13	1.97	2.55	0.1	1.2	2.9	10.7	99.6
14	2.66	2.49	0.0	0.5	3.1	34.3	98.5
15	1.98	2.54	0.0	0.1	1.1	24.0	98.8
16	1.89	2.57	0.0	0.1	0.8	15.0	90.4
17	2.70	2.52	0.2	1.2	3.7	31.7	97.3
18	1.58	2.57	0.0	0.0	0.7	12.7	96.4
19	2.09	2.59	0.0	0.2	0.8	15.7	87.0
20	2.28	2.51	0.0	0.1	1.1	31.0	98.6
21	2.18	2.56	0.0	0.0	0.4	5.0	94.6
22	1.90	2.54	0.0	0.2	0.7	16.8	98.3
23	2.30	2.58	0.2	0.2	0.9	13.8	97.4
24	2.49	2.53	0.3	0.8	2.5	19.5	99.4
25	3.02	2.52	0.0	0.1	0.4	10.7	97.4

<sup>a</sup>Average of a minimum of two tests on each sand, ASTM Designation C 127-59.

in which  $x$  equals the length ratio of the longest to the intermediate axis (reciprocal of flatness),  $y$  equals the length ratio of the intermediate to the shortest axis (reciprocal of elongation), and  $b$  equals the length of the intermediate axis in centimeters.

Measurement was also made of the quantity of water required to bring a standard mortar to a predetermined consistency. These mortars were mixed to a constant volumetric proportion between cement and saturated surface dry sand and the consistency determined by measuring the penetration of a standard cone similar in construction to that reported by Cordon (17).

The results of all descriptive tests on the experimental aggregates are given in Tables 1 through 4.

The mortars for the freeze-thaw study were proportioned for a 1:2.75 ratio of cement to sand by absolute volume and a net water cement ratio of 0.43 by weight. The cement used in this and the drying shrinkage study was from a blend of five cements from different local mills. A fixed amount of liquid neutralized vinsol resin was added to each batch in sufficient quantity to insure the development of a minimum air content of 10.0 percent. Mixing was in 0.21-cu-ft batches in a 24-qt Hobart food mixer. Each batch was tested for air content with a 200-cc pressure-type mortar air meter and for consistency with the cone penetrometer previously described. Each sand was stored for 24 hr before mixing with enough water to bring it to a saturated surface dry state.

TABLE 3  
QUALITATIVE MINERAL ANALYSIS OF MINUS 2-MICRON PORTION<sup>a</sup>

Sand	Calcite	Dolomite	Chlorite	Illite	Kaolinite
1				X	X
2	X			X	
3				X	
4	X		X	X	
5	X	X	X	X	
6			X	X	
7	X		X	X	
8	X			X	
9			X	X	
10	X	X	X	X	
11	X	X	X	X	
12	X		X	X	
13			X	X	
14				X	
15			X	X	
16	X	X		X	
17			X	X	
18	X		X	X	
19	X	X	X	X	
20			X	X	
21			X	X	
22			X	X	
23			X	X	
24			X	X	
25			X	X	

<sup>a</sup>Determined by X-ray diffraction.

TABLE 4  
MISCELLANEOUS DATA RELATING TO  
EXPERIMENTAL SANDS

Sand	Five-Cycle Magnesium Sulfate Soundness	Clay Size in Minus No. 200 Portion <sup>a</sup> (%)	Sand Equivalent	Water Requirement (ml)
1	4.0	5.9	87	198
2	5.9	2.0	92	189
3	12.8	0.9	94	201
4	13.2	1.2	89	195
5	14.0	1.1	93	194
6	14.1	1.1	91	203
7	15.0	1.8	87	191
8	15.4	1.3	92	191
9	17.5	1.7	87	199
10	17.6	5.5	81	205
11	18.3	1.6	93	194
12	18.8	2.0	84	201
13	20.0	2.2	80	202
14	21.6	2.2	81	210
15	22.0	2.0	84	205
16	22.6	5.2	83	198
17	24.2	4.0	80	207
18	25.6	2.9	86	201
19	26.7	3.8	84	198
20	28.1	1.5	80	211
21	29.1	3.6	74	213
22	33.5	2.2	80	210
23	34.2	2.2	74	202
24	40.3	6.4	69	215
25	43.1	6.8	78	233

<sup>a</sup>Smaller than an effective diameter of 2 $\mu$  as determined by hydrometer analysis.

TABLE 5  
SUMMARY OF MORTAR MIXING DATA AND DURABILITY STUDIES

Sand	Mortar Mixing Data			Durability Factors		Surface Condition Rating (lab tests) <sup>d</sup>
	Consistency (mm of pen.) <sup>a</sup>	Unit Wt <sup>a</sup> (gm/cu-cm)	Air Content (%) <sup>a</sup>	@ 300 Cycles (lab tests) <sup>b</sup>	@ 70 Cycles (natural exposure) <sup>c</sup>	
1	65.5	2.08	11.8	99.5	111.3	6
2	74.0	2.06	12.6	98.8	116.3	16
3	69.3	2.07	12.8	93.0	106.3	12
4	72.3	2.03	12.8	100.2	110.8	22
5	72.7	2.05	12.6	100.6	108.8	10
6	65.7	2.01	13.0	96.1	112.4	66
7	74.0	2.05	12.0	99.0	110.3	29
8	73.7	2.03	12.4	97.9	120.2	22
9	68.8	2.05	11.8	102.6	119.3	54
10	65.5	2.07	10.7	97.3	111.8	28
11	72.5	2.08	12.8	100.8	113.7	30
12	67.0	2.05	12.5	100.7	109.0	22
13	70.8	2.02	12.4	103.7	118.5	120
14	65.7	2.02	11.6	99.5	117.3	61
15	66.2	2.06	10.6	100.2	111.4	22
16	74.2	2.05	12.0	98.8	118.4	40
17	71.5	2.07	10.0	97.5	115.9	132
18	69.3	2.05	11.9	97.5	111.8	38
19	73.2	2.06	12.1	96.6	115.8	34
20	64.0	2.05	10.4	95.1	111.9	39
21	67.5	2.05	10.0	95.0	110.2	30
22	62.4	2.01	13.0	97.2	114.8	24
23	69.6	2.06	11.2	95.5	112.0	64
24	63.2	2.07	10.1	98.7	109.2	142
25	61.2	2.07	10.2	98.9	116.5	150

<sup>a</sup>Average of one determination on each of two batches mixed on different days.

<sup>b</sup>Average of values for six prisms, three from each of two batches.

<sup>c</sup>Estimated from daily max. and min. temperatures recorded at Albany Co. Airport, in accordance with the method outlined in reference (18).

<sup>d</sup>Number of "popouts" exceeding 1 mm in any dimension.

Note: Water-cement ratio, by weight, 0.42; cement-sand ratio, by volume, 1:2.75.

Eight 2- by 2- by 11-in. prisms were molded from each mortar, four from each of two batches mixed on different days. All prisms were cured for 14 days at 100 percent relative humidity and  $73.4 \pm 3$  F. After curing, three prisms from each batch were subjected to rapid freezing in air and thawing in water at the approximate rate of 8 cycles per day. The relative performance of each prism was evaluated by the resonance frequency technique and durability factors were calculated after 300 cycles or at 60 percent relative dynamic modulus of elasticity, whichever occurred first (39). The fourth prism from each batch was resonated initially and then placed in an outdoor exposure area for long-term observation. Results of the freeze-thaw study are given in Table 5.

The mortars for the drying shrinkage study were also proportioned for a net water-cement ratio of 0.43 but with no artificial air entrainment. The fractional volume of sand was adjusted slightly from batch to batch to compensate for variations in the water requirement of the different aggregates and to produce a cone penetration of 67 to 71 mm. Mixing was in 0.25-cu-ft batches but otherwise followed the same procedures used for the freeze-thaw specimens. Three series of mortar were prepared, each consisting of one batch from each of the experimental sands. The mixing and molding of each series was completed before another was begun.

TABLE 6  
SUMMARY OF MORTAR MIXING DATA AND LENGTH CHANGE MEASUREMENTS

Sand	Mortar Mixing Data			Length Changes <sup>b</sup>	
	Sand-Cement Ratio (by vol)	Consistency (mm of pen.) <sup>a</sup>	Unit Weight (gm/cu-cm) <sup>a</sup>	Expansion 27-Day Moist Cure (%)	Shrinkage 2 Months Drying (%)
1	2.68	70.0	2.25	0.009	0.0913
2	3.10	67.7	2.25	0.007	0.0847
3	2.80	69.0	2.24	0.010	0.0862
4	2.99	69.5	2.25	0.005	0.1089
5	2.98	67.7	2.24	0.008	0.0923
6	2.82	65.2	2.22	0.009	0.0961
7	3.07	68.7	2.25	0.004	0.1099
8	3.09	68.3	2.22	0.009	0.1057
9	2.90	69.5	2.21	0.008	0.1187
10	2.78	66.5	2.25	0.007	0.1239
11	2.95	69.7	2.26	0.009	0.0985
12	2.70	69.2	2.19	0.007	0.1152
13	2.83	69.2	2.22	0.007	0.1221
14	2.70	72.2	2.19	0.007	0.1180
15	2.75	67.7	2.23	0.011	0.1237
16	2.98	69.0	2.25	0.009	0.1118
17	2.81	70.2	2.23	0.009	0.1377
18	2.82	71.8	2.23	0.007	0.1141
19	3.06	67.0	2.25	0.007	0.1176
20	2.75	67.5	2.21	0.011	0.1239
21	2.82	68.8	2.25	0.007	0.1367
22	2.69	69.5	2.21	0.007	0.1238
23	3.05	68.0	2.26	0.007	0.1519
24	2.78	67.8	2.24	0.005	0.1812
25	2.61	69.0	2.21	0.008	0.1602

<sup>a</sup>Average of one determination on each of three batches mixed on different days.

<sup>b</sup>Average of values for nine prisms, three from each of three batches.

Note: Water-cement ratio, by weight, 0.43.

Six 3- by 4-in. high cylinders and three 1- by 1- by 11-in. prisms fitted with gage plugs were molded from each batch of mortar. Three each of the cylinders were tested for compressive and split cylinder tensile strength after 28 days of moist curing under the conditions previously described. The results of the strength tests were inconclusive and are, therefore, not included. The length of each 11-in. prism was measured when the molds were stripped after 24 hr of moist curing and again after 27 days of additional curing. They were then allowed to dry in the laboratory during which time periodic measurements were made of their length up to two months. During this time, the temperature in the area where the shrinkage specimens were stored was maintained at  $73.4 \pm 2$  F but the relative humidity dropped from 50 percent at the beginning of the drying period to about 20 percent where it remained until testing was completed. Nevertheless, all mortars from the same series were subjected to nearly the same humidity conditioning because they were mixed within a relatively short period of time. This is confirmed by the near perfect correlation among the results from the three series of tests. The variation in relative humidity, therefore, is reflected principally by a high among-batch variance in shrinkage. Results of the shrinkage study are given in Table 6.

#### DURABILITY STUDIES

Any consideration of the influence of variations in fine aggregate, per se, on variations in the freeze-thaw durability of concrete or mortar must recognize, above all, the significance of aggregate particle size. The hydraulic pressure theory set forth by

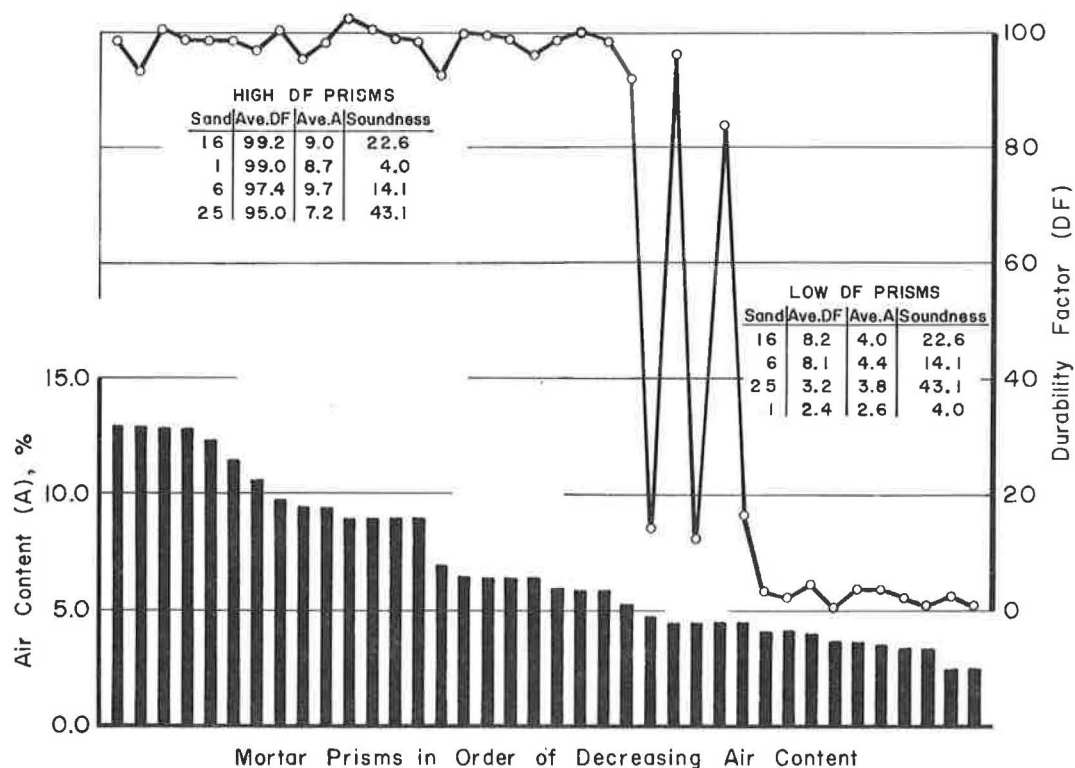
Powers (11) and expanded on by Verbeck and Landgren (19) explains the effect of the physical properties of aggregate on the frost resistance of concrete. The theory argues that the potential for disruption decreases with decreasing aggregate size when other factors remain unchanged.

In order for disruptive hydraulic pressure to develop in a saturated aggregate when water escapes from a zone of freezing, the particle must contain sufficiently long flow paths. Even with stone of high porosity and low permeability, critical sizes are on the order of 0.5 in. (19). It may be shown analytically that any mechanism causing a given percentage increase in the volume of an aggregate embedded in concrete will stress the surrounding paste in direct proportion to its own effective diameter. Likewise, if failure is to occur by expulsion of water into the surrounding paste, it is the size of the particle (i.e., volume-surface area ratio) that determines the critical parameter, volume of water expelled per unit surface area per unit time. Further, the small volume of water expelled by small particles is more readily accommodated by entrained air voids in the paste than the larger volumes expelled by larger particles.

The literature abounds with examples of the effect of particle size on the resistance to frost damage of saturated aggregates both unconfined and in concrete. These are reviewed by Schuster and McLaughlin (20). There are fewer, but equally convincing studies that demonstrate the innocuous character of fine aggregate, as such, in air-entrained concrete and mortar subject to freezing (7, 8).

### Freeze-Thaw Tests of Mortar

Statistical analysis of durability factors obtained from the freeze-thaw studies of mortar prisms (Table 5) indicated that their performance was unrelated to the sand used in their preparation. All performed at an acceptably high level. The variation





in average durability factor among the different mortars was of the same magnitude as the experimental error. These results are, therefore, consistent with the theoretical considerations and the experience of others, cited above.

The conditions of the freeze-thaw test were considerably more severe than what would normally be experienced under natural exposure. The freezing rate, the degree of aggregate and paste saturation, the proportion of sand in the mix, and the number of cycles administered were all excessive. It seems probable, therefore, that even sands of exceptionally low quality can be used in air-entrained concrete without causing deep-seated distress as a result of freezing and thawing. Where magnesium sulfate soundness is the frame of reference, exceptionally low quality would mean approximately 40 percent loss (Table 4).

Four of the experimental sands were mixed into mortar of the same proportions as before but with successively smaller doses of air-entraining agent. The durability factors for these mortars calculated after 300 cycles of freezing and thawing (Fig. 1) demonstrate that the durability of the mortar was not affected by the soundness of the sand even at low levels of air content.

#### Surface Condition Evaluation

The effect of aggregate on the appearance and surface smoothness of concrete is frequently of equal importance as its effect on deep-seated durability. Some distress resulting from unsound aggregate was noted on the surface of nearly all prisms after 300 cycles of freezing and thawing (Table 5). This appeared as either true popouts in which conical spalls of mortar were removed over affected particles or as simple failures of thin paste covers. Distress of this type was not restricted to any one particle size or size range but seemed to depend on a combination of size and proximity to the surface, with the smaller particles requiring greater proximity to the surface to cause disruption.

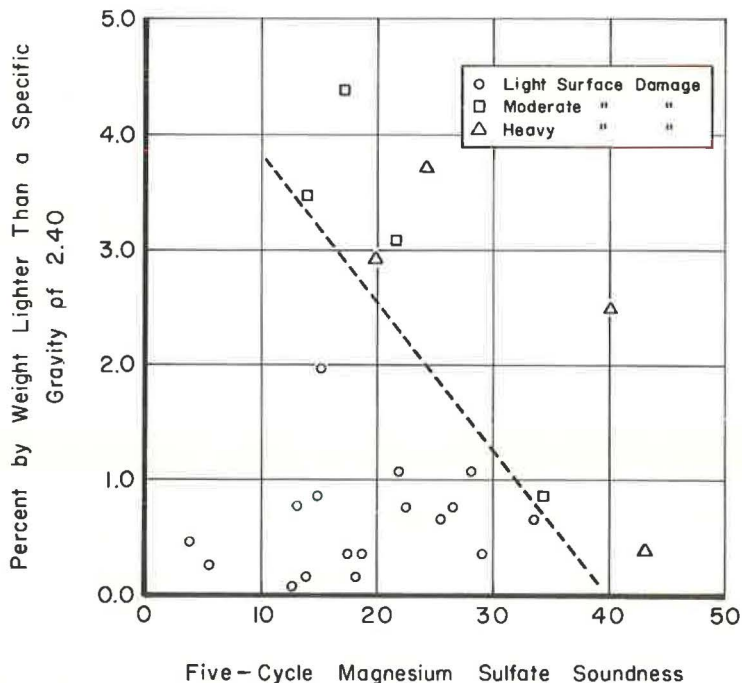


Figure 2. Relationship between sand soundness and proportion of lightweight particles.

TABLE 7

## DELETERIOUS PARTICLES IDENTIFIED ON THE SURFACE OF MORTAR PRISMS

Rock Type	Estimated Frequency of Occurrence (%)	Mortars in Which Observed	Description
Shale	45	4, 6, 7, 13, 15, 16, 17, 20, 21, 22, 23, 25	Typically containing minor amounts of detrital quartz and carbonate; frequently porous and fractured; moderately to extensively weathered.
Siltstone	30	6, 9, 10, 13, 14, 15, 16, 17, 18, 20, 25	10-45% clay or hydromica typically finely dispersed but occasionally as discrete accumulations; smaller quantities of sand-sized quartz, feldspar and carbonate; usually weathered.
Dolomite	5	7, 8, 11, 19, 23, 24	Fine-grained; usually impure with some weathering of impurities; frequently porous and/or fractured.
Calcareous shale	5	24, 25	Typically weathered and porous.
Slate	5	24	Typically dense with slight weathering.
Chert	5	10, 17, 18, 20, 22	Typically impure (clay or hydromica and dolomite); weathered.
Sandstone	< 5	17, 22	Fine-grained, detrital quartz and feldspar in a weathered clay (or hydromica) or iron oxide matrix.
Limestone	< 5	18, 25	Impure, weathered and porous.
Hydrous iron oxide	< 5	1, 4	Intensely weathered and porous.
Hematite	< 5	9	Individual crystal; fresh.

It is difficult to evaluate the significance of distress of this type by scanning 528 sq in. of mortar surface distributed among six small beams. Even in the worst instances (i.e., with sands 13, 17, 24 and 25) damage was not nearly severe enough to result in even moderate attrition of the mortar surface, and it seems unlikely that its depth and areal extent would be sufficient to detract, say, from the riding quality of a concrete pavement.

Concerning appearance, again the significance of this type of distress was difficult to evaluate. The number of particle failures which, in the opinion of the observer, detracted from the appearance of the surface either because of their size or because of high color contrast with the cement paste was counted for each mortar. Ratings by

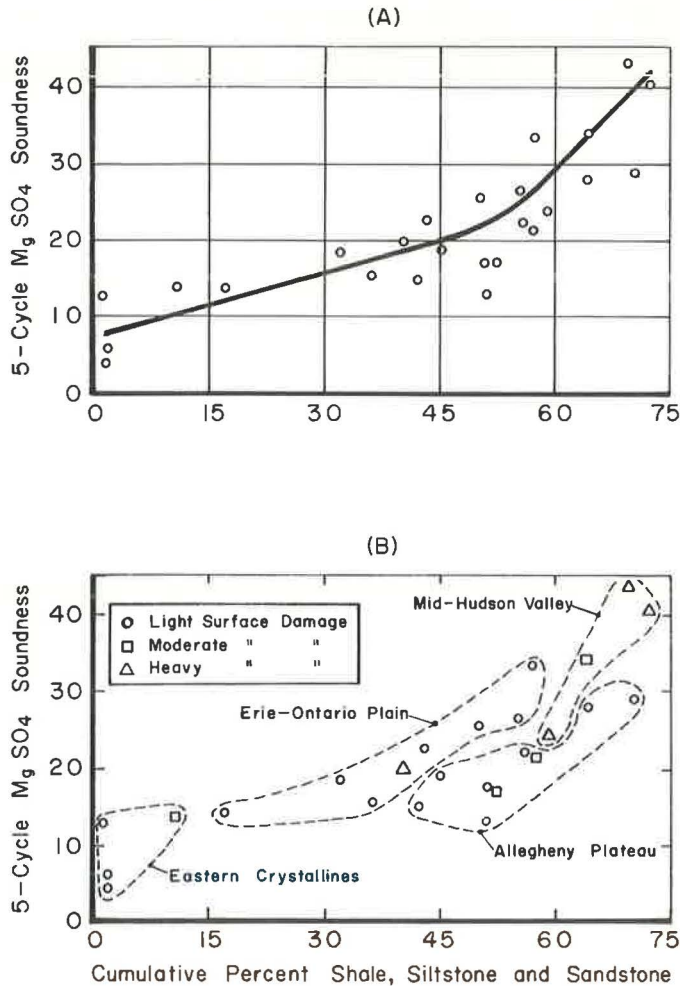


Figure 3. Relationship between sand soundness and its lithologic constituents.

this method correlated well with the surface condition ratings (Table 5). It is probable that the worst cases would detract in an application where appearance was particularly important.

Figure 2 shows the relationship among the surface condition rating of the mortar prisms and the magnesium sulfate soundness and specific gravity distribution of the sands. Mortars exhibiting relatively moderate (rating of 50-100) or heavy (rating of 100-150) surface damage contain sands with magnesium sulfate soundness greater than 34 percent loss or, more commonly, with more than 2.0 percent by weight of material lighter than a specific gravity of 2.40. These data illustrate the well-recognized fact that a gross test such as the one using sodium or magnesium sulfate will frequently not warn the user of the presence of small quantities of material of a deleterious nature.

All individual particles causing "popouts" that were judged to be visually offensive because of size or color contrast were examined in place under a binocular microscope. Representative grains were selected from those examined in each mortar for further examination in thin section under the petrographic microscope. The various materials so identified are described in Table 7 with an estimate of their relative frequency of



occurrence. Two characteristics noted to be common to most of these particles were a relatively high content of clay or hydromica and substantial weathering, both of which would increase the propensity for porosity, softness, lack of strength and, thereby, the observed instability.

Although the magnesium sulfate soundness of the various sands was influenced strongly by the cumulative percent of shale, siltstone and sandstone that they contained (Fig. 3A), popouts on the surface of mortar prisms were associated with shale in about 45 percent of the cases, with siltstone in about 30 percent of the cases but only rarely with sandstone. It is reasonable to infer that this has resulted from the relative preponderance of clay normally found in these rocks. This observation also partially explains why the eight sands which caused the highest incidences of popouts on mortar surfaces were not always the ones with the highest losses in the soundness test. As might be expected, six of the eight were included among the seven sands having together the highest proportion of shale, siltstone and sandstone (greater than 40 percent) and the highest ratios of shale and siltstone to total shale, siltstone and sandstone (greater than 30 percent).

A regional grouping of these same data (Fig. 3B) suggests that although the generality regarding the influence of cumulative percent of shale, siltstone and sandstone on magnesium sulfate soundness may hold true, the exact functional relationship varies somewhat among regions having different geologic histories. This variation apparently results from differences in the character of the particles themselves from region to region as well as from the influence of other materials. The only inconsistency within these groupings is the point represented by the triangle in the zone occupied by sands from the Erie-Ontario Plain. This point represents a sand from the lower Hudson Valley near the Pennsylvania border.

Figure 3B shows that although sands from the Erie-Ontario Plain include about the same range of soundness as sands from the Allegheny Plateau, the former do not cause an appreciable frequency of popouts, whereas the latter, in some instances, do. Should further sampling and testing establish this trend as fact, the knowledge gained could be quite useful in judging acceptability (on the basis of sulfate soundness) in regions such as these where natural sands with low losses in the soundness test are rare.

### Outdoor Exposure Tests

After one winter of exposure to natural weathering in which approximately 70 cycles of freezing and thawing were experienced, prisms similar to those used in the laboratory freeze-thaw test have experienced no loss in dynamic modulus of elasticity (Table 5) and no popouts. The lack of deep-seated deterioration is consistent with results of the laboratory freeze-thaw tests. The absence of surface deterioration undoubtedly reflects the less severe weathering conditions. Annual observation of these specimens will continue.

### Field Performance Surveys

During the summer of 1963, reconnaissance-type surveys were conducted over 1,160 lane-miles of air-entrained concrete pavement and 264 structures incorporating fine aggregate from 37 different sources including 14 from which samples were taken for this study. The sources have produced sand with losses in the magnesium sulfate soundness test of up to about 25 percent. More than half of the concrete examined used sand with soundness losses in excess of 15 percent. The surveys included a detailed inspection of all structures. Pavements were examined while driving the shoulder between 10 and 20 mph with periodic stops for closer observation.

Most of the concrete was in excellent condition. No significant distress was observed that could be related to fine aggregate. Popouts or pits caused by the largest fine aggregate sizes or by smaller sizes where they were close to the surface were observed in some concrete, however, in no instance did they detract noticeably from the appearance of the concrete or from the riding quality of pavements or bridge decks. These field observations are consistent with the laboratory studies. No observations were made of concrete in an application where the effect of deleterious particles on appearance might be more obvious.

### DRYING SHRINKAGE STUDIES

Portland cement paste, which in many respects functions essentially as a hydro-gel, adjusts its bulk volume to changes in the relative humidity of its atmosphere. These adjustments are most apparent when the paste dries from its initially saturated state after mixing and setting, but continue to occur whenever the paste is not in equilibrium with its environment. The unit linear shrinkage of portland cement concrete or mortar during this early period is commonly used as an index of moisture-volume stability. For most applications, high volume stability is a desirable attribute.

The drying shrinkage of concrete or mortar is affected by many factors, chief among which is the unit volume of water (21). The principal effect of aggregate is to provide a rigid internal structure and, thus, to restrain the shrinkage of the paste. It has been shown, however, that aggregates vary in their ability to provide this restraint (15, 21-24). Carlson (21) concluded that their relative compressibility is the most important property in this respect, a view which was later expressed by Pickett (25) in an equation relating shrinkage to the fractional volume and elastic constants of the aggregate.

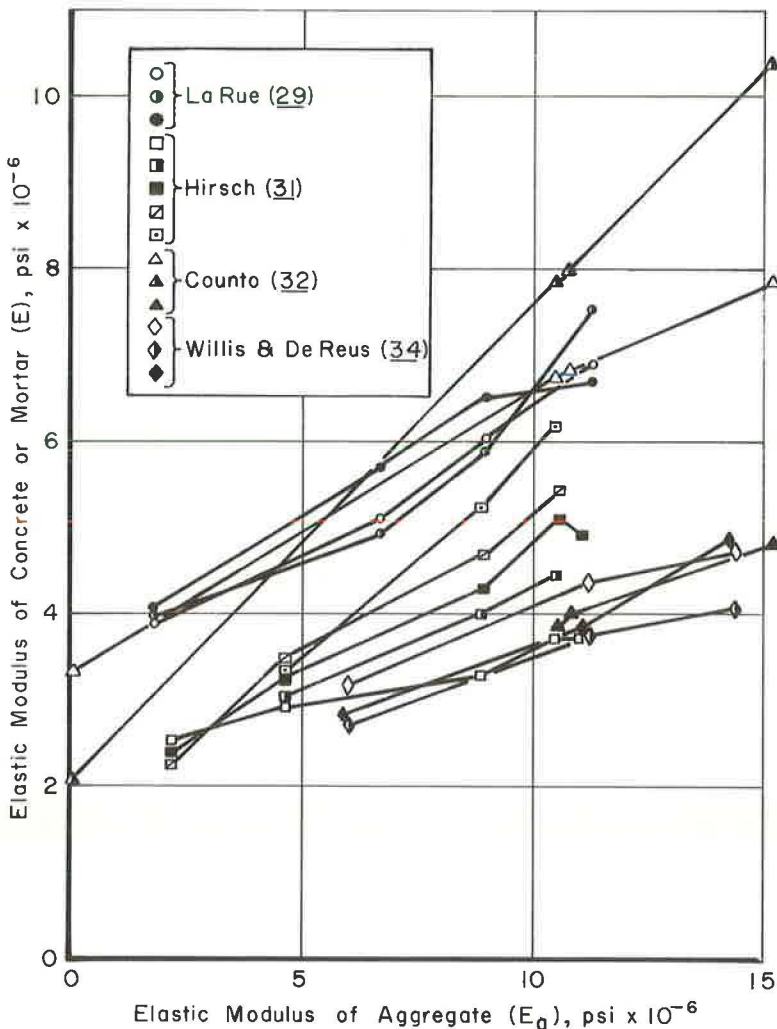


Figure 4. Effect of variations in elastic modulus of aggregate on variations in elastic modulus of concrete or mortar when  $E_m$  and  $V_a$  are constant.

Carlson also suggested that the moisture-volume stability of the aggregate itself is a significant factor. This point was demonstrated by Roper (15) and by Snowdon and Edward (22) in studies of shrinking aggregates and their effect on volume changes in concrete and mortar. Roper, Cox and Erlin (26) stated that "... the moisture-volume instability of the aggregate per se can be significantly more important than the effects of measured elasticity . . . ." This view was supported by Hansen and Nielsen (27) as a result of their extension of Pickett's theory to include, among other things, the effect of aggregate shrinkage.

Hveem and Tremper (12) have concluded that the presence of clay particles in concrete sand, as measured by the sand equivalent test, is a major contributing factor to shrinkage. The results of drying shrinkage tests of mortars made from 248 samples of commercially produced concrete sand yielded a correlation coefficient\* of 0.66 when compared with corresponding measures of sand equivalent. Recognizing the influence of the elastic modulus of the aggregate and its partial dependence on porosity, Hveem and Tremper introduced absorption into their correlation and, thereby raised the coefficient to 0.86. Powers (28) speculated that clay particles associated with aggregate increase shrinkage because they function like cement gel instead of offering restraint, thereby decreasing the effective fractional volume of aggregate.

There is general agreement that aggregate size and gradation affect shrinkage only incidentally as they affect the volumetric proportions of the mix (21, 23, 27).

#### Effect of Elastic Moduli of Sands

The 25 natural sand fine aggregates studied were associated with varying degrees of mortar shrinkage. Some of the mortars shrank more than twice as much as others. As part of the effort to establish the significantly related aggregate properties, elastic moduli of the sands were estimated from the average initial dynamic elastic modulus ( $E_0$ ) of the prisms used in the freeze-thaw test.

Various investigators (29-33) have proposed equations expressing the relationship among the elastic modulus of concrete or mortar ( $E$ ), the elastic modulus ( $E_a$ ) and fractional volume ( $V_a$ ) of the aggregate, and the elastic modulus of the matrix ( $E_m$ ), be it cement paste or mortar. The relationship between  $E$  and  $E_a$  has usually been expressed as a nonlinear function. However, when  $E_m$  and  $V_a$  are constant, as they were in the freeze-thaw prisms, the functional relationship between the two can, for practical purposes, be approximated by a straight line for the range of  $E_a$  normally encountered in concrete aggregate ( $2 - 15 \times 10^6$  psi) without introducing any great error. This is illustrated in Figure 4 by data taken from the literature.

Accordingly,  $E_0$  of each freeze-thaw prism was corrected for the variations in air content ( $A$ ) existing among the different mortars, and the average of the eight resulting values for each mortar was used as an estimate of the relative effective elastic modulus (REEM) of the corresponding sand (Table 8). The corrections were based on equations expressing the average relationship between  $E_0$  and  $A$  derived by the method of least squares from the same data used to calculate the values plotted in Figure 1. Figure 5 shows the derivation of the correction factor.

The high degree of correlation found between REEM and mortar shrinkage is best expressed empirically by a second degree polynomial (Fig. 6). This form is consistent with the nonlinear relationship between the two described by Hansen and Nielson (27). Thus, 83 percent of the variation observed in the drying shrinkage of mortar can be explained by variations among the elastic moduli of the sands. This value corresponds to a correlation coefficient of 0.91. (Strictly speaking, the correlation coefficient has no meaning for a nonlinear fit; it is used here for comparison with other data only.) When the small variations which occurred in the fractional volume of aggregate are taken into consideration, the coefficient of regression is virtually unaffected.

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\*Coefficient of sample linear regression, unless otherwise noted.

TABLE 8  
RELATIVE EFFECTIVE ELASTIC MODULI (REEM)  
OF EXPERIMENTAL SANDS

Sand	Average $E_o$ (psi) <sup>a</sup>	Correction (psi) <sup>b</sup>	REEM (psi)
1	$4.581 \times 10^6$	$-0.099 \times 10^6$	$4.482 \times 10^6$
2	4.562	-0.043	4.519
3	4.412	+0.083	4.495
4	4.036	+0.087	4.123
5	4.215	+0.057	4.272
6	4.180	+0.098	4.278
7	4.143	-0.004	4.139
8	4.185	+0.045	4.230
9	3.816	-0.057	3.759
10	4.080	-0.136	3.944
11	4.051	-0.056	3.995
12	4.043	+0.074	4.117
13	3.921	+0.041	3.962
14	3.723	-0.036	3.687
15	3.917	-0.139	3.778
16	4.068	-0.004	4.064
17	3.897	-0.209	3.688
18	3.975	-0.012	3.963
19	3.919	+0.012	3.931
20	3.974	-0.168	3.806
21	3.939	-0.209	3.730
22	3.874	+0.046	3.920
23	3.814	-0.083	3.731
24	3.678	-0.191	3.487
25	3.662	-0.181	3.481

<sup>a</sup>Average of values for 8 prisms.

<sup>b</sup>All mortars adjusted to 12.0 percent air.

### Effect of Clay

As already noted, other aggregate properties have also been identified with drying shrinkage, specifically, the moisture-volume characteristics of the aggregate themselves and the quantity and type of clay as indicated by the sand equivalent. Interestingly, sand equivalents of the experimental aggregates also correlate with drying shrinkage and to a rather high degree. A coefficient of 0.91 was obtained which is somewhat higher than that reported by Hveem and Tremper (12) for approximately the same range of sand equivalents.

Powers' speculative explanation (28) of the Hveem and Tremper data is a reasonable hypothesis. However, it hardly seems adequate to account for any but minor variations in the shrinkage observed in this investigation when it is recalled that each sand contained only 2 percent by weight of material passing the No. 200 mesh screen, that the proportion of this amount which was of clay size was small and did not vary

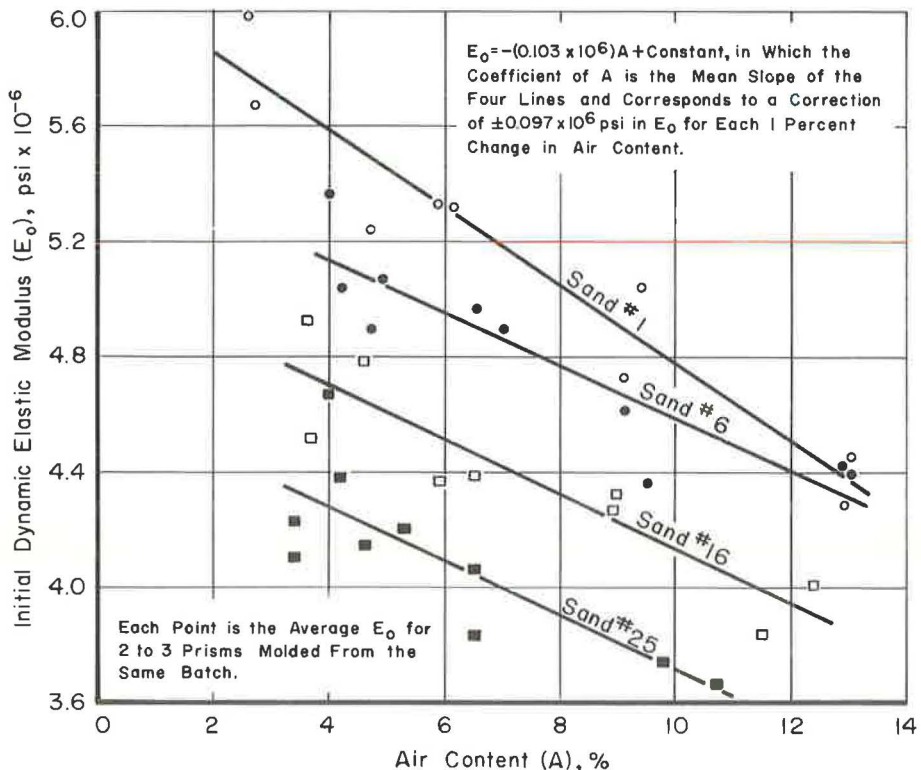


Figure 5. Derivation of the correction factor for variations in air content.

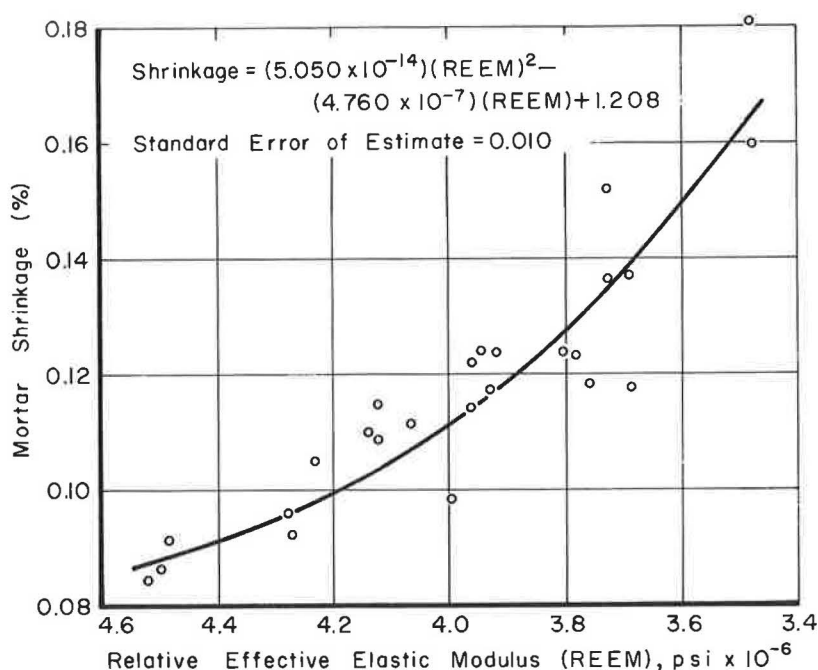


Figure 6. Effect of aggregate elasticity on mortar shrinkage.

TABLE 9  
EFFECT OF MINUS NO. 200 PORTION ON MORTAR SHRINKAGE

Sand	Minus No. 200	Sand Equiv.	S at 56 Days (%)	Average S (%)
3	From Sand 3	94	0.077	0.079
			0.080	
			0.079	
			0.081	
3	From Sand 24	91	0.085	0.082
			0.081	
			0.081	
			0.080	
24	From Sand 24	69	0.161	0.162
			0.160	
			0.163	
			0.162	
24	From Sand 3	68	0.157	0.158
			0.159	
			0.156	
			0.162	

greatly (Table 4), and that the clay minerals identified by X-ray diffraction were nearly all of the same type, illites (Table 3).

The relative contribution of aggregate elasticity and clay content (as measured by sand equivalent) to the observed shrinkage cannot be distinguished by statistical methods alone. This is because the two factors correlate significantly with one another (coefficient of 0.80), that is, sands with low elastic moduli tend also to have low sand equivalents and both, therefore, would be expected to influence shrinkage in the same direction and in unison.

That the observed shrinkage of mortar specimens has been influenced to only a minor degree by variations in clay content is illustrated by the results of supplemental drying



shrinkage tests in which sands 3 and 24 were prepared with their minus No. 200 portions interchanged. These sands are those which, respectively, produced the least and the greatest shrinkage and the highest and lowest sand equivalents. The results are given in Table 9.

The changes in shrinkage accompanying the switching of these fine sizes, if indeed they are significant at all, are in the proper direction and are of the same order of magnitude as might be expected from small adjustments in aggregate volume associated with the probable differences in clay content between the two sands (Table 4), as suggested by Powers (28). Accordingly, it may be concluded that the difference in drying shrinkage between mortars made with sands 3 and 24, and probably the observed variation in shrinkage among all of the mortars, has not been greatly affected by variations in the type or quantity of clay present in the minus No. 200 size fraction. This is in spite of a considerable variation in sand equivalent among the experimental aggregates.

Table 9 is of interest also because it shows that the sand equivalents of sands 3 and 24 were also virtually unaffected by the interchange of fines. A similar exchange between sands 3 and 25 and between sands 2 and 25, likewise, had no significant effect on sand equivalent. It appears, therefore, that while the observed variations in the sand equivalent of the experimental sands do reflect variations in shrinkage of the corresponding mortars, they do not represent significant variations in the type or amount of clay present in the sands at the time the mortars were mixed. Rather, they reflect some other characteristic of the sands which coincides to a high degree with elastic modulus.

A limited number of tests with sands 24 and 25 prepared without the minus No. 200 portion show that fines generated during the shaking portion of the sand equivalent test produce a value of sand equivalent that is only slightly greater than that produced when the minus No. 200 material is included, and that the sand equivalent is higher or lower than this value when the shaking action is continued for a fewer or a greater number of strokes (i.e., for 5 strokes and for 180 strokes). A similar experiment with sands 2 and 3 produced only a small variation in sand equivalent for the same variation in the number of strokes. These results suggest that the variation observed in sand equivalent for the experimental aggregates was caused principally by the tendency of the different aggregates to generate varying amounts of fines during the sand equivalent test and that this tendency generally increases as the elastic modulus of the sand decreases, thus, accounting for the high correlation observed between sand equivalent and drying shrinkage.

### Effect of Aggregate Shrinkage

While no direct measurements were made of the moisture volume characteristics of the experimental sands, an indirect indication of this effect should be obtainable from the shrinkage data by using the equations of Hansen and Nielsen (27), and the estimates of REEM previously described. If the unrestrained linear shrinkage of the paste,  $S_p$ , the linear shrinkage of the aggregate,  $S_a$ , and  $E_a$  are known, the dimensionless parameter  $E_a/E_m$  can be calculated. If  $S_a/S_p$  is assumed to be zero,  $E_a/E_m$  thus determined, is an apparent relative elastic modulus reflecting the combined effect of both the elastic modulus and the shrinkage of the aggregate and, in fact, any other aggregate-related factor affecting shrinkage.

In connection with the mortar shrinkage tests, no measurements were made of the unrestrained shrinkage of the paste. Hansen and Nielsen (27) have shown that the shrinkage of cement mortar and concrete made with different quantities and types of high-quality aggregate is reasonably well accounted for by assuming  $S_a/S_p = 0$  and  $E_a/E_m = 10$ . Their equations also show that as  $E_a/E_m$  exceeds 10, the additional restraint offered by aggregate particles diminishes rapidly. Accordingly,  $S_p$  was estimated by assuming a value of  $E_a/E_m = 10$  for the mortar exhibiting the least shrinkage and the resulting value was used to calculate the parameter  $E_a/E_m$  for the remaining sands under the assumption that  $S_a = 0$  for all sands.

In Figure 7, the resulting apparent relative elastic moduli,  $E_a/E_m$ , are plotted against REEM estimated previously from values of  $E_o$ . These parameters represent

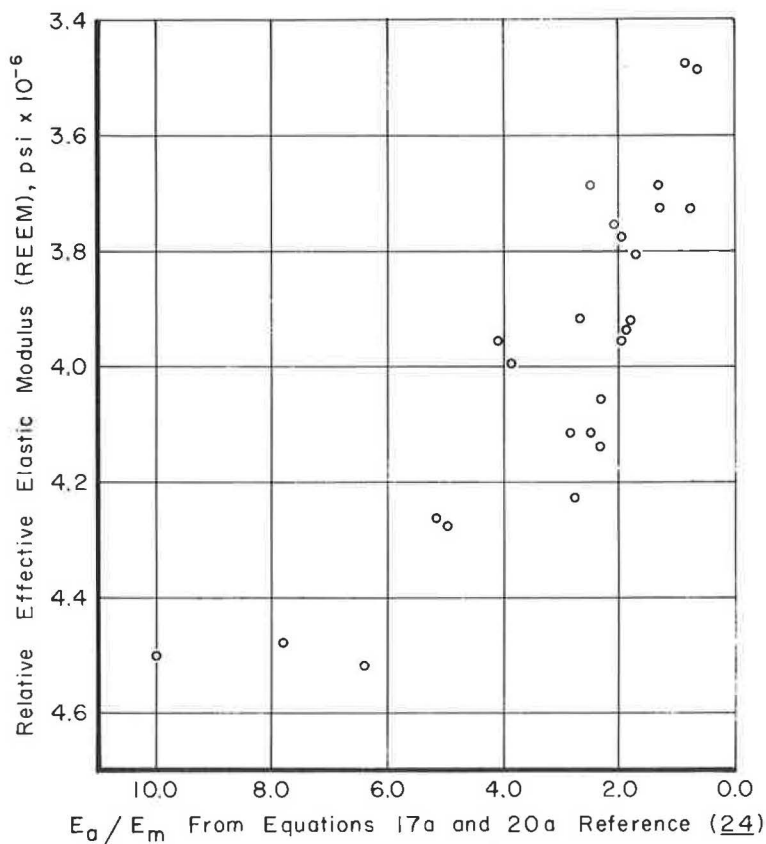


Figure 7. Relative effective elastic modulus (REEM) vs  $E_a/E_m$ .

two sets of independent estimates of the elastic moduli of the experimental sands, and in the absence of other aggregate-related factors affecting shrinkage, would be expected to be linearly related. The correlation between the two is only slightly less (coefficient of 0.84) than that obtained between REEM and mortar shrinkage itself which suggests that if shrinkage of the sands has been a significant contributory factor, variations in aggregate shrinkage have probably coincided in large measure with variations in elastic modulus. If  $E_a/E_m$  of the low shrinkage mortar is assumed to be less than 10, which it may be because of the low water-cement ratio paste, the linearity of the relationship is improved over that shown in Figure 7.

#### WATER REQUIREMENT

It has been shown (14, 35) that when concrete is proportioned to a predetermined fineness modulus of combined aggregate by adjusting the relative volumes of fine and coarse aggregate according to the fineness modulus of the sand (the fineness modulus method), the grading of the sand has no consistent effect on the water requirement of the mix. Under these conditions, water requirement is influenced largely by variations in most of the important concrete properties.

The variations in water requirement observed in this study are only partially predicted by the estimates of specific surface area and sphericity (correlation coefficients of 0.49 and 0.63, respectively). The introduction of roundness index improved the correlation with sphericity by only a small amount. Even allowing for substantial error in the estimates of surface area and sphericity, it would seem that variations in surface texture among the different sands exercise a considerable influence on variations in water requirement, probably equal to that of particle shape.



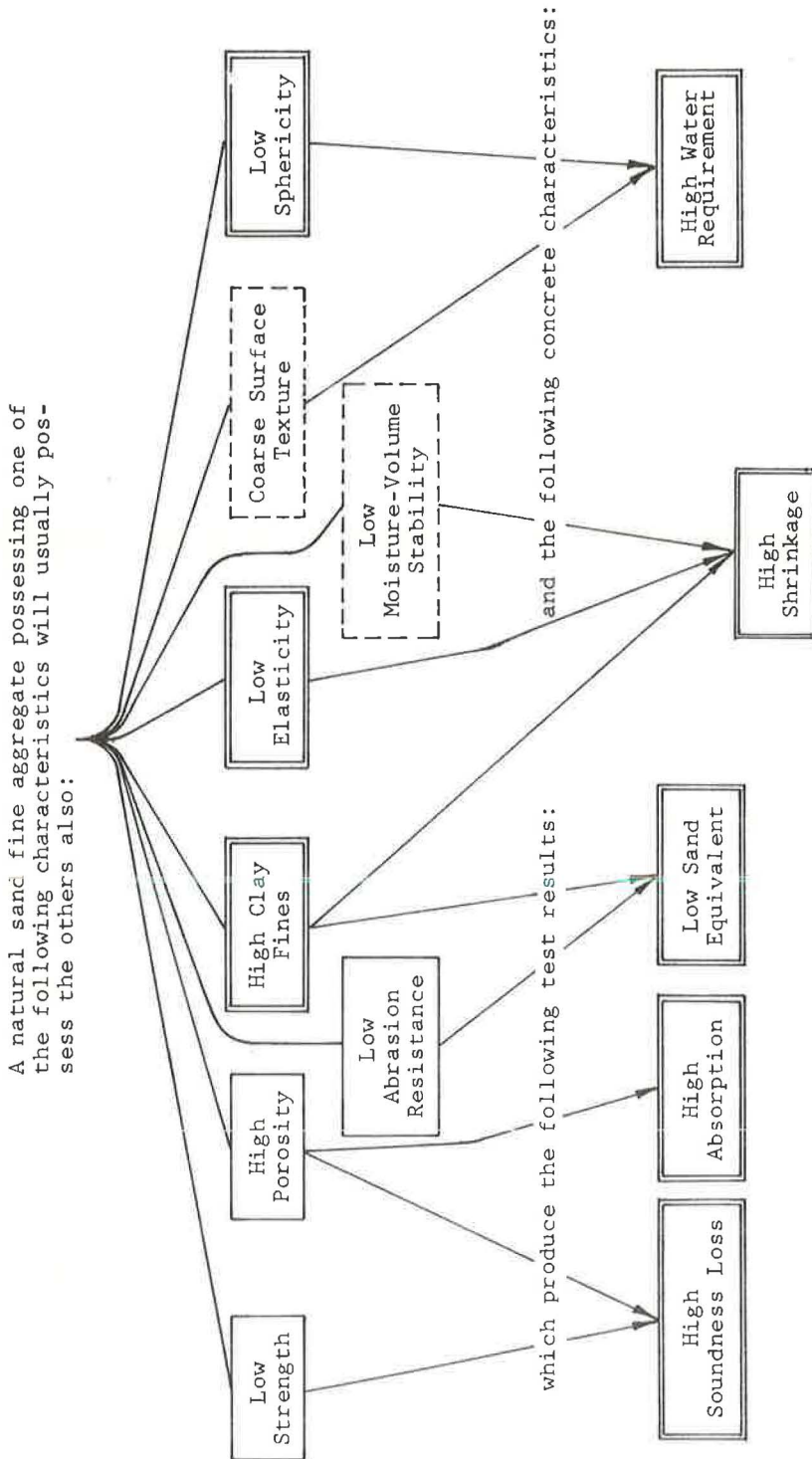


Figure 8. Interrelationship among measured factors.

## INTERRELATIONSHIP AMONG VARIOUS FINE AGGREGATE CHARACTERISTICS

The high degree of mutual correlation among sand equivalent, REEM and mortar shrinkage has already been noted. If the experimental sands are arranged in order of increasing elastic moduli, they are also arranged in approximate order of increasing sand equivalent and decreasing mortar shrinkage. Many of the other measured characteristics of the experimental sands and their corresponding mortars also produce nearly this same array. This observation led to the generality stated in Figure 8, the validity of which, for the experimental sands, is supported by the following coefficients of linear correlation with magnesium sulfate salt soundness:

Percent clay-sized material in minus No. 200	0.59
Elastic modulus of sand	0.85
Sphericity	0.74
Flatness	0.66
Elongation	0.59
Absorption	0.82
Sand equivalent	0.82
Mortar shrinkage	0.92
Water requirement (mortar penetration)	0.78

The foregoing discussion leads immediately to at least one significant conclusion. Results of the magnesium sulfate soundness test are a meaningful index to the relative quality of these particular experimental sands. This is not because the test simulates the growth of ice crystals in aggregate pores (which is not the proper mechanism anyway) or, in fact, has anything to do with freezing at all, but because it is a degradation test which empirically correlates to a high degree with two significant properties of the aggregate—modulus of elasticity and water requirement. Apparently, rock materials which abrade to flat and elongated particles with rough surfaces usually have low elastic moduli, and, by virtue of a high porosity and low strength, break down readily in the salt soundness test.

A sand with a high loss in the sulfate soundness test may contribute to low volume stability in concrete or mortar because of the combined effect of a lower elastic modulus, higher water requirement, more clay in the fine size range, and possibly lower moisture volume stability than a sand with a lower loss in the test (Fig. 8).

Criticism which has properly been leveled against the sulfate soundness test, on theoretical grounds (19, 36), derives from the fact that the test does not measure or reflect those physical characteristics of aggregates involved in the mechanisms of freezing in concrete. That these mechanisms do not completely apply to fine aggregates because of their small particle size partially nullifies this criticism for these materials.

## CONCLUSIONS AND PRACTICAL IMPLICATIONS

The more important conclusions arising from the study of New York natural sand fine aggregates are the following.

1. Extreme variations in the quality of fine aggregate, per se, have an undetectable influence on variations in the freeze-thaw durability of air-entrained concrete or mortar. Where five-cycle magnesium sulfate soundness is the frame of reference, "extreme" includes sands with losses of approximately 40 percent.

2. Different fine aggregates cause varying numbers of popouts on the surface of air-entrained mortar prisms subjected to laboratory freezing and thawing, the worst of which probably detract from the appearance of concrete in some applications. This form of distress was consistently associated with sands having a magnesium sulfate soundness in excess of 34 percent and/or with more than 2.0 percent by weight of material lighter than a specific gravity of 2.40.

3. Most deleterious particles identified on the surface of mortar prisms contained a high proportion of clay or hydromica and were substantially weathered. Seventy-five percent of them were either shale or siltstone.

4. Variations in the drying shrinkage of mortars coincided to a high degree with variations in the effective elastic moduli of the experimental sands.

5. Variations in the clay content of the experimental sands had only a very minor effect on mortar shrinkage in spite of a large variation in their sand equivalent. The sand equivalent test was found to measure, primarily, the tendency of the different sands to generate varying amounts of fines during the sand equivalent test itself. This tendency appears to coincide with a decrease in effective elastic modulus, thus accounting for the high correlation between sand equivalent and mortar shrinkage.

6. The magnesium sulfate soundness of the experimental sands correlates to a high degree with many of the other measured aggregate and mortar factors. In order of decreasing correlation, they are mortar shrinkage, sand elasticity, absorption, sand equivalent, water requirement, sphericity, and percent clay-sized material in the minus No. 200 portion. It probably also correlates well with the abrasion resistance of the sand.

There are several practical implications of these conclusions.

1. Results of the magnesium sulfate soundness test correlate well with several significant properties of some natural sand fine aggregates: modulus of elasticity and water requirement. In geographic regions where these correlations exist, results of the test provide a meaningful index of relative quality.

2. Results of the magnesium sulfate soundness test bear little or no relationship to the effect of fine aggregate, itself, on the frost resistance of concrete. This is because variations in fine aggregates, themselves, have little effect in this respect. Paragraph 5(c) of ASTM Designation: C 33, "Standard Specifications for Concrete Aggregates," which allows acceptance of a fine aggregate which fails the salt soundness test if it gives satisfactory results in concrete subject to freezing and thawing tests, may be misleading because it seeks an effect which probably does not exist (lack of freeze-thaw durability) and overlooks one that may (lowered volume stability with changes in moisture content).

3. A fine aggregate acceptance test based on the resonance frequency of a standard mortar prism expressed as a percent of the resonance frequency of a similar mortar prism mixed to the same volumetric proportions with the same cement but with a standard sand (graded Ottawa, perhaps), would provide a more direct method of testing for the significant property of elastic modulus and may be less time-consuming than the sulfate soundness test.

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# Experiments With Alkali-Silica Reactive Constituents of Sand-Gravel Aggregate

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•CONCRETE made with a certain type of natural aggregate, known as sand-gravel aggregate, from the Kansas-Nebraska area and adjoining states is known to undergo a characteristic cement-aggregate reaction resulting in expansion, cracking, and early deterioration. Such aggregates can be recognized by petrographic examination. Investigations by the Bureau of Reclamation and other organizations have shown that satisfactory concrete can be made with sand-gravel aggregates if a low-alkali cement and a suitable crushed limestone coarse aggregate are used.

Although investigators have cited many factors as involved in this cement-aggregate reaction, the cause of the deterioration is still not well understood. Possible explanations ascribe it to (a) an essentially alkali-silica reaction, (b) an alkali-silica reaction aggravated by various other factors, (c) a variety of other causes with a minor, if any, contribution from alkali-silica reaction, and (d) an as yet undefined chemical or physical phenomenon.

The term "cement-aggregate reaction" as used in this paper refers to the processes responsible for the expansive cracking and deterioration of concrete experienced in the use of sand-gravel aggregate in the Kansas-Nebraska area. The term "alkali-silica reaction" is restricted to the alkali-silica reaction process involving the known potentially deleteriously alkali-reactive siliceous aggregate constituents (including opal, chalcedony, certain microcrystalline and cryptocrystalline forms of quartz, tridymite, cristobalite, and acidic to intermediate volcanic glass).

Previous Bureau of Reclamation investigations (1) have concluded in part:

1. Alkali-aggregate reaction is a major cause of expansive cracking and deterioration of concrete made with the generally highly reactive sand-gravel aggregates from the Kansas-Nebraska area.
2. Percent expansion increases sharply with both moderately and highly reactive aggregates when the alkali content of the cement, computed as  $\text{Na}_2\text{O}$  equivalent, reaches about 0.5 percent.
3. Drying shrinkage is another cause of cracking . . . some of the deterioration can be attributed to enlargement of cracks by freezing and thawing action.
4. A coarse-ground cement is less likely to produce deleterious expansion in field concrete than a fine-ground cement.

Literature on this subject (2 through 9) has mentioned a variety of other factors, including the following.

1. Effects of cement brand and type;
2. Expansion considered as a function of alkali content of cement;
3. Expansion considered as not a function of cement alkali content;
4. Release of alkalies from cement;
5. Release of alkalies from aggregate;
6. Quantity and rate of release of calcium hydroxide during hydration of cement;
7. Delayed hydration of free magnesia in cement;
8. Effect of internal fracturing on volume stability of concrete;

9. Surface texture and bonding characteristics of aggregate;
10. Effects of feldspar in aggregate due to low coefficient of thermal expansion of feldspar, poor bond of smooth surface grains with cement, and high rigidity or low compressibility; and
11. An as yet undefined chemical or physical phenomenon.

Cement-aggregate reaction would, therefore, appear to be due to a combination of factors, rather than to a single causative factor. Alkali-silica reaction is likely to be a major factor in some sand-gravel aggregates; however, not all sand gravels are known to be alkali-silica reactive.

## OBJECTIVE OF PILOT EXPERIMENTS

Since cement-aggregate reaction is associated with a sand-gravel aggregate, the Bureau of Reclamation has undertaken a laboratory test program to study the characteristics of sand-gravel aggregates, and to determine what effect the various rock types and mineral constituents of the aggregate have on the cement-aggregate reaction.

Pilot experiments were conducted to determine the effects of alkali-silica reactive aggregate constituents on cement-aggregate reaction, as measured by mortar bar expansion tests. The sealed moist-storage mortar bar expansion test is used as a measure of alkali-silica reactivity, and the Conrow cycle mortar bar expansion test is used as a measure of cement-aggregate reaction. Both mortar bar expansion tests were made with the natural Republican River aggregate, and with Republican River aggregate processed to remove the alkali-silica reactive constituents. As a control, both mortar bar tests were made with a lithologically somewhat similar nonalkali reactive South Platte River aggregate. Tests were made with the natural aggregate, and with the aggregate blended with the alkali-silica reactive constituents removed from the Republican River aggregate.

## AGGREGATES

### Republican River Aggregate

Republican River aggregate from a source near Indianola, Nebraska, is a typical alkali-silica reactive sand-gravel. This aggregate, used in the past in many Bureau laboratory studies (1), contains alkali-silica reactive particles, has a poor service record, and produces reactive expansion in both the sealed moist-storage and Conrow cycle mortar bar expansion tests.

The aggregate is composed chiefly of individual grains of feldspar and quartz, with some granite, and minor limestones and ferruginous concretions. The fine sand also contains small amounts of hornblende, magnetite, garnet, mica, and, in the pan-size, minor amounts of acidic volcanic glass, chert, and opal. A few particles are partially coated with thin, firmly bonded calcium carbonate, and very few are coated with opal. The alkali-silica reactive constituents—limestones and sandy limestones containing opal, cherts, andesites, and particles of opal and acidic volcanic glass—constitute about 6 percent of the sand. A particle count of the sand retained on the No. 8 sieve showed 35 percent feldspar, 33 percent quartz, 23 percent granite, 7 percent alkali-silica reactive particles, and 2 percent miscellaneous minor constituents.

## SOUTH PLATTE RIVER AGGREGATE

The South Platte River aggregate, from a source near Denver, Colorado, is not considered a sand-gravel aggregate, but bears many similarities to sand-gravels. The chief constituents are similar to those of Republican River aggregates. However, the South Platte River aggregate does not contain opaline particles, is not alkali-reactive, and has a generally satisfactory service record. The aggregate does not produce reactive expansion in either the sealed moist-storage or Conrow cycle mortar bar expansion tests.

The South Platte River aggregate is composed chiefly of granite and coarse-grained quartz and feldspar particles, with minor gneiss, schist, sandstone, devitrified andesite



and rhyolite, shale, and ferruginous particles. The coarse sand contains about 75 percent quartz and feldspar particles in approximately equal amounts. The finer sand contains increasing amounts of quartz and lesser feldspar, with minor detrital minerals that include garnet, mica, and magnetite. Glassy rhyolite is the only alkali-reactive constituent observed, and constitutes only trace amounts, much less than 1 percent in the sand. No opal was detected.

### TEST PROCEDURES

The sealed moist-storage mortar bar expansion test is a routine test employed by the Bureau to test for alkali-reactivity of aggregates. The Bureau's procedure for the test is similar to ASTM Method C-227-61T. Linear expansion of the mortar bars in excess of 0.2 percent in 1 yr indicates a deleteriously alkali-reactive aggregate; expansion of 0.1 to 0.2 percent in 1 yr is potentially deleteriously alkali-reactive; and an expansion of 0.04 to 0.1 percent is regarded with suspicion concerning alkali-reactivity.

The Conrow cycle mortar bar expansion test was devised for the purpose of testing sand-gravel type aggregates in the Kansas-Nebraska area. The Bureau's procedure for the test is similar to ASTM Method C-342-61T. The expansion criteria used by the Bureau are the same as those used for the sealed moist-storage expansion test.

The methods used to remove the alkali-silica reactive particles from the Republican River aggregate included handpicking, hydraulic jigging, heavy liquid separation, and an acid treatment and washing procedure. The alkali-reactive particles are chiefly opaline particles of low specific gravity, and are generally amenable to separation from the denser constituents of the aggregate, chiefly quartz and feldspar. Opal occurs occasionally in limestones and calcareous sandstones. These particles can be decomposed in dilute hydrochloric acid, and the resultant fines, which will include the opal, can be removed by washing.

The weight loss from processing the samples was recorded. The material removed by processing was collected and blended with the South Platte River aggregate in amounts proportional to the amounts removed from the Republican River aggregate.

The mortar bar tests with the processed Republican River and the blended South Platte River aggregates differed from the routine Bureau procedure in two ways:

1. Only one bar for each mixture was used, instead of the customary three bars.
2. The grading was a straight 19 percent by weight retained on sieves No. 8, 16, 30, 50, and 100 plus 5 percent of material passing the No. 100 sieve.

Only one bar for each mixture could be made because of the time spent in processing the Republican River aggregate to remove the alkali-reactive constituents.

### RESULTS OF MORTAR BAR EXPERIMENTS

#### Sealed Moist-Storage Mortar Bar Expansion Test

The natural Republican River aggregate produces deleterious expansion with high- and medium-alkali cements (Table 1). A summary of previous tests, given in Table 2,

TABLE 1  
SEALED MOIST-STORAGE MORTAR BAR EXPANSION TEST  
Republican River Sand

Cement	Alkali Na <sub>2</sub> O Equiv. (%)	Expansion (%)			
		Processed Sand <sup>a</sup>		Natural Sand <sup>b</sup>	
		1 Yr	2 Yr	1 Yr	2 Yr
7488	1.19	0.054	0.070	0.303	0.337
M-3100	0.50	0.045	0.071	0.152	—
M-3727	0.41	—	—	0.165	0.263
9406	0.16	0.013	0.013	—	—

<sup>a</sup>Alkali-reactive particles removed.

<sup>b</sup>Contains alkali-reactive particles.

TABLE 2  
SUMMARY OF PREVIOUS SEALED  
MOIST-STORAGE MORTAR BAR  
EXPANSION TESTS  
Republican River Sand

Cement	Alkali Na <sub>2</sub> O Equiv. (%)	Expansion (%)	
		1 Yr	2 Yr
M-3562	1.23	0.215	0.282
M-3562	1.23	0.183	0.248
M-3562	1.23	0.168	0.200
M-3562	1.23	0.190	0.207
7488	1.19	0.235	0.250
7488	1.19	0.254	—
7488	1.19	0.303	0.337
7488	1.19	0.285	0.327
7488	1.19	0.276	0.286
7488	1.19	0.264	—
7488	1.19	0.264	0.278
7488	1.19	0.307	0.337
7488	1.19	0.270	—
7488	1.19	0.287	—
7488	1.19	0.292	—
7488	1.19	0.308	0.334
7488	1.19	0.228	0.260
7488	1.19	0.225	0.273
7488	1.19	0.293	0.321
7488	1.19	0.342	0.365
7488	1.19	0.251	0.267
7488	1.19	0.291	0.341
7488	1.19	0.176	0.219
7488	1.19	0.262	0.272
M-3527	0.92	0.222	—
M-3052	0.87	0.277	—
2448	0.58	0.340	0.440
2448	0.58	0.330	0.470
2448	0.58	0.303	0.406
2448	0.58	0.286	0.382
M-3100	0.50	0.173	—
M-3100	0.50	0.152	—
8518	0.49	0.058	0.081
M-3727	0.41	0.165	0.263
2735	0.13	0.017	0.026

shows this aggregate to be fairly consistent in producing deleterious expansion with high- and medium-alkali cement (one exception noted).

Removal of the alkali-reactive particles from the Republican River aggregate reduced expansion to within safe test limits (Table 1). The maximum expansion recorded in this series of tests was 0.071 percent at 2 yr, indicating only a mild expansion.

The natural South Platte River aggregate does not produce deleterious expansion with high-, medium-, or low-alkali cements (Table 3). Blending the alkali-reactive particles removed from the Republican River aggregate produced less definitive results, but did show some increase in expansion over that obtained with the natural South Platte River aggregate (Table 3). Results were most pronounced with the mixture using a medium-alkali cement, which showed an expansion of 0.110 percent at 1 yr.

#### Conrow Cycle Mortar Bar Expansion Test

The natural Republican River aggregate produces deleterious expansion with high-, medium-, and low-alkali cements (Table 4). A summary of previous tests shows that the natural Republican River aggregate consistently produces deleterious expansion with high-, medium-, and low-alkali cements (Table 5).

The processed Republican River aggregate (alkali-reactive constituents removed) showed some anomalous results, although

TABLE 3  
SEALED MOIST-STORAGE MORTAR BAR EXPANSION TEST  
South Platte River Aggregate

Cement	Alkali Na <sub>2</sub> O Equiv. (%)	Expansion (%)					
		Blended Sand <sup>a</sup>		Natural Sand <sup>b</sup>		Natural Crushed Gravel	
		1 Yr	2 Yr	1 Yr	2 Yr	1 Yr	2 Yr
7488	1.19	0.067	0.081	0.031	0.037	0.028	0.029
				0.032 <sup>c</sup>	0.040	0.032 <sup>c</sup>	0.037
				0.034 <sup>d</sup>	0.044	0.030 <sup>d</sup>	0.034
				0.020	0.023	0.023	0.022
M-3100	0.50	0.110	0.122	0.010	0.008	0.008	0.008
M-3727	0.41	—	—	—	—	—	—
9406	0.16	0.012	0.032	—	—	—	—

<sup>a</sup>Contains alkali-reactive particles.

<sup>b</sup>No alkali-reactive particles.

<sup>c</sup>Fifty percent of aggregate replaced with innocuous quartz sand.

<sup>d</sup>Seventy-five percent of aggregate replaced with innocuous quartz sand.

TABLE 4  
CONROW CYCLE MORTAR BAR EXPANSION TEST  
Republican River Sand

Cement	Alkali Na <sub>2</sub> O Equiv. (%)	Expansion (%)			
		Processed Sand <sup>a</sup>		Natural Sand <sup>b</sup>	
		1 Yr	2 Yr	1 Yr	2 Yr
7488	1.19	0.056	0.074	0.729	0.754
M-3100	0.50	0.307	0.336	0.753	0.770
M-3727	0.41	—	—	0.206	—
9406	0.16	0.215	0.248	0.328	0.371

<sup>a</sup>Alkali-reactive particles removed.

<sup>b</sup>Contains alkali-reactive particles. See Table 5 for additional test results with these and other cements.

TABLE 5  
SUMMARY OF PREVIOUS CONROW  
CYCLE MORTAR BAR  
EXPANSION TESTS  
Republican River Sand

Cement	Alkali Na <sub>2</sub> O Equiv. (%)	Expansion (%)	
		1 Yr	2 Yr
7488	1.19	0.729	0.754
7488	1.19	0.550	—
M-2595	0.67	0.984	0.993
M-3100	0.50	0.753	0.770
M-3100	0.50	0.683	—
M-3100	0.50	0.500	—
M-3100	0.50	0.442	—
M-3100	0.50	0.763	—
M-3727	0.41	0.206	—
M-2991	0.44	1.208	1.036
9406	0.16	0.328	0.371
9406	0.16	0.194	—
9406	0.16	0.172	—
9406	0.16	0.191	—
2735	0.13	0.874	0.884
2735	0.13	0.514	—
2735	0.13	0.403	—

in all cases the expansion was significantly lower than the corresponding expansion obtained with natural Republican River aggregate. The expansions obtained with medium- and low-alkali cements were still sufficient to indicate deleterious expansion according to the test criteria. The high-alkali cement did not produce deleterious expansion.

The natural South Platte River aggregate does not show deleterious expansion in the Conrow cycle test (Table 6). Mortar bar tests with the high-alkali cement were not made, but would not be expected to show significant expansion.

The blended South Platte River aggregate (containing the alkali-reactive particles removed from the Republican River aggregate) showed deleterious expansion in the Conrow cycle test with medium- and high-alkali cement (Table 6). The low-alkali cement did not produce significant expansion.

TABLE 6  
CONROW CYCLE MORTAR BAR EXPANSION TEST  
South Platte River Aggregate

Cement	Alkali Na <sub>2</sub> O Equiv. (%)	Expansion (%)					
		Blended Sand <sup>a</sup>		Natural Sand <sup>b</sup>		Crushed Natural Gravel	
		1 Yr	2 Yr	1 Yr	2 Yr	1 Yr	2 Yr
7488	1.19	0.478	0.540	—	—	—	—
M-3100	0.50	0.201	0.229	0.082	—	0.022	—
M-3727	0.41	—	—	0.022	—	0.014	—
9406	0.17	0.024	0.027	0.021	—	0.016	—

<sup>a</sup>Alkali-reactive particles added.

<sup>b</sup>Contains no alkali-reactive particles.

TABLE 7  
SUMMARY OF CHEMICAL COMPOSITION OF CEMENTS

Cement No. and Type	3562	7488	M-3257	M-3052	M-2595	2448	M-3100	8518	M-2991	M-3727	9406	2735
Chemical Analysis	High Alkali	High Alkali	High Alkali	High Alkali	High Alkali	Medium Alkali	Medium Alkali	Medium Alkali	Medium Alkali	Medium Alkali	Low Alkali	Low Alkali
Total alkalis as												
Na <sub>2</sub> O:	1.23	1.19	0.918	0.87	0.67	0.58	0.50	0.49	0.439	0.41	0.16	0.13
Na <sub>2</sub> O	1.20	1.16	0.26	0.84	0.27	0.30	0.23	0.22	0.13	0.07	0.03	0.04
K <sub>2</sub> O	0.04	0.04	1.00	0.04	0.61	0.42	0.41	0.39	0.47	0.51	0.20	0.14
MgO	1.51	1.58	3.77	1.95	1.50	1.36	2.69	1.11	1.64	1.0	1.68	1.30
CaO	63.42	63.85	62.05	62.91	62.86	64.46	62.86	63.88	63.46	—	62.89	64.78
SiO <sub>2</sub>	21.39	21.66	21.84	21.63	21.60	23.16	22.72	21.72	22.10	22.2	22.13	22.00
Al <sub>2</sub> O <sub>3</sub>	4.78	5.58	5.31	5.64	5.11	4.39	4.70	5.76	5.16	4.7	5.88	4.52
Fe <sub>2</sub> O <sub>3</sub>	4.08	2.54	2.33	2.89	4.85	3.84	3.28	4.43	4.44	4.1	4.28	3.81
SO <sub>3</sub>	1.67	2.10	2.37	2.72	1.81	1.22	1.86	1.35	1.72	1.9	2.10	1.60
Free lime	0.33	—	—	—	—	0.22	—	—	—	—	—	0.44
Compound composition (%):												
C <sub>3</sub> S	52.74	48.1	40.79	41.09	45.3	46.9	41.60	45.99	44.39	46.0	36.1	54.2
C <sub>2</sub> S	21.56	25.9	31.93	30.51	27.9	31.1	33.84	27.65	29.96	28.0	36.2	22.3
C <sub>3</sub> A	5.75	10.5	10.13	10.07	5.3	5.1	6.91	7.74	6.17	6.0	8.3	5.5
C <sub>4</sub> AF	12.40	7.7	7.08	8.79	14.7	11.7	9.97	13.47	13.50	12.0	13.0	11.6
CaSO <sub>4</sub>	2.84	3.6	4.03	4.63	3.1	2.1	3.16	2.30	2.92	—	3.6	2.70
Mn <sub>2</sub> O <sub>3</sub>	0.19	—	—	—	—	0.06	—	0.27	—	—	—	0.07
TiO <sub>2</sub>	0.26	—	—	—	—	0.25	—	—	—	—	—	0.32
P <sub>2</sub> O <sub>5</sub>	0.10	—	—	—	—	0.20	—	—	—	—	—	0.12

## DISCUSSION

Sand-gravel aggregates from various sources in the Kansas-Nebraska area tested by the Bureau range from highly alkali-reactive to essentially nonalkali-reactive, but generally are alkali-reactive. So far as is known, no studies have been made to determine if the degree of distress in field concrete, or in laboratory concrete, or mortar bars, correlates with the alkali-reactivity of the aggregate. The published literature on the sand-gravel problem is of little assistance, as most references give little, if any, attention to the composition and alkali-reactivity of the aggregate.

Petrographic examination of alkali-reactive sand-gravels has shown the alkali-reactive constituents to consist generally of a very small and variable amount of opaline particles, and also generally small and variable amounts of less reactive chert, volcanic glass, and acidic volcanic rocks.

The various sand-gravels tested by the Bureau usually produce deleterious expansion in the Conrow cycle mortar bar expansion test, but do not always show a deleterious expansion with the sealed moist-storage expansion test. For this reason, the Bureau employs the Conrow cycle test for testing sand-gravel aggregates.

The sealed moist-storage mortar bar expansion tests of Republican River aggregate fairly consistently show deleterious expansion with high- and medium-alkali cements. Deleterious expansions were obtained with two cements having alkali contents as low as 0.41 percent  $\text{Na}_2\text{O}$  equivalent (Tables 1 and 2). Cements with very low alkali contents (0.16 and 0.13 percent  $\text{Na}_2\text{O}$  equivalent) have not produced deleterious expansion. The expansions obtained do not show a linear relationship with alkali content, but do tend to show the sealed moist-storage test as an indicator of alkali-silica reaction. These test results on Republican River aggregate are similar to the experience with tests of other aggregates and mixtures containing small amounts of opal.

The Conrow cycle mortar bar expansion test appears to be fairly reliable in testing sand-gravel aggregates for deleterious expansion. The test produces excessive expansion with highly alkali-reactive sand-gravels, such as the Republican River aggregate under investigation here, and also with much less alkali-reactive sand-gravels from other sources. The Conrow cycle test is sensitive to alkali-silica reaction: the removal of the alkali-silica reactive particles from the Republican River aggregate reduced expansion in amounts ranging from 0.1 to 0.7 percent with the various cements. As a rough approximation, the data suggest that the alkali-silica reaction is responsible for up to about one-half of the expansion in the Conrow test. Conversely, the addition of alkali-reactive particles to the essentially inert South Platte River aggregate produced expansion in the Conrow test.

There are some questions regarding the deleterious expansion in the Conrow cycle test with essentially nonalkali-reactive sand-gravel aggregates, such as the processed Republican River aggregate used in this test. Is the expansion due to the presence of opal, occurring in such small amounts as to be either undetected by petrographic examination, or regarded as insignificant and indicated as innocuous by lack of expansion in the sealed moist-storage test? Or is the reaction due to some other reaction which apparently does not involve the alkali-silica reactive constituents, i.e., the hypothetical as yet unknown and undefined process different from alkali-silica reaction? Furthermore, another source of uncertainty is the fact that in situations such as these it is not well known if the degree of distress experienced in field concrete relates to expansion in laboratory tests.

Some differences in the appearance of mortar bar specimens from the sealed moist-storage and Conrow cycle tests have been observed. Bars from the sealed moist-storage test that develop deleterious expansion generally show cracking and some gel exudations. Bars from the Conrow cycle test that develop excessive expansion are generally of good appearance with very little, if any, visible cracking, despite the rather high expansions frequently obtained with the test. This might indicate that the reaction in the Conrow cycle test, even if it is essentially an alkali-silica reaction, may proceed along a different course than it does in the sealed moist-storage test.



## SUMMARY

A pilot series of experiments investigated the effect of alkali-silica reactive constituents in a sand-gravel aggregate in the sealed moist-storage and the Conrow cycle mortar bar expansion tests. A highly alkali-silica reactive sand-gravel from the Republican River was tested. This aggregate produces deleterious expansion in both the sealed moist-storage and the Conrow cycle mortar bar tests. The alkali-silica reactive particles were removed from the aggregate, and the processed aggregate was tested. The removal of the alkali-silica reactive particles reduced expansion in the sealed moist-storage test to within safe test limits. In the Conrow cycle test, expansion was reduced but a significant amount of expansion remained. The remaining expansion appears to be due to the characteristic cement-aggregate reaction of sand-gravel aggregates; however, the possibility of this expansion being due to an exceptionally small amount of alkali-reactive material remaining in the aggregate should not be overlooked.

As a control, the alkali-reactive constituents were blended with an innocuous South Platte River aggregate. The natural South Platte River aggregate does not produce deleterious expansion in either the sealed moist-storage or the Conrow cycle mortar bar expansion tests. The blended South Platte River aggregate produced some expansion in both the sealed moist-storage and the Conrow cycle mortar bar expansion tests.

These tests indicate that alkali-silica reaction is a major factor in the cement-aggregate reaction with alkali-silica reactive sand-gravel aggregates, but does not answer the entire question. The problem of cement-aggregate reaction with nonalkali-reactive sand-gravel aggregates, if such a problem exists, requires further investigation.

These tests also indicate that the Conrow cycle mortar bar expansion test is sensitive to alkali-silica reaction.

## ACKNOWLEDGMENTS

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# Distribution, Character and Basic Properties of Cherts in Southwestern Ontario

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A study has been made of the cherts occurring in southwestern Ontario with the purpose of establishing a classification system for use in laboratory testing programs, field performance studies, and the subsequent rating of chert for all mineral aggregate uses. Field relationships of nodular and gravel chert were investigated, and laboratory studies made of their mineralogy by petrographic, X-ray, and chemical analyses. Physical and mechanical properties of each type of chert were measured as well as the potential chemical reactivity and resistance to freeze-thaw disruption and volume change.

Cherts were divided into seven types, and it was concluded that the method of classification employed could be used, but an integrated knowledge of the character and basic properties of each variety is required to insure accurate visual identification.

A map is included which shows the approximate locations of gravel pits, the percentage of chert in certain areas of the province, and other data resulting from the study.

•THE INVESTIGATION of chert distribution, both in bedrock and gravels, and the determination of those characteristics and properties significant to the qualitative evaluation of chert, form part of a study designed to correlate the basic properties of chert in southwestern Ontario with simulated laboratory tests of durability and actual field performance. The boundaries of the area of study were chosen to include the chert-bearing bedrock formations of major importance and Pleistocene gravels with measurable quantities of chert.

It has been recongized for some time that chert occurring in Ontario can be divided into different types, but the subdivision into specific groups with similar properties has not previously been attempted. Examples of unsatisfactory performance are common throughout the study area, but it is felt that some varieties of chert may be qualitatively satisfactory. This study is in part directed towards the clarification of this belief.

Failures in asphaltic concrete appear to be due mainly to disintegration by frost action and mechanical instability resulting from poor asphalt adhesion. Figure 1 shows disintegration of a pavement surface course containing most of the recognized varieties of chert, due to little or no adhesive bond between the aggregate and the cement. The disintegration of portland cement concrete is equally noticeable, and it would appear that all varieties of chert are capable of producing surface popouts, although the frequency and severity are much less for some types than others. Severe pattern cracking occurs in some cases (Fig. 2) but alkali-aggregate reactivity has not thus far been determined as the principal cause. Thin sections indicate that alkali-silica gel occurs in minor amounts in these concretes, but surface exudations rarely show more than trace amounts of silica.

It is anticipated that this study will not only elucidate the deleterious interactions of chert, but supply information applicable to the problem of beneficiating many cherty

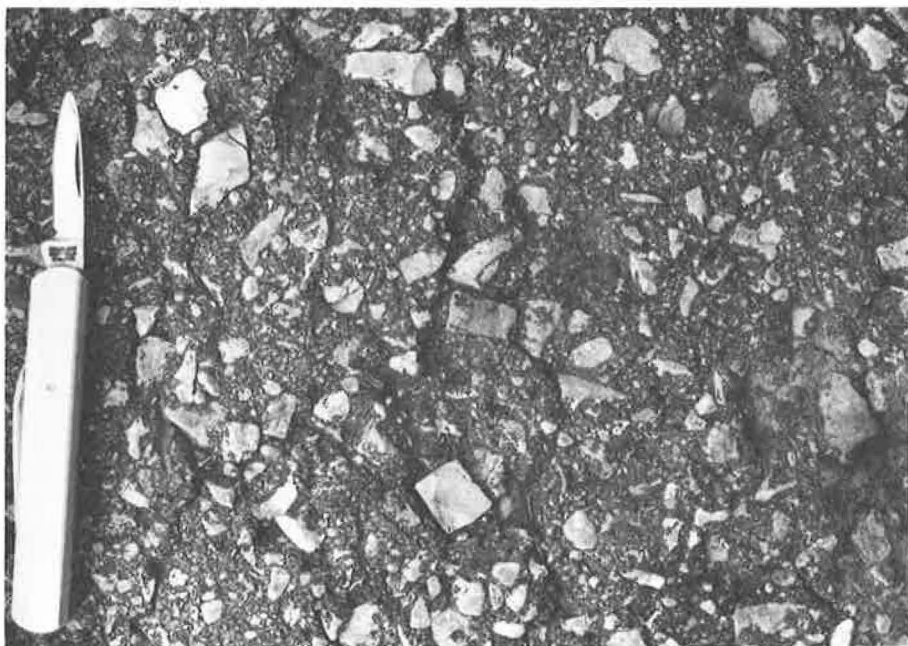


Figure 1. Asphalt disintegration due to little or no adhesion between chert particles and asphaltic cement.

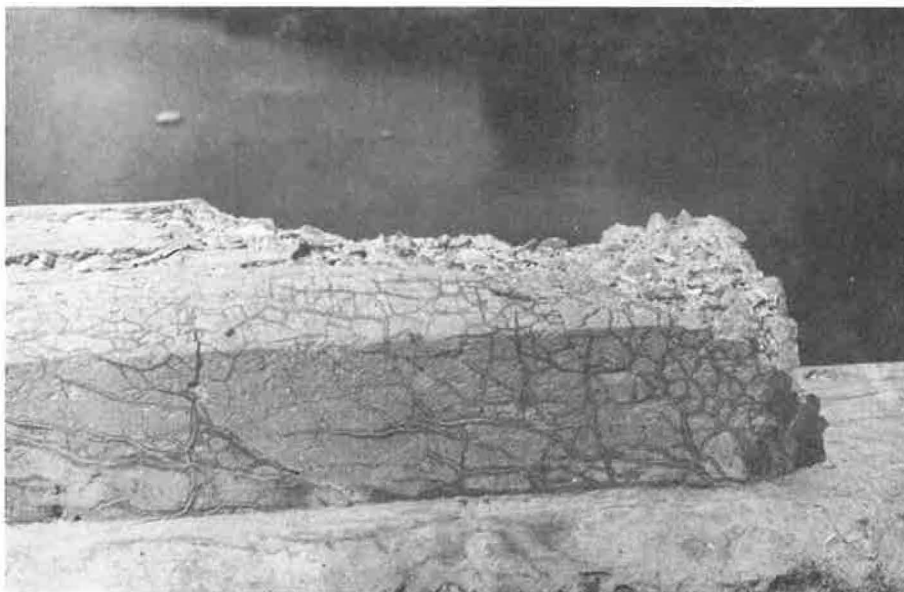


Figure 2. Pattern cracking in concrete.

deposits. The main aim is to devise a method of rating the different types of chert for use in asphaltic and portland cement concrete, based on visual classification and a minimum of laboratory tests.

## OCCURRENCE OF CHERTS IN SOUTHWESTERN ONTARIO

### Palaeozoic Nodular Chert

In the study area, chert occurs principally in limestones of Devonian age. Chert is present less abundantly in Silurian dolomites, and, outside the study area, in limestones of Ordovician age.

Chert is best developed in the Bois Blanc formation, which is the lowermost Devonian present throughout most of southwestern Ontario. The sequence in the study area is lithologically equivalent to the Bois Blanc formation developed in Michigan and similar to the lower Onondaga formation of New York state. The Bois Blanc outcrops along the southern border of the Niagara Peninsula and forms a narrow subsurface-band bordering Lake Erie in this region. From here the outcrop belt stretches in a broad arc across southwestern Ontario to a point on Lake Huron just south of the Bruce Peninsula. The belt averages 5 to 6 mi in width, and, although outcrops are confined primarily to the Niagara Peninsula, the cherty nature of the formation has been proved throughout the area by subsurface drilling. The thickness of the formation increases in a north-westerly direction across the area from 90 to 100 ft in the Niagara Peninsula to more than 200 ft in the vicinity of Lake Huron.

The Bois Blanc formation consists of two members: lower Springvale sandstone, which is locally developed in the Niagara Peninsula and reaches a maximum of only 11 ft, and the overlying main cherty limestone. Chert is relatively abundant in the limestone member, but occurs less frequently in the underlying sandstone except where the sandstone is represented by a sandy lower phase of the upper member.

Fifteen quarry sections and numerous small outcrops of the Bois Blanc formation were examined in detail, and bed-by-bed descriptions of the lithology were prepared. Particular attention was given to the volume and variety of chert and its textural relationship to the limestone host. Sections varied in thickness from 3 to 30 ft with the thickest exposures being in the vicinity of Hagersville. The exposures, which are all low in the Bois Blanc formation, represent only a small portion of the total thickness, but subsurface drilling and the examination of chert particles in gravels confirm the general similarity of the rest of the formation. Ten of the quarries are currently producing crushed aggregate for use as roadstone.

The lithology varies considerably both vertically and horizontally, but certain zones can be recognized and traced throughout the Niagara Peninsula area. The formation is conspicuously fossiliferous and chert-free coral biostromes and crinoid reef mounds are locally developed. These bodies grade laterally into the normal cherty limestone. Comminuted coral and crinoid debris provide most of the fossil content, but coquinoid beds rich in brachiopod and pelecypod valves also occur commonly. Relatively unfossiliferous strata, free from chert, are also a constant feature. A typical section in the Hagersville area consists of an upper fossiliferous cherty zone approximately 10 ft thick, a middle zone of approximately 10 ft of chert-free limestone, and a lower cherty zone approximately 10 ft in thickness, often more shaly with beds of argillaceous limestone. The bottom of the lower zone may be locally sandy and glauconitic.

The chert occurs in nodules of various shapes, sizes, and colors. Only the small nodules are approximately spherical, and most are more or less flattened parallel to their horizontal axes. Adjacent nodules occasionally coalesce to form lenses or beds of limited lateral extent. The thickness ranges from  $\frac{3}{8}$  in. to a maximum of 9 in. with the average size being in the  $1\frac{1}{2}$ - to 3-in. size group. The length varies from  $\frac{3}{8}$  in. to a maximum of approximately 3 ft. Lateral protuberances are relatively common but vertical projections are rare and nodules do not coalesce vertically.

Perhaps the most interesting feature of the Bois Blanc formation is the development of nodular bodies associated with the chert that are silica enriched but lack the gross characteristics of chert. These bodies are generally darker than the limestone host, but may be almost indistinguishable in hand specimen. They have been variously termed

TABLE 1  
RELATIVE ABUNDANCE OF CHERT TYPES IN STRATIGRAPHIC  
SECTIONS EXAMINED IN DETAIL

Map Locality No. <sup>a</sup>	Thickness of Section (ft)	Chert Types (% by vol of total chert)									Tot. Chert Content (% by vol)
		I	Ia	II	III	IV	V	VI	VII	VIIa	
(a) Bois Blanc Formation											
1	4	4.4	—	26.0	—	—	—	69.6	—	—	35.8
2	39	2.4	—	8.6	26.7	—	—	60.6	1.7	—	62.3
3	9 <sup>b</sup>	21.1	—	9.2	13.3	—	—	25.6	30.8	—	40.0
4	10	8.3	—	17.8	39.6	—	—	19.8	14.5	—	43.5
5	28 <sup>b</sup>	2.0	—	28.5	34.1	6.6	0.4	17.7	7.1	3.6	—
6	5 <sup>b</sup>	27.7	—	32.1	29.4	—	—	10.8	—	—	47.2
7	12	45.7	—	27.2	6.9	—	—	20.2	—	—	30.3
8	22 <sup>b</sup>	23.3	—	22.1	15.3	—	—	29.9	9.4	—	45.1
9	35	11.4	—	12.5	13.7	0.8	—	36.3	25.3	—	46.4
10	31	8.4	—	23.4	23.6	0.4	—	29.6	14.6	—	40.8
11	33	—	91.2	—	4.8	—	—	4.0	—	—	24.8
(b) Delaware Formation											
12	16	—	—	9.5	75.0	15.5	—	—	—	—	8.5

<sup>a</sup>See Figure 3.

<sup>b</sup>Including the Springvale sandstone member.

incipient chert (1), embryo chert nodules, and chertified limestone. The first two terms derive from the belief that the nodules are an intermediate stage in the formation of chert. However, whether or not this is the case, the latter term was preferred for the descriptive classification of chert in this study. The chertified nodules, although commonly associated with the true chert nodules, are generally somewhat smaller in size.

The volumes of chert and chertified limestone were obtained at each quarry face by direct measurement of the area exposed in the section. This was performed for each bed and each type of chert within the bed. The total volume of chert and chertified limestone calculated from these data, for each section, agreed very closely with the values determined by analysis of crushed aggregate samples. The volumes of chert are calculated for the total section, i.e., including the chert-free strata as well as the chert-bearing beds (Table 1). In the generalized section given for the Hagersville area, the upper fossiliferous cherty-beds were found to contain 6 to 33 percent nodular chert and 1 to 20 percent nodular chertified limestone. Nodular chert in the lower cherty zone constitutes 8 to 70 percent of the limestone strata and chertified nodules 3 to 26 percent. On the average, nodular chertified limestone is approximately 50 percent by volume of the nodular chert in any bed (Table 1).

Chert also occurs higher in the Devonian succession in the upper member of the Delaware formation. This section may be correlated with the Delaware formation in Ohio and the Dundee formation in Michigan. The minimum thickness has been estimated at 100 ft, and although this is somewhat less than the average thickness of the Bois Blanc formation, the Delaware dips less steeply and outcrops across a broader belt. The outcrop belt, which varies in width from 10 to 30 mi, stretches in a northwesterly direction across the central part of the study area from Lake Erie to Lake Huron. The Delaware also outcrops in the interlake peninsula east of Windsor. Since the Delaware overlaps successively lower horizons to the east and eventually rests directly on the Bois Blanc formation in the southeast part of the study area, the two outcrop belts are adjacent in the vicinity of Lake Erie, but are separated by a belt of noncherty strata along most of the outcrop band.



Chert in the Delaware formation is less commonly developed than in the Bois Blanc formation and exhibits less variety. Chert occurs in flattened nodules throughout the area, but in the south, irregular ragged nodules of white chalky chert are common, sometimes forming an anastomosing network through the limestone host. This type of chert is sufficiently distinctive that its occurrence in gravels can be readily recognized.

Two quarry sections and a number of small outcrops were examined in detail. Particular attention was given to the white chalky chert due to its abundance and distinctive character. Nodules of this type of chert were always highly irregular in shape and constituted 8 to 31 percent of the beds in which they occurred. In general, the Delaware limestone host is relatively uniform and fine grained, and does not contain any highly fossiliferous zones, such as those in the Bois Blanc formation.

The upper member of the Delaware formation is not extensively quarried in southwestern Ontario. However, its contribution to Pleistocene deposits has an important bearing on the provenance of chert in gravels throughout the study area.

In the vicinity of Hamilton, chert is present in the Middle Silurian dolomites of the Lockport formation. Although minor in occurrence, small nodules of white chert and chertified dolomite are nevertheless a distinctive feature of the Goat Island member. Chert nodules occur in this member from Hamilton to Niagara Falls, but only in the Hamilton area are they of any significance. Small chert nodules occur in the Bruce Peninsula towards the base of the Amabel formation, which is the northern equivalent of the Lockport formation. Again developed in dolomites, the cherts are similar to those at Hamilton, but less frequent.

#### Chert in Pleistocene Deposits

The varieties of nodular chert identified in the Bois Blanc and Delaware formations have been recognized in gravels throughout the study area. In addition, minor jasper, which is not indigenous to the area, occurs in some deposits in the northwestern section.

To facilitate the preparation of a map (Fig. 3) showing the distribution of chert in terms of the total chert content of each gravel deposit, the results of analyses from 508 deposits were plotted in the percentage ranges: 0 to 1, 1 to 3, 3 to 5, 5 to 10, 10 to 20, and over 20. Although most of the deposits contain less than 20 percent, there are localized areas with deposits containing in excess of 20 percent and some in excess of 50 percent. In all cases, the mean value was used but the range was taken into consideration in areas where the establishment of boundaries presented some difficulty. The chert fractions from 68 gravel deposits were further separated into their constituent types. Table 2 gives analyses of the chert fractions of eight deposits, traversing the study area from northeast to southwest. Analyses were made on the  $\frac{3}{16}$ - to  $\frac{3}{8}$ -in. fraction of the sample and the samples were obtained from stockpiles, open faces, and in some cases from exploratory test pits. All computed values used in the compilation of data for the map are weight percentages.

Lines of equal chert concentration could be plotted on the map, outlining zones with the ranges of chert content previously mentioned. Definite zones are defined by the boundaries which, although very irregular, approximately parallel the outcrop bands. The greatest concentration occurs along and to the southwest of the Bois Blanc and Delaware outcrop belts in two wide bands with the chert content in the range 10 to 20 percent. Areas with concentrations greater than 20 percent are much more localized and are mainly associated with the Bois Blanc outcrop. In all zones, deposits occur with significantly different concentrations of chert than the surrounding gravels; in some cases eskers had appreciably higher or lower concentrations than associated outwash deposits. However, extensive outwash trains can be traced across different zones with corresponding changes in the chert content and it would appear that minor geological discrepancies do not invalidate the belief that there is a geographical pattern for the distribution of chert. A more accurate pattern could probably be derived from the chert concentration in associated tills, but the zones established are remarkably consistent and concur with the stratigraphic occurrence of chert and the movements of glacial ice through the study area.

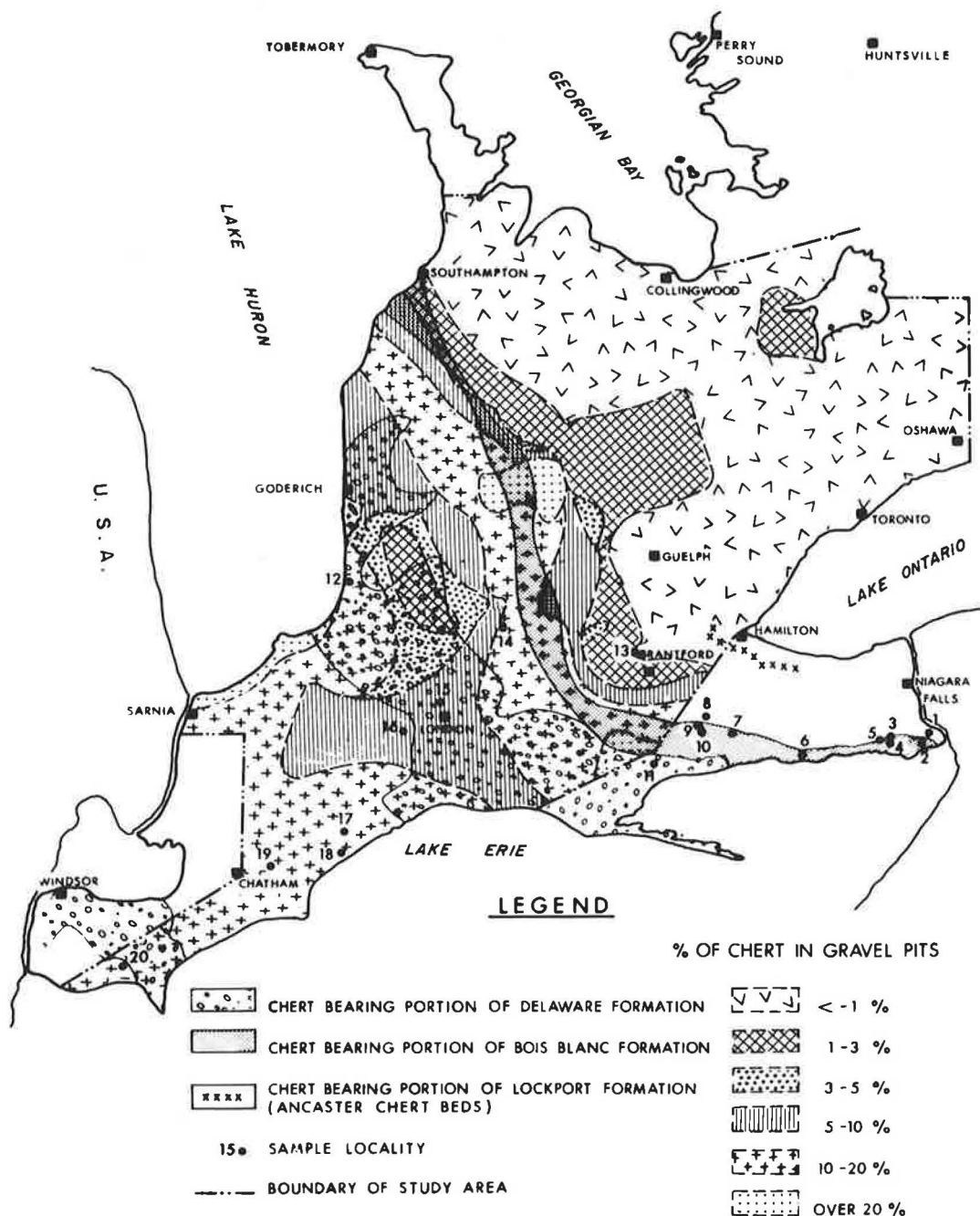


Figure 3. Distribution of chert in gravel deposits in southwestern Ontario.

The provenance of chert is complex, but in many cases it is possible to differentiate between varieties derived from the Bois Blanc formation and from the Delaware formation. Chert from the Lockport formation and Black River group occurs too infrequently to identify in gravels. Chert from both the Bois Blanc and Delaware occurs over a wide area on either side of each outcrop belt. Delaware chert occurs only rarely northeast of the Bois Blanc outcrop and increases proportionately in a southwesterly direction.

TABLE 2  
RELATIVE ABUNDANCE OF CHERT TYPES IN PLEISTOCENE GRAVELS

Map Locality No. <sup>a</sup>	No. of Samples	Chert Types (% by wt of chert fraction)							Tot. Chert Content (% by wt)
		I	II	III	IV	V	VI	VII	
13	1	43.0	—	14.1	—	—	42.9	—	1.7
14	5	44.9	23.8	5.1	—	—	26.2	—	15.0
15	4	32.2	17.7	9.8	1.6	—	38.7	—	5.0
16	3	46.6	23.4	3.0	1.4	—	25.6	—	10.9
17	1	47.3	26.8	14.6	—	—	11.3	—	12.4
18	7	51.4	22.0	4.3	0.9	—	21.4	—	7.6
19	4	27.2	14.2	22.9	—	—	29.8	5.9	9.7
20	1	52.2	20.4	1.4	—	—	26.0	—	11.2

<sup>a</sup>See Figure 3.

The area between the two outcrop belts contains significant proportions of chert from both formations, with Bois Blanc chert predominating and only isolated deposits along the north border of the Delaware outcrop appear to contain equal quantities of both. Gravels in the study area west of the Delaware outcrop contain relatively more Delaware chert. There is apparently no pattern to the distribution of different types of chert except where white chalky cherts from the southern part of the Delaware outcrop are locally concentrated.

Thus the chert content of Pleistocene gravels in southwestern Ontario reflects in a general way the proximity of underlying chert-bearing formations. The variation in concentration within each deposit is also apparently influenced by the local derivation of the chert. In a number of deposits the variability of the chert fraction was examined in relation to the variability of the other components. Two deposits of outwash gravel were examined in detail. In one case, 37 samples from test pits and open faces provided a cross-section of the deposit and gave a coefficient of variation of 27.3 for the chert content and, in the other case, 17 production samples over a 2-yr period gave a coefficient of variation of 23.4. The igneous fractions had coefficients of variation of 9.1 and 9.8, respectively. The variation in these two cases is somewhat greater than the average but due cognizance was taken of this inherent variability in the establishment of boundaries on the map.

Gravel-size particles of chert show only superficial weathering, so that crushed gravel chert is essentially the same as the quarried nodular variety. Incipient alteration by weathering agents is apparent in the brown or reddish brown patina developed on those varieties of chert containing appreciable iron (i.e., types III and IV which are described in the following section of this report). Some varieties of types I and II may be outwardly buff colored from the liberation of smaller quantities of hydrated iron. Particles containing negligible iron are generally somewhat lighter in surface color than their nodular parent material, due apparently to higher porosity. This layer, however, is confined to a surface skin of insignificant thickness, and there is microscopic evidence to suggest a depletion of carbonates, possibly due to leaching. Quarry cherts subjected to unconfined freezing and thawing frequently became lighter surficially after prolonged exposure, and thus this phenomenon may in part be physical in nature.

#### CLASSIFICATION OF CHERTS

Originally the cherts examined were divided into a large number of groups following techniques adopted by other investigators (2, 3). For the most part, classes of chert were based on small differences in color, and it soon became apparent that this system was difficult to apply in practice. Preliminary tests further indicated that some of the groups had sufficiently similar basic properties that they could be combined to facilitate



recognition for bulk separation. Textural relationships with the carbonate host rock were of fundamental importance, insofar as cherts from different locations were similar in appearance when developed in carbonates of the same texture, and played a significant part in the conceptual classification of the chert nodules. While recognizing the heterogeneity of groups based on such characteristics, it was felt that the subdivisions were more or less consistent and provided a reasonable approach to the visual segregation of chert nodules for analysis.

The collection of samples was greatly influenced by the establishment of chert types, for, whereas particular attention was paid to the collection of the most abundant nodules at each exposure, every effort was made to insure that all the varieties present were included. Bulk samples for tests requiring a large amount of material were procured wherever possible from large nodules or lenses, and orientated specimens for microscopic, chemical, and mineralogical study were obtained from nodules considered to be most typical of each variety of chert.

### Type I Chert

In the upper strata of Bois Blanc exposures, zones of white, pale buff or yellowish chalky chert occur. The nodules are somewhat smaller than associated light grey cherts but frequently are a combination of both types. A zone of white chert of this type is locally developed in the lower Springvale sandstone member in the Hagersville area. The most noteworthy development of this type of chert is probably in the southern part of the Delaware outcrop, where it occurs almost to the exclusion of other varieties. Minor zones of small nodules occur in the Lockport formation and Black River group. Hand specimens are soft and fracture very irregularly to display a rough surface texture (Fig. 4). This type of chert is very porous. The Delaware variety is designated I(a) where analytical results are given separately.

### Type II Chert

Light grey or occasionally white or cream colored chert occurs mainly in the abundantly fossiliferous upper strata of the Bois Blanc formation (Fig. 5). Chert of this



Figure 4. Irregular nodules of white chalky chert (type I) in Delaware limestone.



Figure 5. Nodule of type II chert in highly fossiliferous Bois Blanc limestone.

type occurs to a limited extent elsewhere in the Bois Blanc and in the other chert-bearing formations where fossil debris is locally concentrated, and appears to be texturally related to fossiliferous host strata. The outlines of fossil fragments are generally conspicuous and the fragments themselves are paler in color than the rest of the nodule. Hand specimens are hard and dense with a vitreous lustre and conchoidal fracture. Nodules are occasionally splintery and sometimes display what would appear to be a pseudo-cleavage, breaking into prisms along more or less vertical planes with smooth faces. White varieties of this type of chert are differentiated from type I cherts by their hardness and vitreous lustre.

Associated with chert types I and II in the fossiliferous Bois Blanc strata are small solitary corals entirely or partially replaced by chert.

### Type III Chert

Medium gray, hard, dense, conchoidally-fracturing chert with a dull vitreous lustre is commonly found in fine-grained carbonates which are relatively poor in fossil debris. Occurring in all formations, it is most commonly developed in the Bois Blanc limestones and to a lesser extent in the northern outcrop of the Delaware formation. The color is somewhat variable and olive-grey, brownish-grey and greenish-grey nodules have all been described. In some nodules there are conspicuous light and dark spots or patches, giving the chert a mottled appearance. Type III chert occurs primarily in the lower zone of the cherty Bois Blanc member but is also present in the upper fossiliferous beds in relatively unfossiliferous interbedded layers. This type of chert is perhaps less splintery than type II, but a similar pseudo-cleavage is noticeable. In one or two cases, thin elongated nodules split into cubical fragments, and in other cases into thin curved plates more or less perpendicular to the bedding. Further division of these fragments may produce a conchoidal fracture or a similar smooth surface.

### Type IV Chert

This group was established for cherts essentially similar to type III but dark grey in color. The remarks pertaining to type III chert apply equally to this type and in general



Figure 6. Nodules of incipient chert (type VI) in medium bedded Bois Blanc limestone.

the two groups may be considered to comprise a single unit. However, for comparative purposes, the mineralogy, chemistry, and basic properties of this type have been investigated separately to provide more data on the visual identification of chert by color.

#### Type V Chert

Although only of minor occurrence in the Bois Blanc formation and Black River group, this type of chert is sufficiently distinctive to merit separate classification. Black in color, it is hard and dense with a waxy lustre and conchoidal fracture. It occurs in small nodules in fine-grained limestones, frequently with thin carbonaceous shale seams. In the Black River group it occurs in a very fine-grained calcilutite.

#### Type VI Chert

Chertified limestone, prominent as nodular bodies in the Bois Blanc limestone, is a type readily identifiable visually (Fig. 6). The stratigraphic and textural relationships of this type have been described previously. It has an irregular to subconchoidal fracture and in general a surface texture and lustre similar to the limestone host. The lustre may, however, be porcelanous.

Irregular bodies of chertified sandstone occur in the Springvale member of the Bois Blanc but are texturally quite different from the nodular chertified limestones and are insignificantly developed.

Chertified dolomite, where developed, falls within this group.

#### Type VII Chert

Small to medium sized, flattened, ellipsoidal nodules of chert occur in the lower zone of the cherty Bois Blanc in beds of argillaceous limestone (Fig. 7). The nodules are bluish, greenish, or, less frequently, reddish-grey and distinguished by their porcelanous lustre and smooth fracture. They are usually discreet with smoothly curving contacts, and where developed in layers with shale seams the seams bend smoothly above or below the chert. Occasionally nodules of this type are developed in





Figure 7. Type VII nodule in argillaceous Bois Blanc limestone.

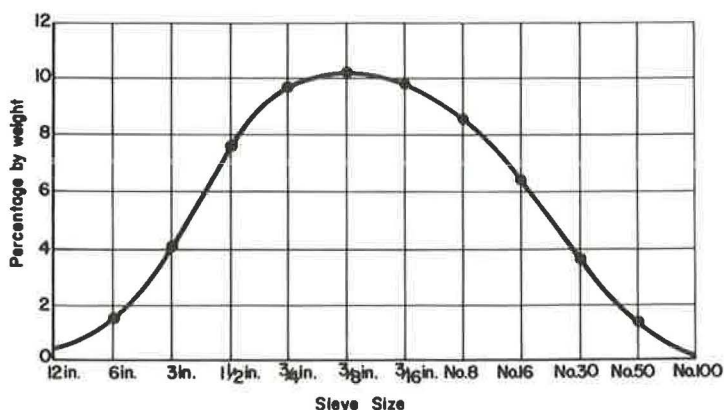


Figure 8. Size-frequency distribution of chert fraction in outwash gravel (map locality No. 16).

black carbonaceous shales at the base of the upper member of the Bois Blanc formation and then have a characteristically earthy lustre. Where analyzed separately this sub-variety is designated type VII (a).

In establishing seven different types of chert, the groups are of necessity based on broad generalities, as the recognition of small differences would create an inordinately large number of categories. If the seven types described are thought of as end members, then the almost infinite variety of intermediate members poses an equally substantial problem of recognition. Fortunately, the types were more or less distinct with relatively few mixtures. In crushed aggregate, however, combinations of chert and limestone were sometimes a problem and in these cases intermediate subgroups must be used for the purposes of analysis.

Frequently, chert nodules of all types display a thin peripheral zone which is dull, porous and generally light grey in color. In one case a zone of small dark grey chert nodules was observed with very thick rim zones which coalesced laterally and vertically to form an anastomosing network throughout the bed. Isolated patches and masses continuous with the peripheral zone have been observed within the nodules, but these are generally of limited extent. The importance of the peripheral zone is that it is an integral part of the chert, mineralogically and structurally different, but, together with the chert nodule, separating from the limestone host.

### Size-Frequency Distribution

Chert is not equally represented in all size fractions of the gravels. To investigate the particle size, distribution samples from several deposits were separated into their constituent size groups and the chert content determined. In general, the distribution curve was similar to that in Figure 8.

The shape of the curve would suggest a log-normal distribution resulting from some natural attribute of the parent chert-bearing formation. That the curve reflects a similar size distribution of nodular chert in both the Bois Blanc and Delaware formations is not the case here, as it would appear to be in the cretaceous flints occurring in Denmark and Britain (4). However, the size-frequency distribution of the parent chert nodules is certainly an important factor and partly controls the resulting gravel-size distribution. The nodules had a log-normal distribution with a size range from 12 to  $\frac{1}{4}$  in. and a mode in the 3- to  $1\frac{1}{2}$ -in. size group.

Crushing curves obtained for all varieties of chert were more or less the same in each case. When bulk samples were reduced, using laboratory techniques to produce a mode in the  $\frac{3}{4}$ - to  $\frac{3}{8}$ -in. size group, the resulting size distribution was approximately log-normal but skewed in the direction of the larger sizes. Good examples of chert nodules crushed in situ have been observed with no subsequent transportation of the resulting particles. Here the distribution is again log-normal but the modal size varies slightly from one type of chert to another. The average size for type III chert is in the  $1\frac{1}{2}$ - to  $\frac{3}{4}$ -in. size group and for types I, II and VII in the  $\frac{3}{4}$ - to  $\frac{3}{8}$ -in. size group. Further, in some cases the particulate materials produced by glacial crushing are remarkably cubical in shape; this is particularly true of type III, and it has been concluded that this pattern of breaking is primarily due to the systems of microcracks in thin section. Frequently nodules of type II chert yield splintery or platy particles, presumably for the same reason, but the relative absence of flat particles in gravels indicates further breakdown by impact in transportation.

Undoubtedly, freezing and thawing were active in glacial environments, producing approximately the same results as the unconfined freeze-thaw tests which form part of this study. The general effect is to reduce the overall partical size and produce a shift in the distribution towards the finer sizes. Superimposing this effect and the crushing characteristics on the natural size-frequency distribution of the parent nodules, all of which are approximately log-normal, would result in the curve shown in Figure 8. Variations, depending on such factors as the type of chert predominating and severity of glacial crushing, do occur, and the modal size ranges between  $\frac{3}{4}$  and  $\frac{3}{8}$  in., but it lies typically in this size group, being closer to the lower limit with increasing abundance of type I chert.

### MINERALOGY

Fifty-six orientated specimens were collected from seventeen localities and thin sections cut normal to the bedding for microscopic study. The samples included all the noticeable varieties from outcrops in the Delaware, Bois Blanc, and Lockport formations and the Black River Group. A portion of each sample was reserved for X-ray spectrographic and chemical analysis.

The essential mineral in all the cherts was quartz. The carbonate minerals calcite and dolomite were also important constituents of all the thin sections, being generally subordinate to quartz but in some sections of equal abundance.

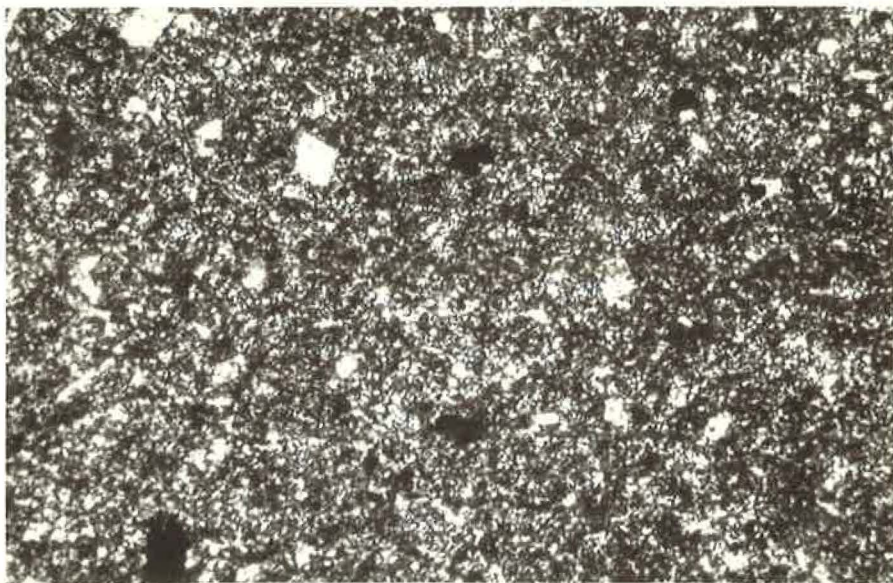


Figure 9. Typical development of microcrystalline quartz (type II chert): euhedral rhomb-shaped dolomite crystals.

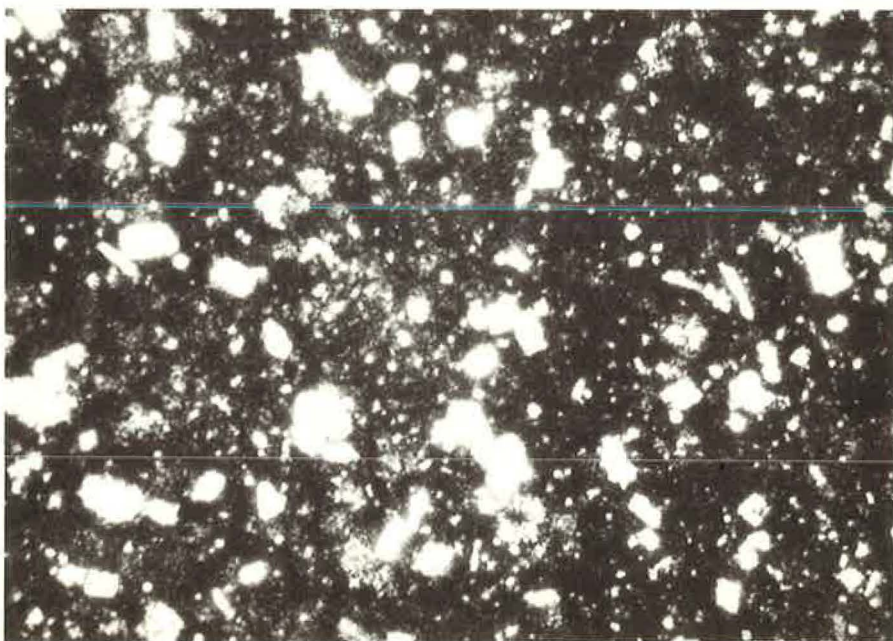


Figure 10. Cryptocrystalline quartz, approximate grain size 0.0009 mm; crossed nicols x 200.

Quartz occurs in two varieties, as equigranular mosaics and as flamboyant or spherulitic aggregates of fibres (generally termed chalcedony.) The textural terms microcrystalline (0.01 to 0.001 mm) and cryptocrystalline ( $<0.001$  mm) have been used for convenience in describing the granular quartz and to facilitate a comparison with other published data. These two types of quartz differ only in grain size. Quartz grains,



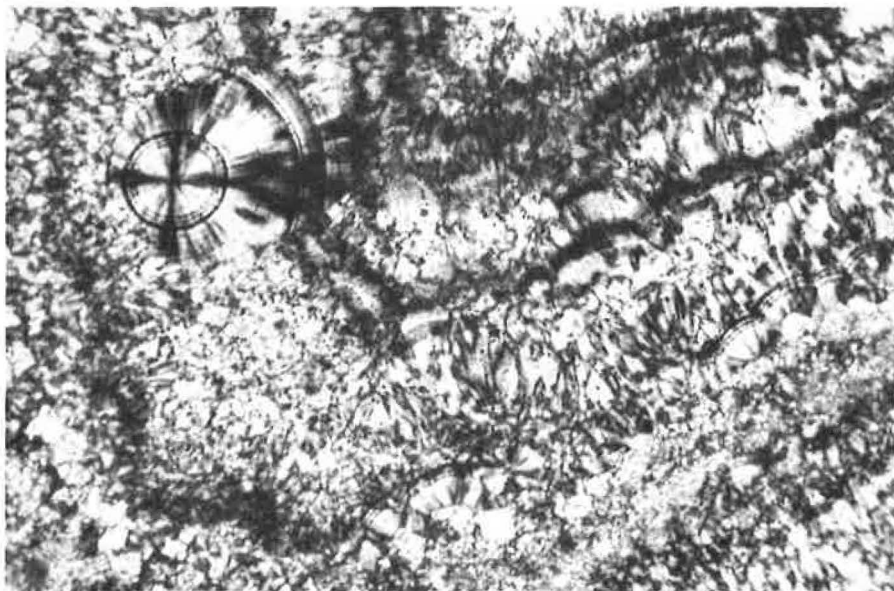


Figure 11. Typical development of plumose fibrous quartz (chalcedony) in type II cherts; the spherulitic development of fibrous quartz is shown in the upper left.

larger than 0.01 mm in grain size, do occur but are not significantly developed in most of the cherts, and where present, are confined to void or crack fillings or small isolated patches in the groundmass, with the general appearance of vein quartz.

Microcrystalline quartz (Fig. 9) is the most common variety, comprising up to 90 percent of some types of chert. The complete range of grain sizes within the textural definition were observed, but the most commonly occurring size is approximately 0.005 mm. In general, size uniformity was most noticeable where quartz had replaced an equigranular groundmass of calcite or dolomite. Microcrystalline quartz replacing large fossil fragments was frequently slightly larger in size than the average. The absence of discrete grain outlines in plane polarized light and the characteristic undulatory extinction between crossed-nicols common to microcrystalline quartz in cherts from other geographic localities (5, 6) were equally true of this type of quartz in the thin sections examined.

Cryptocrystalline quartz (Fig. 10) was present in significant amounts in only one type of chert, where its character was again similar to the same type of quartz found in geographically different cherts. The isotropic appearance of much of the groundmass shown in this figure is found in abundance only in chert type VII.

Fibrous quartz occurs as single rows of fibres lining the faces of small cracks and multiple layers of fibres in larger openings giving a banded appearance and also as small spherulites of radiating fibres. Where banded, successive fibres apparently grow from the free ends of preceding fibres and are generally longer. In one case, three successive layers of fibres, developed in a cavity, were 8, 10, and 40 microns in length. Overlapping fibres from adjacent nuclei give a plumose appearance between crossed-nicols. In plane, polarized light areas of fibrous quartz are frequently pale yellowish-brown in color, or composed of interbanded clear and colored zones. A mosaic of large quartz grains generally occupies the center of voids lined with fibrous quartz (Fig. 11).

Opal was not identified in any of the thin sections. Very minor areas of what appeared to be isotropic silica were observed in some sections, but from considerations of relief and refractive index, it was indicated that these were in all likelihood composed of ultrafine-grained cryptocrystalline quartz.



Figure 12. Microcracks in type III chert showing parallel orientation; reflected light  $\times 150$ .

Both calcite and dolomite occur within the chert nodules. Dolomite is relatively common in cherts developed in limestones, but calcite was not observed in cherts from a dolomite host. Calcite is present as fossil fragments and host rock fragments, both generally deeply embayed by microcrystalline quartz, and as thin irregular veins of clear crystals. Dolomite, which is not uncommon in the limestones, is present as clear rhomb-shaped crystals in the cherts. Iron rich dolomite euhedra (ferrodolomite) or compositionally zoned crystals occur in all the cherts but most frequently in the darker varieties. Dolomite rhombs range in size from 0.01 to 0.05 mm but are most commonly 0.02 mm in diameter. In most cases, there is a concentration of dolomite euhedra at the chert limestone contact and in those cherts with well developed rim zones there is significantly more dolomite in the rim zone than in the chert or limestone host.

Accessory minerals include pyrite, glauconite, limonite, leucoxene, detrital quartz and feldspar, the latter in cherts developed in sandstones or sandy limestones. Limonite appears to be the most important; apparently admixed with some organic matter it is irregularly distributed throughout the groundmass of all types of chert, being concentrated more in the darker cherts than in the lighter varieties. It would seem that limonite and in some cases organic matter are partly responsible for the observed variation in color. Very fine mineral dust is also present in the groundmass of cherts which, from its optical properties, would appear to be a carbonate, probably dolomite.

The microscopic structure of the chert nodules tends to reflect the primary structures of the host rock, and relict shapes are common in cherts from fossiliferous or oolitic strata. However, orientated microcracks are an important original feature present in many of the cherts examined. Microcracks have been described previously in chert nodules from the Hagersville area (7), and it would appear that this is a relatively common phenomenon in nodular cherts from the Bois Blanc formation. Where developed, the cracks are approximately 0.1 to 1.0 mm in length and 0.2 to 0.5 mm apart, parallel to subparallel and at a high angle to the bedding. In reflected light, the cracks dip at more or less the same angle to the plane of section. These short cracks are somewhat different from the irregular or dendritic crack patterns developed in large areas of fibrous quartz (Fig. 12).

### Type I Chert

Microcrystalline quartz is the dominant variety of quartz in this type, constituting 50 to 70 percent of the sections. It is noticeably larger in grain size than in most other varieties of chert ranging between 0.005 and 0.008 mm, with an average size of 0.007 mm. Fibrous quartz is generally not present in significant amounts but may reach 12 percent in some sections. It is developed as plumose varieties in the larger voids and as small spherulites in the groundmass.

Carbonates are important constituents of this variety of chert, in many cases comprising almost as much of the section as the quartz minerals. Calcite is a variable constituent generally in the form of ragged masses of granular limestone, showing all stages of conversion to microcrystalline quartz. In contrast, dolomite is a constant constituent, comprising approximately 20 percent of all sections as euhedral rhomb-shaped crystals.

Accessories are limited to minor limonite and mineral dust as coloring matter in the groundmass.

### Type II Chert

Equigranular microcrystalline quartz, average size 0.005 mm, is the major component of the light-grey fossiliferous cherts in this group. Some fossil fragments appear to have been replaced by microcrystalline quartz of slightly larger grain size, but this is not a general condition. Fibrous quartz is a more significant component of this type than any of the other chert varieties examined. Plumose varieties of fibrous quartz are well developed in large voids created by fossil debris, in the cells of coral and bryozoa fragments and lining the inside of brachiopod and pelecypod valves, where both ventral and dorsal valves are present. Coarsely crystalline quartz is invariably developed in the centers of voids not completely filled by fibrous quartz. Spherulitic varieties are confined to minor occurrences in the groundmass and irregular cracks.

Fossil remnants are common, together with fragments of granular host rock and coarsely crystalline clear calcite cement in all stages of replacement by microcrystalline quartz. Clear dolomite euhedra are again constantly developed, but are only occasionally iron rich or compositionally zoned. Less important than in type I, dolomite comprises approximately 10 percent of most sections.

Limonite and minute black rounded bodies of organic material occur sparsely and in irregular patches throughout the groundmass. Minor pyrite, occurring in small crystals, is the only other accessory of any consequence.

Microcracks are moderately common and occasionally occur in swarms 0.02 mm apart (developed in 3 out of 8 sections).

The fossiliferous limestone host in which light-grey chert is developed frequently displays small cherty spots that are peripherally milky-white with darker centers. In thin section these are voids and fossil molds with plumose fibrous quartz lining the walls of the cavity and coarsely crystalline quartz occupying the center. The matrix is comminuted fossil debris showing negligible replacement by microcrystalline quartz, even close to the chert limestone contact.

### Types III and IV Chert

Microcrystalline quartz, as the major quartz mineral, constitutes 75 to 86 percent of chert types III and IV. The grain size ranges from 0.003 mm to 0.007 mm and averages 0.005 mm. Fibrous quartz is not significantly developed and, where present, occurs as small spherulites in the groundmass and short fibres lining cracks. Coarsely crystalline quartz is confined to veinlets traversing the microcrystalline quartz and passing into the host rock.

Calcite occurs as small randomly distributed fragments of granular limestone partially replaced by quartz. Dolomite euhedra are uniformly developed but proportionally the same as in the limestone host.



Limonite with minor admixed organic material is the most common accessory and is apparently responsible for most of the megascopic color variation. It is very unevenly distributed and occurs frequently in clots with finely divided mineral dust. Pyrite and leucoxene are less common but also occasionally occur in small segregations.

Microcracks are probably more often present (9 out of 18 sections) than in the variety of chert classed as type II. Generally spaced 0.1 to 0.4 mm apart, they may occur as close as 0.02 mm, in swarms. In one section, two sets of cracks almost normal to each other were observed.

#### Type V Chert

The black resinous or waxy cherts contain the highest percentage of microcrystalline quartz, 90 to 95 percent. The grain size varies little and is generally close to 0.005 mm. Spherulitic fibrous quartz is more important than in types II, III, and IV, and is found completely filling small spherical organic bodies, probably spores or pollen cases, as minute spots in the groundmass and filling cracks. Coarsely crystalline quartz is confined to minor irregular areas unconnected with the fibrous quartz.

Calcite is not important, and, as with other varieties of chert, is much corroded by microcrystalline quartz. Dolomite euhedra, although the same as those in most cherts, are less commonly developed.

Iron is a relatively insignificant impurity and most of the coloring matter appears to be finely divided brown organic material, irregularly distributed and frequently concentrated in areas of fibrous quartz.

#### Type VI Chert

In thin section, the matrix is approximately equal proportions of granular calcite and microcrystalline quartz (average grain size 0.005 mm). The microcrystalline quartz is uniformly distributed and there are apparently no areas of preferential replacement within the nodule. The chert limestone contact is relatively sharp as is the case with all of the nodular cherts. Where fossil fragments are of importance they are moderately or deeply embayed. Large unit crystals of calcite, usually pelecypod prisms, may be replaced by slightly coarser microcrystalline quartz, but frequently the replaced portions cannot be differentiated from the microcrystalline groundmass. Minute areas of cryptocrystalline quartz probably result from the replacement of segregations of shaly material and are of negligible proportions. The larger voids are lined with plumose fibrous quartz and coarsely crystalline quartz fills the central cavity. Smaller voids are completely filled by spherulitic or plumose fibrous quartz.

Dolomite euhedra are concentrated near the chert limestone contact, but within, the nodules are developed only to the same extent as in the surrounding host rock.

Pyrite, and in some cases glauconite, are minor accessories. Limonite is present as irregular patches of coloring matter, but is not common in this type of chert.

#### Type VII Chert

This is the only variety of chert in which cryptocrystalline quartz is significantly developed. The average grain size is slightly less than 0.001 mm with minor areas finer than this that appear isotropic between crossed-nicols. Microcrystalline quartz is relatively infrequent and occurs replacing fossil fragments. Fibrous quartz is confined to small spherulites in the groundmass and spherulitic aggregates in cracks.

Calcite is present as fossil debris in all stages of replacement by microcrystalline quartz. Euhedral dolomite rhombs are common and some of the larger grains show slight to moderate corrosion by microcrystalline quartz.

An intimate mixture of clay and organic matter occurs throughout the groundmass, giving the thin section a brownish, clouded appearance. However, the nature of the clay minerals and their textural relationship with the matrix cannot be resolved with the optical microscope. Limonite is also apparently present in the reddish or brownish nodules.

The most common accessory is silt size detrital quartz with minor feldspar. Minute grains of pyrite and glauconite are also locally common. Detrital biotite is a rare accessory.

TABLE 3  
MICROSCOPIC ANALYSES

Chert Type	Constituents							No. of Sections
	Calcite	Dolomite	Micro-Crystal-line Quartz	Fibrous Quartz	Crypto-Crystal-line Quartz	Quartz	Accessories	
I	16.5	26.5	48.5	4.0	—	3.5	1.0	10
II	8.0	10.0	70.0	9.5	—	1.5	0.5	8
III	7.0	9.5	80.0	1.5	—	1.0	1.0	11
IV	6.5	8.0	82.5	1.0	—	0.5	1.5	7
V	1.0	1.0	88.5	5.0	—	3.0	1.5	3
VI	18.5	30.0	48.0	1.0	—	2.0	0.5	11
VII	5.0	16.0	9.0	1.0	66.0	1.5	1.5	6
VIIa	5.5	21.0	7.0	10.0	48.5	0.5	7.5 <sup>a</sup>	2

<sup>a</sup>Mostly detrital quartz, some glauconite and pyrite.

Table 3 indicates the relative abundance of the major varieties of silica and carbonate minerals constituting the types of chert recognized megascopically. The results given are averages of individual measurements made on the number of thin sections indicated.

#### X-RAY ANALYSIS

X-ray diffraction patterns were obtained for the same powders used in the measurement of the true specific gravity. A total of 21 samples were analyzed, which included representatives of all the chert types previously described. The recorded spectra included reflections characteristic of  $\alpha$ -quartz, calcite, and dolomite together with other minor constituents. A study of the weak reflections registered by the accessory minerals indicated the presence of trace amounts of feldspar and a mica-like mineral or minerals, and in one case chlorite. Amorphous silica is completely disordered and does not produce any recorded spectra, but opal generally consists of a cristobalite phase and an  $\alpha$ -quartz phase in addition to the truly disordered amorphous phase (8).

TABLE 4  
X-RAY SPECTROGRAPHIC ANALYSES

Chert Type	Constituent					
	Quartz	Calcite	Dolomite	Feldspar	Mica-Like Clay Minerals	Chlorite
I	41	22	37	Trace	Trace	—
Ia	52	25	23	—	—	—
II	64	11	25	Trace	—	—
III	78	14	8	Trace	—	—
IV	89	—	11	Trace	—	—
V	90	10	—	—	—	—
VI	49	26	25	Trace	Trace	—
VII	71	4	25	Trace	Trace	Trace
VIIa	45	5	50	Trace	Trace	—

No cristobalite reflections were recorded, and thus it was concluded that opal did not occur in any of the samples in measurable quantities (Table 4).

Semiquantitative determinations of the percentages of  $\alpha$ -quartz, calcite and dolomite were made from the relative intensities of the appropriate reflections. The values given in Table 4 are calculated as percentages on the assumption that these three minerals represent 100 percent of the sample. This assumption is not strictly valid, but in most cases minor minerals constitute less than 1 percent and the discrepancy introduced by omitting them from the computation is slight. The most conspicuous feature is the presence of  $\alpha$ -quartz as the only silica mineral and dominant constituent of each type of chert. Obviously, the varieties of quartz determined petrographically all have the same crystalline structure, that of  $\alpha$ -quartz, regardless of form or grain size. For this reason the use of the term chalcedony to denote a distinct mineral species has been avoided here, and the designation fibrous quartz used in preference.

In general, the correspondence between petrographic and X-ray analysis, in terms of the major minerals, is good. However, in some cases there appears to have been an overestimation of the silica content at the expense of dolomite in the thin section determinations. From the greater abundance of dolomite registered by X-ray diffractometry it may be concluded that the fine mineral dust clouding these sections is for the most part dolomite in character.

Trace amounts of micaceous minerals were recorded in samples of types I, VI, and VII. Although not determined directly, glauconite is the principal mineral, and if any illitic clay minerals are present they are undoubtedly of very minor occurrence.

#### CHEMICAL ANALYSIS AND REACTIVITY

There are now numerous records of analyses of Paleozoic cherts in the literature, all of which emphasize the basically siliceous nature of this material. The chemistry of the cherts studied in this investigation illustrates the dominance of silica and the importance of lime and magnesia as recognized elsewhere, and also the relative unimportance of soda and potash.

Twenty-five chemical analyses were made on the same powder samples used for specific gravity and X-ray diffraction measurements, and the results given in Table 5 are averages for each type of chert. As anticipated, silica is the essential constituent and all the varieties are noticeably calcareous. In general, the silica content is in accord with the microscopic or X-ray determinations. However, the chertified limestone (type VI) appears to be more siliceous than the microscopic or X-ray results would suggest. The range, 56 to 63 percent  $\text{SiO}_2$ , is significantly different from that reported by Laird (1) of 45 to 50 percent  $\text{SiO}_2$  for selected nodules of incipient-chert. Probably the reason for this, as mentioned previously, is the difficulty encountered in making a

TABLE 5  
CHEMICAL ANALYSIS

Content	Chert Type								
	I	Ia	II	III	IV	V	VI	VII	VIIa
L.O.I.	14.35	15.0	7.97	7.33	4.10	3.90	16.75	7.20	15.50
$\text{SiO}_2$	65.25	64.40	79.83	81.53	85.40	89.50	59.35	75.95	60.90
$\text{Fe}_2\text{O}_3$	0.95	0.70	0.97	1.15	1.80	0.40	1.10	1.70	1.10
$\text{Al}_2\text{O}_3$	1.25	0.90	0.60	1.17	2.60	1.40	1.85	4.45	1.90
CaO	13.20	16.10	7.27	7.22	3.00	4.30	15.30	5.70	12.30
MgO	3.80	2.90	2.26	1.30	1.70	Trace	4.45	3.15	6.50
$\text{K}_2\text{O}$	—	—	0.23	0.39	0.82	—	0.39	1.69	1.46
$\text{Na}_2\text{O}$	0.03	0.03	—	—	—	—	—	0.01	—
Total	98.83	100.03	99.13	100.09	99.42	99.50	99.19	99.85	99.66



TABLE 6  
POTENTIAL ALKALI-AGGREGATE REACTIVITY BY  
RAPID CHEMICAL TEST

Chert Type	Reduction in Alkalinity ( $R_c$ ) (millimoles/litre)	Dissolved Silica ( $S_c$ ) (millimoles/litre)	$\frac{S_c}{R_c}$	No. of Samples
I	160	598	3.75	4
Ia	192	530	2.76	2
II	133	583	4.39	2
III	132	555	4.21	3
IV	165	520	3.15	1
VI	159	485	3.05	3
VII	217	500	2.30	1
VIIa	185	570	3.08	1

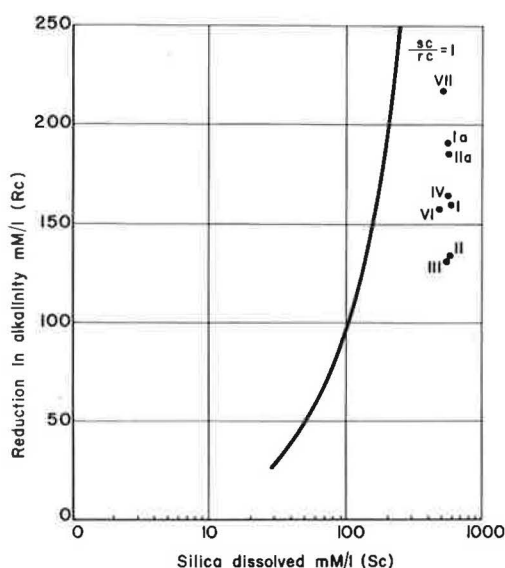


Figure 13. Potential reactivity of chert type I, II, III, IV, VI and VII by means of rapid chemical test.

on 17 samples by means of the rapid chemical method (ASTM Designation C289-57T). The samples included all varieties of chert from four localities in the Bois Blanc formation, one locality in the Delaware formation, and two Pleistocene gravel deposits. Table 6 gives the amount of dissolved silica ( $S_c$ ) and the concomitant reduction in alkalinity of the NaOH solution ( $R_c$ ). Figure 13 shows the same results.

All of the cherts gave results indicating a high degree of chemical reactivity. The  $S_c/R_c$  ratio varied between 2.30 and 5.20 with most of the higher values registered by the more quartz-rich cherts. However, the results are variable and there is no apparent correlation between the mean value for each type of chert and either the quartz content or the porosity. It must be concluded that a complex relationship exists between both of these factors and probably between the grain-size and pore-size distribution also.

suitable visual separation between particles designated as type VI and type VII in crushed aggregate.

Alumina is the most abundant minor constituent. In the chert varieties I, II, III, and IV, it is probably present in feldspar together with the potash. In type VII, the somewhat greater quantities of alumina and potash reflect the presence of glauconite as well as feldspar in the thin sections. More or less glauconite has been noted in all varieties of chert from the Bois Blanc formation, but it occurs most commonly in type VII. Iron, the remaining minor constituent of importance, is present in all the analyses and illustrates the general distribution of limonite and pyrite. Some of the analyzed iron, in type VII chert, is present in the glauconite lattice.

#### Chemical Reactivity

The possibility of alkali-aggregate interactions exists for all types of chert. To make a preliminary evaluation of the potential reactivity, tests were performed

## PHYSICAL PROPERTIES

Specific Gravity

The specific gravity of chert has been used as a measure of its durability and thus as a means of classifying cherts into groups of equal soundness rating. Disruption by frost action is dependent on many factors, but specific gravity is particularly useful as it is a readily determined quantity and forms the basis for certain methods of beneficiation. Specific gravity measurements were made for all the varieties of chert present in the stratigraphic sections described in detail.

Selected specimens of each variety were used to determine the bulk specific gravity and true specific gravity. The true specific gravity is essentially the specific gravity of the mineral constituents and in this study it was determined by the pycnometer method. The specimens were pulverized by grinding under water to minimize the effects of heat generated during reduction. For the purposes of comparison, selected particles of each type of chert, from a number of gravel deposits, were also used for specific gravity determinations. The gravel particles had values within the range of specific gravities for the corresponding type of nodular quarry-chert.

The appropriate mean specific gravity and range are given for each type of chert in Table 7. Differences in specific gravity exist between individual groups, although there is some overlap in certain cases. Statistical analysis indicates that at each sample locality the chert types are significantly different on the basis of specific gravity. However, examination of all the localities demonstrates that although all types are significantly different on this basis, absorption is a more reliable criterion for the differentiation of individual groups.

The true specific gravities, although consistent within each chert group, were generally lower than might be expected, and in the case of types II, III, IV and V, actually lower than the specific gravity of quartz. The phenomenon is too systematic to have occurred by chance and of sufficient magnitude to suggest a cause other than the very slight decrease in the density of quartz due to prolonged fine grinding. Possibly the failure to remove all the en-

TABLE 7  
BULK DRY SPECIFIC GRAVITY  
OF CHERT

Chert Type	Mean	Range
I	2.175	1.841-2.368
II	2.482	2.452-2.536
III	2.582	2.541-2.598
IV	2.603	2.577-2.612
V	2.574	— <sup>a</sup>
VI	2.623	2.594-2.634
VII	2.446	2.409-2.488
VIIa	2.439	2.416-2.478

<sup>a</sup>One sample only.

TABLE 8  
SPECIFIC GRAVITIES OF CHERT AND HOST ROCK

Type	Bulk Dry Sp. Gr.	Apparent Sp. Gr.	True Sp. Gr.	Theoretical Sp. Gr.
I	2.175	2.653	2.657	2.701
II	2.482	2.638	2.645	2.673
III	2.582	2.616	2.631	2.689
IV	2.603	2.626	2.638	2.686
V	2.574	2.588	2.616	2.658
VI	2.623	2.686	2.696	2.726
VII	2.446	2.668	2.670	2.712
VIIa	2.439	2.692	2.701	2.741
Bois Blanc limestone	2.655	2.721	2.731	2.730
Delaware limestone	2.645	2.725	2.735	2.733

trapped air from the mineral powder in the pycnometer determinations may partly account for the effect, as small discrepancies have been noted in density measurements of this type with very fine materials. In this case, the powders had a maximum particle size of 50 microns and mean diameter of 25 microns. It is probable that the larger particles, which are still several times the average grain size of the main quartz variety, microcrystalline quartz, contain some sealed intergranular pores, but this is unlikely in the finer sizes, raising the possibility that the minute quartz crystals contain even smaller micropores, as suggested by other investigators (6, 9, 10, 11).

To investigate the gross effects of porosity in a general way and to act as an approximate check of the true specific gravity values, a theoretical specific gravity was calculated for each type of chert from the mineralogical composition. The thin section mineralogy supported by normative mineral analyses and X-ray data was sufficiently precise to permit this. Specific gravity values for the constituent minerals were taken from the Handbook of Physical Constants, 1963 (12). Minor amounts of limonite, organic matter, and clay minerals were not allowed for in the computations, but other accessories were included where present in sufficient quantity.

The bulk-dry, apparent, true, and theoretical specific gravities for each type of chert are compared in Table 8. With the exception of the two limestones, the true specific gravities are 1 to 2 percent lower than the theoretical values for all the chert types. The theoretical calculations are of necessity approximate, but there appears to be a general relationship between the content of microcrystalline quartz and the calculated difference. So much so that at a microcrystalline quartz content of 50 percent, the discrepancy is approximately 1 percent, and at a content of 90 to 100 percent microcrystalline quartz it is approximately 2 percent. These observations are consistent with similar findings by other investigators and suggest that the differences are characteristic of quartz in this finely crystalline form.

### Porosity

All varieties of chert are more or less porous, and sources of variation exist in the total pore volume and individual pore size, both within and between the chert types. Due to difficulties in measuring the size of interconnected pores, no accurate information is available concerning the size-frequency distribution of the pore systems in each variety of chert, but pertinent data on maximum pore size and the volume of pores greater than 5 microns in diameter were collected during the thin section examinations.

TABLE 9  
POROSITY AND PORE SIZE OF CHERT AND HOST ROCK

Type	Tot. Porosity	Effective Porosity	Theoretical Porosity	Max. Pore Size Microns	Pores < 5 Microns (% by vol) <sup>a</sup>
I	18.141	18.017	19.474	50	50
II	6.163	5.914	7.146	50	80
III	1.862	1.300	3.980	10	95
IV	1.327	0.876	3.090	10	95
V	1.606	0.541	3.161	10	90
VI	2.708	2.345	3.778	50	70
VII	8.390	8.321	9.808	5	100
VIIa	9.700	9.398	11.018	5	100
Bois Blanc limestone	2.783	2.426	2.783	50	50
Delaware limestone	3.220	2.803	3.220	70	40

<sup>a</sup>Difference between total pore volume and estimated proportion of pores >5 microns (thin section) expressed as percentage of total porosity.



Significance has been attached to the proportion of pores smaller than 4 to 5 microns in size, and thus for the purposes of comparison with other research, the appropriate values are given in this way in Table 9.

The total pore volume, i.e., the volume of permeable and impermeable pores, was calculated for each specimen using the bulk-dry specific gravity and true specific gravity. The effective porosity (also termed apparent porosity or available porosity) being the volume of pores that can be vacuum saturated with water, was also calculated using the apparent specific gravity instead of the true specific gravity. The total porosity was calculated for comparison using the theoretical value for the true specific gravity, and is included in Table 9 as the theoretical porosity.

Total porosities as high as 22 percent were obtained for some specimens of type I chert which is by far the most porous variety. It also has the greatest proportion of larger pores, 50 to 5 microns, and, probably by virtue of this, shows the closest correspondence between the total and theoretical porosity of any of the cherts. The porosity of type VII, although less than that of type I, is still moderately high and conspicuous for the abundance of very fine pores. Type II, is moderately porous and is again characterized by a large proportion of fine pores. Types II, IV, V, and VI, have relatively low porosity but the majority of pores are relatively small. It appears that the maximum pore size in types I, II and VI is controlled by the residual limestone texture, and that in types III, IV, V and VII by the grain size of the quartz. However, the quartz crystallinity is a reflection of the original texture, and in this sense the maximum pore size in these types is also controlled by the texture of the host. The unusually coarse porosity of type I chert cannot be fully explained in terms of original texture. It has been suggested that the high porosity is due to the leaching of carbonates from nodules in strata within the zone of weathering. As this type is found far below the weathering surface, it would seem that this is not the sole cause or even the most important. In addition, nodules of this type are not enriched in silica as might be expected. The mineralogical similarity between types I and VI may have some bearing on the problem, but at this time its significance is obscure.

### Absorption

The property of absorption is common to each class of chert, and the total absorption, in terms of the quantity of water absorbed in a fixed interval of time, varies considerably from type to type. In general, however, the higher the porosity the greater the absorption. Most varieties of chert exhibit noticeable suction, and again, the magnitude of this phenomenon increases with increasing porosity and absorption.

The same specimens used for the determination of bulk and apparent specific gravity were used for absorption measurements. This quantity, normally called the vacuum saturated absorption, was calculated for each type from the specific gravity experiments; the mean values are given in Table 10. In addition, the 24-hr and 1-mo absorptions were determined for each specimen by immersion in water, in an initial oven-dry condition, for the appropriate period of time. The mean values are given in Table 10. In all cases, the absorption is a percentage by volume of the bulk volume of the specimen.

Degree of saturation, which is the ratio of the amount of water absorbed in a specified time under specified conditions to the amount required to saturate the pore system of the specimen completely, has been used as a measure of physical soundness (13). In this study, the volume of water absorbed under conditions of vacuum saturation has been taken as complete saturation, and the degree of saturation at 24 hr and 1 mo is the corresponding amount of water absorbed during these periods expressed as a percentage of the former quantity. Also, the degree of saturation has been calculated as the quantity of water absorbed under vacuum saturation expressed as a percentage of the volume of water that would be theoretically required to saturate the total pore system completely. The latter value is the total porosity in Table 9. After 24-hr soaking, only chert types I, II, VII, and VII(a) have exceeded the theoretical limit of 91.7 percent for critical saturation. At 1 mo, type III has also reached this critical limit. Soaking for longer periods than 1 mo produces no change in the volume of water absorbed other than random variations that are within the limits of experimental error. The degree of

TABLE 10  
ABSORPTION AND DEGREE OF SATURATION

Type	Absorption Percentage by Vol			Degree of Saturation Percentage by Vol		
	Vacuum Saturated <sup>a</sup>	24 Hr Soaking	1 Mo Soaking	Vacuum Saturated <sup>b</sup>	24 Hr Soaking <sup>c</sup>	1 Mo Soaking <sup>d</sup>
I	18.02	17.44	17.71	99.32	96.82	98.29
II	5.91	5.54	5.68	95.96	93.69	95.98
III	1.30	1.15	1.20	69.82	88.23	92.61
IV	0.88	0.72	0.77	66.01	82.32	87.38
V	0.54	0.44	0.46	33.69	80.92	84.30
VI	2.35	1.99	2.00	86.60	84.84	85.16
VII	8.32	8.20	8.24	99.18	98.68	99.01
VIIa	9.40	9.10	9.10	96.89	96.85	96.84
Bois Blanc limestone	2.43	1.87	1.97	87.17	77.23	81.01
Delaware limestone	2.80	2.22	2.39	86.96	79.19	85.35

<sup>a</sup>Quantity absorbed by 1 hr saturation under vacuum and 23 hr soaking.

<sup>b</sup>Volume of water absorbed by vacuum saturation expressed as percentage of volume required to fill total porosity.

<sup>c</sup>Volume of water absorbed in 24 hr as percentage of volume absorbed by vacuum saturation.

<sup>d</sup>Volume of water absorbed in 1 mo as percentage of volume absorbed by vacuum saturation.

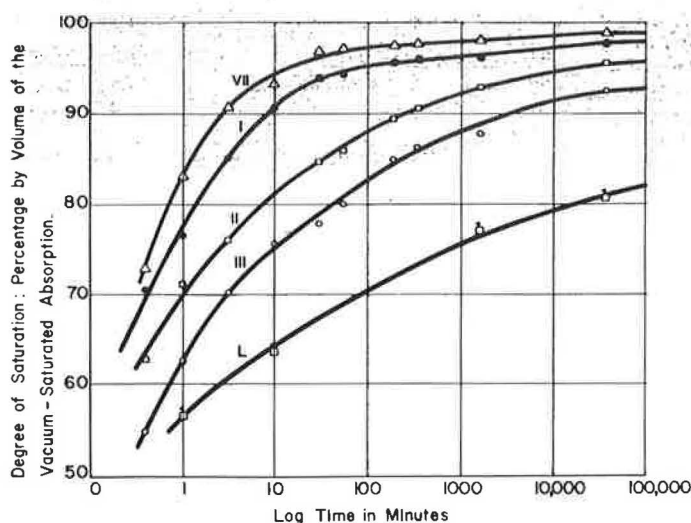


Figure 14. Rate of absorption for chert types I, II, III and VII and Bois Blanc limestone, L.

saturation in terms of the total porosity again shows types I, II, VII, and VII(a) to be critically saturated. Types III, IV and V, however, show relatively low degrees of saturation, apparently a further indication of the disparity between total calculated porosity and measured effective porosity for these varieties.

To investigate the rate of absorption, selected specimens of types I, II, III and VII were oven-dried, and then immersed in water for specific time intervals. The specimens were then removed from the water and weighed to determine the quantity of water



TABLE 11  
AGGREGATE CRUSHING STRENGTH

Chert Type	I	II	III	VI	VII
Aggregate crushing value	19.0	19.5	19.5	15.0	18.8

rapidly for the first 10 min, and the relationship is approximately linear over this period of time. Thereafter, absorption is less rapid, decreasing to zero at 1 mo. Within 3 min, types I, II, III and VII have reached approximately 85, 76, 70 and 87 percent saturation, respectively.

### Crushing Strength

Efforts to measure the compressive strength of cylindrical test specimens of chert yielded highly variable results, probably due to the heterogeneity of the specimens. To obtain some data on the mechanical strength of different types of chert,  $\frac{3}{4}$ - to  $\frac{1}{2}$ -in. aggregate samples of each variety were subjected to the Aggregate Crushing Test BS, 812. No great differences are indicated between the respective losses in Table 11, although type VI has a value somewhat smaller than the rest. Perhaps of more significance is the number of cracked particles not counted as loss, which are, except for type VI, equally as abundant as the loss. In addition, types II, III and to some extent type VII, produce large numbers of flakes or splinters on fracturing which may be due, in part, to the abundance of microcracks present in many specimens of these types.

## PERFORMANCE TESTS

### Stripping of Asphaltic Cement

The evaluation of asphalt stripping for each variety of chert was made by means of ASTM Test, DD1664, 59T—for dry aggregates—at a water immersion temperature of 120 F for 24 hr, using 85/100 penetration grade asphaltic cement. The results given in Table 12 indicate that only type VI chert is sufficiently resistant to asphalt stripping to be suitable for asphaltic cement concrete construction.

### Freeze-Thaw Resistance

The measurement of physical soundness formed an integral part of the evaluation of basic properties of different varieties of chert. Soundness is the ability to withstand disintegration or destructive volume change as a result of freezing and thawing in a wet condition. Increase in volume without particle disintegration can normally be tolerated in asphaltic concrete, but is permissible only within narrow limits in portland cement concrete. Consequently, measurements of volume change are important in the correlation of basic properties and field performance. At first the magnesium sulfate test was used to determine the disintegration, but many of the results proved to be anomalous, and the cyclical freezing and thawing of saturated unconfined particulate samples was a more suitable method.

Bulk aggregate samples of each type of chert were vacuum saturated and subjected to cyclical freezing in air and thawing in water. The duration of freezing was established at 3 hr at a cooling rate of 23 F/hr, and the period of thawing at  $\frac{1}{2}$  hr at a water temperature of 70 F. The testing was performed on the  $\frac{3}{4}$ - to  $\frac{3}{8}$ -in. size group but the samples were separated into two fractions for the test,  $\frac{3}{4}$  to  $\frac{1}{2}$  in. and

TABLE 12  
STRIPPING OF ASPHALTIC CEMENT

Chert Type	Percentage Retained Coating After Test	Remarks
I	10	Very poor
II	28	Poor
III	10	Very poor
IV	10	Very poor
VI	89	Good
VII	5	Very poor

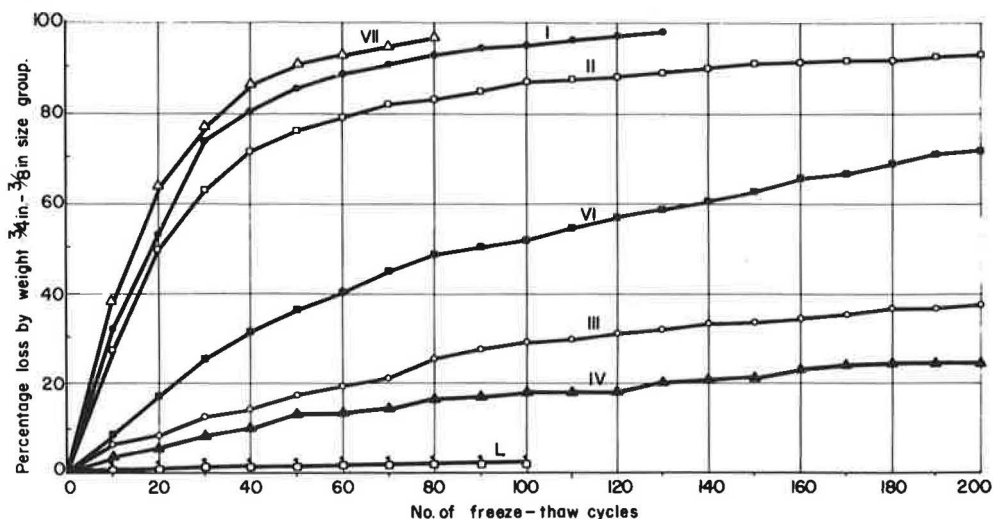


Figure 15. Disruption by unconfined freezing and thawing of chert types I, II, III, IV, VI, and VII and Bois Blanc limestone, L.

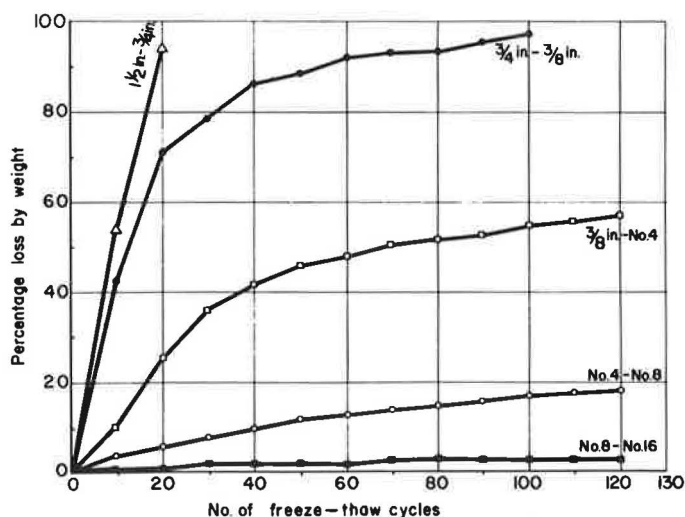


Figure 16. Disruption by unconfined freezing and thawing of individual size groups of type II chert.

$\frac{1}{2}$  to  $\frac{3}{8}$  in., and the results were subsequently combined. The percentage loss was determined on the finer sieve, in each case, at 10-cycle intervals. The test was carried to 300 cycles or until a loss of 95 percent was achieved. Beyond 200 cycles, however, further losses were negligibly small, and, in the case of the reference Bois Blanc limestone, no additional loss was noted past 90 cycles. The cumulative losses are plotted as freeze-thaw curves in Figure 15.

Some correlation exists between absorption and freeze-thaw loss but there is no direct relationship between the magnitude of the loss and either the porosity or the amount of absorption. A closer parallel is apparently seen between the rate of absorption and freeze-thaw losses; however, here again, although those cherts reaching the theoretical degree of critical saturation sooner have higher losses, the relative losses at a specific



number of freeze-thaw cycles are not directly proportional to the rate of absorption. The specimens with high capillary suction, showing the most rapid rates of absorption and exhibiting the highest degree of water retention under vacuum drying, i.e., types I, II, and VII, form a series of closely related freeze-thaw curves of similar shape. Disintegration is very rapid for the first 30 to 40 cycles; the rate of loss then decreases abruptly and thereafter continues at a much reduced level. Types III and IV and the Bois Blanc limestone form a series of curves of somewhat different shape that show a progressively decreasing rate of loss after initial disintegration produced during the first few cycles. In view of the severity of the test, which continually exposes the particles to freezing in a critically saturated state, it is evident that a marked difference in behavior exists between those cherts represented by the two series of freeze-thaw curves. The curve representing type VI is apparently intermediate between these two types of curve. However, subsequent examination of the test samples indicated that they were a heterogeneous mixture of types VI and VII, further emphasizing the difficulty of making a precise bulk separation of these two varieties in crushed aggregate.

The shape of fragments produced by freeze-thaw disintegration is of some interest inasmuch as the microstructure of certain varieties of chert may directly influence the type of breakdown and, consequently, the recorded loss. Most of the type I particles break down immediately into small irregular fragments appearing to disintegrate by crumbling. Type II also breaks into irregular fragments, but the fragments are more angular. It is not, however, reduced immediately to fragments as small as type I, and in the majority of cases, individual particles are disrupted by only one or two fractures. Chert type VII again breaks down rapidly into smaller fragments, but in this case, predominantly thin platy fragments which may be the result of fractures along planes of structural weakness inherited from the limestone. Types III and IV, the cherts with conspicuously developed systems of microcracks, do not generally fracture through the particles, although the loss portion is composed of thin flakes, slivers, and curved plates. These minute fragments appear to originate by spalling from the surfaces of individual particles, and from the quantity it would seem that many of the particles are involved.

The concept of critical size has received much attention in literature (13, 14); it is the particulate size of a rock type of given porosity and permeability below which disruption due to freezing will not occur under a given set of conditions. To evaluate this concept in a practical way and provide data on the physical soundness of different sizes of chert, the seven size groups from  $1\frac{1}{2}$  to  $\frac{3}{4}$  in. through No. 30 to No. 50 were subjected to cyclical unconfined freezing and thawing using the same procedure as previously outlined. Figure 16 shows the freeze-thaw curves for each size group of crushed material from selected nodules of type II chert. The curves are all essentially the same shape but the ultimate magnitude of the loss diminishes abruptly with decreasing size. At 100 cycles the  $1\frac{1}{2}$ - to  $\frac{3}{4}$ -in. size group has registered a 100 percent loss, and the  $\frac{3}{4}$ - to  $\frac{3}{8}$ -in. group, 97 percent. The  $\frac{3}{8}$  in. to No. 4, No. 4 to No. 8, and No. 8 to No. 16 groups have proportionately smaller losses which are 56, 18 and 3 percent, respectively, of the  $\frac{3}{4}$ - to  $\frac{3}{8}$ -in. loss. Sizes below No. 16 had approximately the same loss curve as the No. 8 to No. 16 size group.

Obviously, there is no critical size under the conditions of unconfined freeze-thaw testing used here, but there is a marked difference in the disintegration characteristics of different sizes. So much so that the fine aggregate fraction of a graded sample of this type of chert can be expected to show an integrated loss of only 14 percent of the coarse aggregate fraction. The freeze-thaw loss, in terms of size, is essentially the same for the other varieties of chert, modified by their relative susceptibility, as shown in Figure 15.

## DISCUSSION

It is pertinent to inquire whether or not the data collected concerning the character and basic properties of the cherts are sufficiently definitive to provide a framework for classification.

In terms of the mineralogy, the content of silica, calcite and dolomite, and the variety and texture of the silica minerals, significant differences do exist. These differ-

ences apparently reflect specific textural relationships between the host limestone and the chert nodules, and support the initial subdivision into seven groups. Nodules of different types are visually recognizable by variations in color and texture. A possible exception to this generalization is type VII, which is texturally earthy to porcelaneous and may be confused in bulk separation with particles of incipient chertified limestone (type VI). Field relationships are probably more diagnostic in this case. The differences between types III, IV and V may seem less significant than between the other varieties, but in view of the variation in silica content it is probably worthwhile to retain these arbitrary subdivisions until performance tests indicate otherwise.

The system of classification should provide an adequate starting point from which to assess the correlation between the basic properties and performance of chert, and subsequently a suitable scheme for the petrographic recognition and qualitative rating.

## CONCLUSIONS

The occurrence of chert in southwestern Ontario has been studied both in nodular, chert-bearing limestones and Pleistocene gravels. The nodules are principally developed in the Bois Blanc and Delaware formations of Devonian age, which underlie glacial deposits in two broad arcuate outcrop belts stretching from the Niagara Peninsula to Lake Huron. In the Bois Blanc formation, chert constitutes 30.3 to 62.3 percent by volume of the limestone, and 8.5 to 24.8 percent in the Delaware formation. Particles of gravel chert derived from these two parent formations constitute from 1 to more than 50 percent of Pleistocene gravels in the area, and it was possible from the analysis of the deposits to compile a map of chert distribution showing zones of equal chert content and relating maximum concentration to the outcrop belts.

The primary purpose of the study was to establish a classification of chert, based on its fundamental character and properties, whereby different varieties can be recognized visually. Seven types of chert, numbered I to VII, were recognized on the basis of color and texture. Microscopic study revealed that microcrystalline quartz was the dominant silica mineral with cryptocrystalline quartz less common and fibrous quartz a minor constituent. Calcite and dolomite, the latter in the form of euhedral rhomb-shaped crystals, are always present and may constitute up to 50 percent of types I, VI and VII. Type VII chert is the only variety in which cryptocrystalline quartz occurs and here it is the major silica mineral. Types I to V show increasing silica content to a maximum of 95 percent. Type VI, although similar mineralogically to type I, is texturally a chertified limestone. Clay minerals are unimportant and minor pyrite, glauconite and detrital quartz are the only conspicuous accessories. Chemical analysis and X-ray diffractometry confirm the microscopic findings. Oriented microcracks were a pronounced feature in many nodules of types II and III and may be correlated with the tendency of the nodules to break into thin splinters or curved flakes.

The bulk, apparent and true specific gravities were measured for each type of chert, and the appropriate porosities calculated. An estimation of the proportion of pores smaller than 5 microns was made petrographically. Porosities ranged from 1.3 to 18.1 percent by volume and the corresponding 24-hr absorptions from 0.7 to 17.4 percent. The degree of saturation in each case was determined, and to clarify further the absorptivity, the rate of absorption was measured.

A parallel is seen between the rate of absorption and freeze-thaw soundness inasmuch as those cherts reaching critical saturation rapidly also exhibit rapid early loss in unconfined freeze-thaw tests. Although some cherts show relatively little disintegration in this test, all varieties increased in volume when subjected to frost action, approximately in relation to their porosity. This is true even for types that cannot be critically saturated.

The crushing strength and chemical reactivity were investigated, but no major differences were noted between the various types of chert. The rapid chemical test for alkali-reactivity gave results suggesting that all types are highly reactive. The content and type of silica minerals has little effect on the reactivity. Conversely, the resistance to stripping of asphaltic cement was found, in some cases, to increase with decreasing silica content.

It is concluded that different types of chert existing in southwestern Ontario can be classified visually. Color alone is not sufficiently diagnostic, and accurate classification presupposes a knowledge of the mineralogy, character, and basic properties.

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