

Tests for Freeze-Thaw Durability of Concrete Aggregates

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Tests of aggregate in air-entrained concrete have been made by methods suggested by T. C. Powers (1) for resistance to freezing and thawing. The procedure differs from that of currently used test methods in several important respects. Among these are (a) maintenance of the original moisture in the aggregates, (b) testing of the largest particle sizes to be used in the work, (c) subsequent conditioning of the cured concrete by drying to a degree found appropriate to exposure conditions at the site of construction, and (d) freezing at a rate commensurate with natural conditions. Methods and apparatus used in conducting the tests are described, and results of variations in test procedure are shown.

Specifications based on the test procedure have been used for the acceptance of aggregates in construction work that is subject to severe winter conditions at high elevations in California. Many of the aggregates would not be considered to be acceptable under commonly used freeze-thaw methods. One hundred and seventy-three lane-miles of pavement have been subjected to one or two winters of severe exposure. At present, the concrete is judged to have withstood the effects of exposure without evidence of distress due to freezing and thawing.

● THIS REPORT describes test methods used to evaluate the frost resistance of aggregates when incorporated in air-entrained concrete. The concept of the test procedure was provided by Powers (1, 2). As far as known, the acceptance under contract procedure of aggregates based on this concept has not previously been undertaken.

In California most of the concrete pavements have been constructed at relatively low elevations where freezing weather is of rare occurrence. Until recently, the Division of Highways has had little occasion to study the ability of locally available aggregates to produce frost resistant, air-entrained concrete.

A decision to pave 70 mi of Rt 80 between Colfax at an elevation of 2,500 ft and the Nevada state line near Reno with portland cement concrete, provided the impetus for the extensive investigation of available sources of aggregates. This road reaches an elevation of 7,135 ft at Donner Pass, then drops to an elevation of 5,000 ft at the Nevada state line. Precipitation is heavy on the western slope. The annual snowfall near the summit is among the heaviest in the United States. Temperatures as low as -25 F are not uncommon near the Nevada state line.

In considering methods of making freezing and thawing tests of concrete, the two papers by Powers (1, 2) were studied carefully. They were believed to contain a number of proposals of distinct merit. Equipment to perform tests by the Powers procedure as well as that for making the four ASTM tests was obtained.

Six points made by Powers are considered to be of importance. These are given

as follows, along with the test procedure used to put them into effect.

SIZE OF AGGREGATE

There is a critical size of aggregate with respect to its frost resistance. In general, the larger its size the greater its probability of being vulnerable. Therefore, test specimens should contain the largest size of aggregate to be used in the work and the dimensions of the specimen should be adequate for this purpose.

Specifications for the projects under consideration required that the concrete contain 1½-in. maximum size aggregate. This size range was used in preparing laboratory mixtures for test. Cylinders 4½ in. in diameter by 9 in. high were molded for the Powers test. While it might have been possible to consolidate specimens of smaller size, the number of the large size particles of aggregate in each specimen would have been reduced. Since the larger particles are more likely to be the critical ones, it was not thought advisable to use a smaller test specimen. Specimens for ASTM rapid freezing and thawing in water were 4- by 5- by 18-in. prisms, on which dynamic E was measured, or 4½- by 9-in. cylinders, on which length changes were measured.

MOISTURE IN AGGREGATES

Aggregates that have been dug from locations below the water table if subsequently allowed to dry may not regain their full amount of water by simple soaking for a reasonable length of time. If the aggregates as incorporated in test concrete are not saturated to a degree comparable to the condition in which they are used in the work, the test results can be very misleading.

Specifications require that aggregates be washed before use. In practice, aggregates are usually dug, screened, washed, and batched without any opportunity for drying. Preliminary test samples of pit run material were taken below the water table. Samples from manufactured stocks were taken only where free surface moisture was visible. They were placed in metal cans with tight fitting covers. Additional water was placed in each can. In the laboratory, the aggregates were maintained in a thoroughly wet condition during screening and other processing and were introduced into the mixer while wet. It might have been possible to resaturate dried aggregates under vacuum but uncertainty as to completeness of saturation led to the adoption of the first procedure.

AIR BUBBLE SPACING

For tests of air-entrained concrete, the paste should be protected with bubbles, and adequate protection requires that the calculated spacing factor not exceed 0.01 in.

The laboratory is not equipped to make linear traverse measurements of polished sections. It was assumed that the use of neutralized Vinsol resin in an amount to result in a measured air content of 4.5 ± 0.5 percent would fulfill the bubble spacing requirement.

RATE OF COOLING

The rate of cooling in the laboratory test should not be greatly higher than the rate experienced under natural conditions of exposure. The use of high cooling rates in the laboratory as required in some current methods subjects the concrete to internal hydraulic pressures of a magnitude much greater than experienced in nature and may produce misleading results. Such a test may serve to reject aggregates that would perform satisfactorily in service.

Prior to starting tests, an experimental slab 12 ft square had been installed with thermocouples at Donner Pass, the highest elevation of the proposed construction. Temperature measurements were recorded during one winter. In the range below 32 F, the greatest rate of temperature drop within the concrete did not exceed 3 F per hour except on one day when a drop of 6 F in one hour was recorded at a point just below the surface of the slab. Powers has suggested a cooling rate in the test of 5 F per hour and this rate was adopted in the work.

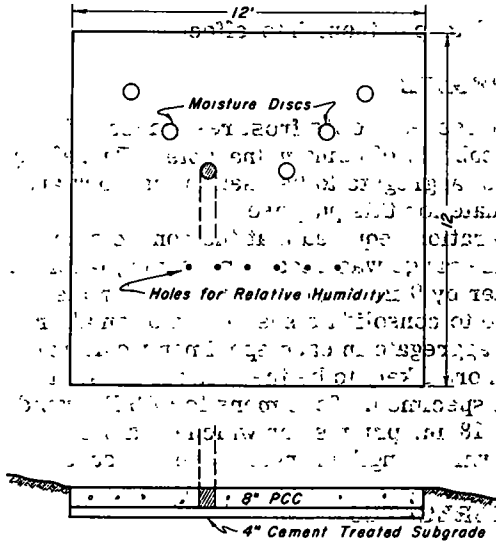


Figure 1. Typical test slab for moisture measurement.

Unless the concrete is to be exposed in such a way that it will never have an opportunity to dry, it will dry to some extent during each summer season. Concrete specimens should be conditioned by partial drying to a degree comparable to field conditions before subjecting them to freezing and thawing.

The four ASTM methods require that freezing and thawing tests be started after 24 hr of moist curing in the fog room followed by immersion in water saturated with lime to the age of test. Corps of Engineers method C.10-54 has a similar requirement except that storage is in the fog room until 48 ± 4 hr prior to test when they are stored in saturated lime water. None of these methods provides for preliminary drying of the concrete.

California Division of Highways has conducted tests of concrete slabs for the purpose of estimating the distribution of

moisture in concrete pavements throughout annual cycles of weather. The first of these experiments was conducted in Sacramento during 1953-4. Subsequently test slabs have been installed at two locations at high elevations on Rt 80. Although the slabs have differed somewhat in design detail, a typical layout is shown in Figure 1. The test slabs are 12 ft square and 8 in. thick. They are supported by a cement treated subgrade with bituminous seal in accordance with prevailing practice in California. The slabs are located in driveways leading to maintenance stations. These sites were selected to provide assurance that snow would be removed at the frequency occurring on the highway proper.

Holes 6 in. in diameter extending from the surface to the subgrade, were formed ahead of placing the concrete. During placing, a number of 6- by 12-in. cylinders were molded. A central rod in the mold provided a cast-in-place hole at the longitudinal axis of the cylinder. After curing for 14 days, the cylinders were sawed into discs 1 in. thick which were then lapped to provide intimate contact between them when stacked. Each disc while still in an undried condition, was weighed to the nearest gram and then subjected to drying at 220 F to 230 F to essentially constant weight. The loss in oven drying is called the "evaporable water." The value is not constant between discs because of non-uniformity in distribution of coarse aggregate in specimens of such size. Typically, each disc contained about 50 g of evaporable water. A change of 1 g in weight therefore, represented a change of about 2 percent in evaporable water content.

After drying, the discs were soaked in water for several days and then bolted together to form a stack 8 in. high as shown in Figure 2. The assemblies were then inserted in the precast holes of the test slab. A small amount of calking compound was placed in the annular space at the surface to prevent access of surface water. The diameter of the discs was about 0.02 in. less than that of the hole. The clearance was so small that considerable practice was required in assembling the stacks so that they could be inserted in the slab. Regardless of the moisture content of the discs relative to that of the slab at the time of insertion, it is believed that equilibrium with the slab proper becomes established after a period of time.

Test slabs were constructed in August. Discs were inserted in September and first removed for weighing in October. They were weighed subsequently at monthly intervals up to some date in late November or early December when the slab became frozen to its entire depth. Periodic weighings were resumed in the spring and continued until

freezing weather the following winter. Changes in weight of each disc were recorded as percentages of evaporable water.

Each of the test slabs contained a series of metal-lined holes with the concrete at the bottom exposed at varying depths. Metal plugs remained in place at the top of the sleeves except when electric hydrometers were inserted to measure the relative humidity of the air within the concrete.

Thermocouples were installed at varying depths within the slab and temperatures were recorded automatically during the winter season.

The first of such test slabs, containing only moisture discs, was installed in a field at Sacramento (elevation 25 ft) in August, 1953. Actually there were two smaller slabs each containing two stacks of discs. One slab was on a cement-treated subgrade with bituminous seal, the other on natural earth. The presence or absence of a cement-treated subgrade with bituminous seal made no significant difference in the measured moisture changes in the slabs during the ensuing year. Figure 3 shows the moisture changes during an annual cycle at Sacramento as the average of changes in the four stacks of moisture discs. It will be noted that the top disc, 1 in. in thickness, became progressively drier starting in March, and by September contained only 26 percent of its evaporable water. Changes in the lower discs were progressively less. The bottom disc lost only 8 percent of its evaporable water despite the fact that less than 0.2 in. of rain fell between the middle of May and the first of November. Summer weather at Sacramento is hot and the relative humidity is low.

Figure 4 shows moisture changes at Donner Summit (elevation 7, 135 ft) as the slab approached two winters. Changes in the top set of discs are shown independently. The balance are grouped within the band as shown to avoid confusion. The significant fact to be noted is that although the top disc responded to short changes in weather conditions, the balance of the concrete slab entered the period of severe winter freezing in 1956 with an evaporable water content of 85 to 89 percent. The slab contained slightly more moisture in December-1957.

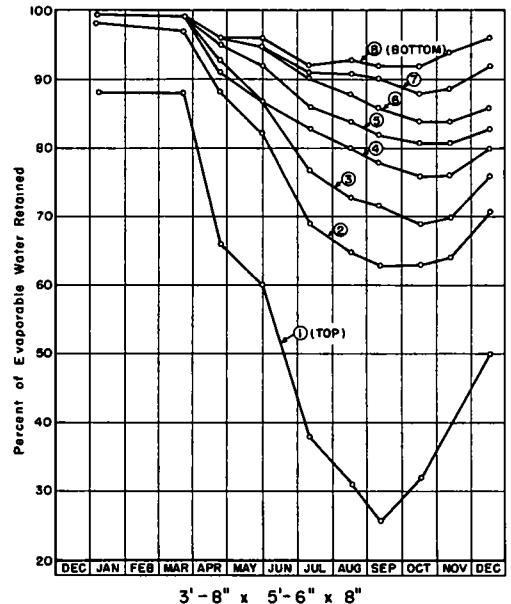
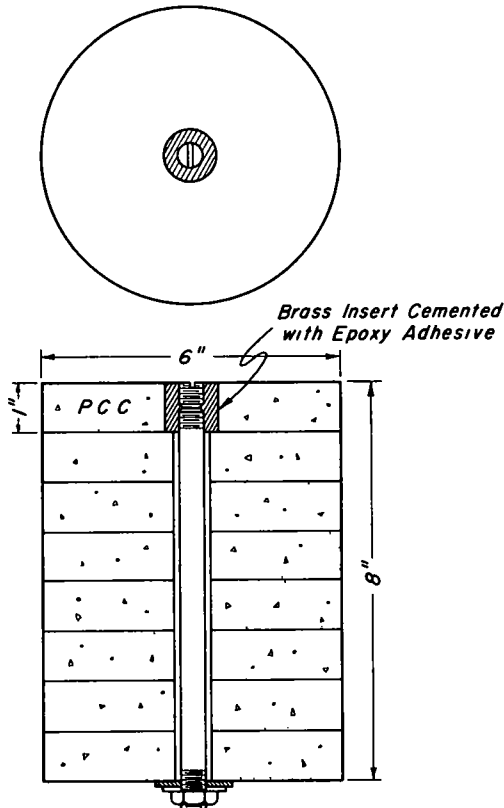


Figure 3. Test slabs at Sacramento (placed Nov., 1953).

Figure 2. Assembly of moisture discs.

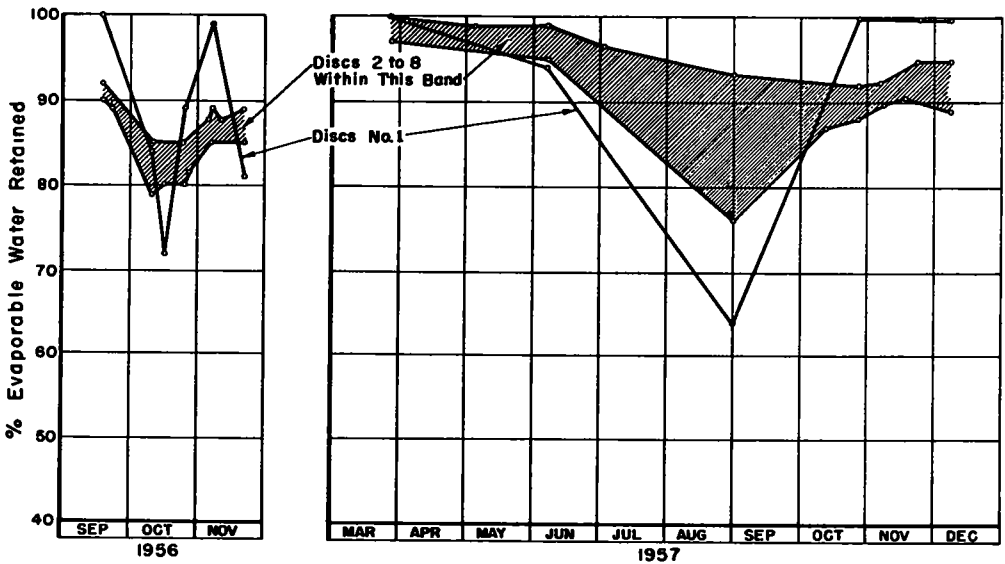


Figure 4. Test slab at Donner Summit (placed Aug. 9, 1956).

A third test slab at Yuba Gap (elevation 5,700 ft) yielded results similar to those at Donner Pass, except that a higher degree of saturation resulted. The site, however, was at a poorly drained location which is not considered to be representative of a modern highway. The results are not given in this report.

Measurements of relative humidity within the slabs were not completely reliable particularly when the temperature was below 45 F. However, it is believed that relative humidities (given in Table 1) at the start of the winter season are reasonably correct.

From the data obtained, it was concluded that specimens containing the kind of concrete present in the test slabs would be suitably conditioned for freeze-thaw tests if, after fog-room curing for 14 days, they were allowed to dry until the evaporable water content reached about 85 percent and the relative humidity was also about 85 percent. Experiments with test cylinders containing aggregates similar to those of the Donner Pass test slab indicated that they lost 15 percent of evaporable water when subjected to drying at room temperature and about 50 percent relative humidity for 48 hr. It was expected that concrete containing other aggregates might lose water at different rates. Also, it was believed that the best representation of site conditions would be obtained by a constant period of drying rather than by drying to a predetermined loss in weight. Therefore, the time of conditioning test specimens by drying was established at 48 hr. In order to effect a more uniform distribution of moisture within the specimen and to approach a relative humidity of about 85 percent within the concrete, the air-dried specimens were placed in sealed containers above a saturated solution of sodium acetate for 5 days. The theoretical relative humidity of the atmosphere surrounding the specimens was 76 percent at 68 F. It was assumed that after 5 days, the relative humidity within the specimen was about 85 percent. Subsequent tests, however, have shown that this procedure frequently produced a degree of drying somewhat greater than was intended. Later in this report, data are presented to show the effect of different degrees of drying.

TEST PROCEDURE

The proposed approach is to determine the change in length of concrete while it is being slowly cooled below the normal freezing point. If the concrete shrinks normally

in the freezing range, it is immune at the time of test. If it dilates, it is not immune; the process that eventually causes disintegration has begun. It is proposed to make such a test after each two weeks of water-soaking a specimen that previously has been conditioned in air to represent job expectations. Soaking should be continued until the longest safe period has been found. Loss of water from the specimen during freezing should be prevented. If the safe period of soaking exceeds the probable duration of freezing weather each year at a specific construction site, the concrete may be considered to be safe from the danger of damage from freezing at the site.

Specimens were molded from concrete containing $5\frac{1}{2}$ sacks of cement per cubic yard to which sufficient Vinsol resin solution was added to produce $4\frac{1}{2} \pm \frac{1}{2}$ percent air and water to give a slump of 2 in. Originally, duplicate specimens were molded in the form of $4\frac{1}{2}$ - by 9-in. cylinders with gage studs and 4- by 5- by 18-in. prisms. The cylinders were tested by the Powers procedure. The prisms were used in rapid freezing and thawing in water (ASTM C 290) on which changes in dynamic E were measured. Later $4\frac{1}{2}$ - by 9-in. specimens were used in the rapid freezing and thawing in water test and deterioration was measured by change in length.

Originally each specimen for the Powers procedure contained a thermocouple, but it soon became evident that all specimens in a cooling bath cooled at the same rate. Thereafter, a single dummy specimen with a thermocouple was used for temperature measurement. Prior to testing by the Powers procedure, the specimen was placed in a rack made of Invar steel, except for a brass insert at the top, on which was mounted a linear variable differential transformer (see Figs. 5 and 6).

Length changes indicated by the transformer were recorded continuously on a strip chart. Temperature changes were recorded on another chart. Records were obtained while the specimens were being cooled from about 50 F to 0 F. Equal time intervals were laid off on each chart and a plot of length change versus temperature was constructed for each specimen. The characteristics of the plotted curve were used to evaluate the behavior of the specimen as it was cooled above and below the freezing point.

Cooling was accomplished in a bath of water-saturated kerosene as a means of preventing gain or loss of moisture from the specimen. Cooling equipment consisted of two small commercial household freezing boxes which were lined with

TABLE 1
RELATIVE HUMIDITY WITHIN TEST SLABS AT
START OF SEVERE WINTER WEATHER

Date	Depth Below Surface of Slab (in)	Relative Humidity (%)
(a) Donner Pass—First Year, 1956		
11/7/56	1	83
	2	85
	3	90
	5	98
	7	89
	9 (in subgrade)	82
11/20/56	Slab temperature below 32 F Readings not reliable	
(b) Yuba Gap—First Year, 1957		
11/22/57	$\frac{3}{4}$	76
	$1\frac{1}{2}$	89
	3	74
	5	94
	7	98
	$8\frac{1}{2}$ (in subgrade)	89
12/12/57	Slab temperature below 32 F Readings not reliable	

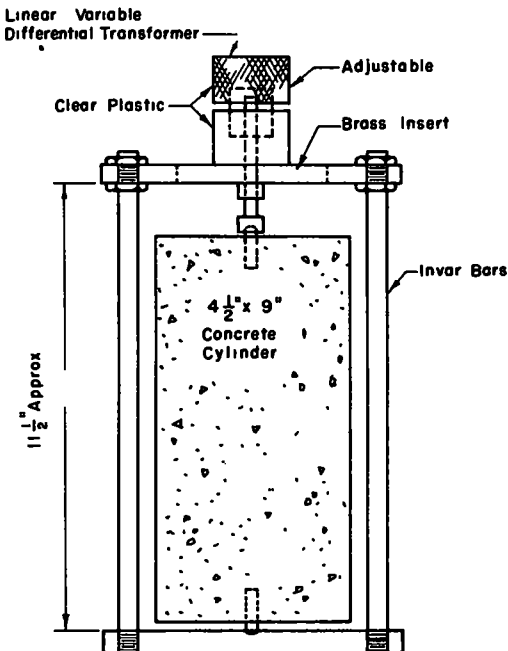


Figure 5. Frame for continuous measurement of dilation.

copper sheeting to prevent leakage of kerosene to the insulation. The units have inside dimensions of 14 by 26 by 18 in. deep and each provides space for four test specimens plus a dummy specimen containing a thermocouple. The kerosene was agitated by a small circulating pump, which was used only to establish equilibrium conditions at the start of a test. Rate of cooling was adjusted to 5 F per hour quite successfully without automatic control. Views of cooling units in operation are shown in Figures 7 and 8.

INTERPRETATION OF RESULTS

Discussions with other engineers revealed that methods of making freezing and thawing tests, and the establishment of test limits generally have been adjusted in accordance with service experience in the locality. California did not have an extensive service record of air-entrained concrete in severe climates and thus was handicapped with respect to a basis for judgment of locally available materials.

A concrete pavement of non-air-entrained concrete had, however, been constructed at Donner Pass in 1937. This pavement suffered surface scaling early in its history but the concrete otherwise has remained in excellent condition. Concrete aggregates from the American River near Sacramento had been used in this construction. In this report, aggregates from this source will be designated as No. 1. It was learned that a considerable number of minor structures had been constructed in Nevada over a period of several years using air-entrained concrete containing aggregates produced from the Truckee River in the vicinity of Reno. Examination of these structures led to the conclusion that this aggregate (No. 2) was capable of producing durable concrete.

Two examples of known durable aggregate which could be obtained for testing purposes were thus provided. Samples of an aggregate of known poor service history—a limestone from the Rapid formation in Iowa—were obtained through the courtesy of the Iowa State Highway Commission.

Results of test with the three aggregates described above provided guide marks in establishing quantitative limits of performance in the Powers procedure.

Figure 9 shows idealized cooling curves. Curve 1 represents thermal contraction above the freezing point. The measured slope is not strictly proportional to the thermal coefficient for two reasons. The first is because the over-all thermal coefficient of the frame supporting the specimen is not zero. The second is because movement of water within the paste and aggregate does not have time to reach complete equilibrium when the temperature is being lowered continually. Curve 2 represents the contraction after ice begins to form in the concrete. Ice crystals under progressive cooling, tend to attract moisture at the expense of that in the paste, causing the latter to shrink at a rate greater than that due to thermal contraction alone. The point of intersection of Curves 1 and 2 indicates the temperature at which ice begins to form. If experimental curves could be obtained with the precision of those shown in Figure 9, the freezing point could be determined accurately.

Curve 3 represents the type of result that has been obtained in certain instances. In this case, there is little or no change in length while the specimen is being cooled several degrees below its freezing point. Eventually the curve resumes a downward slope. Dilation has occurred as measured by the distance, a , which is the greatest distance between Curves 3 and 2. Dilation of this type is extremely difficult to measure from the plotted curves obtained in the study.

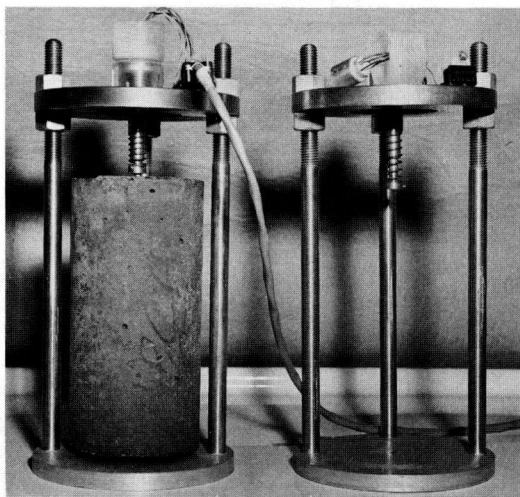


Figure 6. Frames for continuous measurement of dilation.

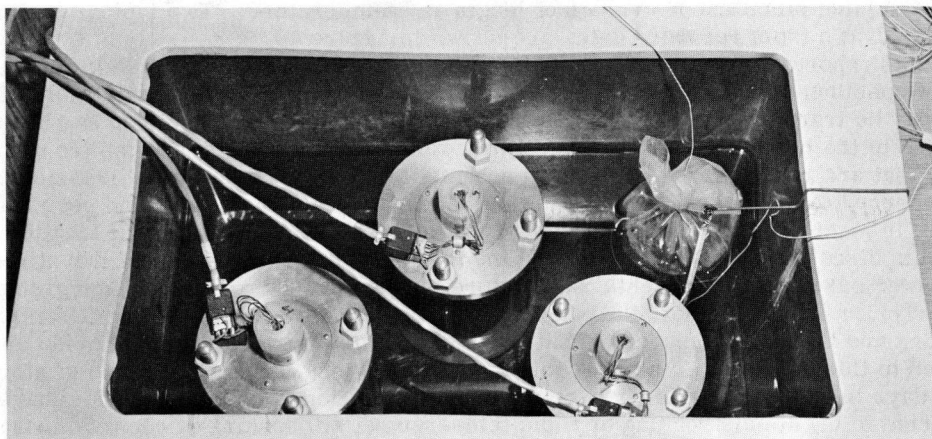


Figure 7. Specimens in cooling unit.

The most common type of curve that is obtained in the work when the concrete is not immune, is represented by Curve 4. Here there is an abrupt expansion at the freezing point. The curve rounds quite sharply and then assumes a downward slope. The distance, b , is easily measured; however, it does not represent the entire dilation because of its nearly horizontal trend over a few degrees of cooling. The distance, b' , represents the complete dilation but is difficult to measure in practice.

It is evident that for the purpose of acceptance or rejection, the selected limitation on dilation must be one that can be measured with reasonable assurance. The chart on which length changes are recorded can be estimated to the nearest 0.000025 in. The gage length of the test specimen is $7\frac{1}{2}$ in., therefore, the recorded length change can be estimated to the nearest 0.00003 in. per in. The chart on which temperatures are recorded can be read to the nearest 1 F. This is the temperature near the center of the specimen and the outside is slightly cooler. Although separate charts are used for recording length and temperature, it is believed that there is no significant error

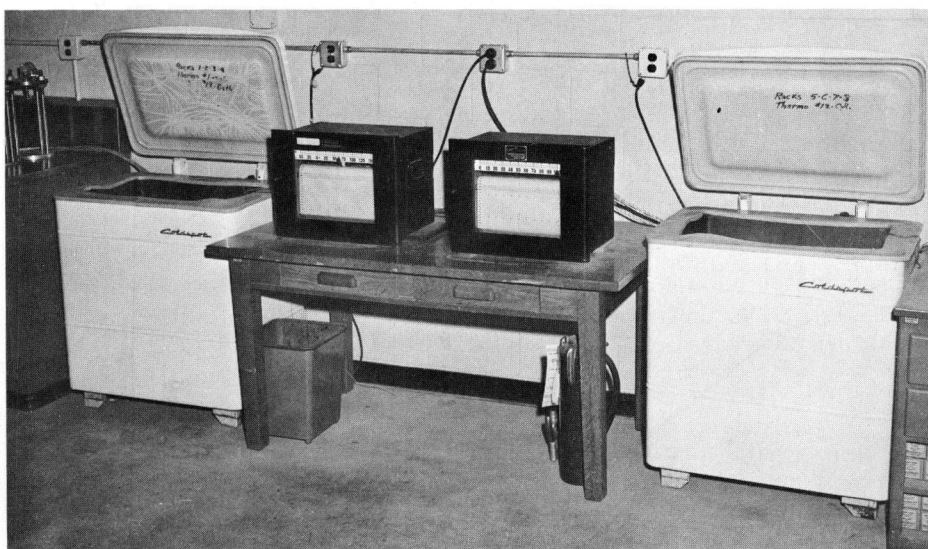


Figure 8. Cooling units and recorders.

in determining simultaneous values of length and temperature. Two typical cooling curves, drawn from recorded data, are shown in Figure 10.

In this report, the expression "dilation" is used to denote an increase in length of test specimens as they are cooled into the freezing range of contained water. Dilation may be transitory; that is, it may not be retained after the specimen has been warmed to the temperature at which cooling was started. Length changes from any cause that are retained when measured at equal temperatures above the freezing point, are referred to as "permanent changes in length." Values of dilation and permanent change in length are reported in terms of millionths (unit change per unit length).

In his discussion, Powers (1) raised the following question and stated that it required an answer based on experimental data before the test procedure can be interpreted properly: In the absence of dilation, is the absorption rate increased significantly by freezing and thawing compared to that obtained by simple soaking? To provide an answer to this question, similar specimens, after drying (drying consisted of storage for 7 days in a closed container over a saturated solution of barium chloride) have been subjected to (a) simple soaking at room temperature, (b) soaking at room temperature with intervening cycles of temperature variation in water in the range of 70 F to 120 F at the rate of 5 cycles per week, and (c) soaking in water at room temperature with intervening cycles of freezing in water-saturated kerosene at the rate of 5 cycles per week. During procedure (c) the temperature was lowered at the rate of 5 F per hour. After the specimens were cooled to 0 F, they were transferred manually to a water bath where they remained at all times except while being frozen. (Test results are shown in Figs. 11 and 12.) Although differences in the amount of water absorbed after drying were not large, schedule (c) produced the greatest dilation and the greatest final permanent change in length. Schedule (c) was adopted as standard for acceptance testing of aggregates.

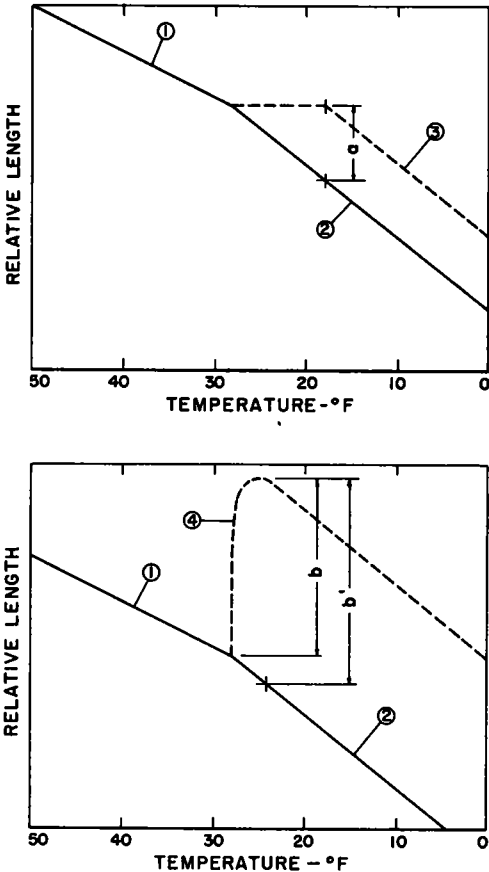


Figure 9. Idealized cooling curves.

DURATION OF TEST

The length of the soaking period appro-

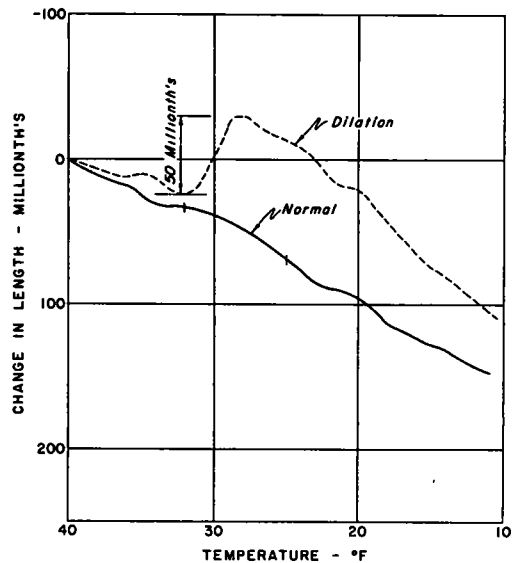


Figure 10. Powers cooling curves illustrating dilation as measured.

priate to the construction site was determined from temperature records of the experimental test slab at Donner Pass. The great majority of freezing-thawing cycles occurred during a 10-week period. For test purposes, therefore, it was concluded that a soaking period of 10 weeks would be appropriate.

NUMBER OF CYCLES

Data of the number of freezing and thawing cycles for the winter of 1956-7 are given in Table 2. The freezing point of water in concrete is somewhat less than 32 F. It was concluded that an effective freeze-thaw cycle occurred each time the temperature dropped below 23 F and then rose above 28 F. The data indicate that an estimate of 40 to 50 cycles per year would be amply severe as a criterion for establishing a test procedure. For test purposes, therefore, 40 or 50 cycles of freezing and thawing at the rate of 5 per week were introduced during the 10-week soaking period.

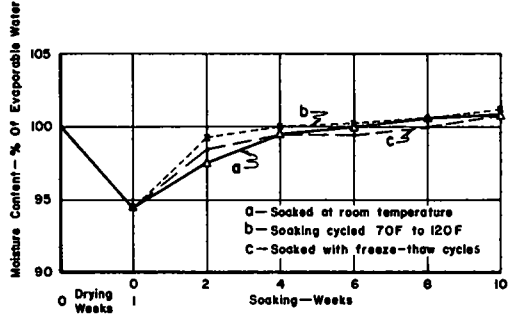


Figure 11. Effect of soaking treatment on absorption (aggregate No. 7).

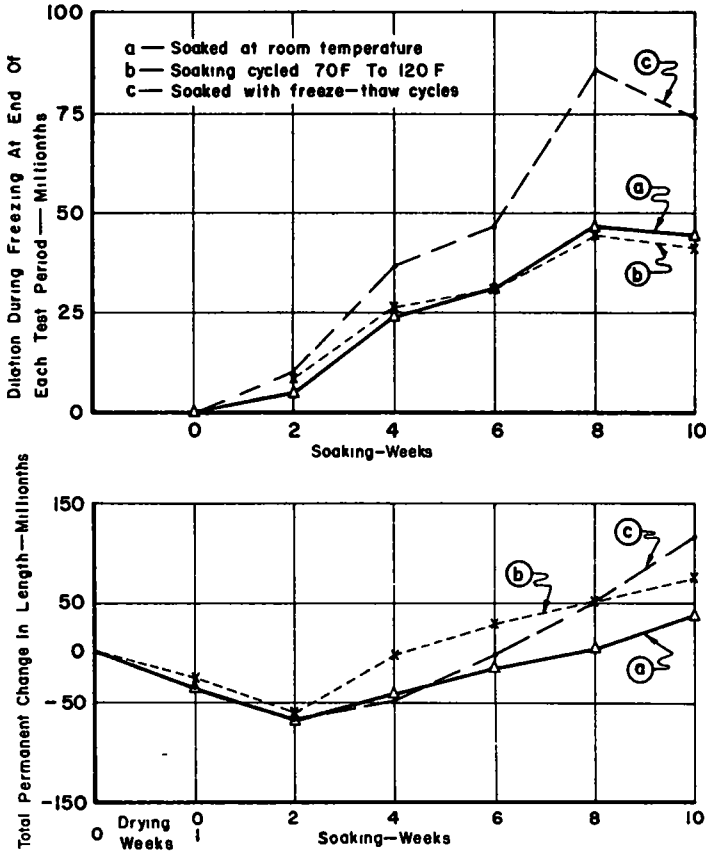


Figure 12. Effect of soaking treatment on dilation and permanent length change (aggregate No. 7).

TABLE 2
SUMMARY OF TEMPERATURE CHANGES WITHIN
TEST SLABS^a

Temperature Range	Depth of Thermocouple Below Surface		
	1/4 in	4 in	8 in
(a) Donner Summit, Winter of 1956-7			
Below 28 F	74	41	4
Above 33 F			
Below 23 F	55	12	5
Above 28 F			
Below 18 F	30	6	1
Above 23 F			
(b) Yuba Gap, Winter of 1957-8			
Below 28 F	49	17	7
Above 33 F			
Below 23 F	23	2	0
Above 28 F			
Below 18 F	13	0	0
Above 23 F			

^aValues shown are number of cycles completed during winter between temperature ranges shown

Except when length changes were being measured during cooling by the Powers procedure, freezing was performed in a larger bath containing water-saturated kerosene, the temperature of which was lowered from about 50 F to 0 F at the rate of 5 F per hour. After the specimens reached 0 F, they were transferred manually to a water bath where they remained at all times except while being frozen. The use of a separate freezing bath for intermittent freezing made it possible to utilize apparatus for the Powers procedure effectively. By properly staggering the program, it was possible to test about 80 specimens concurrently in addition to other specimens receiving simple soaking in water.

END POINT OF TEST

Powers (1) raised the following question which he felt required experimental evidence to answer: Should the end point be the first occurrence of permanent dilation after thawing or the occurrence of dilation (rather than shrinkage) during the freeze, even though the dilation may be small and transitory? The question has been studied by recording length changes during the warming period of the freeze-thaw cycles. Although evidence of hysteresis was noted, it was found that after returning to the starting temperature no permanent increase in length occurred when the measured dilation during freezing did not exceed 50 millionths. It thus appears that dilations of this magnitude or less were the results of stresses within the elastic range, that either no damage occurred to the concrete or that if the damage did occur, it was quickly repaired by autogenous healing.

Later in this report examples will be given of the relationship between dilations in excess of 50 millionths and permanent changes in length.

KEROSENE COOLING BATH

At the start, considerable concern was felt as to the effect of immersing partially dry test specimens in water-saturated kerosene during freezing. A few auxiliary tests indicated the probability that some kerosene was being absorbed. It may be argued that a minor amount of absorbed kerosene would not affect the performance of specimens during freezing since the liquid kerosene should develop hydraulic pressure as does the unfrozen portion of water. Data have been presented to show that regardless of the possible presence of absorbed kerosene, the over-all effect of freeze-thaw cycles during the water soaking period was somewhat more severe than was simple soaking in water.

It is considered to be essential that gain or loss of moisture be prevented during the freezing cycle. Since it is necessary to provide means of contact with the gage studs, the use of a non-aqueous bath is much more convenient than would be a watertight envelope.

In earlier work, specimens at the conclusion of the drying period were immersed at once in kerosene for the measurement of dilation by the Powers procedure. They were then immersed in water except while being frozen in kerosene at the rate of 5 times per week. In later work, the specimens at the conclusion of the drying period were immersed in water at room temperature where they remained for two weeks before they were subjected to freezing and thawing. The probability of absorption of kerosene was thus minimized. This procedure reduced the number of cycles during the 10-week soaking period from 50 to 40. It is not believed that the severity of the test was reduced appreciably by this change.

RAPID WATER TEST RESULTS

During the early stages of the investigation, aggregates from several prospective sources were tested both by the Powers procedure (1) and by rapid freezing and thawing in water, ASTM Designation: C 290-52 T. The aggregates were stream wet when incorporated in the concrete. Specimens were not allowed to dry before subjecting them to the test procedures (Table 3).

It will be noted that aggregates 1 and 2 gave good resistance and aggregates 3 and 7 poor resistance, as measured by both test procedures.

Although the results could be interpreted as indicating adequate resistance to freezing for aggregates 1 and 2, the haul distances to the site of the proposed work were so great as to make their use extremely costly.

In order to explore the effect of drying the specimens subsequent to curing and prior to commencing freezing, another series of rapid water tests was made in which ASTM Designation: C 290-52 T was followed except that test specimens were 4½- by 9-in. cylinders and deterioration was measured by expansion rather than drop in dynamic E. Also part of the specimens were tested without any drying and part were subjected to drying after curing according to schedule E which will be described later. The aggregates tested included numbers 1 and 3 of the first series and several others (Fig. 13).

Data derived from results reported by Kleiger (3) indicate that an expansion of 0.08 percent is approximately equivalent to a reduction of 40 percent in dynamic E. Aggregate 1 exhibited good resistance whether the concrete was given a preliminary drying or not. The remaining aggregates in concrete that were not dried, and therefore tested in this respect in accordance with ASTM Designation C 290-52 T, failed rapidly. Also, a moderate amount of drying produced a marked improvement in resistance.

Individual specimens subjected to the ASTM rapid water test were quite erratic in performance. The reliability of average results (Table 3 and Fig. 13) is therefore subject to question. Uncertainties in interpretation of the ASTM rapid water test led to its discontinuance. Subsequent studies were devoted to the development of Powers procedure (1).

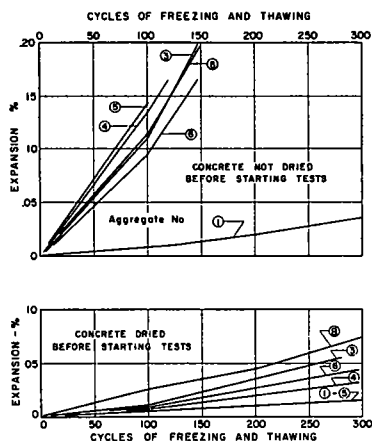


Figure 13. Rapid freezing and thawing in water. Similar to ASTM Designation: C 290-52T except using 4½- x 9-in. cylinders which were measured for change in length.

TABLE 3
TESTS OF AGGREGATES BY ASTM DESIGNATION
C 290-52 T AND POWERS PROCEDURE

Aggregate No	Average Durability Factor C 290-52 T, 4x5x18-In Prisms	Average Maximum Dilation During Powers Test (10 weeks of soaking) 4½- x 9-In Cylinders
1	90	0
2	77	7
3	21	280
7	43	68

Notes: Aggregates were stream wet when incorporated in concrete. Specimens were not allowed to dry subsequent to moist curing and prior to start of tests. Specimens for both types of test were molded from same batches. Five and one-half-sack concrete, air-entrained. Each value is average of 6 or more specimens for the rapid freeze-thaw test and an average of 3 or more specimens for the Powers test.

CORRELATION WITH FIELD PERFORMANCE

A measured dilation in excess of 50 millionths (in. per in.) above the length at the apparent freezing point of water in the specimen has been adopted as the criterion of unsatisfactory dilation. Concrete that produces less dilation during the selected time of the test is reported to be satisfactory. The validity of the selected criterion can be confirmed or rejected by the performance in service of concrete so tested.

To date, 1,242,000 sq yd or 180 lane-miles of pavement have been constructed under six contracts on the Donner Pass route between Colfax and the Nevada state

line. Of this amount, 173 lane-miles have been subjected to one or two winter's exposure. Salts have been applied during the winter to maintain the pavement in a substantially ice free condition. Aggregates used in this work were accepted on the basis of the Powers procedure as described earlier (details of the latest specification requirements are given in the appendix). The aggregates used in three of the projects, (aggregates 4, 7 and 8) were tested by rapid freezing and thawing in water, ASTM Designation: C 290-52 T and, (Table 3 aggregate 7 and Fig. 13 aggregates 4 and 8) would not be considered by this test to be resistant under any reasonable interpretation of the data.

The authors have examined all of these pavements in detail on several occasions. It is their conclusion that there is no significant evidence of distress attributable to the action of freezing and thawing. This statement warrants further amplification because of the possibility that others who might have occasion to inspect the work might consider that certain defects are indications of distress due to freezing and thawing. In a project between Boca and Floriston, (a few miles west of the Nevada state line) rather severe raveling occurred at several locations during the winter of 1959-1960; the first winter after construction. In other locations on this project, surface mortar has become detached to a depth of about $\frac{1}{16}$ in. Most of the distress is in the outer lanes. It has been observed that raveling starts abruptly at locations such as bridge approaches or at the start of a days work. The pattern is such as to suggest that local differences in performance are due to variation in construction methods, possibly erratic curing, rather than to the quality of the materials that were used. The authors have concluded that surface abrasion, where it has occurred, is the result of mechanical action of tire chains.

A section between Hampshire Rocks (elevation 5,800 ft) and Soda Springs (elevation 6,800 ft) west of Donner Pass constructed in 1959, is of particular interest. This pavement is believed to be in the most severe location of the route. It traverses a mountain meadow with heavy vegetation giving evidence of abundant precipitation. Freezing and thawing cycles are substantially the same in number as those at the summit. Aggregate No. 7 was used in the concrete. This aggregate, although meeting the selected criterion by the Powers test, performed poorly in the ASTM rapid water test. The pavement has been examined carefully at many closely spaced locations. Three types of defects have been noted. One is the presence of pieces of wood in the surface of the pavement. The second is a pit 1 in. or larger in diameter which was caused by a mechanically weak particle of rhyolite tuff. The pit edges are sharply defined by the surrounding concrete. These pits appear at the rate of 0 to 5 per 12- by 15-ft slab. The third defect is a typical popout produced by an unsound particle a short distance below the surface. Rupture of the overlying concrete has produced a crater-like depression. This type is absent in many areas and when present, has not appeared with a frequency greater than one per 12- by 15-ft slab.

Except as described above, the authors have observed nothing in any of the pavements that indicates freeze-thaw distress due to the materials used.

VARIATIONS IN DRYING

The concept of preliminary drying of cured test specimens before subjecting them to cycles of freezing and thawing is based on the expectation that pavements would be constructed during the summer or early fall and would have some opportunity to dry before the onset of severe weather. If construction were to be completed so late in the fall that a lesser degree of drying occurred before severe weather, the adopted schedule of drying the test specimens would produce unrealistic results.

The effect of varying degrees of drying of test specimens has been explored with aggregate No. 7. Five degrees of drying after standard curing were investigated.

After moist curing for 4 days, the specimens were conditioned by placing them in sealed containers over saturated solutions of salts which produced atmospheres of the relative humidities given in the following five schedules of drying:

- A. No drying (cured 3 weeks under standard fog conditions).

- B. Seven days over sodium sulfate, 97 percent relative humidity.
- C. Seven days over barium chloride, 87 percent relative humidity.
- D. Seven days over sodium acetate, 70-75 percent relative humidity.
- E. Two days in air at 50 percent relative humidity and 5 days over sodium acetate.

Schedule E was used in acceptance testing for the construction work completed to date. The different schedules resulted in varying losses in water during drying in grams per specimen as follows:

- A. + 3 grams (gain),
- B. -15 grams,
- C. -18 grams,
- D. -53 grams, and
- E. -87 grams.

As shown in Figure 14, schedules A, B and C resulted in dilation of 50 millionths or greater after $\frac{1}{2}$, $3\frac{1}{2}$ and 6 weeks of soaking, respectively. Schedules D and E did not result in dilation as great as 50 millionths during the 10-week soaking period.

Total permanent changes in length are shown in Figure 15. Increases in length above that at the conclusion of moist curing resulted from schedules A, B and C in decreasing order of magnitude at the end of 10 weeks of soaking. Schedule D resulted in a length equal to that at the conclusion of moist curing. The length produced by schedule E, while greater than at the conclusion of the drying period, was less than the "as-cured" length.

The results show that relatively small variations in drying procedure cause greatly different degrees of resistance to freezing and thawing. As a result of this investigation schedule D is now being used for acceptance purposes for new construction in the Donner Pass area in lieu of schedule E which has been used formerly.

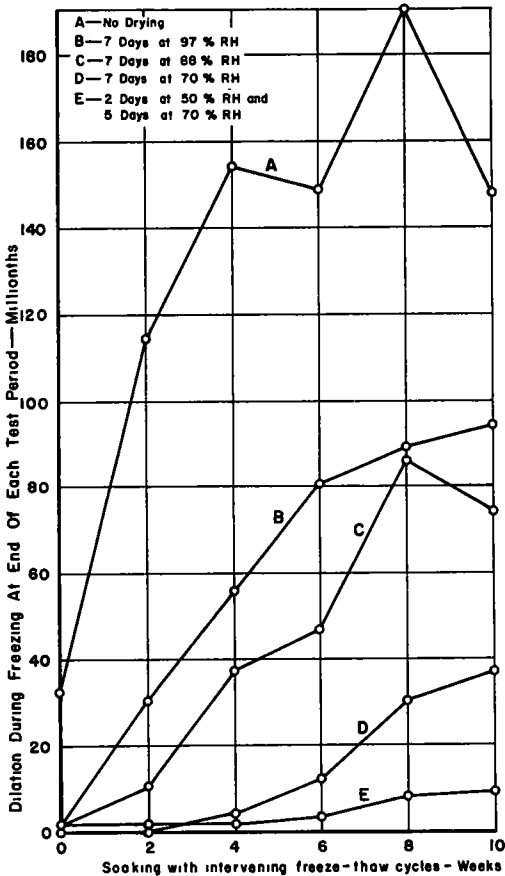


Figure 14. Effect of partial drying on dilation (aggregate No. 7).

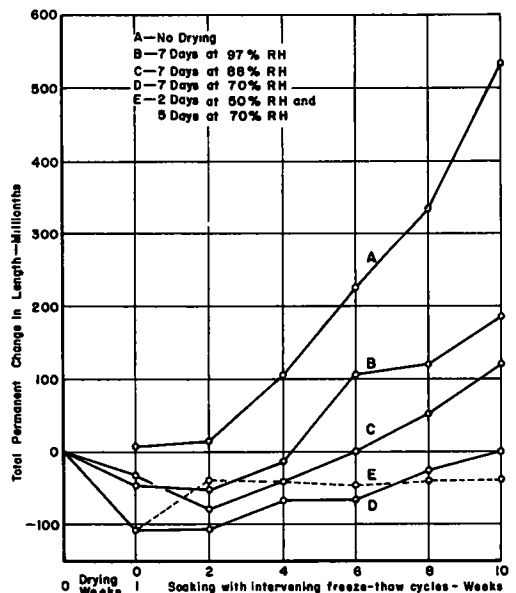


Figure 15. Effect of partial drying on permanent change in length (aggregate No. 7).

IOWA LIMESTONE

The test performance of limestone from the Rapid formation in Iowa was explored in concrete in which the fine aggregate consisted of sand from the American River (aggregate No. 1). The limestone as received was a crushed product in a dry condition. It was sieved and recombined to 1½ in. to No. 4 sieve. The limestone was prepared for incorporation into concrete by two methods: (1) soaking in water at room temperature for 24 hr, and (2) saturation under vacuum.

After the specimens were fog cured, they were subjected to conditioning schedules A (no drying) and E (drying).

Figure 16 shows the observed dilations during the Powers cooling cycle. Vacuum saturation produced more rapid distress than did simple soaking but either treatment resulted in definite indications of poor durability even when the concrete was subjected to preliminary drying.

Figure 17 shows permanent changes in length of the specimens. The curves again show a rapid loss in durability as the concrete was soaked.

With the limestone, when incorporated into concrete after 24 hr of soaking, specimens that were not dried performed nearly as well as those that had been partially dried after curing. This is in direct contrast with results obtained under similar treatments with aggregate No. 7 which was incorporated in the concrete in a stream-wet condition. It appears that the limestone was not completely saturated by 24 hr of soaking, and therefore, was in a condition approaching that resulting from drying the concrete after curing. It also appears that the limestone slowly absorbed water from the paste as evidenced by continued shrinkage for several days after the specimens were immersed in water, as shown in Figure 17 (concrete not dried).

LONG-TIME SOAKING

On the completion of tests by the Powers procedure (1) which were discontinued at the end of the 10-week soaking period, the specimens were placed in water storage at room temperature where they remained for periods up to three years. A cooling curve was again recorded for some of these specimens. (Tests results are shown in Figure 18.) Specimens that had received no preliminary drying were rendered more vulnerable. This can be explained by the

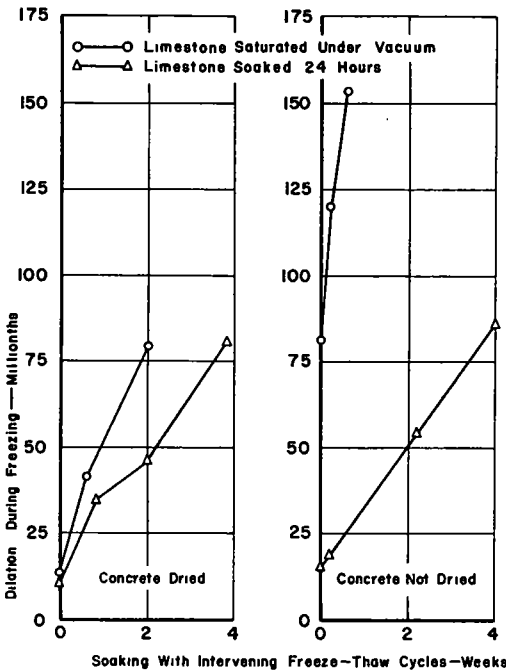


Figure 16. Effect of aggregate condition and drying of concrete on dilation—Iowa limestone coarse aggregate.

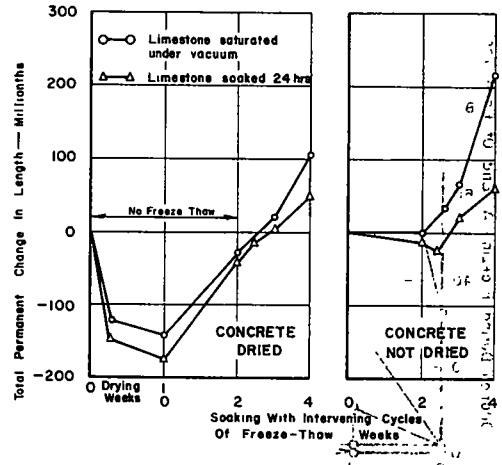


Figure 17. Effect of aggregate condition and drying of concrete on permanent changes in length—Iowa limestone coarse aggregate.

theory that the cement during hydration extracted some of the water from the presumably completely saturated aggregate. During prolonged soaking, part or all of this water was restored. Specimens that had been subjected to preliminary drying were also rendered more vulnerable but at a lower rate. None of the dried specimens reached a critical dilation of 50 millionths during the period of soaking, but the trend of results points to the conclusion that concrete is not likely to remain permanently resistant to freezing if it is exposed in such a way that it is continuously immersed in water.

LONG-TIME SOAKING FOLLOWED BY A SHORT DRYING PERIOD

A few specimens containing aggregate No. 7 were soaked in water for 9 mo following completion of tests by the Powers procedure. Cooling curves then showed dilations of 80 and 110 millionths for specimens dried originally in atmospheres of 70 and 87 percent relative humidity, respectively. The specimens were again dried for 8 days in an atmosphere of 70 to 75 percent relative humidity. Cooling curves then showed that dilations were reduced to 43 and 49 millionths. These results show that even short periods of exposure to mild drying conditions provide substantial relief to the build-up of vulnerability resulting from long-time soaking.

EVALUATION BY PERMANENT LENGTH CHANGE

Examples of permanent changes in length of specimens are shown in Figures 12, 15 and 17. The feasibility of using such length changes as criteria of the performance of aggregates in concrete has been studied. Concrete shrinks when drying and swells when soaking. During all cycles of simple soaking, swelling is less than the original shrinkage. When subjected to freezing and thawing cycles in conjunction with soaking at room temperature, the amount of swelling may be greater than under simple soaking. An increase above normal swelling may indicate damage as a result of freezing.

In many cases, those specimens that developed a dilation in excess of 50 millionths during cooling have attained a permanent length approximately equal to the as-cured length. If this were a universal rule, it would be possible to eliminate the test for dilation during cooling and to use only the measurement of permanent length change as a criterion of performance. To do so, would permit the elimination of expensive and complicated apparatus for recording the cooling curve. However, some specimens have not swelled to the as-cured length at the time a critical dilation has been found in the cooling curve. In other cases, the length of the specimen has exceeded the as-cured length before a dilation of 50 millionths has been indicated by the cooling curve. Attempts have been made to relate permanent changes in length as referred to the as-dried length and dilations as indicated by the cooling curve. The results were negative.

It appears therefore, that evaluation of performance in accordance with the Powers concept must include the measurement of dilation during cooling. Manual, in place of automatic measurement, could be used with considerable saving in the cost of apparatus.

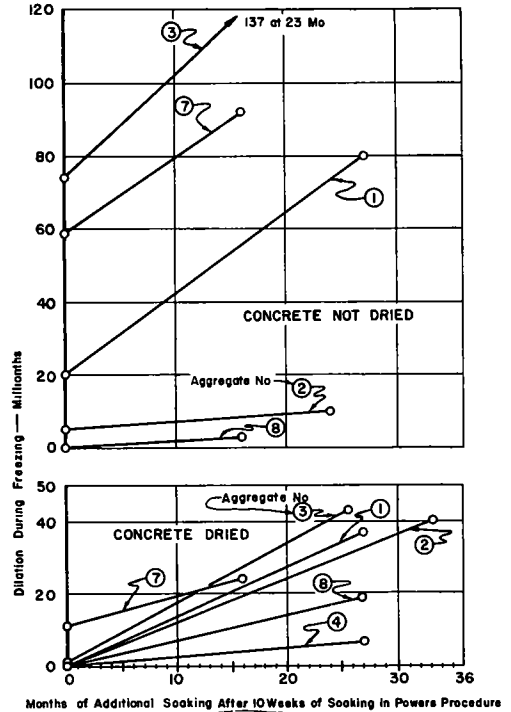


Figure 18. Effect of long soaking on dilation.

These results show that even short periods of exposure to mild drying conditions provide substantial relief to the build-up of vulnerability resulting from long-time soaking.

EFFECT OF CEMENT FACTOR

The movement of water from and into aggregate particles is restricted by the surrounding cement paste. The more impervious the paste, the greater should be the restriction on movement of water into and out of the aggregate. Richer mixtures (of lower water-cement ratios) therefore, should extend the period during which partially dried concrete can be soaked before it becomes vulnerable to the effects of freezing and thawing.

If the above assumptions are true, higher cement factors should produce concrete that is more resistant to freezing and thawing under natural exposure. Reported examples of field performance as affected by cement factor are few in number; however, certain projects of the "Long Time Study of Cement Performance of Concrete" (4) have yielded such comparisons. In the "Ten Year Report" (4), it is stated that one row of boxes at the Illinois test plot containing 27 test cements and an aggregate of good service record, had developed considerable distress. These boxes were constructed with non-air-entrained concrete containing $4\frac{1}{2}$ sacks of cement per cubic yard and having a slump of 8 in. Boxes constructed of similar concrete except with a cement factor of 6 sacks per cubic yard, were in excellent condition. At the Saugerties, New York test site (5) concrete piles of 7-sack concrete have been more resistant to freezing and thawing than comparable piles containing 5-sack concrete.

A good laboratory test procedure should be capable of exhibiting improved performance of richer (lower water-cement ratio) concrete. The performance of the Powers procedure in this respect has been investigated. Specimens were made of concrete containing 4, $5\frac{1}{2}$ and 7 sacks of cement per cubic yard using aggregate No. 7. The corresponding water-cement ratios were 7.5, 5.1 and 4.3 gal per sack, respectively.

After moist curing for 14 days, groups of 6 specimens were subjected to four schedules of drying; namely, schedules A and E as previously described and two intermediate conditions which were similar to, but not exactly the same as, schedules B and C. The intermediate drying conditions are designated as schedules B' and C'. After curing and drying according to these schedules, the specimens were subjected to 14 weeks of soaking, of which the first two weeks were in water at room temperature and the remainder were with intervening cycles of freezing and thawing at the rate of 5 per week.

The results of the tests are summarized in Tables 4 and 5.

Referring first to the data on change in weight (Table 4) which is used as a measure of moisture movement, the results are reported to the end of 8 weeks of soaking only, because at later periods, the mechanical loss of solid material became great enough to obscure the moisture change relationship. It will be noted that the loss of moisture during drying increased with decreasing

TABLE 4
EFFECT OF CEMENT FACTOR ON MOISTURE MOVEMENT
(Aggregate No. 7)

Cement Factor (sk/cu yd)	Drying Schedule			
	A'	B'	C'	E
(a) Change in Weight (grams per specimen) Due to Drying				
4	4	-38	-66	-88
5.5	3	-29	-49	-67
7	3	-24	-37	-52
(b) Change in Weight (grams per specimen) Relative to the As-Dried Weight After 8 Weeks of Soaking				
4	-	40	58	77
5.5	-	43	52	67
7	-	37	41	54
(c) Change in Weight (grams per specimen) Relative to the As-Cured Weight After 8 Weeks of Soaking				
4	20	2	- 8	-11
5.5	18	14	3	0
7	15	13	4	2

¹Schedule A specimens were soaked in water for one week while remaining specimens were subjected to drying.

TABLE 5
EFFECT OF CEMENT FACTOR ON DILATION AND
PERMANENT CHANGE IN LENGTH (Aggregate No. 7)

Cement Factor (sk/cu yd)	Drying Schedule			
	A	B'	C'	E
(a) Time of Soaking (weeks) to Produce 50 Millionths Dilation				
4	5	5	8	7
5.5	3	4	11	12
7	2	9	12	14+
(b) Total Permanent Change in Length (millionths) Relative to As-Cured Length After 14 Weeks of Soaking				
4	160	105	40	15
5.5	240	65	-45	10
7	230	-40	-25	-55

cement factor, a result that is in accordance with theory. Upon soaking, the leaner concrete absorbed more water. At the end of 8 weeks however, the leaner concrete that had been subjected to drying schedules B', C' and E contained less water than the richer concrete.

Judged on the basis of final moisture content, it might be concluded that the leaner concrete, after drying and then soaking, should be more resistant to the effects of freezing and thawing. That this is not true, however, is shown by the data of cooling curves (Table 5). These data show that with increasing cement factor, the concrete withstood increasingly longer periods of soaking before the specimens became sufficiently vulnerable to produce a dilation of 50 millionths. A reversal of this trend is shown for specimens that were not dried (schedule A). The reversal in trend may be explained by the fact that since the aggregate as incorporated in the concrete was thoroughly saturated, further soaking did not affect its vulnerability greatly. All cement factors under this condition produced highly vulnerable concrete and the slight differences in safe periods of soaking are of little practical consequence.

The data of permanent change in length (Table 5) show that after drying, the richer concrete was more resistant. For specimens that were not dried (schedule A), the data indicate somewhat better performance for the 4-sack concrete; however, its resistance was of low order and the differences between cement factors are of little practical consequence.

On the whole, the data give convincing evidence that the test procedure exhibited improved performance with increasing cement factor, and decreasing water-cement ratio. The fact that this behavior is in accordance with observed performance under natural exposure lends added evidence of the validity of the basic concepts of the Powers method.

REPEATABILITY

The degree to which test results can be duplicated in the same laboratory is a matter of considerable importance in assessing the value of any test method.

The aggregates that have been tested, with one exception, are sands and gravels of igneous origin and are composed of intrusive and extrusive acidic, intermediate and basic rocks. Some of the particles have absorptions as high as 5 percent. The problem of making like specimens is therefore, connected intimately with that of getting representative amounts of various rock types into them. When large dilations have occurred they have been accompanied frequently by rupture of the specimen apparently caused by abrupt expansion of one or more large particles (Fig. 19). The fact that the larger particles are the more vulnerable further complicates the fabrication of uniform test specimens.

For this reason, specifications have been written on a go-no-go basis. That is, acceptance has been based on the condition that the majority of the individual specimens pass the test requirement. A number of aggregates have been accepted as a result of tests of six specimens, two each from three batches mixed on different days. Usually more than one sample from the same deposit has been tested.

Six is certainly the minimum number of specimens that should comprise one test. Twelve or more is desirable. The reason for using a number as small as six in past work has been to provide extra

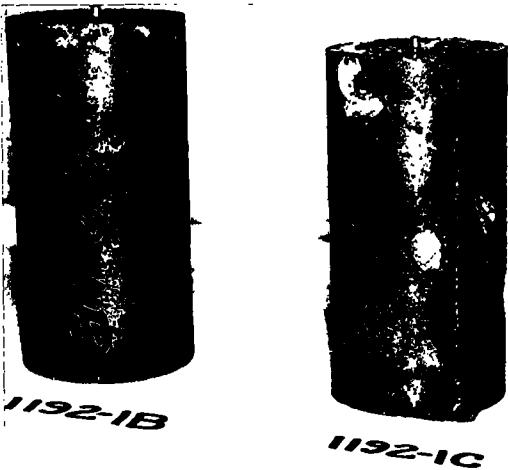


Figure 19. Effect of drying concrete on condition of specimens subjected to freeze-thaw (1192-1B dried after moist curing, and 1192-1C not dried).

specimens for study purposes on the effect of variations in drying or soaking procedures. The volume of the $4\frac{1}{2}$ - by 9-in. cylinder that has been used is about 0.083 cu ft. A test batch of 0.6 cu ft is ample to determine slump, air content and yield, preparatory to making four tests specimens. Twelve specimens can, therefore, be obtained from three batches of this size.

The measure of repeatability should be based on (1) the variation in time of soaking required to exceed a selected degree of dilation, or (2) the percentage of specimens that reach or do not reach the critical dilation in the specified period of soaking.

A measure of the degree of variation between individual specimens under category (1) is obtained by comparing the soaking time of individual cylinders to reach specified amounts of dilation measured during the Powers procedure. Since the cooling curve has been recorded at two-week intervals, it is possible that dilations during intervening freezings may exceed those at the time of measurement. Tests of individual specimens have shown instances of large dilations being followed by smaller ones. Nevertheless, by plotting the data obtained, the time of soaking can be fairly well estimated to about $\frac{1}{4}$ week for each increment of dilation considered. A few sets of test data were selected from those groups in which all of the test cylinders dilated more than 50 millionths during the 10-week soaking period, and the weeks of soaking to reach dilations of 30, 40 and 50 millionths were determined. The standard deviation for each group was calculated also, and the results are given in Table 6.

The above method of evaluating precision is severe because the aggregates tested were composed of a variety of rock types, some of which could produce a small and transitory dilation at the time of measurement largely as a matter of chance inclusion in different specimens. As the dilation selected for consideration becomes larger, and more significant, the variation in time between specimens becomes smaller percentage-wise.

A more useful measure of repeatability is obtained by considering the results on a go-no-go basis as suggested under category (2).

As mentioned earlier, some sources were tested more than once at different times. Aggregate No. 7 was tested early in 1958 and again late in 1958. Two test pits were sampled for the early series, and three for the latter, bringing the total number of samples tested to five. The tests for development of dilation were made under the same conditions for each sample. The results of the test are given in Table 7 which shows the number of samples from each group that pass the test. Six specimens were used in each test in which the concrete was dried. With the exception of the second sample of the not-dried groups, five specimens were used. Only four specimens were used in the second sample. The results are amply discriminatory on the basis of evaluation on the performance of at least two-thirds of the specimens.

That the test method, even with the relatively small number of test specimens, effectively distinguishes between vulnerable aggregate and nonvulnerable aggregate for the test conditions used, is given in Table 8. The sources listed are those tested recently under the current specification requirements, and also without drying. In all cases involving drying of the concrete, at least 5 of the 6 specimens definitely passed the test. Of the not-dried group, the separation was positive except for source 11 in which 1 of 5 passed and sources 14 and 15

TABLE 6
VARIANCE IN RATE OF DILATION OF SELECTED
TEST GROUPS

Test Group	Time to Reach Specified Dilation (weeks) and Standard Deviation for Each Group					
	30 Millionths		40 Millionths		50 Millionths	
	Wks	Std Dev	Wks	Std Dev	Wks	Std Dev
A	1 6	1 6	2 6	1 5	3 6	1 5
B	0 6	0 6	1 3	0 6	1 6	0 5
C	0 6	0 2	1 0	0 3	1 4	0 4
D	3 6	2 3	6 5	1 6	7 2	1 8

TABLE 7
GO-NO-GO EVALUATION OF FIVE AGGREGATE
SAMPLES FROM SAME SOURCE (Aggregate
No 7)

Sample No	Condition of Concrete	
	Dried, Then 10 Wks of Soaking	Not Dried, 11 Wks of Soaking
1	6 of 6 pass	0 of 5 pass
2	5 of 6 pass	1 of 4 pass
3	6 of 6 pass	0 of 5 pass
4	6 of 6 pass	0 of 5 pass
5	6 of 6 pass	0 of 5 pass

in which 3 of 5 passed in each case. It is evident that better repeatability was obtained with dried specimens than with undried ones.

The foregoing discussion indicates that the test procedure, based on the Powers concept, is sufficiently precise to render it usable as a basis for purchase specifications for concrete aggregates.

CONCLUSIONS

The method of performing freezing and thawing tests to determine performance of aggregates in air-entrained concrete as suggested by Powers (1), is adaptable to different methods of producing and handling aggregates and to varying exposure conditions. Results in service show that when the test conditions were adjusted to the aggregates as they were furnished and to the exposure conditions to which pavements were subjected, aggregates that did not produce a dilation in excess of 50 millionths have performed satisfactorily through one and two winters of severe weather. There is no present evidence to warrant an expectation that the concrete will not continue to be durable.

On the other hand, had acceptance of the aggregates been based on tests by ASTM Designation: C 390-57 T, or Corps of Engineers Methods CRD-C 20-55, Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water, and had the concrete been mixed with aggregates containing the full amount of original moisture, several of the aggregates that were used would have been rejected. Rejection of such aggregates would have increased the cost of the work substantially.

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Appendix

METHOD OF TEST FOR FREEZE-THAW RESISTANCE OF AGGREGATES IN AIR-ENTRAINED CONCRETE (Powers Procedure)

Values shown for maximum size of aggregate, size of test specimen, conditioning by drying and length of soaking period are those used by California Division of Highways for construction work in a particular locality. Other values should be substituted when construction or exposure conditions so warrant.

Samples

Samples of aggregates shall be secured under the direct supervision of the engineer

TABLE 8

GO-NO-GO EVALUATION OF SEVERAL AGGREGATES

Aggregate No	Condition of Concrete	
	Dried, Then 10 Wks of Soaking	Not Dried, 11 Wks of Soaking
9	6 of 6 pass	5 of 5 pass
10	6 of 6 pass	5 of 5 pass
11	5 of 6 pass	1 of 5 pass
12	5 of 6 pass	0 of 5 pass
13	6 of 6 pass	0 of 4 pass
13A ¹	5 of 6 pass	0 of 5 pass
14	6 of 6 pass	3 of 5 pass
15	6 of 6 pass	3 of 5 pass

¹Aggregate No 13A contains coarse aggregate No 13 and fine aggregate No 15

in charge of the work. Samples from existing stockpiles of processed aggregate shall be taken from washed materials and shall be visibly damp. Samples from materials in place in a proposed source shall be taken at depths from the surface that will insure the presence of the full quantity of ground water. Excavations for the purpose of securing samples shall be made to the full depth of intended operations. Samples shall be protected against loss of contained water until they are delivered to the engineer.

Samples shall be shipped to the laboratory in metal containers with tight fitting covers. Water shall be added to each container before it is sealed.

In the laboratory, samples shall be so handled to prevent loss of absorbed water. They shall be processed by washing if required, separated into sieve sizes and recombined to the required grading in the presence of excess water. If particles larger than $1\frac{1}{2}$ in. are present in the sample, they shall be crushed and added to the finer material unless it is proposed to waste the oversize particles during manufacture.

Apparatus

The following apparatus is required in addition to that needed for making and curing concrete specimens in the laboratory, ASTM Designation: C192.

One or more refrigerated baths of a size and depth required to contain the test specimens immersed in kerosene and with suitable controls to provide a lowering of the temperature at a rate of 5 ± 1 F per hour from room temperature to 0 F. Household type deep freezing chests with a copper liner have been found to be satisfactory.

One or more water baths of a size and depth to contain the specimens immersed in water, equipped with water supply and overflow outlet.

A supply of frames with linear variable differential transformers attached, for supporting test specimens for automatic measurement of length changes during cooling. A satisfactory design is shown in Figures 5 and 6.

A supply of $4\frac{1}{2}$ - by 9-in. cylinder molds, equipped with top and bottom detachable plates for holding gage studs.

A comparator for measuring permanent length changes of test specimens, meeting the requirements of Section 2(b) of ASTM Designation: C 157-54 T and with a standard reference bar.

A balance sensitive to 1 g and having a capacity of about 6,000 g.

Closed corrosion resistant containers large enough to contain one or more $4\frac{1}{2}$ - by 9-in. test specimens with free space of at least $\frac{3}{4}$ in. from all faces, equipped with means of supporting the specimens at least $2\frac{1}{4}$ in. above the bottom.

A strain recorder with suitably ruled paper. It shall consist of a multipoint displacement recorder having one channel for each specimen being tested for dilation during cooling. The switching sequence from one channel to the next shall be accomplished automatically at a rate such that the time interval between prints for a particular channel does not exceed 5 min. The system shall have a sensitivity sufficient to indicate displacements of 25 millionths inch equal to one chart division through a range of 0.004 in. Displacements indicated by the linear variable differential transformers in contact with the studs of test specimens shall be recorded. A calibration bar of accurately known thermal coefficient of expansion shall be furnished. (Manual means of measuring length changes and temperature may be substituted for automatic recorders provided the apparatus has a sensitivity equal to that of the automatic apparatus.)

An automatic temperature recorder connected to thermocouples imbedded in companion concrete cylinders of the same size as the test specimens, one in each bath in which dilations are measured. The temperature of each thermocouple shall be printed on a chart within an accuracy of 1 F through a range from room temperature to -10 F. Prints for each channel shall be recorded at a frequency of not more than 5 min.

Procedure

Concrete mixtures shall be proportioned with $1\frac{1}{2}$ -in. maximum size aggregate graded to conform to the specifications for the work. The cement factor and slump shall be within the limits specified for the work. An air-entraining agent consisting of neutralized

Vinsol resin solution shall be incorporated in the quantity required to result in an air content of 4.5 ± 0.5 percent air in the fresh concrete. Slump, air content and cement factor shall be determined for each batch. The size of batch shall be sufficient to provide at least four $4\frac{1}{2}$ - by 9-in. test specimens.

Four or more $4\frac{1}{2}$ - by 9-in. cylinders shall be molded from each batch. Stainless steel gage studs 1 in. long shall be embedded in the fresh concrete so as to project $\frac{1}{8}$ in. at each end of the longitudinal axis of the specimen.

For each aggregate or combination of aggregates to be tested, at least three batches of concrete shall be mixed, each on a different day, providing a minimum of 12 test specimens. The specimens in the molds shall be cured under standard moist conditions for 24 ± 4 hr and then be removed from the molds. Standard moist curing shall be continued to the age of 14 days.

At the end of the moist curing period, the specimens shall be weighed to the nearest 1 g in a surface-dry condition and shall be measured for length to the nearest 0.0001 in. (The method of inserting gage studs does not insure that they coincide absolutely with the vertical axis of the specimen. For this reason the needle of the dial gage may not remain stationary as the specimen is rotated. Reproducible results are obtained by rotating the specimen until the minimum reading is found.) They shall then be placed in closed containers over a saturated solution of sodium acetate with an excess of salt for 7 days at a temperature of 73.4 ± 3 F. The solution shall be at least $1\frac{1}{2}$ in. in depth and shall not be closer than $\frac{3}{4}$ in. to the bottom of the test specimens. The specimens shall be removed, weighed and measured for length. They shall then be immersed in water at room temperature for 14 days.

The specimens shall then be removed from the water bath (total age at this point is 5 weeks). They shall be wiped free of surface water and be weighed and measured for length.

The specimens shall then be placed in the frames used to measure and record strains during cooling. The specimens in the frames shall be immersed in water-saturated kerosene in the refrigerated baths and connected to the displacement recording instrument which shall be placed in operation. A companion specimen containing a thermocouple shall also be placed in the bath and be connected to the temperature recording instrument which shall be placed in operation. The kerosene shall be agitated mechanically until the temperatures of the specimen and the bath are equal. The temperature at this point shall be above 45 F. The time shall be recorded on the charts. The bath shall be cooled at a rate of 5 ± 1 F per hour and cooling shall be continued until the temperature reaches 0 ± 5 F. The specimens shall then be removed and the time recorded on the charts.

The specimens shall be immersed in water and allowed to warm to room temperature, then weighed and measured for length. The specimens shall remain in the water bath continuously except that they shall be cooled in kerosene as described above, at the rate of five times per week until a total soaking period of 10 weeks has elapsed. (The total age of the specimens at this point is 13 weeks.) Cooling to 0 F except during the first cycle and each succeeding 10th cycle may be performed without connecting the specimens to the strain-recording device. At the end of each two-week soaking period, the specimens shall be weighed and measured for length and then be connected to the strain-recording device during one cycle of cooling.

At the conclusion of each recorded cooling cycle, the charts shall be marked to show equal time periods of 15 min. The recorded strain, estimated to the nearest one-half chart division, divided by the gage length, $7\frac{1}{2}$ in., shall be plotted against the temperature recorded at the same time. The curve shall be examined for evidence of dilation at the approximate freezing point. If dilation is evident, it shall be measured as the distance between the start of dilation at the apparent freezing point and the greatest succeeding length. The result shall be recorded as the dilation in millionths.

Report

Specimens shall be reported to have "passed" the test if:

1. The dilation at any measured period did not exceed 50 millionths.

2. If the permanent length at the conclusion of the soaking period does not exceed the length at the conclusion of the 14-day moist curing period by more than 0.006 percent of the gage length (measured between inner ends of the gage studs).

Specimens shall be reported to have failed the test if they fail to meet any of the requirements set forth above to qualify as "passing".

The aggregate or combination of aggregates under test shall be reported to have "passed" the test if 65 percent or more of the individual specimens of the group passed the test. If less than 65 percent of the individual specimens of the group passed the test, the aggregate or combination of aggregates shall be reported to have "failed" in the test.