CORPS OF ENGINEERS, U. S. ARMY

COOPERATIVE FREEZING-AND-THAWING TESTS OF CONCRETE SPECIMENS

TECHNICAL MEMORANDUM NO. 6-395

CONDUCTED FOR

OFFICE OF THE CHIEF OF ENGINEERS

BY

WATERWAYS EXPERIMENT STATION

VICKSBURG, MISSISSIPPI

ARMY-MRC VICKSBURG, MISS.

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PREFACE

The investigations reported herein were conducted in accordance with authority provided by the Office, Chief of Engineers, and form a part of Civil Works Investigations Item CW 602 "Research on the Properties of Aggregates for Concrete." The basic authority for initiating the investigations was provided in January 1949.

The work has been conducted as five separate investigations (Phases I through V). Six of the Corps of Engineers Division Laboratories cooperated with the Waterways Experiment Station in one or more of these phases. This report includes details of the results of Phases III and V which have not previously been reported, and summarizes the entire study including the results of Phases I, II, and IV reported previously.

Phase III was authorized in June 1950, and was participated in by all the laboratories. Detailed instructions therefor were given in memoranda dated 28 June, 28 July, and 2 November 1950, distributed by the Waterways Experiment Station. Representatives of the Waterways Experiment Station visited the cooperating laboratories as shown below while the Phase III specimens were being made:

South Atlantic Division: November 1950, January 1951

Southwestern Division: December 1950, May 1951

Missouri River Division, South Pacific Division,
North Pacific Division: February 1951

Ohio River Division: June 1951

Phase V was authorized in September 1953 and was conducted by the Waterways Experiment Station.

This report was prepared by Mr. E. E. McCoy of the Waterways Experiment Station.

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COOPERATIVE FREEZING-AND-THAWING TESTS OF CONCRETE SPECIMENS

PART I: INTRODUCTION

Need for and Purpose of Investigations

1. The usefulness of a laboratory freezing-and-thawing test in providing data applicable to the evaluation of aggregates proposed for use in construction projects is generally acknowledged. However, the comparability of the results of such tests made in different laboratories at different times has been the subject of controversy and investigation for many years. Seven Corps of Engineers Division Laboratories have one or more freezing-and-thawing test installations. The apparatus at each laboratory necessarily differs somewhat from that at the others. However, the principal object of the test is the same at each laboratory -- to subject the specimens to a standard cycle of freezing-and-thawing, as measured by a thermocouple at the center of a representative specimen. The investigations described herein were conducted to develop data that would:

- a. Indicate the magnitude and causes of variation in results of tests of aggregates by freezing-and-thawing using a standard test method*.
- b. Permit suitable revisions of the test method that would reduce variations, both within any one laboratory and between laboratories, to acceptable limits.

Scope

2. The studies conducted to date have comprised five phases, each supplementing the previous ones to some extent. The principal elements of each phase are summarized in the following tabulation.

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* Item CRD-C 114, "Method of Test for Soundness of Aggregates by Freezing and Thawing of Standard Concrete Specimens," Handbook for Concrete and Cement, Waterways Experiment Station, 1949.

Aggregates Tested	Participating Labs (a)	Lab Making Test Specimens	No. Specimens Tested Per Laboratory
		Phase I	
Coarse: crushed trap rock Fine: natural siliceous sand	WES, SPD SWD, SAD	WES	9 or more
		Phase II	
Same as Phase I, shipped from WES	WES, SPD, SWD, SAD	Laboratory performing tests thereon	9 or more
		Phase III	
Coarse: crushed trap rock Fine: rod-milled trap rock	WES, SPD, SWD, SAD, MRD, ORD, NPD	Laboratory performing tests thereon	18 or more
		Phase IV	
Same as Phase III	SAD, MRD	Laboratory performing tests thereon	27, 9 for each of 3 air content ranges
		Phase V	
Coarse: crushed limestone Fine: manufactured limestone	WES	WES	90, 30 for each of 3 coarse aggregate gradings

(a) SAD, South Atlantic Div. Lab; SPD, South Pacific Div. Lab; SWD, Southwestern Div. Lab; NPD, North Pacific Div. Lab; MRD, Missouri River Div. Lab; ORD, Ohio River Div. Lab; WES, Waterways Experiment Station

3. The tests and results of Phases III and V are described in detail in parts II and III of this report. The results of Phases I, II, and IV,

which have been included in earlier reports*, are summarized together with the results of Phases III and V in part IV.

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* The results of Phases I and II were discussed in "Progress Report on Cooperative Freezing-and-thawing Tests" issued by the Waterways Experiment Station on 20 April 1950. Copies of this report were submitted to the Office, Chief of Engineers, and were distributed to all Division Laboratories on 27 June 1950. The results of Phase IV were included in Laboratory Technical Memorandum, "Effect of Air Content upon Reproducibility of Test Results and Durability of Standard Concrete Beam Specimens When Subjected to Accelerated Freezing-and-thawing," dated November 1952, prepared and distributed by the Missouri River Division Laboratory to all Division Laboratories.

PART II: PHASE III -- VARIABILITY OF RESULTS OF SPECIMENS SUBJECTED TO FREEZING-AND-THAWING

4. As in the previous programs, the purpose of this investigation was to determine the variability of the test results when concrete specimens were tested for resistance to freezing-and-thawing by the various Corps of Engineers Division Laboratories. The test method (CRD-C 114-48) has been revised from time to time. In Phase III, particular emphasis was placed on the use of "tempering" tanks in compliance with the details covered in the first revision of the test method.* The tempering tank provides more equable temperature conditions for the test beams when they are out of the freezer for sonic testing. Other modifications and recent developments in the method were also considered in Phase III as described below.

Controls

5. Every effort was made to insure that the instructions to the cooperating laboratories would be clear and concise so that all items of the testing procedure for Phase III would be clearly understood by all. A representative of the Waterways Experiment Station observed and carefully checked mixture design, mixing, and specimen molding in each laboratory. Other operations in connection with the program were closely inspected at the same time. Divergences from the normal or desired procedure were either corrected at each laboratory or reported for consideration in the final analysis.

Description of Investigation

Materials

6. Trap rock from the supply in stock at each laboratory was used for both fine and coarse aggregate. The coarse aggregate was graded in

* Item 6 of Distribution No. 1 (December 1949) of "Supplements, Corrections, and Revisions" to the Handbook for Concrete and Cement.

accordance with requirements of Method CRD-C 114, as revised, to a maximum size of 1 inch. The fine aggregate was produced by wet-process rod-milling, graded to conform to requirements of the test method. Two laboratories, SWD and ORD, which did not possess facilities for production of wet-process rod-milled fine aggregates, conducted the tests in duplicate. One set of tests was made using fine aggregate produced by locally available processing equipment, the other using wet-process rod-milled fine aggregate supplied by the WES. Type II cement, Serial No. RC 177, was distributed by the WES for use in the tests. The air-entraining admixture was commercially produced, neutralized Vinsol resin solution, and was also distributed by the WES.

Procedures

7. Test methods contained in the "Handbook for Concrete and Cement," including the most recent revisions at the time of tests, were followed exactly. Particular emphasis was placed on several items pertaining to the tests, and several new features were incorporated in the test method. Some of these items were already in the Handbook, some were under consideration as revisions, while others were refinements proposed for this program only. The items of particular emphasis in the test requirements were as follows:

a. That the curve for the time-temperature history of the thermocouple specimen agree closely with the curve in fig. 1*, and that the temperature cycle be maintained as close to 0-42 F as possible.

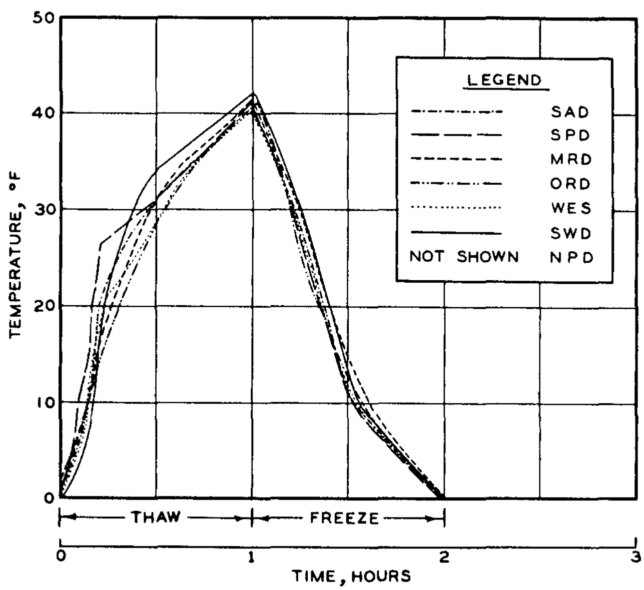


Fig. 1 Time-temperature curves

* Fig. 1 is a reproduction of fig. 1 of progress report on Phases I and II issued by WES 20 April 1950.

b. That a special thermocouple specimen be fabricated from a mixture similar to that of the test specimens.

c. That the cycling of the freezing-and-thawing apparatus be continuous, and that all interruptions be noted and explained in the report.

d. That the dimensions of the specimens be within ± 1/16 in. of those specified in the test method, and that sixteen linear measurements be made to determine the actual size of each specimen. (An illustration showing desired linear measurements was distributed to the participating laboratories.)

e. That the inside dimensions of each container and its capacity be measured before and after its use in the testing of a specimen, and that containers be provided with tight-fitting covers.

f. That a record be kept of the amount of water required to immerse each specimen, so that its top would be covered to a depth of 1/2 in., each time the water was changed.

g. That the mixture design for all laboratories be provided by the Waterways Experiment Station.

h. That the batch size be 0.9 cu ft instead of 0.75 cu ft.

i. That mineral oil be used as form oil.

j. That specimens be removed from forms at 48 ± 4-hr age.

k. That all specimens be cured after stripping by immersion in a saturated lime solution.

l. That on the ninth day, specimens be removed from the curing tanks, kept moist at all times with minimum exposure at ambient temperatures above 73.4 F, measured in all dimensions, weighed in air and in water to ± .02 lb, placed in the 46 ± 4 F tempering tank for not less than 1 hour, then placed in specimen containers, and after water is added, placed in the freezing-and-thawing apparatus.

m. That air in the hardened concrete be determined by weight measurements on the ninth day.

n. That specimens be oriented from left to right in the three-gang specimen molds when labeled, and that specimens be turned end for end after each testing.

o. That the position of specimen-rack spacer bars be noted, and that notes be taken of cracking, pits, and cavities on the specimens at locations near specimen-rack spacer bars.

p. That an examination be made of specimens giving anomalous results, or preferably a petrographic examination be made of all specimens after testing, and,

q. That sonic readings be taken after one cycle in freezing-and-thawing, and that relative E (dynamic modulus of elasticity) and DFE (durability factor) be determined based on the fundamental frequency at one cycle, in addition to the usual calculations provided for in CRD-C 114.

Equipment

8. The equipment used by the various laboratories for both preparation and testing of the specimens differed in some, mostly minor, respects. There were also minor differences in the types of concrete mixers. During the inspection tours, representatives of the WES made detailed notes of differences in equipment and technique which, though apparently trivial, might in some obscure way influence the testing of the specimens. In one instance personnel were advised to place ingredients in the mixer by the scoopful instead of by total quantity lot. Considerable information pertaining to the various items of test equipment and to specimen preparation and testing was received at the WES for analysis to isolate possible causes of variation of test results. No clearly defined influence was found. All laboratories reported instances of deviation beyond the specified limits for specimen size; however, any deleterious effect due to this source was overshadowed by the effect of other variations.

9. The principal requirement of a freezing-and-thawing test apparatus is that it produce the specified intensity of freezing or thawing in one hour. Test Method CRD-C 114 specifies that the specimens be frozen to 0 ± 2 F in one hour and thawed to 42 ± 2 F in one hour. The apparatus at each laboratory fulfilled this requirement. Temperatures during cycle change agreed fairly well and those at the termination of the cycle showed excellent agreement. Temperature curves for six of the participating laboratories are shown in fig. 1, page 5. The thaw curves for the SAD, SPD, and SWD apparatus are the steepest; and the freeze curves for these laboratories are also as steep as any others.

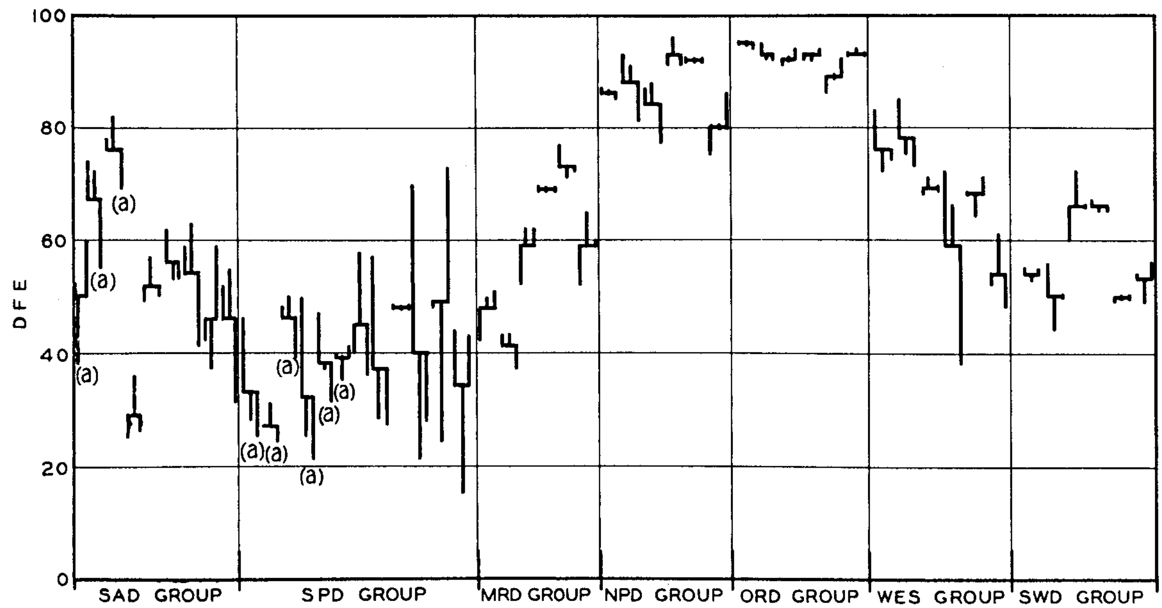
10. Variation in rate of temperature change, especially at or near the freezing point, as occasioned by variation in rate of flow of the cold or thaw fluid, quantity of fluid in the tank, power of compressor, or wattage of heaters, might very well be one of the more important

factors influencing test results. A low rate of flow may require a lower initial fluid temperature to produce the necessary amount of freezing in one hour.

Test Results

11. DFE of individual beams, air content, actual cement factor, and air loss are presented in table 1. The air loss shown is the average difference between air content in the plastic concrete and in the hardened specimens. Air content for all batches falls within 4.5 ± 0.5 per cent, as specified by CRD-C 114. There was no cause for rejection of any DFE data, and statistical analysis was thus simplified. Cement factor varied slightly, not more than 2 per cent among laboratories and 1 per cent at any one laboratory. Air loss was as great as 2.4 per cent (subtracted percentage values), but no consistent relationship between air loss and lower DFE is evident from a close inspection of the tabulated data. Air loss was not considered a sufficient cause for rejection or special handling of data in the statistical analyses.

12. Individual values of DFE are plotted in fig. 2, which affords a good general comparison of results from the various laboratories. A



(a) SUPPLEMENTARY SPECIMENS, NOT USED IN STATISTICAL ANALYSIS.

Fig. 2. Summary of results, Phase III

short horizontal line is drawn at the position representing the average DFE of each group of three specimens and vertical lines are drawn from the average line to points representing individual results. Extra sets of beams were made by the SAD and SPD laboratories. Results of tests of these specimens are also given in table 1 and shown in fig. 2, but were not used in the control chart analysis described in a subsequent paragraph, as it was desired to keep the weight of the data the same for each laboratory. As a uniform practice, the last 18 beams made at each laboratory were used in all statistical analyses.

13. Control charts are presented in figs. 3-6. Each of these charts conveys a slightly different interpretation of the outcome of the tests. The statistics used are average DFE, standard deviation, range, and coefficient of variation. These charts were prepared in accordance with the ASTM Manual on Presentation of Data, 1951 edition.

Discussion of Results

Control charts

14. The control charts (figs. 3-6) were produced from the results of tests on 18 beams from each laboratory. The control chart for average DFE, fig. 3, illustrates the type of reproducibility among laboratories that was desired. If close agreement were achieved among laboratories for average DFE, then further studies might be attempted to improve precision at any given laboratory. While the control chart for averages involves accuracy (as compared with precision) whether absolute or relative, the other control charts, figs. 4-6, deal largely with precision as attained in each laboratory. In these three

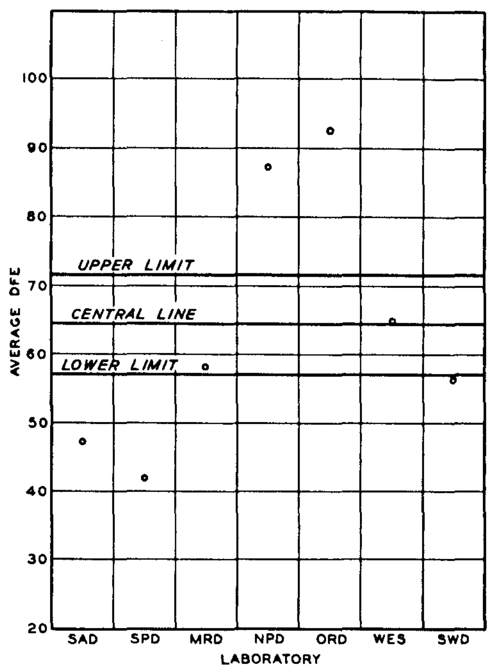


Fig. 3. Control chart for averages

charts, low values, rather than proximity to the central line, generally represent greater precision; that is, better agreement among results for individual specimens tested by a single laboratory. These charts are useful principally in demonstrating that the best apparent precision is not necessarily the result of adequacy of testing, at least as compared with the average adequacy of testing. This point is discussed further when each chart is considered separately in succeeding paragraphs.

15. The control chart for averages shows that the desired reproducibility was not attained among the laboratories. The central line represents the most probably accurate value, tests from all laboratories considered. Compared to this value, the SAD and SPD averages were too low while the NPD and ORD averages were too high. The SWD average was very near the lower control limit. Since instructions were developed and technical assistance was given by the WES, it was not improbable that WES results would most often be near the central line of the control charts, as is the case.

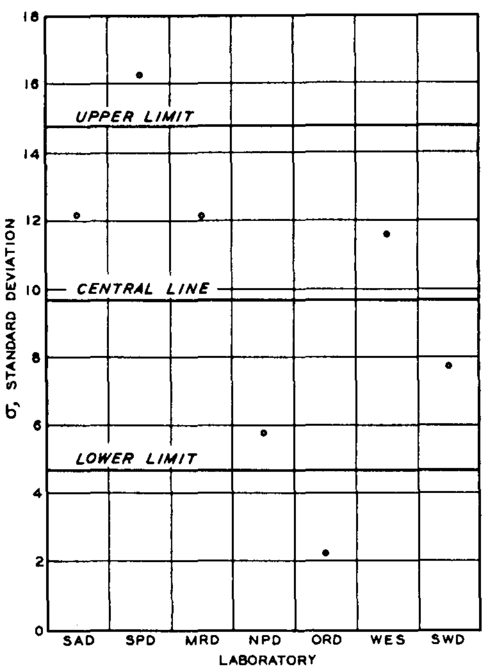


Fig. 4. Control chart for standard deviations

16. In the comparisons of standard deviation, illustrated by means of the control chart in fig. 4, a high value would ordinarily reflect lack of uniformity of fabrication or testing of individual specimens, and a low value would tend to indicate precision in all operations. This observation can be made only with considerable reservation. Again the central line represents the most probable, best value. The low value calculated for ORD, for instance, simply reflects to a considerable extent the effect of the grouping of individual results around an average DFE of 93, in a region where great variability is not to be expected. With results from all laboratories considered, it is noted that either the ORD concrete was much more durable

than the average, or the ORD freeze-thaw equipment produced less deterioration. ORD data are used only for illustration; the same reasoning applies to SPD results.

17. High and low values of range (SPD and ORD), fig. 5, should be considered with the reservation respecting standard deviation discussed in the preceding paragraph.

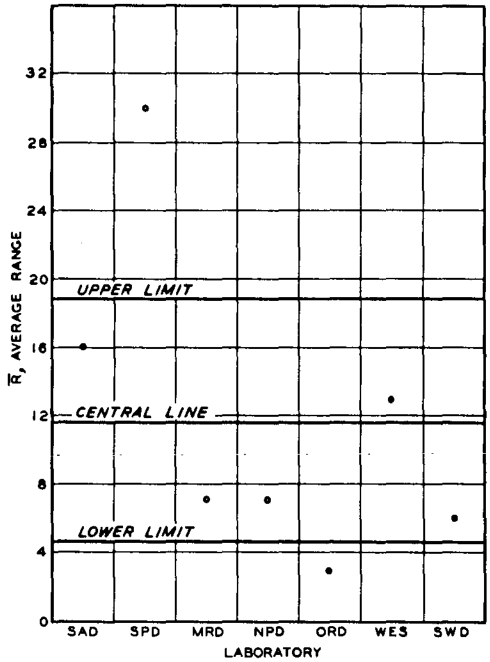


Fig. 5. Control chart for ranges

18. Coefficient of variation relates the measure of dispersion to its average and converts to percentage form, fig. 6. It is implied by this statistic that variation is likely to be proportional to the numerical value of the variable. The freezing-and-thawing test tends to confine data below 100 per cent as the upper limit (see fig. 7); therefore, coefficient of variation is no more reliable as a measurement of precision than is standard deviation or range.

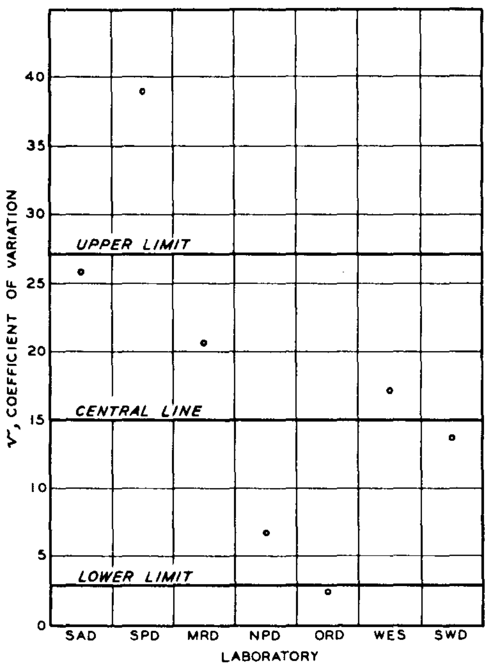


Fig. 6. Control chart for coefficient of variation

19. The results of tests at five laboratories fell within limits of control charts designed to compare precision.

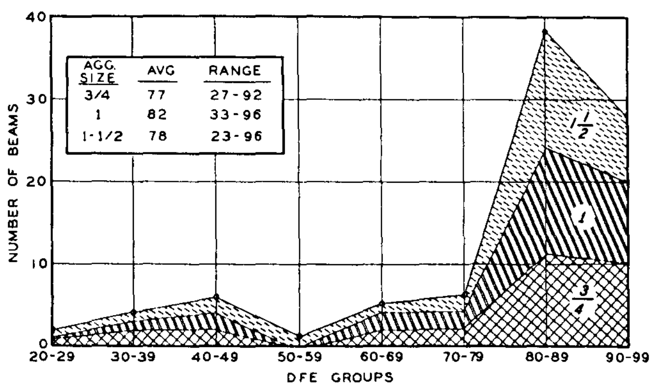


Fig. 7. Frequency distribution of DFE

The more important criterion, reproducibility among laboratories, was not so well satisfied. Only two laboratories, with a close third, produced results within control limits on the chart for averages. This chart was designed to compare results on the basis of reproducibility or accuracy, with absolute accuracy assumed to be at or near the average for all laboratories.

Cause of variation

20. The same mixture proportions were used by all laboratories and considerable care was exercised to have personnel perform each operation in exactly the same manner in each laboratory. Variation in specimen fabrication is therefore not regarded as being a probable cause of lack of reproducibility. Also there are no known differences of sufficient magnitude in freezing-and-thawing equipment to explain the variation in final average DFE values of 42 for SPD and 93 for ORD. Severity of the freezing-and-thawing operation in the temperature region of 32 F is perhaps the most nearly direct cause of abnormal reduction in DFE. SAD, SPD, and SWD had steep freeze-thaw curves over the region in question, and the average DFE's (see fig. 3) were low for these laboratories. The temperature curve (fig. 1) for the ORD apparatus was less steep than any other during the thaw cycle and the average DFE for ORD was highest of all. Several variables determine the rate of temperature change in the vicinity of 32 F. It has been suggested that rate of flow might affect results to the extent observed. A slow rate of flow might require that a lower initial cold fluid temperature be used in order to produce the required temperature change in one hour. Large differences in cold solution temperature might determine such an important factor as whether the specimen froze from bottom upward or from top downward. There is evidence that flow rate alone does not account for the observed variation. For instance, SAD and SPD had the greatest deterioration (if specimens were alike), although SAD had a flow rate of 75 gpm as compared with 15 gpm at SPD.

Comparisons of Phase III data with Phases I and II data

21. Four laboratories participated in Phases I and II, and seven

in Phase III. In Phase I all specimens were made at WES. In Phases II and III the specimens were made in the various laboratories where they were tested. In general, precision at each laboratory improved but reproducibility between laboratories apparently worsened. Since new conditions were imposed for each phase of tests, the lack of improvement in reproducibility is not difficult to understand. The improvement in over-all precision is measured by a reduction in average standard deviation from approximately 11 in Phase I, and slightly less than 11 in Phase II to 9.7 in Phase III.

Additional Tests

22. Paragraph 26 of the progress report dated 20 April 1950 stated that additional tests were in progress to determine, if possible, the cause of cracking near the ends of specimens. The results of these tests were inconclusive.

Conclusions and Recommendations

23. Reproducibility of results from the various participating laboratories is not adequate. This inadequacy is clearly illustrated on the DFE chart, fig. 2, and on the average DFE control chart, fig. 3. The direct cause of lack of reproducibility is not readily apparent, although there is considerable evidence that severity of the actual freeze-or-thaw process, that is, temperature variation in the region of 32 F, may be the principal factor. It is recommended that each laboratory make an examination of its operating procedures and equipment using the information gained by this analysis of data from all laboratories.

24. The ORD and NPD laboratories should attempt to determine whether there is reason to believe that specimens made in those laboratories were more durable than the average or whether the freeze-thaw test was less severe. On the other hand, the SAD and SPD laboratories should try to account for the apparent severity of tests conducted in those laboratories.

25. There is evidence, as shown in fig. 2, of considerable disparity among individual results in groups of three beams tested at SPD, possibly at SAD, and one group tested at WES. These laboratories should give attention to obtaining better uniformity of specimen fabrication or testing.

26. It is recommended that beams fabricated from the mixture design distributed by WES for use in Phase III be tested, as work load permits, at each laboratory where further study is desired. The mixture proportions should be regarded as a standard with DFE of beams equal to 65, at least until such time as further comparison of data from all laboratories warrants a change in this value. Each laboratory should conduct tests to determine why the procedure or equipment at that laboratory yields results different from the average value of 65.

PART III: PHASE V -- EFFECT OF MAXIMUM SIZE OF COARSE AGGREGATE ON DURABILITY OF 3-1/2- BY 4-1/2- BY 16-IN. BEAMS

27. This investigation, conducted in 1953 by the WES, was designed to determine the effect of size of coarse aggregate on the durability of concrete tested in the freeze-thaw apparatus. A total of 90 specimens was tested, in which each of three maximum sizes of the same aggregate was represented by 30 specimens. The three sets of specimens were as nearly identical as possible except for aggregate size. The specimens were made in accordance with applicable standard procedures, except as noted subsequently. It is possible to conclude from these tests, as will be seen below, that fortuitous variation in the test specification respecting maximum size of aggregate probably has no measurable effect on test results. There was a pronounced tendency for the specimens in this series to fail by pitting and fracture approximately 3 to 5 inches from the ends. A significantly large number of the specimens made on the first casting day failed. No variation in specimen fabrication or testing procedure is recorded to account for excessive failures in this group. The results of these tests, as do the results of tests described in part II, serve to place emphasis upon the fact that constant exercise of care is necessary in the performance of even the apparently minor steps of the procedures.

Description of Investigation

Materials

28. The cement was laboratory stock type II. The aggregate, both fine and coarse, was commercially produced crushed limestone. The fine aggregate was graded and recombined to give the following grading:

Sieve	% Passing
No. 4	100
No. 8	85 ± 3
No. 16	65 ± 5
No. 30	45 ± 5
No. 50	21 ± 5
No. 100	7 ± 2

The three gradings of coarse aggregate, to comply with the special requirements of this particular test, were as follows:

3/4-in. Max Size		1-in. Max Size		1-1/2-in. Max Size	
Sieve	% Passing	Sieve	% Passing	Sieve	% Passing
3/4-in.	100	1-in.	100	1-1/2-in.	100
1/2-in.	66 ± 3	3/4-in.	75 ± 5	1-in.	20 ± 5
3/8-in.	33 ± 3	1/2-in.	50 ± 5	3/4-in.	55 ± 5
No. 4	0 - 3	3/8-in.	27.5 ± 2.5	1/2-in.	35 ± 5
		No. 4	0 - 3	3/8-in.	20 ± 3
				No. 4	0 - 3

Mixtures

29. The concrete mixtures were made according to provisions of Test Method CRD-C 114-48 except that: (a) three aggregate combinations were used, i.e., the fine aggregate with each of the coarse aggregate gradings shown above; (b) calculated air content in the mortar phase varied from 9.3 to 12.4 per cent, the air content of the hardened mortar, calculated from density measurements, varied from 5.1 to 10.1 per cent; and (c) the specimens were cured for 14 days.

Specimens

30. The size of the specimens was 3-1/2 in. by 4-1/2 in. by 16 in. Ten rounds of 3 beams each for each aggregate grading were made. A set of 3 beams for each aggregate size (9 beams) constituted a round; one or more rounds with each aggregate size were made on each of five days. Nine beams were made on the first day, 18 each on the second, third and fourth days, and 27 on the fifth day. A total of 90 specimens was thus made, 30 for each of the three coarse aggregate gradings.

Tests

31. After curing for 14 days the specimens were subjected to the accelerated freeze-thaw test in accordance with method of test CRD-C 114-48. It has been noted previously that a considerable number of beams of this series broke or cracked severely near the ends although the specimens appeared to be firm and gave good relative E measurements at the time of last measurement prior to failure. The DFE for these broken specimens was extrapolated according to the formula given for the purpose in the above test method.

Test Results

32. Results of the tests are reported in terms of DFE of individual specimens in tables 2 to 4. The air content of the plastic concrete is given in these tables for each batch. Three beams were made from each batch. The air content of the hardened concrete is given for individual beams. Standard deviation and coefficient of variation were calculated for the 90 specimens, for each group of 30 specimens representing an aggregate size, and for each group of 30 beams excluding certain beams, as shown in table 5. The average DFE's are also given in table 5. In table 6 the DFE's are grouped to show frequency distribution with class intervals in steps of 10. These data are plotted in fig. 7 to show the contribution of each group of 30 to the total frequency distribution. This chart illustrates the characteristic skewness of DFE. The ordinary types of statistical analysis can be applied only with caution to data showing such a distribution. Fig. 8 is a scatter diagram illustrating the effect of air content on DFE. The beams, as they appeared after testing, are shown in figs. 9-11. The failed specimens are shown as a separate group in fig. 12.

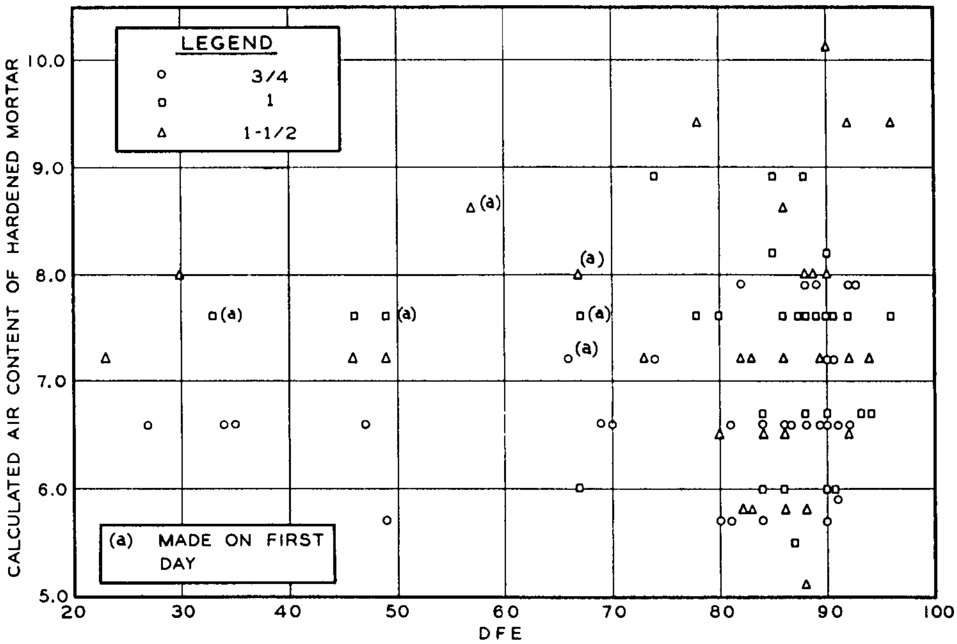


Fig. 8. Scatter diagram, DFE vs air content

Fig. 9. Beams after testing, 3/4-in. maximum-size aggregate

Fig. 10. Beams after testing, 1-in. maximum-size aggregate

Fig. 11. Beams after testing, 1-1/2-in. maximum-size aggregate

Fig. 12. Beams having DFE less than 80 per cent

Discussion of Results

33. It was originally proposed that the air content of plastic mortar and of the mortar of specimens in the hardened condition be within certain specified limits. The specified minimum amount of air in hardened mortar was 9.0 per cent. Early in the course of specimen fabrication it became apparent that this condition could not be met. The program was continued with good uniformity of air content, but only a very few specimens had as much as 9.0 per cent air in hardened mortar. Air content data from tables 2, 3, and 4 are summarized as follows:

Maximum Aggregate Size	Air Content: Plastic Mortar	Air Content: Hardened Mortar
3/4 in.	9.3 to 10.3 (avg 9.7)	5.7 to 7.9 (avg 6.7)
1 in.	9.4 to 10.8 (avg 10.0)	5.5 to 8.9 (avg 7.3)
1-1/2 in.	9.7 to 12.4 (avg 10.3)	5.1 to 10.1 (avg 7.4)

Of the 90 beams tested, the range in DFE was 23-96 (average 79). DFE values ranged as follows according to aggregate size:

Maximum Aggregate Size	Range
3/4 in.	27 to 92 (avg 77)
1 in.	33 to 96 (avg 82)
1-1/2 in.	23 to 96 (avg 78)

Of the 90 beams, 66 had DFE's of 80 or above. These are grouped according to aggregate size as follows:

Maximum Aggregate Size	DFE Range 80-89	DFE Range 90-99	Total
3/4 in.	11	10	21
1 in.	13	10	23
1-1/2 in.	14	8	22

Of the 90 beams, 24 had DFE's of less than 80, grouped as follows:

Maximum Aggregate Size	DFE of Individual Beams	Total No.
3/4 in.	27,34,35,47,49,66,69,70,74	9
1 in.	33,46,49,67,67,74,78	7
1-1/2 in.	23,30,46,49,57,67,73,78	8

Of the 24 beams with DFE less than 80, 5 suffered failure by the breaking off of one or both ends; 16 had major cracks. There is no apparent correlation between DFE less than 80 and (a) aggregate size, as essentially equal numbers of failures occurred in all three groups, or (b) air content of mortar calculated from determinations made either before or after the concrete had hardened (see fig. 8). There was a noticeable correlation between day of making and number of specimens having DFE's less than 80, summarized as follows:

Making Day	Total Made	Specimens with DFE Less than 80: 3/4-in. Aggregate	1-in. Aggregate	1-1/2-in. Aggregate	Total	Per Cent
1st	9	1	3	2	6	67
2d	18	1	0	1	2	11
3d	18	2	1	2	5	28
4th	18	2	0	1	3	17
5th	27	3	3	2	8	30

The proportion of specimens with DFE $<$ 80 is 2 to 6 times greater for specimens made on the first making day than for those made on later days. It can be seen from table 5 that aggregate size had very minor, if any, effect on average DFE; the average for 90 beams was 79 and other averages of groups of 30 specimens were 77, 82, and 78. Since failure in most instances was attributable to an abnormal cracking condition, DFE's were averaged for separate groups (see table 5) from which specimens with DFE less than 80 were removed. After exclusion of these specimens a better agreement was obtained, and DFE's averaged 87, 88, and 87 for the three groups.

34. Eleven of the 90 specimens were subjected to microscopic examination after removal from freezing-and-thawing. These specimens included those having the highest and the lowest:

a. Calculated air content of freshly mixed mortar,

b. Calculated air content of hardened mortar,

c. Difference between a and b, and

d. DFE.

All three coarse aggregate gradings were represented. The examination included a determination of void content using the point-count method. Differences were found between values for air content determined by point-count and those calculated from pressure tests of freshly mixed concrete and displacement tests of hardened concrete. These differences, however, do not indicate that a correlation exists between air content and DFE within the range illustrated by these tests. Four of the eleven specimens examined had DFE values lower than 80 (23, 27, 57, and 67). In some cases it is possible that lowered DFE may have resulted from a larger-than-normal number of particles of lower-than-average soundness occurring in the particular group of coarse aggregate particles included in the specimen. It may be noted that while the aggregate used in this work is of exceedingly uniform composition, it nevertheless includes variable amounts of several recognizably distinct varieties of limestone of significantly different quality.

Conclusions and Recommendations

35. The following conclusions and recommendations appear to be justified on the basis of the results of the tests and statistical studies.

a. The durability of concrete, as represented by the specimens fabricated for this series of tests and as measured by the accelerated freeze-and-thaw test, is not affected by: (1) aggregate size, or (2) variation in calculated air of plastic or hardened mortar, within the ranges encountered.

b. It is recommended that cause of the development of severe cracks at or near the ends of the specimens be investigated in further studies designed to improve reproducibility of the freeze-and-thaw test method.

PART IV: SUMMARY OF RESULTS OF PHASES I-V

36. Phases I and II of the cooperative freezing-and-thawing program served to emphasize the necessity of obtaining better reproducibility among laboratories. Four laboratories participated in Phases I and II; seven laboratories participated in the Phase III program. Results of the tests show conclusively that better reproducibility is required in order that results from several laboratories be comparable.

37. Phase III of the program of cooperative freezing-and-thawing tests involved tests of only one type of specimen. Eighteen or more specimens were tested by the seven different laboratories. The results show that reproducibility among laboratories is not sufficiently adequate to permit reliance to be placed on comparative tests when several laboratories are involved. The results of this series of tests illustrate that care must be exercised in the execution of even the minor steps of the procedure and in the observation of the effect of every detail of improper operation of machinery or equipment. Cooperative tests of this type frequently clarify procedures, and result in ultimate revision of the test method.

38. The cooperative tests designated Phase IV were conducted jointly by the MRD and SAD Laboratories. This program of tests is a good example of the type of investigation that will lead eventually to acceptable reproducibility among results from different laboratories, as the results show excellent agreement between the laboratories. The combined results illustrate remarkably well the relationship between durability and air content. However, conclusions regarding reproducibility of any freezing-and-thawing results must be very carefully examined before they are accepted as forming a basis for a major revision of the test method. The data reported by MRD and SAD show a range of 34 DFE units at 4.5 per cent air (average DFE approximately equal to 65) compared with a range of only 8 units at 10 per cent air (average DFE approximately equal to 92). Actually, no better reproducibility is represented by the latter data, with a range of only 8 DFE units. The reproducibility in each case is almost exactly identical, as shown below. What actually

is produced by a freezing-and-thawing apparatus is a measurable deteriorating effect. The effect is measured by the reduction in DFE and not by the remaining DFE, the value commonly reported. The range of 34 divided by the reduction in DFE (100-65) is very nearly equal to the range of 8 divided by the corresponding reduction (100-92), as follows:

$$(34)/(100-65) \cong (8)/(100-92)$$

Even if better reproducibility were obtainable at a higher specified air content, the view may be taken that a mortar matrix can be so durable as to overshadow effects due to "unsoundness" of the aggregate; the information sought would therefore be lost. A change to higher air content, if a general increase of DFE could be assumed for most types of concrete, would undoubtedly result in a smaller range in DFE results, as illustrated by results of the MRD-SAD tests, without regard to soundness of aggregates. A lesser air content and a wider range of results should be regarded as preferable until the advantages and disadvantages of a change to higher air content can be more thoroughly evaluated.

39. In Phase V, 90 specimens representing three gradings of the same aggregate were tested in the WES freezing-and-thawing equipment under conditions that might be regarded as typical for this type of testing. The test conditions were considered adequate even though the equipment was inoperative for short periods of time due to breakdowns. Also there was an unexpected, though previously experienced, breakage of some of the specimens near the ends. Partly concurrent with the testing of this series of 90 specimens, several hundred specimens of another series of a different type of concrete were tested with less than the usual occurrence of fracture near ends. The exact cause of this type of failure has not as yet been determined. When the cause is known a further revision of the test method may be indicated. The results of this series of tests illustrate the agreement among results that may be obtained when groups of specimens are tested in the same freezer. (Also illustrated is the fact that some type of unexpected problem relating to the behavior of the various specimen types may arise at the outset or during the course

of any series of tests using this type of equipment.)

40. Representatives from all Division Laboratories attended the 1952 Division Laboratories Concrete Conference at Omaha, where various problems confronting the laboratories were discussed, including problems relating to the freezing-and-thawing tests. Suggestions made at this conference and comments received from all laboratories since the conference have been considered in the formulation of proposed revisions to Test Method CRD-C 114.

Table 1

DFE, Air Content, and Cement Factor of Specimens Tested in Cooperative Freezing-and-thawing Program, Phase III

Beam No.	Tested by	DFE	Plastic Air Content %	Actual Cement Factor bags/cu yd	Average[a] Air Loss %
399A, B, C[b]	SAD	52, 38, 60	4.5	6.30	0.7
400A, B, C[b]	SAD	74, 72, 55	4.5	6.30	1.0
401A, B, C[b]	SAD	78, 82, 69	4.9	6.28	0.3
402A, B, C	SAD	25, 36, 26	4.5	6.30	1.3
404A, B, C	SAD	49, 57, 50	4.4	6.30	0.5
438A, B, C	SAD	62, 53, 53	4.9	6.30	0.6
438D, E, F	SAD	59, 63, 41	5.0	6.30	0.6
438G, H, I	SAD	42, 37, 59	5.0	6.30	1.3
439D, E, F	SAD	52, 55, 31	5.0	6.30	1.9
4, 5, 6[b]	SPD	46, 28, 25	4.7	6.40	0.6
1A, 2A, 3A[b]	SPD	27, 31, 24	4.8	6.40	0.2
7, 8, 9[b]	SPD	48, 50, 39	4.8	6.40	0.6
10A, 11A, 12A[b]	SPD	50, 25, 21	5.0	6.40	0.2
13, 14, 15[b]	SPD	47, 37, 31	4.9	6.40	0.5
16A, 17A, 18A[b]	SPD	40, 35, 41	5.0	6.40	0.4
184, 185, 186	SPD	40, 58, 36	4.5	6.40	0.0
187, 188, 189	SPD	57, 28, 27	4.9	6.40	0.8
193, 194, 195	SPD	48, 48, 48	4.9	6.40	0.2
196, 197, 198	SPD	70, 21, 28	5.0	6.40	0.3
199, 200, 201	SPD	47, 24, 73	4.9	6.40	-0.1
205, 206, 207	SPD	44, 15, 43	4.4	6.40	0.2
A-1, A-2, A-3	MRD	42, 50, 51	4.6	6.40	1.2
B-1, B-2, B-3	MRD	43, 43, 37	5.0	6.40	1.5
C-1, C-2, C-3	MRD	53, 62, 62	5.0	6.40	1.3
D-1, D-2, D-3	MRD	69, 69, 69	5.0	6.40	0.3
E-1, E-2, E-3	MRD	77, 71, 72	5.0	6.40	0.3
F-1, F-2, F-3	MRD	52, 65, 60	4.8	6.40	0.6
3118-1, 2, 3	NPD	87, 86, 85	4.7	6.40	1.8
3118A-1, 2, 3	NPD	93, 91, 81	4.7	6.40	2.0
3118B-1, 2, 3	NPD	87, 88, 77	4.5	6.40	2.0
3118C-1, 2, 3,	NPD	91, 96, 91	4.5	6.40	1.5
3118D-1, 2, 3	NPD	92, 92, 92	4.9	6.40	2.4
3118E-1, 2, 3	NPD	75, 80, 86	4.9	6.40	1.5
54Z-1, 2, 3	ORD	95, 95, 94	5.0	6.30	1.6
54Z-4, 5, 6	ORD	95, 92, 92	4.8	6.30	1.4
54Z-7, 8, 9	ORD	91, 92, 94	4.6	6.30	1.6
54Z-10, 11, 12	ORD	92, 92, 94	4.9	6.30	1.1
54Z-13, 14, 15	ORD	86, 89, 92	4.9	6.30	1.8
54Z-16, 17, 18	ORD	93, 94, 93	5.0	6.30	1.3
4946, 47, 48	WES	83, 72, 74	4.9	6.37	1.4
4949, 50, 51	WES	85, 75, 73	5.0	6.36	1.0
4952, 53, 54	WES	68, 71, 69	4.9	6.37	1.7
4955, 56, 57	WES	72, 66, 38	4.5	6.40	1.6
4958, 59, 60	WES	68, 64, 71	5.0	6.36	0.4
4961, 62, 63	WES	52, 61, 48	4.9	6.37	2.2
1560, 61, 62	SWD	55, 53, 55	5.0	6.29	2.1
1563, 64, 65	SWD	56, 44, 50	5.0	6.29	2.1
1566, 67, 68	SWD	60, 72, 66	4.7	6.30	2.3
1569, 70, 71	SWD	67, 65, 65	5.0	6.29	1.9
1579, 80, 81	SWD	49, 50, 50	5.0	6.29	1.6
1582, 83, 84	SWD	54, 49, 56	4.6	6.31	1.7

[a] Plastic air content minus hardened air content.
[b] Supplementary specimens, not used in statistical analysis.

Table 2

DFE of Concrete Specimens Containing 3/4-in. Maximum-size Coarse Aggregate

Spec No.	Making Date	Air Content a	Air Content b	DFE[c]	Remarks
5161			7.2	(66)[d]	End broke off at 268 cycles
5162	10/8/53	10.3	6.6	92	
5163			7.2	90	
5176			7.9	89	
5177	10/9/53	10.0	7.9	92	Cracked on end at 277 cycles
5178			7.9	92	
5179			6.6	(27)	Cracked on end at 156 cycles
5180	10/9/53	9.3	6.6	91	
5181			5.7	84	
5188			6.6	(35)	Cracked 3 in. from end at 210 cycles
5189	10/13/53	9.6	6.6	81	
5190			5.7	81	
5197			6.6	84	
5198	10/13/53	9.3	5.7	90	
5199			6.6	(34)	Cracked on ends and sides at 210 cycles
5206			5.9	91	Cracked on side at 241 cycles
5207	10/14/53	9.5	6.6	(47)	Cracked across end at 272 cycles
5208			5.7	(49)	Cracked on top and side at 220 cycles
5215			6.6	86	
5216	10/14/53	9.6	6.6	90	
5217			6.6	90	
5224			7.2	90	
5225	10/15/53	9.6	6.6	86	
5226			6.6	88	
5233			6.6	(69)	Cracked on end and top at 261 cycles
5234	10/15/53	9.6	5.7	80	
5235			6.6	(70)[d]	Both ends broke off at 282 cycles
5242			7.9	82	
5243	10/15/53	10.0	7.9	88	
5244			7.2	(74)	
	Average	9.7	6.7	77	

[a] Air content of batch, plastic mortar.
[b] Air content of individual beams, hardened mortar.
[c] Relative modulus of elasticity at 300 cycles, per cent.
[d] DFE was extrapolated after end broke off.

Numbers in parentheses are DFE values less than 80.

Table 3

DFE of Concrete Specimens Containing 1-in. Maximum-size Coarse Aggregate

Spec No.	Making Date	Air Content a	Air Content b	DFE[c]	Remarks
5164			7.6	(33)	End broke off at 223 cycles
5165	10/8/53	10.8	7.6	(49)[d]	Cracked 4 in. from end at 289 cycles
5166			7.6	(67)[d]	End broke off at 247 cycles
5173			7.6	89	
5174	10/9/53	9.9	6.7	94	
5175			6.7	93	
5182			7.6	96	
5183	10/9/53	9.7	7.6	90	
5184			6.0	91	
5191			5.5	87	
5192	10/13/53	9.6	6.0	86	
5193			6.0	90	
5200			8.9	(74)[d]	End fell off at 283 cycles
5201	10/13/53	10.6	8.9	85	
5202			8.2	85	
5209			6.7	90	Cracked on end at 272 cycles
5210	10/14/53	9.7	6.7	84	Cracked on side and end at 241 cycles
5211			7.6	88	
5218			7.6	92	
5219	10/14/53	9.4	7.6	80	
5220			7.6	88	
5227			8.2	90	
5228	10/15/53	10.4	8.9	88	
5229			7.6	90	
5236			6.7	88	
5237	10/15/53	10.1	7.6	86	
5238			7.6	(78)	
5245			6.0	84	
5246	10/15/53	10.2	7.6	(46)	Cracked on end and top at 282 cycles
5247			6.0	(67)	Cracked 4 in. from end at 282 cycles
	Average	10.0	7.3	82	

a Air content of batch, plastic mortar.
b Air content of individual beams, hardened mortar.
c Relative modulus of elasticity at 300 cycles, per cent.
d DFE was extrapolated after end broke off.

Numbers in parentheses are DFE values less than 80.

Table 4

DFE of Concrete Specimens Containing 1-1/2-in. Maximum-size Coarse Aggregate

Spec No.	Making Date	Air Content a	Air Content b	DFE[c]	Remarks
5167			8.6	(57)	Cracked on end and side at 289 cycles
5168	10/8/53	12.4	8.0	(67)	
5169			8.0	88	
5170			8.6	86	
5171	10/9/53	10.4	7.2	(73)	Cracked 3 in. from end at 235 cycles
5172			7.2	94	
5185			10.1	90	
5186	10/9/53	10.7	9.4	96	
5187			9.4	92	
5194			5.1	88	
5195	10/13/53	9.7	5.8	88	
5196			7.2	90	
5203			7.2	(49)	Cracked 4 in. from end
5204	10/13/53	9.7	7.2	(23)	Cracked on side at 142 cycles
5205			6.5	84	
5212			7.2	82	
5213	10/14/53	10.2	9.4	(78)	Cracked 4 in. from end at 293 cycles
5214			6.5	92	
5221			7.2	82	
5222	10/14/53	10.2	7.2	92	
5223			8.0	88	
5230			8.0	90	
5231	10/15/53	10.4	7.2	86	
5232			8.0	(30)	Cracked on side at 167 cycles
5239			7.2	(46)	Cracked 3 in. from end at 261 cycles
5240	10/15/53	9.7	5.8	82	
5241			6.5	86	
5248			5.8	82	
5249	10/15/53	9.7	5.8	86	
5250			6.5	80	
	Average	10.3	7.4	78	

[a] Air content of batch, plastic mortar.
[b] Air content of individual beams, hardened mortar.
[c] Relative modulus of elasticity at 300 cycles, per cent.

Numbers in parentheses are DFE values less than 80.

Table 5

Average DFE, Coefficient of Variation, and Standard Deviation, According to Aggregate Size

Specimen Group	No. of Spec	Coefficient of Variation %	Standard Deviation of DFE	Average DFE
Total series	90	21.8	17.2	79
3/4-in. max size	30	24.1	18.6	77
1-in. max size	30	15.1	12.4	82
1-1/2-in. max size	30	24.8	19.4	78
Selected groups[a]				
3/4-in. max size	21	11.4	9.9	87
1-in. max size	23	10.7	9.5	88
1-1/2-in. max size	22	11.3	9.9	87
Selected groups[b]				
3/4-in. max size	27	27.0	20.5	76
1-in. max size	27	12.9	11.0	85
1-1/2-in. max size	27	24.1	19.0	79

[a] DFE's less than 80 are excluded from these groups.
[b] DFE's of beams made on first making day are excluded.

Table 6

Frequency Distribution of DFE

Maximum Size of Aggregate	Class Interval 10-19	20-29	30-39	40-49	50-59	60-69	70-79	80-89	90-100
3/4 in.		27C	35C 34C	47C 49C		66E 69C	70EE 74	89, 84, 81 81, 84, 86 86, 88, 80 82, 88	92C, 90 92, 92 91C, 90 91, 90, 90 90
	(0)	(1)	(2)	(2)	(0)	(2)	(2)	(11)	(10)
1 in.			33E	49C 46C		67E 67C	74E 78	89, 87, 86 85, 85, 84C 88, 80, 88 88, 88, 86 84	94, 93, 96 90C, 91 90, 90, 92 90, 90
	(0)	(0)	(1)	(2)	(0)	(2)	(2)	(13)	(10)
1-1/2 in.		23C	30C	49C 46C	57C	67	73C 78C	88, 86, 88 88, 84, 82 82, 88, 86 82, 86, 82 86, 80	94, 90, 96 92, 90, 92 92, 90
	(0)	(1)	(1)	(2)	(1)	(1)	(2)	(14)	(8)
Total	0	2	4	6	1	5	6	38	28

E An end broke off.
EE Two ends broke off.
C Crack developed.

Numbers in parentheses are totals for the class and aggregate size indicated.