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## Durability Test on Iowa Limestones and Dolomites

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*Iowa State Highway Commission*

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## DURABILITY TESTS ON IOWA LIMESTONES AND DOLOMITES

LYMAN W. WOOD

### *Introductory*

The purpose of this paper is to summarize recent durability investigations of Iowa limestones and dolomites by the Iowa State Highway Commission, and to relate these investigations to the more general subject of the durability of Portland cement concrete. A subject as broad as the title indicates can be presented only very briefly at this time. The evidence discussed is for the most part from the Highway Commission records, supplemented by information from other sources including the Iowa Geological Survey publications and files. Acknowledgement is made to Bert Myers, Engineer of Materials and Tests, and to other employees of the Highway Commission who have assisted with information and suggestions, and to the Iowa Geological Survey for permission to use unpublished material in their files.

Laboratory tests for the purpose of predicting durability of structural materials are of long standing and any comprehensive discussion of the history of such tests cannot be undertaken here. From the earliest time, the attempt has been to reproduce and accelerate natural weathering processes, operating through temperature and moisture variations, freezing and thawing, or chemical changes. Of these, freezing and thawing seem to play an important part in Iowa latitudes, and this has at all times received a large portion of investigators' attention. Other weathering processes, particularly the chemical changes which may take place in this humid climate, have perhaps not had the attention they deserve. Freeze and thaw studies have received great impetus in recent years from the development of mechanical refrigeration.

Durability or soundness tests on concrete materials may include those performed on the cement, the fine aggregate, and the coarse aggregate. Of these, cement soundness is a most interesting subject in itself, and no attempt will be made to include it here. Iowa is fortunate in the possession of an abundant and well-distributed supply of sand for fine aggregate, and this sand, being composed mainly of quartz grains, is felt to have uniformly good enough durability characteristics to make a fine aggregate soundness test generally unnecessary. Iowa gravel coarse aggregates have had a long history of transportation and weathering

before their utilization by man, and they display generally good soundness. This paper will therefore be confined principally to a consideration of durability determinations on limestone and dolomite coarse aggregates.

The first coarse aggregate soundness test in the Highway Commission laboratory consisted of repeated crystallizations of sodium sulphate in saturated specimens, with accompanying volume changes which disrupted the rock in much the same manner as do growing ice crystals during freezing. This test was in common use on masonry stone, and when concrete pavements were first built extensively in Iowa, it was applied to the crushed stone coarse aggregates. With the development of mechanical refrigeration, the direct freeze and thaw test was introduced as a check on the sodium sulphate test, both determinations at first showing fairly good agreement. As time went on, and concrete pavements grew older, there began to appear some evidence that the unsoundness of certain coarse aggregates previously accepted was responsible for some observed failures of small sections of pavement. The freeze and thaw test was therefore modified in the direction of greater severity, finally reaching the present form outlined in a later part of this paper. More recently the sodium sulphate test on concrete aggregates has been entirely discontinued.

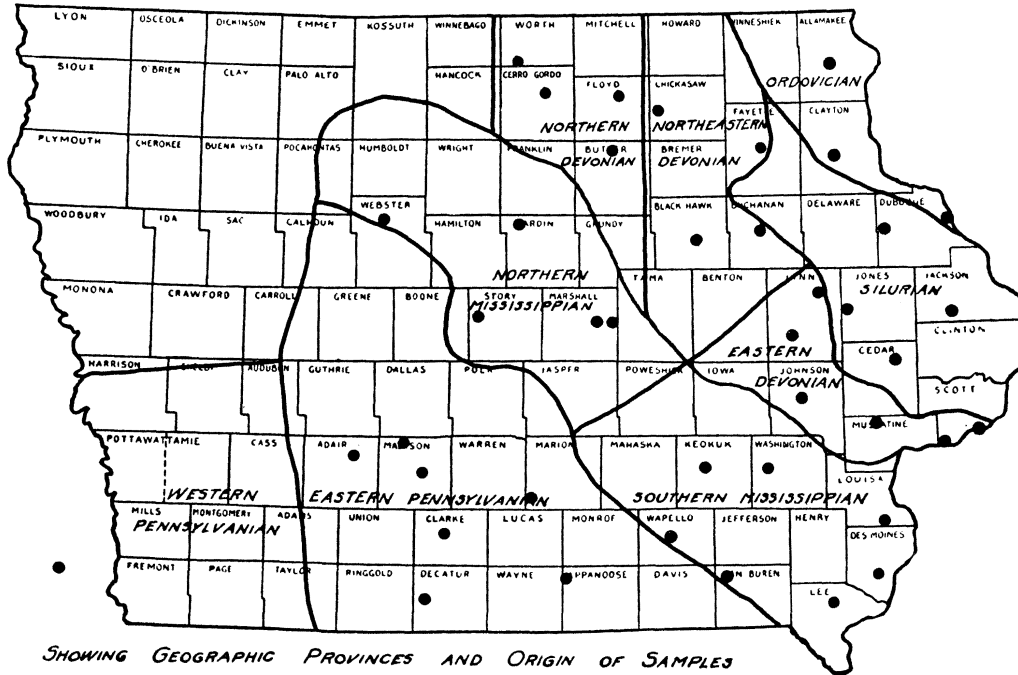
At the end of the 1940 construction season, it appeared desirable to resurvey the whole body of knowledge on Iowa concrete aggregate durability, and to reconsider the evidence, from the service record and other lines of information, as well as from the modified freeze and thaw test. In this resurvey, forty-one representative sources or potential sources of concrete aggregate were chosen. Most of these were represented by one laboratory sample, but some by more, so that a total of fifty-two samples was studied. Figure 1 shows the locations of the sources sampled and the boundaries of nine geographic and geologic provinces into which the samples are grouped.

#### *Laboratory Tests*

Earlier freeze and thaw tests were performed on ten pieces of stone of approximately  $1\frac{1}{4}$ -inch cubical shape. The present series of samples was crushed, either at the source or in the laboratory, and then divided into four sizes; plus 1-inch, 1-inch to  $\frac{3}{4}$ -inch,  $\frac{3}{4}$ -inch to  $\frac{3}{8}$ -inch, and  $\frac{3}{8}$ -inch to  $\frac{3}{16}$  inch. This crushing is believed to increase the accuracy of the test by increasing the number of pieces involved, and to increase its severity by the increased surface area exposed. Each size was tested separately.

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SHOWING GEOGRAPHIC PROVINCES AND ORIGIN OF SAMPLES

All samples were frozen and thawed in pans, partially or entirely immersed in water at all times, and thoroughly saturated. Freezing was in an alcohol bath at a temperature of 0 to  $-10^{\circ}$  F, and thawing in a water bath at  $70^{\circ}$  F, each being accomplished in about an hour's time. Unsoundness was measured after 16 cycles and 50 cycles of freezing and thawing as a percentage disintegrated to a size which passes the Number 8 sieve (0.093-inch square openings).

The matter of saturation deserves further comment. It has been found in the Highway Commission laboratory, and verified in other laboratories, that when water is removed by evaporation from the minute pores in a stone, the air which takes its place becomes partly entrapped so that it permits the re-entrance of much less water with subsequent immersion. This seems to be true even when immersion is continued as long as seven days. The entrapped air acts as a cushion which greatly reduces the destructive effect of water expansion from freezing. Since laboratory samples nearly always have more or less opportunity for evaporation before testing, experiments were made on various means of accomplishing complete saturation, the following being finally adopted. The samples were placed under a 28-inch vacuum for 15 minutes to exhaust the air, water was then admitted into the vacuum slowly until the material was totally immersed, and the vacuum then continued for another 15 minutes. Consideration of the cushioning effect of entrapped air leads to the thought that all stones in construction work will withstand weathering better if allowed first to dry out thoroughly. Quarry men of masonry stone have long recognized this fact by their insistence upon a period of 'seasoning' before use of the material.

The type of failure brought on by the freeze and thaw test described is a disintegration and sloughing, starting at the surface and continuing thence inward toward the center of the particle. In any one type of sample of stone, this disintegration is approximately proportional to the surface area, so that the smaller particles suffer most. Since it seemed desirable in the recent study to express the degree of disintegration by one figure, an average concrete aggregate grading was assumed, and disintegration percentages on each size combined into an average percentage weighted according to the proportions of each size in the average grading. This is the average disintegration percentage used in comparing freeze and thaw test results with other lines of evidence.

Comparison of the average disintegration percentage at 16 cycles of freezing and thawing with that at 50 cycles, showed that in the majority of cases the curve of disintegration was a nearly straight line, or that its slope became flatter as the test progressed. In other words, the larger number of cycles showed no unsoundness tendency which could not be predicted from the smaller number. A few exceptions to this generalization seem to be connected with high moisture absorption percentages, an effect not unexpected from the nature of failure by freezing and thawing.

#### *Chemical and Mineralogical Composition*

The chemical and mineralogical compositions of limestones and dolomites probably have an important effect on their durability. These factors, however, have had very little study. A few generalizations can be made at this time.

It has been pointed out by Grim, Lamar, and Bradley(1), that the amount and character of included clay minerals have much to do with the resistance of limestone to weathering, and particularly to repetitions of wetting and drying. Mineralogical studies on the clay minerals in Iowa stones have not been made, but some indication of the amount of clay mineral present can be had from the percentage of  $R_2O_3$  in the chemical analysis. Stones with a high content of  $R_2O_3$  are believed to be less durable.

Limestones of high calcium carbonate content are more easily soluble in natural waters than those containing magnesium carbonate or silica. In the humid climate generally prevailing in Iowa, solubility effects on some stones may be important. Very little is known of this type of failure, although Gwynne(2), pointed out its occurrence in sandstone used in the State Capitol Building at Des Moines. It appears that concrete aggregate stone, being largely protected from circulating water by the surrounding mortar, is not much subject to this type of attack.

Since the minerals calcite and dolomite are isomorphous, and rather closely related chemically, there is no reason for expecting any important difference in their soundness characteristics. Experience in highway concrete pavements shows no appreciable difference. Dolomite may be a little more durable on account of lesser solubility, but this is not believed to be important.

(1) Grim, R. E., Lamar, J. E., and Bradley, W. F.; *The Clay Minerals in Illinois Limestones and Dolomites*, Journal of Geology, Vol. 45, No. 8, pp. 842-843, 1937.

(2) Gwynne, C. S., *Weathering of Sandstone in the Iowa State Capitol Building*, Iowa Academy of Science, Vol. 41, pp. 177-190, 1934.

*Natural Weathering*

No quantitative measure of the effect of natural weathering on Iowa stones has been made, as it includes such a complexity of factors as to permit only the most general conclusions. Briefly, it can be said that stones which stand out in natural weathering without evidence of serious cracking or disintegration are the most durable. Put in another way, the sound stones are cliff makers, while the unsound stones weather to a steep slope strewn with broken fragments or other products of decay, but showing little or nothing of the original ledge in place. This judgment, however, must be modified by certain factors, such as thickness of ledge affected, topographic relief, presence or absence of softer material above or below, and amount of overburden. Some of the stones studied occur in localities where there is almost no natural outcrop, so that there is no evidence on their durability from natural weathering. Others are widely exposed and their weathering characteristics are well known.

*Service Record*

Cement concrete highway pavements in Iowa were built as early as 1913, and a considerable mileage is now about 20 years old. Systematic observation of condition, especially in recent years, has yielded a large body of information on durability characteristics. Such condition survey information has considerable value, and yet, it is affected by so many variables, some known and some partly or entirely unknown, that only general conclusions can be drawn. For example, the present condition depends much upon age, and intensity and character of traffic. It is affected by the design of the slab, whether thicker at the edges or the center, and how reinforced. The character of subgrade support is important; this depends much upon topographic conditions and nature of subgrade materials. Climatic variations, even in a state no larger than Iowa, seem to have had some effect. Nevertheless, it is true in spite of all these and other variables, that pavements built with certain coarse aggregates have remained in outstandingly good condition, while pavements built with certain others have already shown alarming signs of failure.

Concrete pavement failures may be of many kinds, unrelated or only partly related to aggregate soundness. Most Iowa pavements are carrying a traffic much faster and heavier than contemplated in the original design, and the resulting increases in stress have brought on or aggravated most pavement failures. On the

other hand, many sections of road are successfully carrying the heavy traffic so that additional causes of failure must be sought. Occasional pitting or spalling results from individual unsound particles in a coarse aggregate otherwise satisfactory. Scaling and surface disintegration are caused by excessive salt applications to remove ice, or in part by repetitions of freezing and thawing of small quantities of surface water in winter. Hairchecking seems to be related to moisture or temperature conditions at the time of construction. Cracking is the result of temperature or moisture contraction, aggravated by inadequate or uneven subgrade support. There are, however, some examples of disintegration, beginning usually at the interior slab corners or other vulnerable places, and progressing thence almost indefinitely, which seem to be related to unsoundness of the concrete. It has not been definitely proved that the unsoundness of concrete in these instances is the result of coarse aggregate unsoundness, but this type of failure is frequent with some aggregates and conspicuously absent with others, and the thought of a relation to some aggregate characteristic seems inescapable.

Many of the sources used in the present resurvey have little or no service record. In some cases, this is because the material has always been considered unsound and has been excluded by engineers' specifications. Others have been considered acceptable but not used because of competition from other materials. Exposure conditions in road or bridge structures are believed to be more severe than in most masonry walls, and the latter use has for that reason had only minor consideration.

#### *Rating of Stone Sources*

From the foregoing lines of evidence, the rating table, Table 1, has been prepared. The forty-one sources included in the most recent study are grouped in accordance with the provinces in which they occur. The geological formation represented, the location of the sample by section and township, and the nearest named locality are shown.

On the basis of laboratory evidence from the freeze and thaw test, three ratings are made. The "A" group shows 0% to 5% average disintegration at 16 cycles of freezing and thawing; the "B" group 5% to 12%; and the "C" group over 12%. Ratings in individual cases have been modified by the disintegration percentage at 50 cycles, or by results on earlier tests.



In the chemical and mineralogical rating, only one factor, the percentage of  $R_2O_3$ , has been used. The validity of this factor is open to question, and yet, nothing better is known to the writer at this time. The "A" group has 0% to 4% of  $R_2O_3$ , the "B" group 4% to 8%, and the "C" group over 8%. No account is made of solubility, and calcite and dolomite are considered equal.

Table 1. Durability Rating of Limestone and Dolomite Sources

ORIGIN OF MATERIAL EXAMINED						Lab. Freeze & Thaw Rating	Chemical & Mineralogical Rating	Natural Weathering Rating	Service Record Rating
Province	Formation Represented	County	Section	Township	Locality				
Ordovician	Oneota	Allamakee	19	98:4	Waukon	A	B	A	A
	Dubuque	Clayton	17	92:4	Elkader	C	--	B	B
	Prosser	Dubuque	7	89:3E	Dubuque	A	A	A	A
Silurian	Hopkinton	Dubuque	26	89:2W	Dyersville	A	A	B	--
	Hopkinton	Jackson	18	84:3E	Maquoketa	A	A	A	--
	Anamosa	Jones	33	85:4	Anamosa	B	A	B	--
	LeClaire	Cedar	33	82:1W	Lowden	A	A	A	--
Eastern Devonian	Otis	Linn	12	85:6	Central City	B	A	B	--
	Otis	Linn	34	83:7	Cedar Rapids	A	A	B	B
	Davenport	Scott	26	78:4E	Bettendorf	A	A	A	A
	Davenport	Scott	23	77:2E	Linwood	A	A	A	--
	Linwood	Muscatine	8	78:2W	Moscow	C	--	C	--
	Coralville	Johnson	33	80:6	Iowa City	A	A	A	A
North-eastern Devonian	Linwood	Buchanan	2	88:9	Independence	C	--	C	--
	Littleton	Black Hawk	17	88:12	Millerdale	A	B	--	B
	Littleton	Fayette	29	93:8	Fayette	B	--	B	--
	Littleton	Chickasaw	16	95:14	Bassett	C	--	--	--
Northern Devonian	Coralville	Butler	25	93:17	Greene	C	--	A	--
	Coralville	Floyd	16	96:16	Floyd	B	A	A	A
	Mason City	Cerro Gordo	3	96:20	Mason City	B	A	--	A
	Mason City?	Worth	35	98:22	Fertile	B	--	--	--
Southern Mississippian	Burlington	Washington	20	76:8	Westchester	A	--	B	--
	Burlington	Louisa	33	73:2	Oakville	A	A	B	--
	Burlington	Des Moines	25	70:3	W. Burl'ton	A	A	B	--
	St. Louis	Lee	36	69:6	West Point	C	--	B	--
	St. Louis	Van Buren	25	70:11	Douds	A	A	A	A
	St. Genevieve	Wapello	16	72:14	Ottumwa	C	A	--	--
	St. Louis	Keokuk	23	76:12	Sigourney	A	--	--	--
Northern Mississippian	Hampton	Marshall	1	83:17	LeGrand	A	A	B	C
	Hampton	Marshall	21	84:17	Marshall'tn	A	A	B	B
	Gilmore City	Hardin	18	89:21	Alden	A	A	A	A
	Gilmore City	Webster	24	89:29	Fort Dodge	A	A	--	--
	St. Louis	Story	23	84:24	Ames	B	--	C	--
Eastern Pennsylvanian	Chariton?	Marion	21	74:21	Melcher	C	--	B	--
	Cooper Creek	Appanoose	21	70:19	Plano	B	--	--	--
	Winterset	Decatur	32	69:26	Decatur City	B	--	B	--
	Bethany Falls	Clarke	12	72:26	Osceola	C	--	C	--
	Bethany Falls	Madison	33	76:27	Winterset	B	C	C	C
	Bethany Falls	Madison	4	77:28	Earlham	B	B	C	C
	Deer Creek	Adair	12	76:31	Howe	C	--	C	--
W. Penn.	Deer Creek?	Cass (Neb.)	--	--:--	Weeping Water	A	A	--	--

The chemical analyses are from the Iowa Geological Survey<sup>(3)</sup> supplemented in some cases by information from other sources. In many instances the data is too meager to permit any rating.

<sup>(3)</sup> Vol. 24, pp. 659-664, 1913. Also unpublished data.

From natural weathering evidence a very arbitrary rating of three groups is made. The "A" group includes the cliff makers, and those in which natural exposures show little or no cracking or disintegration. The "C" group includes those formations which weather to a steep rock-strewn slope, with little or no exposure of rock in place. The "B" group is intermediate. In a few cases weathering evidence is not sufficient to permit any rating.

Many of the sources listed have little or no service record and no rating is attempted. Others have been used in highway cement concrete pavement, bridge structures, riprap, or masonry stone. Some which were accepted under earlier conceptions of soundness are now believed to be questionable. Consideration of all known evidence gives three very general ratings, "A" for good, "B" for questionable, and "C" for a few which appear to be bad. Any service record is a constantly growing and changing thing, and the temporary nature of the present rating must be emphasized.

#### *Comments on Ratings*

The first fact emerging from the foregoing rating table is that some of the stones examined are consistently satisfactory and that some are unsatisfactory in all lines of evidence. In many instances, however, the evidence is indefinite, confusing, or even contradictory. In comparing laboratory with field evidence, there are cases where the laboratory evidence is more favorable and other cases where the opposite is true. Some of these discrepancies may result from inaccuracies in sampling, but there still remain some cases where samples are believed to be accurately parallel. The whole assemblage of data indicates that while laboratory freeze and thaw evidence is fairly satisfactory in predicting field behavior, there are important individual instances where this is not so.

Two lines of evidence are rather seriously incomplete; the service record, and the chemical and mineralogical composition. Only time can complete the service record. There remains, however, a body of chemical and mineralogical analysis which might well be made. It would seem particularly desirable, along the lines suggested by Grim, Lamar, and Bradley, to examine the character as well as the amount of the clay minerals present in these stones. This examination will require apparatus and techniques not yet in general use and will call upon the services of the chemist and the physicist, as well as the geologist.

Another important line of evidence, insofar as durability of concrete is concerned, is a study of the interaction of the various concrete constituents upon each other. This can be and is now being done empirically in the Highway Commission laboratory by means of freeze and thaw tests and natural exposure on concrete specimens made up with a large number of material combinations. Progressive effects of the weathering processes are estimated at regular intervals from volume changes and reductions in strength. The latter determination is made by the sonic method in which the modulus of elasticity is computed from the period of vibration of a prism of definite size and shape. This permits repeated observations without destroying the specimen. The present series, when thus observed over a period of time, should yield a considerable body of valuable information.

More fundamental, however, is a further study of the basic properties of the materials which enter into concrete and of the reactions which take place between those materials. Such properties and reactions are already partly known from quantitative chemical analysis, but more knowledge of molecular structures and of molecular interactions such as base exchange is needed. This study may require methods quite different from those now in general use. The concrete analyst may have to follow the lead of the metallurgist in using polarized light or the X-ray on thin sections of concrete or its constituent materials. If this paper can encourage some scientist or group of scientists to pursue this line of investigation, its purpose will have been served, and its preparation well justified.

IOWA STATE HIGHWAY COMMISSION,  
AMES, IOWA.