REPORT OF COMMITTEE ON CORRELATION OF RESEARCH IN MINERAL AGGREGATES

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THE PRESENT STATUS OF THE SULFATE CRYSTALLIZATION TEST FOR SOUNDNESS OF AGGREGATES

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SYNOPSIS

During the past few years many testing organizations have given considerable attention to accelerated soundness tests for aggregates Some have concentrated upon the freezing and thawing test, which is thus far not standardized, but probably most have used either the sodium or the magnesium sulfate crystallization test The Committee has attempted to determine the present status of the latter type of test Letters were addressed to all State Highway Departments and recent literature was reviewed in an attempt to determine to what degree the test is considered useful and reliable

The report discusses the factors which influence the magnitude and uniformity of test results and shows that the test method is not adequately defined or explored With respect to the choice between the two salts, it is not indicated that the more consistent results which might be expected, from theoretical considerations, with magnesium is actually obtained

The study developed into a summary of the conflicting opinions and practices which exist with respect to the criterion of failure, the extent to which correlation with service behavior has been attained, and the general reliability of the test

The Committee feels that the test must be more thoroughly investigated if it is to serve a generally useful purpose, that the two salts, magnesium and sodium sulphates are about equally satisfactory, that the test should not be used for purposes of rejection unless, under well defined local conditions, positive correlation with service behavior has been established, that failures of aggregates in the soundness test should be regarded as a warning but that well authenticated records of service behavior should be given greater consideration in determining acceptability

Two types of test to determine the probable resistance to weathering of aggregates are in use The first is conducted by alternately freezing and thawing specimens of the aggregate which are immersed in water This is an attempt to simulate the natural conditions under which aggregates are most likely to deteriorate Although regarded favorably by most engineers, this test has certain disadvantages, chief of which are lack of standardization, the considerable number of exposure cycles required to produce deterioration even of aggregates of known poor characteristics, and, finally, the difficulty of describing, interpreting and correlating results

The second type of test is similar in conception to the freezing and thawing test, but solutions of salts are substituted for plain water A complete test is a number of exposure cycles each composed of a period of soaking in the salt solution, during which time the solution

is supposed to penetrate and saturate the aggregate particles, and a period of drying at an established temperature, when the salt is precipitated During the cycle the crystalline pressure produces stress in the same manner as does freezing water

As approximately equivalent degrees of deterioration are produced in considerably fewer cycles than with the freezing and thawing test, and as, moreover, it seems the easier to perform, there has been a marked tendency to apply the results of the sulfate crystallization soundness test in the enforcement of the indefinite "sound and durable" requirement of aggregate specifications Formerly chiefly used in connection with coarse aggregate, it is now often, although less frequently, applied to fine aggregates

Much study has been given the test, and it seemed that an appraisal of its present status might well be undertaken Letters to state highway departments and to other users and investigators brought interesting and instructive replies From these, in addition to the considerable volume of literature which has been developed, it seems possible to give a fairly definite picture of present attitudes and practices, to indicate some of the weaknesses of the test, and to draw a few conclusions

Use of the soundness tests These tests receive the most attention and are most used in those regions where freezing and thawing is experienced or other exposure conditions are severe and where aggregates of questionable durability are known to exist The southern states are but little interested and the same is true of those with more rigorous climates but which draw their aggregate supplies from sources of well known characteristics Elsewhere, and much more generally, the need of a basis for judging the durability of aggregates is recognized and usually finds expression in, at least, tentative adoption of the sulfate soundness test

Test procedures and effects of testing conditions Until the last few years sodium sulfate, either crystalline or anhydrous, was the only salt used to any considerable extent It still is the one principally used, but of late magnesium sulfate has been proposed and adopted by certain states The reasons for the substitution, with substantiating evidence, have been presented by Paul (1)[1] and by Garrity and Kriege (2) Comparing the two salts, it seems to be quite well established that a broader testing range is obtained by the use of magnesium sulfate and that extremely accurate control of temperature is less necessary than with the sodium salt Although it appears that the use of magnesium sulfate should produce greater differentiation between materials and closer duplication of results, these advantages have not always been obtained, and practical difficulties in the conduct of the test with magnesium sulfate are believed by some users to outweigh any theoretical preference to which that salt may be entitled Most investigators report that with the same number of test cycles the action of magnesium sulfate is more severe, but there is no fixed relation between test results obtained by the use of the two salts

The American Society for Testing Materials and the American Association of State Highway Officials have adopted methods of testing These methods are virtually the same and permit the use of either salt Each organization recommends that the test be considered as a guide in judging aggregates rather than as a positive basis for rejection

Various investigators have found uncertainties or deficiencies in the prescribed test methods Paul (1) points out that sodium sulfate exists in three crystalline forms within the temperature range of preparing and using the solution Garrity and Kriege (2) state that the amount of magnesium sulfate necessary

[1] Numbers in parentheses refer to list of references at end

to produce a condition of saturation is greater than that suggested These authors further clearly show the difficulty of securing and maintaining saturation, not only due to temperature changes but also to variations in concentration at various depths in the liquid layer It appears that saturation of a volume of solution requires a considerable period of stirring and can be maintained only by frequent agitation in the presence of a large excess of the salt

The same authors, as well as Walker and Proudley (3), call attention to the marked effect upon results of changes in concentration of the salt solution As the extent of variations is unpredictable, the necessity of maintaining the solution in the condition of saturation is fundamental and should receive the most careful attention

The time of drying to constant weight at the specified temperature between immersions and at the end of the test seems to affect results, according to Garrity and Kriege Another variable, investigated to a limited extent by Walker and Proudley, is the type of container which holds the specimen during immersion Different results were obtained by using pans, sieves and cheese cloth sacks They also found some effect from the use of salts of different degrees of purity Not yet thoroughly investigated are the effects of various methods of removing the salt solution from the aggregate and of various conditions of immersion, as, for instance, the possible difference in results obtained when specimens are immersed in a large body of the solution as compared to immersion in individual containers

It is evident that the effects of variables in the testing procedure are incompletely understood and that standardization of the test is likely to be difficult

Determination of degree of failure of specimens The prescribed methods of testing state that deterioration of coarse aggregate may occur as disintegration, splitting, cracking, flaking and crumbling Such effects may be observed visually but are difficult to describe in reporting Therefore, such statements of condition are supplemented by a determination of the percentage of the original sample which passes the sieve or screen upon which it originally was retained

The smaller sizes of fine aggregate prevent a thorough visual examination and the result of the test is expressed by the percentage of loss in the sieve test

Throughout the country opinion varies and practice differs regarding the degree and extent of deterioration which should be regarded as significant In connection with coarse aggregate, some engineers prefer to judge on the basis of a visual examination only Illinois, for instance, has not adopted a percentage loss standard for the evaluation of results In that state, slight chipping or spalling is ignored Specimens which break up badly or are reduced virtually to powder are considered to have failed Iowa considers disintegration of a particle to have occurred when its strength is impaired either on the surface or throughout the mass From the specification standpoint, a rock type is considered unsound if any particle disintegrates or if more than 20 per cent of the particles of that class are split Missouri considers an individual piece to have failed when it breaks into two pieces or loses one-third of its weight The degree of surface disintegration is also considered

On the other hand, California has adopted, in principle, the practice of the prescribed specifications Aggregates, both coarse and fine, are tested in a number of sizes and percentages of loss are determined at the end of the test However, sieve sizes somewhat smaller than those upon which the specimens were originally retained are used in the determination of loss

It is obvious that testing engineers are

not in accord regarding a criterion of soundness Since materials and conditions of exposure vary so widely over the country and, as indicated, the test itself is not adequately explored or defined, this state of affairs is not to be wondered at

Consistency of test results Many data are available which indicate that considerable variation in test results may be expected even in the same laboratory Walker and Proudley (3), on repeated tests of a single size of the same material extending over a period of several months, found that the average deviation from the mean was, on the No 8-4 size, 19 1 per cent with sodium sulfate and 16 7 per cent with magnesium sulfate Five cycle losses in the individual tests ranged from 10 to 25 per cent with sodium sulfate and 13 2 to 30 7 per cent with magnesium sulfate On the No 4—¾-in size the average deviation was 12 9 and 14 9 per cent with the sodium and magnesium salts respectively The five cycle losses ranged from 8 7 to 18 7 per cent with the former and 9 5 to 29 7 per cent with the latter salt Similar difficulties in reproducing results are reported by Garrity and Kriege (2) and by the Michigan State Highway Laboratory, which latter organization feels that the test has been found quite incapable of yielding reproducible results

Correlation with other properties of materials There have been many attempts to determine relationships between the sulfate soundness test results and the composition or physical properties of aggregates Kriege and Garrity (2) assembled data on 338 samples of sedimentary materials from widely different localities and found that no relationship existed between soundness and the properties of absorption, hardness, toughness and resistance to abrasion There was shown, however, a fair degree of agreement between the percentage of loss in the soundness test and the percentage of non-carbonate content (silica, alumina, iron oxide, etc) of the specimen For these sedimentary rocks, soundness decreased with the increase in non-carbonates L O Hanson, in an unpublished report on a considerable number of Wisconsin limestones, was able to show a general relationship between soundness and geological origin, but found no connection between soundness and other physical characteristics Paul (1) reports a general correlation between the absorption of fine aggregates and the results of the magnesium sulfate test, but no relationship between mortar strength and soundness of the aggregate

On the other hand, several states have observed that, in their localities, aggregates which are likely to give trouble from lack of durability are likely also to fail in physical tests, either abrasion or toughness It has been noted many times that deleterious materials which are structurally weak, soft, and highly absorptive, such as shales, loosely bonded sandstones and highly altered rocks, often will be found deficient in both the physical and the soundness tests In such cases of radically unsound materials, it is doubtless preferable to depend for protection upon some other than the sulfate soundness tests

There is a prevalent feeling that the sulfate crystallization tests do not duplicate natural exposure conditions closely enough and that a proper freezing and thawing test supplies more reliable data As previously stated, the latter test for aggregates is not standardized and usually requires a much longer time to perform However, some states depend primarily upon some form of the freezing and thawing test as the laboratory measure of soundness, as is the case in Kansas Some others apparently prefer the freezing and thawing test but resort to it only in case a material fails to meet the more convenient sulfate test This

is the case in Illinois, which finds that a coarse aggregate passing the sodium sulfate test is extremely unlikely to fail in freezing and thawing Iowa states that with its materials, except cherts, the A S T M freezing and thawing test for drain tile (C4-24) checks the 5-cycle sodium sulfate test, and the former is, in fact, specified as an alternate test for soundness Some Iowa cherts fail under freezing and thawing and are sound in the sodium sulfate test

Regardless of correlation with other tests, authorities agree that laboratory determinations of soundness by any method require judgment in interpretation Moreover, the weathering characteristics of the material as observed in the quarry or in service over a considerable period of time, are in most instances considered more reliable than the results of laboratory tests

Correlation with service behavior The degree of confidence with which the sulfate soundness test is regarded naturally depends upon the extent to which correlation with service behavior is believed to have been obtained

New York feels that the magnesium sulfate test is a reasonably satisfactory means of excluding sands, the unsoundness of which, it is believed, causes rapid disintegration of concrete pavements It is stated that sands showing a loss of less than 17 per cent have, in general, proved satisfactory That this figure cannot be applied indiscriminately is shown by the fact that in Michigan natural sands which show losses of 20 per cent or more have given apparently successful service

California uses the sulfate crystallization test where accelerated weathering may be anticipated due to the climate or to other conditions such as sea coast exposure In such cases definite percentages of loss are specified for both fine and coarse aggregates Either magnesium or sodium sulfate is used in the test and the same limiting percentage of loss applies to both salts Stanton states that in certain counties along the coast severe cracking of pavements and spalling of walls occurred in construction where aggregates were used which showed high soundness test losses Later studies indicate that some of these aggregates weather badly even though not showing excessive losses in the soundness test He concludes, therefore, that the test is unreliable in detecting all unsuitable aggregate, although the fact remains that the most extensive failures due to the weathering of aggregates have occurred when aggregates were used which fail in the sulfate test

Iowa, while agreeing that it is not infallible, feels considerable confidence in the sodium sulfate test for all of the aggregates, which they encounter, except chert A failed section of pavement is cited in which a coarse aggregate was used which contained some 20 per cent of unsound argillaceous limestone At the same time, two other pavements containing at least 20 per cent of coarse aggregate particles which fail to meet either the sulfate crystallization or the freezing and thawing test are 25 and 15 years old, respectively, and except for pitting in the former, are in good condition

Tennessee regards the sodium sulfate test as a valuable means of detecting potentially unsound shale in limestones Texas, although free from concrete failures due to unsound aggregates has found that certain limestones have proved unsound in open type asphaltic mixtures and as cover materials on the same types of construction In this state, as in Washington, the 5-cycle sodium sulfate test is regarded as unnecessarily severe in view of climatic conditions and the observed behavior of aggregates

Other states cite failures for which it appears that aggregates which gave poor results in the sulfate soundness tests are responsible

Hanson, in the study of Wisconsin limestones previously referred to, made tests on 38 deposits He also observed the resistance to weathering of exposed ledges and gathered some information regarding the behavior of aggregates prepared from them He states that the average losses suffered by the specimens under the three tests (magnesium sulfate, sodium sulfate, 100 cycles freezing and thawing) were in fair agreement with the weather resistance rating of the materials. However, he felt that a prediction of the resistance to weathering of any specimen could not be predicted definitely from the results of any one test.

In contrast with those organizations which employ the sulfate soundness test with at least some degree of assurance, there are many who assert that its present uncertainties render it quite unsatisfactory New Jersey feels that many aggregates, especially gravels, which have given good service in pavements would fall in the test Michigan believes it is useless seriously to attempt service correlation until testing inconsistencies are cleared up Ohio and others feel that the test is a source of confusion

In connection with attempts to tie up the results of the test with observations in the field, it may be that too close correlation is sometimes expected The soundness test is a test for durability under the conditions of the test Aggregates are used in unprotected situations such as traffic-bound roads, and with coatings of widely varying protective qualities, as in concrete and bituminous mixtures. Also the dimensions of the structures may affect the severity of exposure Further, the surface characteristics of the aggregate particles may influence the efficiency of protective coatings of bituminous materials or cement It is asking much of any one laboratory test to predict the reaction of an aggregate under any or all of these conditions of use

Thus, while correlation is desirable and should be determined when possible, it may be that it does not exist in any particular instance Strong emphasis should be placed on the preamble to the test methods of the A S T M and the A A S H O :

> It is recommended that the results obtained with this test method shall be considered only as a guide in the selection of aggregates It is not intended that the test shall be used as an arbitrary basis for rejection of the material without taking other factors into account, consideration should be given to the results obtained with the material when exposed to actual weathering conditions

CONCLUSIONS

From the foregoing digest of current attitudes and practice with respect to the sulfate soundness test, it is evident that there is no general agreement on the test method, the interpretation of results, the degree of correlation obtainable with the service behavior of aggregates, or, in fact the desirability of using the test Certain it is that those who feel that it serves a useful purpose will continue to use it, and those who find it lacks significance will not adopt it

The committee feels, however, that the evidence points to certain conclusions as follows.

1. The test method must be more thoroughly investigated and defined in order that more consistent results may be obtained and in order that the test may serve a more generally useful purpose
2. For sulfate soundness tests either sodium or magnesium sulphate appear to be about equally satisfactory. While the test made with the magnesium sulphate solution would appear to be easier to control than that made with the sodium sulphate solution, on account of the greater stability of the solution under variable temperatures, there is no clear evidence of greater concordance of results

3 Complete correlation of present soundness test results with service behavior of aggregates has not been attained

4. Even if refined to a greater degree than at present, it is doubtful if the test under consideration will constitute an entirely satisfactory measure of soundness The search for a better soundness test should be continued

5 The sulphate crystallization test should not be used for the purpose of rejecting aggregates unless, in connection with some local condition, it is very definitely established that the results of the test are significant

6 In all usual cases of failure in the soundness test, such results should be regarded as a warning that the source of supply of the material and the record in service should be investigated thoroughly

7 Service records of aggregates over a reasonable period of time under conditions similar to those to which it is proposed again to subject the material should be given greater weight than the results of soundness tests in determining acceptability

REFERENCES

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(3) Progress Report on Studies of Sodium and Magnesium Sulfate Soundness Tests, Stanton Walker and C E Proudley, Report of Committee C-9, *Proceedings* A S T M , Vol 36, p 327