

# STUDIES OF THE ACCELERATED SOUNDNESS TESTS

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## SYNOPSIS

The sodium and magnesium sulfate soundness tests have been accused of unreliability because of difficulties experienced in obtaining check results between several laboratories or within one laboratory using the same materials and test methods. This condition justifies a critical study of the test technique. Beginning with the theory of the test, the authors have given attention to the several steps in the preparation of the sulfate solutions and the specimens to be tested, and the care necessary to keep the solutions at their proper concentrations. New data are given for the dissolution rates of the hydrous and the anhydrous forms of both sulfates, together with a review of the literature dealing with the solubilities of these salts. Crystallographic data are also given to aid in the easy identification of the crystal forms which may appear under the conditions of the soundness test. Illustrations are given showing the observed crystals and thin rates of growth.

Such factors as time of drying, temperature variations, aggregate containers, etc., on the constancy of results are considered, with supporting evidence. Distilled water as an immersing fluid is compared with sodium and magnesium sulfates. The constancy of check determinations with materials from several sources is shown. The interpretation of the present test is discussed and certain changes are recommended.

The testing of fine and coarse mineral aggregates by the accelerated soundness methods is discussed frankly in view of field observations and other established facts. Several recommendations are made with the hope of fixing the status of the test. A bibliography is appended which includes the most important chemical references as well as the researches in the realm of the test engineers.

The literature on the accelerated weathering tests dates back over 100 years to the procedure employed by Brard (1)<sup>1</sup> in 1828 with subsequent modifications by D'Héricart and deThury. This early test consisted of boiling individual specimens in a saturated solution of sodium sulfate for one half hour in order to complete saturation, cooling them under fresh solution for several hours, and then suspending them by

<sup>1</sup> Numbers in parentheses refer to bibliography at end

threads for a twelve hour crystallization period in a dark room. Since that time many modifications and abrupt changes have been introduced. Today, the test procedure accepted tentatively by the A. S. T. M., A. A. S. H. O., and numerous other organizations consists essentially of immersing sieved fractions of fine or coarse aggregates in a saturated solution of sodium or magnesium sulfate for 18 hours at 70°F plus or minus 2°, and drying them to constant weight at approximately 105° for each cycle. Interpre-

tation is then based on the amount of material passing through the minimum sieve over which each size was originally prepared

#### STATUS

For quite a number of years engineers in the sanitary field have employed the sodium sulfate test as a predetermination of the service behavior of mineral aggregates in exposed uses like trickle and contact filter media. Such usage calls for a maximum of durability, since this aggregate is exposed to many cycles of freezing and thawing per day in winter. Other characteristics such as hardness, toughness and abrasion loss are of much less significance, since sewage filter media are not subjected to impact, crushing, rolling action or wear. All the aggregate is supposed to do in a sewage treatment bed is to *stay intact* and provide safe anchorage for the bacterial colonies which feed upon and convert the raw liquid sewage.

For such a purpose, the sodium sulfate soundness test certainly has a place. The test sample usually consists of 10 or 20 pieces of the size to be used as the filter medium. The concentration of the solution, period of immersion and of drying and temperatures maintained in the bath and the oven are all practically the same as found in A S T M. C-88-32 T and A A S H O T-75. A major difference is that the test is run for 20 cycles instead of the 5 now generally required for aggregates intended for highway construction. However, the interpretation is based upon the number of pieces broken into three or more parts or having lost more than a stated percentage of their weight as chips and flakes instead of a sieving separation. A significant investigation of the reliability of this soundness test for sanitary purposes was reported (2) in 1930. While

by no means constant results were obtained, it was found that the classification of the materials into sound, unsound and questionable was quite definitely accomplished, as shown by their weather resistance in the field. This is the whole purpose of soundness tests as conducted and interpreted by sanitary engineers.

It is not surprising that highway engineers, in seeking for some measure of durability and permanence, have rather hungrily grasped at accelerated soundness methods recently. We have not hesitated to make some drastic changes, such as imposing it upon aggregates whether intended for exposed uses or not, cutting down the number of cycles from twenty to five, including fine aggregates as well as coarse, and applying a mechanical analysis as a criterion of acceptance or rejection, instead of a visual examination which could have its counterpart in the bank or quarry face, and even going to such lengths as setting up arbitrary rejection limits for wide sweeps of territory without regard to values established by satisfactory service behavior over many years. The unfortunate thing is that we pressed to our bosom some friendly looking trick, pinning our faith upon it only to find that we knew little of its principles or purposes and that it had played us false. Thus, lately, comparative tests between several laboratories using the same materials and supposedly the same procedures have shown that too many irregularities appear in the check results to promote complacency regarding the accelerated soundness tests. This is particularly true of fine aggregate testing, to which this soundness test has been applied but recently. Since in any analytical work the value of a method primarily is its ability to reproduce results, it seems entirely proper to subject the

sodium and magnesium soundness procedures to a rather critical study, to determine in what phases of the methods now generally accepted there may lurk unsuspected dangers or causes for the variations in the test results that have become all too common of late.

#### THEORY

While many points involved in the accelerated weathering test are under dispute, it seems that the purpose or theory involved is commonly accepted as being the following. The 18-hour immersion of the dried specimen in a saturated solution of sodium or magnesium sulfate is presumed to fill the pores of the immersed substance with this liquid. During the drying portion of the cycle the moisture is supposed to be removed from the solution within the pores, leaving a deposit of anhydrous salt in the pore spaces and walls. The second immersion of the specimen in the solution brings fresh saturated solution in contact with the solid anhydrous salt deposited in the pore, thereby producing crystallization of the hydrated salt. Since this hydrated form occupies a larger volume than the dehydrated or anhydrous salt does, there follows an expansive action of the confined salt within the pores. This disrupting action is supposed to be of sufficient severity, that under the control of the test procedure, it serves as a measure of the resistance of the mineral aggregate to the natural weathering forces, particularly the expansive action of water freezing within its pores and fissures.

#### TEST ESSENTIALS

From even a preliminary consideration of the theory behind this test procedure, it is apparent that obtaining and main-

taining *saturation of the solution* is essential if crystal growth is to be promoted. The second important item is the complete penetration of the solution in an undiluted form into the innermost parts of the specimen. Third, since the temperature very largely determines the solubility of the salts involved, accurate temperature control of the immersing solution is required. Fourth, a drying period sufficient to dehydrate the absorbed solution is important. Fifth, it is highly desirable that the interpretation of the test results be on an easily understood and indisputably accurate basis.

While not every phase of the accelerated soundness test is covered in this paper, the following subjects will be treated:

- Preparation of solution, including rate of dissolution, quantities of salt used, effect of temperature, layering out tendencies (graphs and illustrations)
- Preparation of the sample
- Immersion period—time, temperature and action (illustrated)
- Drying period—length, temperature and action
- Cooling period—effect on filling pores in aggregates
- Kind of crystals formed—optical and specific gravity determinations (illustrated)
- Interpretation—sieve versus 1/2 minimum sieve versus visual methods, field observation of deposit faces and examples of finished structures, effect of size upon soundness
- Relation to other physical characteristics of abrasion, absorption, hardness, toughness, chemical composition and appearance
- Discussion
- Recommendations

## THE SOLUTION

The most widely accepted test methods permit the use of either the anhydrous or the hydrous sodium sulfate form, suggesting that about 500 g of the anhydrous or 1,000 g of the decahydrate be used for each liter of water at a temperature of 75 to 80°F. Furthermore, it is suggested that "the solution be thoroughly stirred during the addition of the salt and sufficient excess salt shall be added to insure saturation." The conditions for the preparation of the magnesium sulfate solution are similar with the exception that 325 g of the anhydrous form or 650 g of the crystalline form are required for each liter of water. Following this preparation the solution shall be allowed to cool to room temperature before use, which shall not be lower than 20°C (68°F).

A review of the literature reveals that the solubilities of the anhydrous and hydrous sodium and magnesium sulfates have not been frequently determined within that narrow range of temperatures in which our test procedure is confined, namely 68 to 72°F. There is also a lack of information regarding the *rates of dissolution* of these several salts and salt forms, as well as their effect upon the temperature of the resulting solution during the dissolving process. It therefore seemed desirable to determine the solubilities in this temperature range, the dissolution rates over a sufficient period of time to insure saturation, and to note the changes in temperature under certain conditions during this dissolving period.

The following method was found most applicable to the study of these dissolution rates: A 3-liter round bottom pyrex flask, as a solution chamber, was equipped with a mechanical stirring device of sufficient strength to keep all the suspension

in thorough agitation and the salt particles in complete distribution throughout the liquid. A constant temperature bath surrounded the flask, when this was desired. A measured quantity of distilled water, usually 1.5 liters, at 70°F plus or minus 0.1° was placed in the flask. The amount of salt (U S P) suggested in the A S T M and A A S H O methods for the preparation of the solutions was then added to the water in the flask under constant agitation and its temperature recorded at frequent intervals. At these same intervals samples were withdrawn from the solution so as to take a truly representative portion of both liquid and suspended solids at any instant. This was accomplished by means of a  $\frac{3}{8}$ -in diameter glass tube extending from within the flask to a Gooch crucible outside the flask which was attached to the suction pump. By quickly exhausting the system, a portion was drawn over within a second or two to the Gooch crucible, where the liquid was separated from the suspended salt particles by filtration through a dried asbestos mat. The specific gravity of the filtered solution was then immediately determined by means of a pycnometer. The concentration and percentage of saturation of the solution was thus determined from the specific gravity values at the temperatures desired.

An illustration of the apparatus used is shown in Figure 1.

The data obtained from these rate of dissolution determinations are shown in Figures 2 and 3. Starting with a temperature of 70°F, it is quite apparent that the temperature of the solution is widely different for the anhydrous or the hydrous form of the sodium salt. Thus, a temperature drop of some 13°F was observed for the decahydrate while a

temperature increase of 15°F occurred for the anhydrous form of the sodium sulfate under the conditions of the test

In the case of the anhydrous sodium sulfate the evolution of heat observed by the increase in tem-

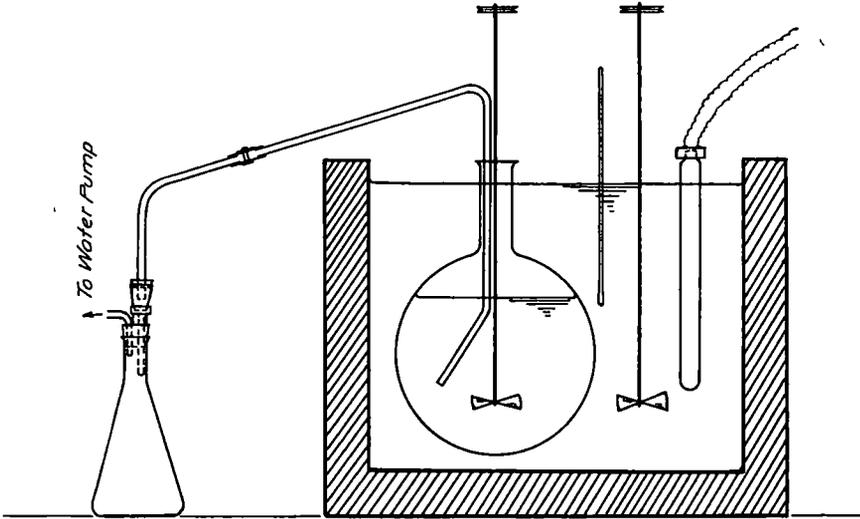


Figure 1. Rate of Dissolution Apparatus

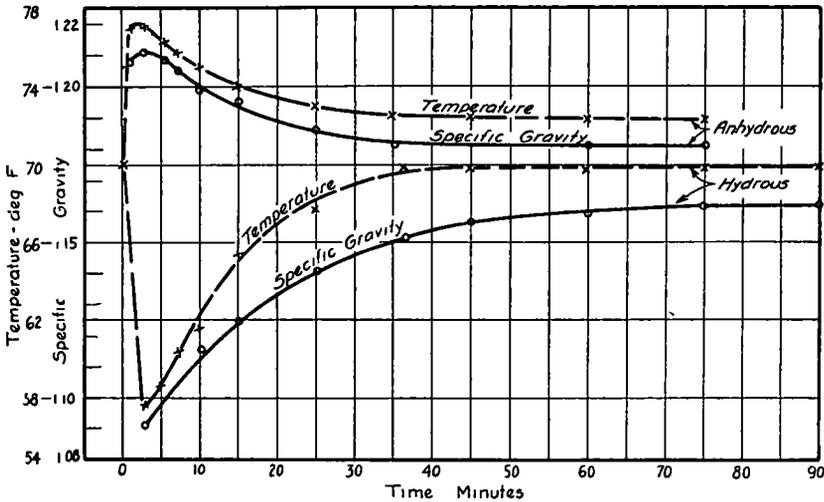


Figure 2. Rate of Dissolution of Sodium Sulphate

is quite apparent, also, that the curve representing the concentration (shown here as specific gravities) follows, with interesting regularity, the changes in the

perature is due to the rapid hydration of the salt. If the solution is not stirred, the change is readily observed to be that of an originally loose powder forming a prac-

tically solid cake of a well crystallized character. With thorough agitation, such as was maintained in the rate of dissolution study, this caking effect could not occur so that dissolution began almost instantly and continued undisturbed. It will be seen that within a very few minutes, three or less, practically complete saturation is obtained under these conditions.

In the case of the decahydrate crystals, however, the strong drop in temperature

is allowed to stand for some little time in the presence of excess crystals, complete saturation does not occur unless thorough agitation is maintained over a considerable period of time. After the temperature is held constant there is a definite increase in the specific gravity of the solution indicating further approach to saturation.

Figure 3 shows the dissolving properties of the heptahydrate magnesium sulfate ( $MgSO_4 \cdot 7H_2O$ ). Several facts are

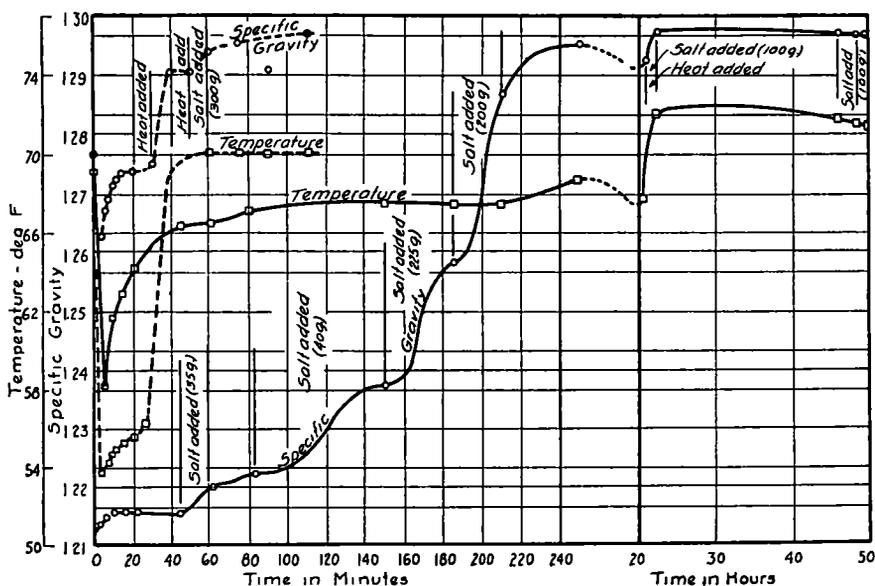


Figure 3. Rate of Dissolution of Magnesium Sulphate

is accompanied by a lower dissolving rate. The degree of saturation attained at these lower temperatures is not as great as that at 70° or more. We note here the dual effect of time and temperature on the dissolving tendencies. It is very easy to understand, therefore, why it is so difficult to obtain saturation of the solution as commonly made up for the soundness test if the decahydrate crystals of sodium sulfate are used. Even though the solution

noted: (1) the strong cooling effect, (2) the much lower effect of temperature on the amount of salt dissolved, (3) the requirement of more salt for saturation than recommended in the present test instructions. The successive additions of salt during the run are responsible for the steps and abrupt rises in the lower specific gravity curve.

An easy verification of this fact is had by the following test. Add to flasks con-

taining 100 cc portions of distilled water the indicated quantities of  $MgSO_4 \cdot 7H_2O$  (U. S. P.), shake for a few minutes, bring to  $70^\circ F$ , and check the specific gravities of the solutions. The results tabulated below are typical:

	Weight of $MgSO_4 \cdot 7H_2O$			
	65 g	75 g	85 g	100 g
Resultant Sp Gr at $70^\circ F$	1.2306	1.2527	1.2737	1.2969 (94 g)
Resultant Saturation	69.1%	79.8%	90.4%	100%

Each solution except the last is complete and clear. It is therefore evident that our present magnesium sulfate procedure is not at all correct in the amount of salt required for the preparation of the saturated solution. The stated specific gravity shown in the A. S. T. M. and A. A. S. H. O. procedures is likewise 0.007 below that found at the point of saturation.

*Layering Out Effect* In order to maintain saturation during the period of time involved in the soundness test, the usual suggestion has been to make sure that crystals of the salt are present in the container during every cycle. It is our firm belief that this is not sufficient to insure saturation as can be shown very readily from the accompanying data on the "layering out" effect of these solutions. The test conditions were the following: Four gallons of sodium sulfate were prepared in the usual manner, using  $Na_2SO_4 \cdot 10H_2O$ , allowed to stand 48 hours at  $75^\circ F$ , decanted into a container until the solution stood four inches deep. A pound of the decahydrate salt was then added. Portions of the solution were withdrawn from the upper half inch, the middle and the bottom half inch of the solution, filtered through dry asbestos and the specific gravities determined by the pycnometer method. The data are given in Table I.

From these data it is evident that not only is the dissolving effect of the decahy-

drate continued over a protracted period of days and weeks when no positive stirring is introduced but also that there is lag in the upper and central portions of the solution in acquiring the concentration of the lower portion in immediate

contact with the crystalline phase. This is true in spite of the periodic disturbance or slight stirring effect caused by the removal from or the insertion into the solution of the baskets twice a day. If we expect to maintain a condition of saturation within the immersing liquid, it is essential that ample agitation and the presence of a large excess of the crystalline phase be maintained. Mere quiescent

TABLE I

Age of Solution in days	Percentage of Theoretical Saturation at $69^\circ F$		
	Upper $\frac{1}{2}$ in	Middle	Bottom $\frac{1}{2}$ in
4	86.7	86.8	87.3
5	88.4	88.9	89.4
9	91.5	91.8	91.8
11	92.0	92.2	95.9
12	92.8	93.0	99.9
13	93.8	94.1	99.9
16	97.3	97.3	99.9
20	Entire solution stirred 40 min		99.9

contact with a few crystalline particles of the decahydrate is not sufficient to maintain 100 per cent saturation throughout its volume.

*Solubility Data* The relation between temperature and the concentration of sodium sulfate solutions has been given careful study by numerous chemists. There occurs in the vicinity of  $32.5^\circ C$  a transition point due to the change in the

solid phase ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ) to the anhydrous form  $\text{Na}_2\text{SO}_4$ . This point has been set at  $32.46^\circ$  by Matsui and Oguri, (3) and  $32.383^\circ \pm 0.001^\circ$  by T. W. Richards and R. C. Wells (4) and H. C. Dickinson and E. F. Muller (5). Several investigators have called attention to the rapid change in concentration with temperature

tion, and  $t$  = temperature in degrees centigrade. Then

$$X = 10.102 - 0.37145t + 0.0335t^2$$

$\text{MgSO}_4$  in the range  $0^\circ$ – $40^\circ\text{C}$ . Let  $X$  = grams  $\text{MgSO}_4$  per 100 grams of solution, and  $t$  = temperature in degrees centigrade. Then

$$X = 20.5726 + 0.29461t - 0.0006602t^2$$

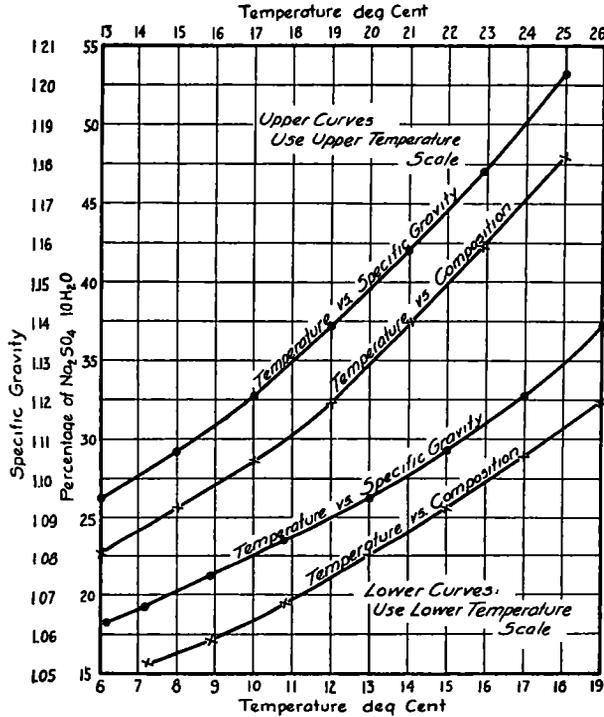


Figure 4. Solubility and Specific Gravity Curves for Saturated Solutions of Sodium Sulphate

below the transition stage of sodium sulfate and the slower change for magnesium sulfate, and have given us specific data covering these changes. A helpful summary of these facts is found in the work of Montillon and Badger (6) giving the following expressions for the concentrations of the two solutions to be the following:

$\text{Na}_2\text{SO}_4$  in the range  $20^\circ$ – $32^\circ\text{C}$ . Let  $X$  = grams  $\text{Na}_2\text{SO}_4$  per 100 grams of solu-

The density values of the sodium sulfate solutions are those by the Earl of Berkeley (7) and Dawson and Williams (8). Those for magnesium sulfate are given by Barnes and Scott (9) while the values for the solubility of this salt are found in Landolt-Bornstein Tabellen (10). Figure 4 is presented to refresh our memories on the characteristics of the solubility-specific gravity versus temperature relationships of sodium sulfate

solutions These data were obtained by following the concentration changes of a large quantity of solution saturated at above 90°F as it cooled to below 50°F, constant agitation being maintained Temperature and hydrometer readings were made periodically At the same time portions were pipetted off for chemical analysis in accordance with standard methods The values obtained were checked against the International Critical Tables and were found to be satisfactory.

Others, including one of the present authors (11) have shown that an increase in the destructive effect of crystallization of a salt within the pores of a specimen can be secured either by a forced drop in the temperature of the solution or by maintaining the temperature at a higher point than the customary 70°F Thus, we have within our power the means of making the accelerated test more severe than at present This thought will be given some amplification

To illustrate these conditions we have carried through the calculation of the amounts of salt deposited in the pores of a material during the sodium sulfate soundness test Assume an aggregate having 3 per cent absorption by weight in an 18-hour soaking period in water Using the values found for the saturation concentration of sodium sulfate at several temperatures as shown in Figure 4, we can calculate the deposition of salt within the pores of the aggregate in the manner shown in Table II.

This tabulation of the amounts of the two crystalline phases developing emphasizes several facts which have been more or less vague in previous considerations. In the first place, there is produced a four fold increase in the volume of the salt when a given weight of the anhydrous  $\text{Na}_2\text{SO}_4$  hydrates to the decahydrate

( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ) form This is due to the combined effect of more than doubling the weight and almost halving the specific gravity The second fact emphasized is the strong effect of temperature of the saturated solution upon the amount of crystallization taking place during successive cycles, even though that temperature is held constant A third fact is also apparent, namely, that with each cycle less of the original volume remains to be filled with the mother liquor There must also be an increase in the difficulty with which the pore is filled, since much of the anhydrous salt left after the drying period must line the entrance and outer portions of the pore or crevice The hydration of this salt will tend more or less effectually to block the further entrance of the solution

It should be stated that our present procedures are *not* conducive to complete filling of the pores of the immersed specimen We know full well from the usual absorption tests that the 5-hour boiling procedure gives higher values than the 24-hour soak at room temperatures Table III gives the comparison of a few 500 g portions of aggregates examined to determine the relative absorptive abilities under the conditions stated These tests are to be considered merely indicative

Another observation was made which seems worthy of statement in this connection Several coke bulbs, as formerly used for the determination of coke in creosote oils, were employed to represent that type of pore having a relatively small aperture for its volume These bulbs were carried through the usual sodium sulfate soundness procedure to follow the progress of crystallization visually and by increase in weight It was immediately noted that mere immersion of a bulb into

TABLE II

	15°C 59°F	20°C 68°F	25°C 77°F
a Temperature—Centigrade			
b Temperature—Fahrenheit			
c Specific gravity of saturated solution	1 107	1 148	1 203
d Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O in grams per cubic centimeter—saturated solution	0 2834 g	0 3984 g	0 5774 g
e Equivalent Na <sub>2</sub> SO <sub>4</sub> in grams per cc	0 1250 g	0 1757 g	0 2566 g
f Grams (or cc ) water in each cc of saturated solution	0 824 g	0 750 g	0 507 g
<i>1st cycle</i>			
g Weight of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O absorbed in 3 cc pore space (assuming same absorption as for water)	0 85 g	1 20 g	1 74 g
h Equivalent weight Na <sub>2</sub> SO <sub>4</sub> after oven drying	0 37 g	0 53 g	0 77 g
i Corresponding volume of Na <sub>2</sub> SO <sub>4</sub>	0 14 cc	0 20 cc	0 28 cc
j Original pore vol filled with Na <sub>2</sub> SO <sub>4</sub>	4 7%	6 7%	9 3%
<i>2nd cycle</i>			
k Weight of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O formed by hydrating the Na <sub>2</sub> SO <sub>4</sub> left from the 1st cycle drying period (assuming perfect hydration)	0 85 g	1 20 g	1 74 g
l Volume of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O formed by the hydration of the Na <sub>2</sub> SO <sub>4</sub> from the 1st cycle	0 57 cc	0 80 cc	1 17 cc
m Volume in cc of water drawn from remaining solution by the above hydration process	0 48 cc	0 67 cc	0 97 cc
n Weight of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O forced from solution by the above removal of water	0 165 g	0 358 g	1 107 g
o Vol of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O forced from solution by the above removal of water	0 11 cc	0 24 cc	0 74 cc
p Total vol of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O formed during the 2nd cycle (l) plus (o)	0 68 cc	1 04 cc	1 91 cc
q Percentage of original pore vol (3 cc ) now filled with Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O after immersion period	22 7%	34 7%	63 7%
r Percentage of original pore vol (3 cc ) now filled with Na <sub>2</sub> SO <sub>4</sub> after drying period	5 6%	8 5%	15 7%
<i>3rd cycle</i>			
s Weight of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O formed during the immersion period (3rd cycle) of the Na <sub>2</sub> SO <sub>4</sub> left from the drying period of second cycle	1 015 g	1 558 g	2 847 g
t Vol of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O formed by the hydration of the Na <sub>2</sub> SO <sub>4</sub> from the second cycle immersion period	0 68 cc	1 04 cc	1 91 cc
u Vol in cc of water drawn from remaining solution by the above hydration process	0 57 cc	0 87 cc	1 59 cc
v Weight of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O forced from solution by the removal of water for the above hydration	0 196 g	0 462 g	1 817 g
w Vol in cc of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O forced from solution by the above removal of hydration water	0 13 cc	0 31 cc	1 22 cc
x Total vol of Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O formed during the third cycle (t) plus (w)	0 81 cc	1 34 cc	3 13 cc
y Percentage of original pore vol (3 cc ) now filled with Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O during immersion period—third cycle	27 0%	44 7%	104 3%
z Percentage of original pore vol (3 cc ) now filled with Na <sub>2</sub> SO <sub>4</sub> after the drying period, assuming no loss of solid material	6 6%	11 0%	25 7%

the solution, both at room temperatures, did not fill the bulb. It was necessary to remove the air within it by some artificial means, such as boiling, in order to have anything like complete filling of the bulb. This indicates that the present process does not guarantee that the pores of our immersed specimens are filled.

TABLE III

Material	5-hour boiling with cooling to 70°F		18-hour soaking at 70°F	
	Water	Sat Sodium Sulfate Solution	Water	Sat Sodium Sulfate Solution
	%	%	%	%
Stone A	6.47	6.13	2.60	2.39
Stone B	1.69	2.84	1.55	1.20

Note the values for sodium sulfate have been corrected for volume of solution.

Upon drying the bulbs after artificial filling, it was noticed that the evaporation at the orifice caused salt to crystallize out, frequently blocking the neck of the bulb. Hence, with continued heating, very considerable losses of both solution and salt occurred as entrapped vapor from the contained solution freed itself with some violence. We have all noted the glazed appearance most aggregates assume after several cycles of the soundness test. Undoubtedly, most of the salt causing this glaze is expelled from within the pores either by vapor pressure during evaporation or by the intumescence of the salt itself. In any event, the difficulty experienced in filling pores of restricted opening and the losses of salt to the outside during the drying process both show that the present procedures for immersing and drying specimens do not permit anything like the desired destructive effect, based on the ideal crystallization conditions.

It was further noted visually and microscopically that not all of the anhydrous salt left within the pore hydrated when new solution was introduced, some inner portions being protected by the growth of crystals surrounding them.

Let us now turn to the consideration of another effect of temperature on the sodium sulfate soundness test. Previously we mentioned that any drop in temperature below that at which the solution was saturated adds crystals to the growth already begun within the pores of an immersed sample. While this statement may appear obviously correct to many, it will be worth while discussing the extent of this additional crystal growth and corresponding disrupting force. From the solubility chart, Figure 4, we get the following values in grams of salt per 100 cc of saturated solution.

Temperature, degrees		Na <sub>2</sub> SO <sub>4</sub> 10H <sub>2</sub> O (Decahydrate)	Difference per 5°C or 9°F	
Centigrade	Fahrenheit			
15	59	28.34 g	59-68°F	11.5 g
20	68	39.84 g	68-77°F	17.9 g
25	77	57.74 g		

Let us take again our specimen of 100 g weight, sp gr 2.55 and 3 per cent absorption. Suppose the immersing solution was saturated with respect to Na<sub>2</sub>SO<sub>4</sub> 10H<sub>2</sub>O at 77°F and was then allowed to cool to 68°F after the specimen was immersed in it. The decrease in solubility of Na<sub>2</sub>SO<sub>4</sub> 10H<sub>2</sub>O at 68°F instead of at 77°F would force out of solution 17.9 g of crystals per 100 cc. The solution within the pores of the specimen would undergo the same changes in temperature and hence in concentration. Thus, in the 3 cc. of absorbing pore volume 0.54 g of the salt will *crystallize due to temperature drop alone*. Comparing the effect of this

77–68°F. drop with the residual salt of the first immersion in a solution at 68°F., we find nearly half as much salt (0.54 g. vs. 1.20 g.) crystallizing, due to the temperature drop. Thus, the normal room temperature fluctuation of solutions saturated at higher temperatures may be nearly as destructive as the action of an additional cycle in which the immersing solution is saturated and kept at a lower constant temperature. These calculations should explain the observation that practically all operators of this test have made, namely, when working under uncontrolled temperature conditions that, after a considerable drop in temperature in the solution, much more distress is registered by the immersed specimens than if the temperature remains constant.

#### CRYSTALLIZATION PERIOD

The usually accepted soundness procedure calls for an 18-hour immersion period in the saturated solution. This time is assumed to be adequate to permit the growth of crystals within the pores of the immersed substance. Since no data could be found pertaining to the rate of crystal growth of these salts, observations were made with the aid of a microscope and camera. The conditions maintained for the observed crystal growth were the following: A small portion of the anhydrous salt was placed on a microscopic slide, placed on the stage, and brought into focus with low magnification (75 diameters). A drop of saturated solution of sodium sulfate was brought in contact with the anhydrous powder and immediately covered with a cover slip. It was noticed that the apparent white powdery anhydrous salt became clear and visibly crystalline the instant it was touched with the fluid, indicating that hydration had occurred. Very soon crystals of consider-

able size began to form at the numerous points of contact between salt and solution. When the conditions of temperature were maintained constant so that the heat from the illuminator used did not raise the temperature of the specimen above its original temperature, it was noted that the change in crystal size was quite rapid. Under certain conditions the field became "frozen over" within a few minutes, the size of the crystals

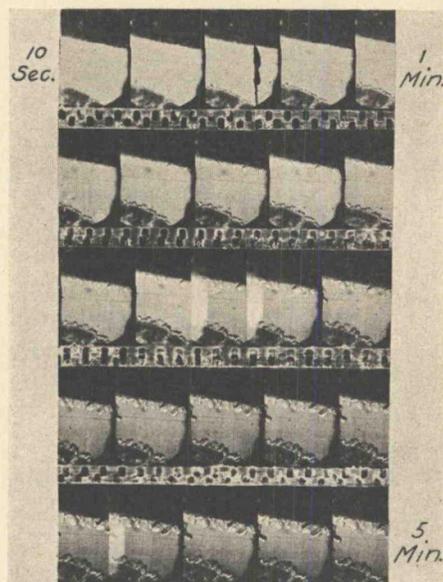


Figure 5. Microphotographs of the Rate of Crystal Growth of Sodium Sulfate

formed being determined by the distance between crystallization points, etc. The micro-photographs in Figure 5 indicate the kind of crystals formed and their rate of growth, the photographs being taken at 10 sec. intervals. The growth was complete in 10 min. Evidently we are allowing much more time than is necessary for the immersion period in our present procedures.

It is apparent that decahydrate crystals of obviously different shapes appear

simultaneously from the same drop of solution. This is by no means to be interpreted as indicating that the solid phase crystallizing out is different. Crystals of many shapes were examined microscopically immediately after their formation and were identified as the decahydrate salt. Furthermore, thirteen crystalline shapes produced from solutions of sodium sulfate by slow and rapid temperature drop, stirring, evaporation, in or out of contact with mineral particles, containers of several sorts, etc., were examined. Some of these crystals were 3 to 4 in in length, others were elongated to 30 times their width, while still others were thin and thick plates. However, the specific gravity and the ignition loss determinations on each specimen showed the same crystalline phase, namely  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ , to be the only one present at the temperatures observed, i.e., between  $10^\circ$ – $32^\circ\text{C}$ . These data are presented because there seems to be still a hesitancy on the part of some engineers to accept the fact long known by chemists that only one crystalline phase is in equilibrium with this saturated solution at these temperatures. An illustration of the difficulty of obtaining a super-saturated solution which alone can produce the heptahydrate, is given by Tantzov (12).

*Crystallographic Data.* It will probably be of some value to state the means of identifying the several crystal forms which may appear in the accelerated soundness test as commonly recommended. It is a recognized fact that the crystals exhibit characteristic optical properties when viewed microscopically with the aid of polarized light and various microscopic attachments such as the quartz wedge and the selenite plate.

In Table IV the optical and other characteristics of the crystal phases which

can occur under the conditions of the accelerated soundness test using magnesium sulfate or sodium sulfate are taken from the U S G S Bulletin 679 by Esper S Larson.

*Identification.* To anyone familiar with the optical properties of these minerals it becomes an easy matter to distinguish between them and to identify a crystalline phase under observation. In the case of the two sodium sulfate crystalline phases, a very simple and positive means of identification is the Becke line reaction. This test depends upon the fact that a crystal phase immersed in a liquid of different refractive index exhibits a fringe of light at its surface in contact with the liquid, when the field is in sharp focus. When the tube of the microscope is raised slightly to bring the field out of focus, this fringe of light moves in the direction of the more refractive substance. Thus, if the crystal has a higher refractive index than the liquid, this bordering line of light moves into the crystal as the tube is raised. If the liquid has the higher refractive index, the line of light moves into the liquid. Since the refractive index of thenardite, the anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) is 1.474 for beta while that for mirabilite, the decahydrate phase, is 1.396, and the refractive index of kerosene is 1.448, it is very easy to distinguish positively and quickly between these two or to identify either by the Becke line reaction, using kerosene as the immersing fluid.

In a similar way the two crystalline phases of magnesium sulfate can be identified with cloveoil, or valvolene or any good clean lubricating oil.

If a further identification of crystalline phases is desired, the extinction angles, optical figures with polarized light and

TABLE IV

	Mineral			
	Thenardite	Mirabilite	Kieserite	Epsomite
Composition	$\text{Na}_2\text{SO}_4$	$\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$	$\text{MgSO}_4 \cdot 1\text{H}_2\text{O}$	$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$
Group	Biaxial Pos	Biaxial Neg	Biaxial Pos	Biaxial Neg
Indices of Refraction				
alpha	1 464	1 394	1 523	1 433
gamma	1 485	1 398	1 586	1 461
beta	1 474	1 396	1 535	1 455
2 V	84°	76°	57°	52°
Dispersion	p > v weak	p < v	p > v mod	p < v weak
Optical Orientation	X = b Y = c	X = b or Y = b Z $\wedge$ c = 30° Disp Strong	Y = b Z $\wedge$ c = 76 5 Disp dist	X = a Y = b
System and Habit	Orthorhombic	Monoclinic near pyroxene	Monoclinic	Orthorhombic Elong C
Cleavage	(001) dist	(100) perf (001) (010) tr	(111) (113) perf (101) (012) (111) good	(010) very perf (011) less perf
Color	White Brown	White	White	White
Hardness	2 to 3	2	3 5	2
Specific Gravity	2 69	1 481	2 57	1 68
Remarks	Very sol in $\text{H}_2\text{O}$ F = 1 5 to 2	Sol in $\text{H}_2\text{O}$ F = 1 5 Effloresces rapidly	Slowly sol in $\text{H}_2\text{O}$ F = 2 to 3	Sol in $\text{H}_2\text{O}$ Tastes bitter and salty F = 1

other crystallographic characteristics may be employed

#### CONTAINERS

As long as the soundness test is being applied to coarse mineral aggregates such as are used in sewage disposal beds, the question of containers for the specimens

is not important, since the individual pieces can be handled as such. However, it is advantageous even with large aggregates to have some convenient means of transferring the samples from their bath to the oven for the drying period and back again. The most convenient form suggested thus far is a wire

basket, in size large enough to accommodate the entire fraction or sample under test and of a shape to fit most conveniently the container used for the immersing solution. The size of the mesh in the wire basket should be no more than one half the size of the smallest particle contained in the original sample. If any loss occurs due to breaking down of the aggregate which allows particles less than one half their original size to escape, such material could certainly be considered real loss.

When the soundness test was first applied to fine and coarse aggregates in concrete and other types of construction, the need of suitable containers became paramount. Judging from our own experience and the experiences of numerous other laboratory technicians, we believe that wire baskets are the most adaptable and most satisfactory form of containers. The advantages are three: first, the solution has ready access to all parts of the immersed sample, second, drying is greatly facilitated by the free circulation of air around and through the mass of particles contained in a wire basket and third, there is no need of transferring samples to and from solid walled containers or trying to dry samples in enclosed containers. We believe that these advantages are sufficient to justify the use of wire baskets wherever possible over any other type of container thus far suggested.

Just by way of illustration, it may be stated that several laboratories have tried to use cloth bags of more or less uncertain weave and have found that the crystallization and the effect upon the contained mineral particles is very greatly reduced by the surrounding cloth. The reasons for this are not altogether obvious nor is their discussion of prime

worth here. Any individuals who have attempted to use any container of solid walls and bottom have experienced difficulties either in separation of the solution from the particles or inadequate drying within reasonable periods of time.

It will be of interest to record the following observation. One of our laboratories used 400 cc Pyrex beakers as containers for the individual fine aggregate fractions, decanting the supernatant liquid and placing the beaker with its contents in the oven for the drying period. It was found with the magnesium sulfate tests that nearly all of the beakers broke during the second cycle, the walls being cut off just above the bottom, at the contact of the salt crust. This did not occur with sodium sulfate.

#### DRYING PERIOD OF THE CYCLE

At present the test methods call for either drying for 4 hours or drying to constant weight. Recently, in a comparison between several laboratories testing the same sands for soundness by the A S T M method, it was found that two laboratories used the 4 hour period while another dried the samples for 48 hours for each cycle. In view of the wide discrepancies in results obtained, two further investigations were conducted. In the first the samples were dried for 4 hours and weighed and then were dried for another hour and reweighed, to determine how nearly constant weight was obtained in the original four hour period. The results are given in Table V.

The beaker samples were stirred three times during the drying period, while those in the baskets were left untouched. These data show that with a properly maintained oven temperature and wire baskets, the drying period of four hours

is sufficient for fine aggregate testing. Had the beaker samples not been stirred their weight would not have been constant, due to the incrustated surfaces

In the second series, a direct comparison was made of the effect of four versus forty-eight hour drying periods. Two sands were run in duplicate, their losses in five cycles being

Sand	4 hour	48 hour
"O"	8 23	18 27
"G"	7 18	10 11

It appears that prolonged drying adds a destructive action apart from that of the solution. In order to throw some

In line with the destructive action produced by alternate immersion and excessive drying, samples of two natural sands, "G" and "N" and one stone sand "O" were prepared as outlined above and boiled for 8 hours in distilled water and dried for 48 hours. The samples were cooled to room temperature before each immersion. This treatment was carried on for five cycles and the results obtained are shown in Figure 6. It is interesting to note that the loss from this test is greater than that obtained by the sodium sulfate test in the case of sand "N".

TABLE V

	Determinations		
	75	70	79
Containers used	Wire baskets	Wire baskets	Beakers
When weighed	Immediately on removal	Cooling to room temperature	Cooling to room temperature
Per cent showing constant weight	20 0	30 0	53 2
Per cent showing some further loss (amount)	50 7 (0 04)	41 4 (0 04)	34 2 (0 08)
Per cent showing some further gain (amount)	29 3 (0 04)	29 6 (0 04)	12 6 (0 04)

Note: Oven temperature 105-110°C

light on this condition several more fine aggregates, both natural and stone sands, were subjected to the soundness test except that *distilled water* was used as the immersing fluid instead of saturated sodium sulfate and the drying periods were either four or forty-eight hours. The results follow.

Fine Aggregate	Hours in oven	Loss	Average
"O"	48	5 31	7 67
		9 16	
"O"	4	3 41	
"G"	48	10 78	10 11
		9 45	

*Check Results* The discussion thus far has dealt with the factors affecting the test procedure. It becomes pertinent to know from the results of check or multiple tests how various the values may be, in spite of observing all precautions recommended by the present tentative methods. The results of one recognized laboratory are given as an example in Table VI.

At another laboratory the same sand "O" showed an average magnesium sulfate loss of 16.15 per cent and a sodium sulfate loss of 13.22 per cent in four tests.

each, using supposedly identical procedures.

To state that irregularities like these are disconcerting is putting it mildly. Other laboratories that have run a sufficient number of repeat determinations have found similar disquieting variations in results. It might be thought that the irregularities are all due to the sodium sulfate and that magnesium sulfate is

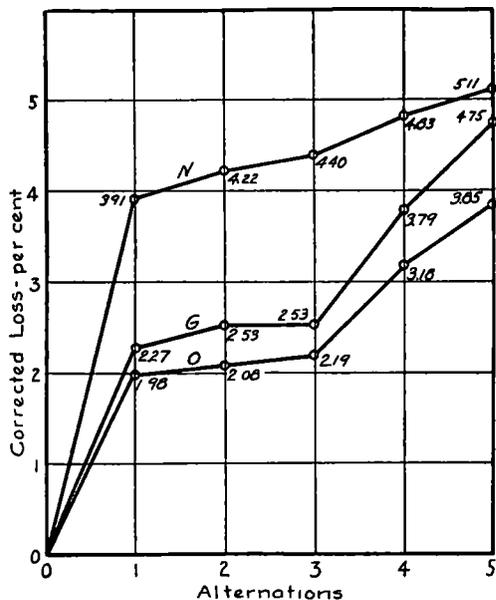


Figure 6. Fine Aggregate Breakdown. Sample prepared as per A. S. T. M. C-88-32 T. Fractions boiled 8 hours in distilled water, dried in oven for 48 hours; double boiler used.

free from theoretical and practical uncertainties. A few statements regarding this salt are therefore pertinent.

MAGNESIUM SULFATE

Lest there be any misunderstanding regarding the forms in which magnesium sulfate occurs with its several hydrates, a quotation from one of the outstanding chemical authorities, namely, J. W. Mellor (13) should be included here

TABLE VI

Fine Aggregate	No of Tests	Corrected Loss Average	Max Per cent Above Avg	Min Per cent Below Avg
Natural Sands—Sodium Sulfate (A S T M C-88-32T)				
A	2	4 13	1 45	1 45
B	2	3 21	16 5	16 5
C	3	7 83	20 4	12 8
D	4	8 22	11 8	22 8
E	2	9 63	16 7	16 7
F	2	6 72	44 0	44 0
G	8	7 18	50 0	57 2
H	2	7 79	28 5	28 5
I	4	7 25	15 2	10 7
J	3	8 99	9 3	14 2
K	4	6 98	5 7	4 4
L	5	9 35	29 4	33 0
M	2	9 36	4 8	4 8
N	3	3 57	30 5	25 5

Stone Sands—Sodium Sulfate (A S T M C-88-32T)				
O	10	8 23	97 6	45 0
P	3	5 53	31 6	43 8
Q	2	6 71	8 2	8 2

Natural and Stone Sands—Controlled Sodium Sulfate (Check of solution, strength, constantly stirred)				
A	4	6 83	9 3	14 2
G	4	11 88	9 9	11 9
O	3	19 40	15 5	22 0
P	2	6 30	1 3	1 3

Natural and Stone Sands—Magnesium Sulfate (A S T M C-88-32T but checked Sp Gr)				
O	1	7 84		
G	2	15 36	3 6	3 6
Q	2	13 00	9 4	9 4
A	2	4 93	5 7	5 7

“Hydrates of magnesium sulfates,  $MgSO_4 \cdot nH_2O$ , have been reported, with  $n = 1, 1.5, 2, 4, 5, 6, 7,$  and  $12$ . The hexa- and hepta-hydrates each have two different crystalline forms. The mineral

kieserite occurring in the Stassfurt salt beds, Hallstadt, etc., and named after M Kieser of Jena, is the native form of the monohydrated magnesium sulfate,  $MgSO_4 \cdot H_2O$  "

"Magnesium sulfate obtained by crystallization from aqueous solution is usually in the form of epsomite, i e., the heptahydrate, "

"According to G J Mulder the percentage of water which is lost after heating (the heptahydrate) to various temperatures is as follows.

	40°	60°	82°	101°	119°	172°	201°	Theoretical
Water lost in %	8.7	33.2	34.7	34.7	40.5	40.8	41.5	51.2
Mol equivalent	1 $H_2O$	3.5 $H_2O$	—	5 $H_2O$	—	—	6 $H_2O$	7 $H_2O$

"C Schmidt made the anhydrous sulfate by warming the heptahydrate to 200°C. H C F Schroeder found all the water is expelled from the hydrated salt in vacuo at 180°C "

From these data it is evident that in the magnesium sulfate soundness tests we are not dealing with the simple changes of the anhydrous to the heptahydrate and back again. In fact, the anhydrous form never appears in the properly conducted test, but rather the monohydrate, which is the phase lowest in water that may be attained under the test conditions. It is unfortunate that our test literature is in error on this point, and corrections should be made as promptly as possible.

The thought has often been expressed that with magnesium sulfate soundness testing we may do away with accurate temperature control, since the solubility remains constant regardless of the temperature. Using the expression of Montillon and Badger the values of the

solubility of  $MgSO_4$  in grams per 100 grams of solution are 26.65 g at 20°C and 30.02 g at 30°C. Even though there is much less change in concentration with change in temperature than in the case of sodium sulfate, it is still great enough to require positive control of temperature as well as every other phase of the procedure, if satisfactory duplication of results is to accrue.

One further thought should be recorded, which applies to both sodium and magnesium sulfate tests. Most operators dealing with some very unsound materials like ochres, shales and cherts, have seen destructive action begin in the second or third cycles, sometimes even in the first. That this is not due to the true crystallization phenomenon described in our earlier calculations is obvious, since not enough crystals can form to fill any pores and crevices. Perhaps entire filling is not necessary to cause disruption of the enclosing mass, but we have repeatedly observed microscopically that the crystals apparently followed the path of the least resistance and hence tended to follow the *length* of the containing pore tube rather than across its width. We have watched a single crystal elongate itself 4 or 5 mm down the length of a 1 mm bore glass tubing before any lateral growth was evident. This indicates that little or no pressure will be exerted against a containing pore until it is practically full of crystalline salt, since the crystals will tend to develop first in the direction of least restraint.

#### SOUNDNESS VERSUS CHARACTERISTICS

Many attempts have been made to coordinate soundness tests with some other physical or chemical property of the material. The relationships con-

sidered have been usually quite vague We are presenting the data gained from our experience with aggregates from many sources in the sedimentary deposits of the basin between the Appalachian and Rocky Mountains Some 338 samples are included on which soundness and other tests had been made It will be seen from Figure 7 that the only relationship apparent is with the total non-

not within the scope of this paper to give a complete bibliography and review of the investigations in this phase of testing, it would be unwise not to refer to some of the more interesting of the recent researches The work of Kessler (14, 15), McMillan (16), Gonnerman (17), Jackson (18), Paul (19) and Walker (20) are of particular worth either in their contribution to the test methods themselves

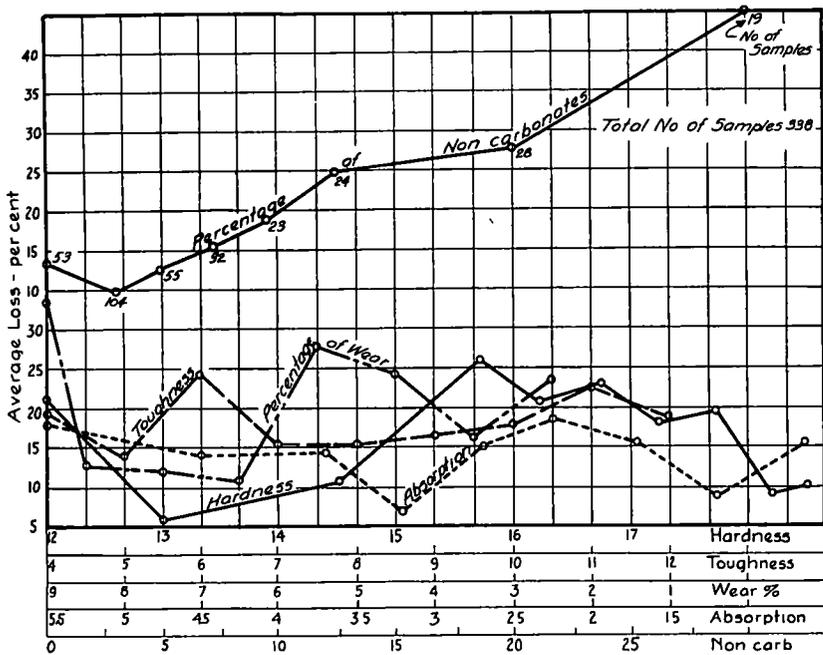


Figure 7. Relation Between Sodium Sulphate Test Losses and Certain Properties of Limestones 10 Cycles

carbonate content, namely, the silica, alumina, iron oxide, carbon, etc of the materials. As these impurities increase the unsoundness likewise increases with fair regularity The numerals at each point of the curve indicate the number of samples combined to establish that value

The literature on accelerated soundness tests and durability of materials has become quite voluminous While it is

or in their interpretation and attempted correlation with observed field behavior of materials

All careful operators of the soundness tests as now applied to aggregates must have observed that very often the material (called "loss") which passes the sieve originally used to size that fraction, is composed of particles or pieces which barely pass that sieve and are in no wise truly disintegrated Thus, for example,

sand fractions of 4-8 mesh size, which have passed through the test and are sieved again over the 8-mesh sieve, frequently show many coarse particles which are caught on the 10-mesh or even the 9-mesh sieve. To consider them as unsound is quite a stretch of the imagination.

Furthermore, if survival of the fittest means anything in the degradation of materials in Nature, it is reasonable to assume that the coarsest sand particles (4-8 and 8-16 mesh fractions) should be the most sound. It is, however, the usual thing to find these fractions showing decidedly the highest loss of the five taken for the test. Such unreasonable results tend to make the thoughtful operator suspicious of the test and its present interpretation.

#### INTERPRETATION OF TEST RESULTS

Any interpretation of the accelerated soundness tests must include, or at least admit, the evidence of exposed masses of the materials or known structures containing them, since the whole purpose of the tests is to determine the *weather resisting properties* of materials. To interpret failure on any other basis is a departure from the real purpose. If, then, our present method introduces a factor which measures friability, gradation or any other property apart from its weather resistance, our interpretation is in error.

Furthermore, it has been explicitly stated by numerous specifications that these tests are to be considered merely as guides in the selection of aggregates correlated with their known behavior in the field. This, in view of the added facts presented in this paper, should make it quite obvious that we are at the present time in no position to fix hard and fast numerical limits for the acceptance or

rejection of material based on the present test procedure. Further reason for hesitancy to affix specific limits, is that known deleterious materials, such as some cherts, will withstand these accelerated tests, but will cause concrete disintegration in service. A safer means of keeping such admittedly unsound materials as shales, cherts, iron bearing concretions, etc., from our aggregates is by the limits on deleterious substances based on visual observation and count. Most specifications now include such limits and hence we have some protection.

#### CONCLUSIONS

While admitting the desirability of establishing definite limits of acceptance for all characteristics of materials, our observations have forced us into the conclusion that the time has not yet come for this to be done with the present accelerated soundness test results. The present sodium sulfate procedure permits entirely too many uncertainties, even with refinements of technique not now required. The newer magnesium sulfate procedure contains serious errors, in concept and in practice, and furthermore lacks the correlation of known field behavior over periods of years without which test results can be of little practical value. It has not taken a great deal of effort to find mistakes in the theory and manipulation of our present methods and to indicate how they tend to vitiate results when applied to highway aggregates, both fine and coarse. We have shown that, in their present form, these procedures are unreliable. Both procedures and interpretation of control are necessary to make them reliable as a test for acceptance or rejection of material, and whether the care needed in this control is justifiable. We believe, therefore, that at present these

accelerated tests dare not be used for more than indicative purposes where field observations are impossible

#### RECOMMENDATIONS

Because of the difficulty of maintaining proper conditions of saturation, stirring and temperature control, it is suggested that the sodium sulfate test be dropped from highway procedures, except for *purely indicative purposes*

Magnesium sulfate offers a somewhat better chance for practical refinement and is usually more severe in its effect. For these reasons it is recommended in place of the sodium sulfate alternate, but with the reservations, since it requires the previously noted corrections and has very little background of experience and correlation with field behavior

Specific recommendations are:

1. Sufficient salt must be added to produce saturation at the temperatures desired (68–72°F.) The amounts now prescribed are inadequate

2. The proper salt ( $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ ) should be substituted for the anhydrous form ( $\text{MgSO}_4$ ) since the latter is not commonly available and is not formed in the drying period of the test cycle.

3. Mechanical stirring should be used not only in preparation of the original solution but in maintaining saturation in the solution during the tests. At least ten minutes of thorough agitation of the solution should precede the immersion of the test specimen.

4. The presence of considerable quantities of the solid salt is essential to maintain saturation

5. The concentration of the solution, both in preparation and in use, must not be taken for granted, but must be determined by means of chemical analysis or specific gravity determinations by hy-

drometer or preferably pycnometer measurements

6. No attempt should be made to set fixed limits for acceptance or rejection of material until a fund of knowledge has been built up, based on field experience of known weathering characteristics

7. Further study should be made to determine if the time for each cycle cannot be shortened, since it has been shown that the 18-hour period now given to immersion and crystallization process is much longer than necessary. Any saving of time over the six-day requirement at present would be very welcome

8. When fine aggregates are to be tested, it seems advisable to omit the fractions below the 50-mesh sieve, since results on them are highly difficult to obtain and are of questionable value

9. Whenever possible, wire mesh baskets should be used as containers, the size of the mesh being one half the smallest size of the fraction contained

10. The drying period should not be prolonged beyond that time in which the samples are brought to constant weight. Four to five hours time is ample with oven temperatures maintained at 105–110°C when wire mesh containers are used for the samples

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### DISCUSSION ON SOUNDNESS TESTS

MR IRA PAUL, *New York State Department of Public Works* The purity of the sodium sulphate salt is important in making the sodium sulphate soundness test on aggregates. The technical grade containing various impurities may introduce other variables which are likely to give erratic test results, and it is therefore important that only chemically pure salts either in anhydrous or crystalline form be used in making the solution to its proper degree of saturation.

In reference to using sodium sulphate solutions at higher temperatures than that called for in the A S T M Tentative Method of Tests, our laboratory has conducted such tests with saturated solutions at elevated temperatures. Contrary to our expectations we found that the average loss of the aggregate with the saturated sodium sulphate solution at higher temperature was less than that obtained under the temperature called for in the Tentative Method of Test.

The tentative method of testing aggregates with magnesium sulphate, adopted by the A A S H O and the A S T M is definite on the degree of saturation of the magnesium sulphate solution. The method states definitely that the specific gravity of the magnesium sulphate solution before using shall be not less than

1.29 at 25°C (77°F) as determined with a hydrometer. In our laboratory we are very careful in controlling the degree of saturation of this solution. A weekly check up is made for control purposes with a pycnometer.

PROF EDWARD E BAUER, *University of Illinois*, AND FRANCIS R GRANT<sup>1</sup>, *Research Engineer, The Barber Asphalt Company* There is one phase of the sodium sulphate procedure as outlined by the American Society for Testing Materials,<sup>2</sup> which seems to us to need greater detail in the instructions for the performance of the test. During the soaking period the A S T M specifications suggest that the sample be placed in the moist room used in the curing of portland cement concrete specimens, with only sufficient coverage to prevent extraneous material from getting into the container.

In making some tests not long ago in connection with our student work at the University of Illinois, we had this experience. A sample of rock, which was sent to the laboratory by one of the state high-

<sup>1</sup> Student assistant to Professor Bauer at the time the tests were made.

<sup>2</sup> Tentative Method of Test for Soundness of Fine Aggregate by Use of Sodium Sulfate, Designation C88-32T and the companion procedure for coarse aggregate, C89-32T.

way departments as a rock known to be unsound, showed no disintegration when the A S T M procedure of placing the samples in the moist room during the soaking period was followed. Knowing that the rock should have gone to pieces, we looked for a possible reason for this unexpected behavior. The idea that appealed to us most was that the sodium sulphate solution might be taking on moisture from the air and becoming less concentrated than when first placed in the moist room. In such a case, due to the decrease in concentration, sodium sulphate would remain in solution instead of forming crystals as is necessary if any disintegration is to take place. With this thought in mind the test was repeated the soaking being done in normal laboratory air. Unfortunately, the relative humidity of the air at that particular time was not determined but the tests were made during the winter months when the relative humidity is rather low. The samples of rock disintegrated considerably when the soaking was done in the drier laboratory air. In one of the samples, some disintegration was noted during the first soaking period.

It seems to us that if this test is to be of any value to engineers, it must be thoroughly standardized and that the relative humidity of the atmosphere in which the soaking is done must be very carefully controlled. It seems to us, further, that the relative humidity selected for this test should be such that the formation of the proper crystals will be encouraged in order that the number of cycles may be kept at a minimum. We feel that this detail in testing procedure may cause quite a variation in the results that are secured.

PROF H H SCOFIELD, *Cornell University*. Some time ago we were asked to

make sodium sulfate tests on sand from a deposit near Ithaca, New York. At that time we were interested enough to investigate the uniformity of our own test procedure and also to compare our results with those of tests on the same sand by other laboratories. We believe the results are discordant enough to be worthy of comment with the view of emphasizing the necessity for control of test procedure or possibly for the substitution of some other type of soundness test.

*Results of the Tests*

Sample Number	Laboratory	Total Weighted Loss (per cent)	Description of Sample
1	A	3.83	Sample taken by owner
2	A	4.54	Sample taken by owner, same pit
3	A	3.89	Check test on sample No. 2
4	B	10.5	Same pit, same location as No. 1 and 2 but taken by the laboratory
5	B	5.94	Check test on sample No. 4
6	C	9.9	One of four samples obtained from sample No. 2 by quartering
7	D	5.94	Sample same as No. 6
8	A	5.44	Sample same as No. 6

Note: Laboratory C used 18-hour drying periods.

In sample No. 8, Laboratory A used 24-hour drying periods.

In all of these tests, made by well known laboratories unusual care in procedure was taken. Five alternations of immersion in the sodium sulfate solution and drying were made. Identical sieves were used before and after. Solutions were made at proper temperatures and the temperature was regulated within the proper limits during test.

It is to be noted that in the case of laboratory B the check test result varied 43 per cent from the first test. In the case of laboratories A and C samples Nos 6 and 8, the longer time of drying seemed to produce higher losses.

One laboratory reports that they find it necessary at the end of the cycles to submit the sample to circulating hot water for 24 hours to be sure that no sodium sulfate is left in the sample.

The sieving operation of the several

group sizes is very sensitive. The particles that just fail to pass the square mesh sieve require only a very slight surface deterioration by the sodium sulfate to allow them to pass at the final sieving at the end of the test.

In view of the above results on account of the extreme sensitivity of the test, it seems to the writer that considerably more study of this test should be made before applying specifications so rigidly as is being done in some cases.