

It is concluded that in concrete of the proportions used in this investigation the effect of the oil coating on the stone was to reduce the strength. This conclusion applies to stone from one particular quarry and therefore has no general application but it suggests that oil coatings on coarse aggregate for concrete may be viewed with some suspicion until it has been shown by investigation that they are not harmful.

MAGNESIUM SULPHATE ACCELERATED SOUNDNESS TEST ON CONCRETE AGGREGATES

By IRA PAUL

Associate Laboratory Engineer, New York State Department Public Works

SYNOPSIS

Although several accelerated tests of aggregates have been devised to simulate weathering, no true laboratory measure of soundness has yet been evolved. The use of magnesium sulphate solution is proposed as a substitute for the commonly used sodium sulphate, because the solubility of the former only varies from 26.2 per cent to 29 per cent for temperatures from 68 to 86°F, whereas the solubility of sodium sulphate varies from 19.4 per cent to 40.8 per cent within the same range of temperature. Moreover the magnesium sulphate has only one crystalline form at these temperatures while sodium sulphate has three. A direct relationship was found between a freezing and thawing test and the magnesium sulphate test. The data show that the loss in a five cycle magnesium sulphate test was about twice the loss under a 60 cycle freezing and thawing test. The absorptive power of sand was also found to bear a general relation to the results of the soundness test. The results of the proposed test have been correlated with the service records of pavements in New York State. A number of concrete roads built between 1921 and 1931 with fine aggregate having a magnesium sulphate test loss of 20 per cent or more showed poor service records. On the other hand, concrete pavements in which the fine aggregate showed losses of 17 per cent or less in this test have good service records.

This paper is a discussion of the magnesium sulphate accelerated soundness test for the quality and durability of fine aggregates. I shall attempt to show how and why this salt test is superior to the sodium sulphate soundness test, what our laboratory results mean in the light of field service records obtained with these aggregates, and finally what the future specification on aggregates will be.

Several accelerated tests simulating weathering have been developed by various investigators, but no true laboratory measurement has been devised for determining the quality of concrete aggregates. Inorganic salts such as sodium chloride, calcium chloride, sodium nitrate, and sodium sulphate have been tried but each has had its shortcoming because of chemical activity or physical inactivity. As a result of our

investigation, magnesium sulphate was found to have certain desirable characteristics. In the first place this salt has an almost straight line solubility curve within the normal range of temperatures prevalent in the laboratory. Solubility of the magnesium sulphate varies from 26.2 to 29 per cent between 68 and 86°F while the solubility of sodium sulphate varies from 19.4 to 40.8 per cent within the same range of temperatures. Secondly, sodium sulphate assumes three different crystalline forms, whereas, the magnesium sulphate has only one crystalline form within the range of 68 and 86°F.

The method of making the magnesium sulphate soundness test on aggregates is the same as the sodium sulphate test with one exception, namely, that temperature control of the saturated solution of magnesium sulphate is unnecessary under average laboratory conditions. With the sodium sulphate, rigid temperature control must be maintained in order to secure the maximum effect of that salt on the aggregate. Elimination of temperature control, such as is possible with the magnesium sulphate is quite an important feature in a routine laboratory where a large number of samples are under test.

We now have record of over 300 complete tests on fine and coarse aggregates, obtained from commercial sources of supply in New York and neighboring states. These tests cover a very wide range of materials, including our best quartz, feldspar and limestone aggregates as well as the poorest aggregates containing shale, argillaceous materials, water lime and the like. Some very illuminating information has been uncovered by analysis of the large number of data collected.

The rational chemical analysis which we adopted about two years ago gives an idea of the mineralogical composition of the aggregate, but fails to show how firmly the mineral particles are held together. Minerals which in themselves are durable and sound, when held together with such a binding constituent as clay or kaolin, would prove to be non-durable. Neither can the presence of water lime in such aggregates be determined by a chemical analysis.

The sodium sulphate test has not correctly measured the soundness of aggregates, because a large number of aggregates which passed the test failed according to the field service records of the concrete in which they were used. On the other hand the magnesium sulphate soundness test used in New York has checked with the field service records of the materials.

In New York State we have practically every kind of aggregate known to the testing engineer. We have good quartz sands in the southeast end of the state, hard durable feldspar sands in the northern region and limestone bearing sands in the central portion, ranging from the better quality limestone to the poor quality containing water lime. In the southern tier of counties we have shaly, argillaceous and calcareous sands all of which are classified as poor to very bad. On materials from the

southeast region of the state, where the fine aggregates contain a high percentage of quartz, the magnesium sulphate results on the five cycle test range from 1.2 to 6.2 per cent loss. The sodium sulphate test on the same materials varies from 0.7 to 4.5 per cent loss. In the northern part of the state where the sands are composed of granites, gneisses, syenites, and durable feldspars, the magnesium sulphate results range from 4.5 to 17 per cent loss, while the corresponding sodium sulphate losses vary from 2.3 to 7.4 per cent.

In another section we find an entirely different picture which illustrates the superiority of the magnesium sulphate test over the sodium sulphate test. In this particular region several commercial sources of supply were examined by rational chemical analysis and found to contain argillaceous sandstone, calcareous sandstone and shale. The magnesium sulphate test losses were from 20 to 30 per cent, whereas the sodium sulphate test gave losses ranging from 2.9 to 7.6 per cent. In another locality the chemical examination of several sources of supply detected a considerable percentage of kaolin in the form of shale and shaly sandstone. The magnesium sulphate test losses ranged from 37.5 to 63 per cent and those by the five cycle sodium sulphate test ranged from 3.4 to 15 per cent. From the above evidence it can be seen that the magnesium sulphate scale, on the five cycle test, ranged between 1 and 63 as compared to the sodium sulphate scale with a range between 1 and 15. With such a scale of results as that given by the magnesium sulphate test, the engineer is in a better position to segregate the good sands from the poor or bad ones.

Before showing how the results of the magnesium sulphate test have checked up our field service records, other laboratory test data will be presented substantiating the reliability of the magnesium sulphate test.

Freezing and thawing tests were made on some of the same samples used in the sodium and magnesium sulphate tests. The method of testing the fine aggregates consisted in first freezing 100 grams of each of the separated sizes in a shallow pan of water, then thawing at laboratory room temperature. This procedure was repeated for 60 cycles with a sieving at the end of each ten cycles. Computations of the total loss were made in the same manner as in the sulphate tests.

The results indicate that there is a direct relationship between freezing and thawing and the magnesium sulphate soundness test. Very little or no relationship between freezing and thawing and the sodium sulphate soundness test was found. From the test data thus far gathered it was found that the loss on a fine aggregate after its subjection to 60 cycles of freezing and thawing was equivalent to about one half the loss from the five cycle magnesium sulphate test. A quartz sand with 6.2 per cent magnesium sulphate loss had a 2.8 per cent loss after 60 cycles of freezing and thawing. A chert bearing sand with a 20.7 per cent magnesium sulphate loss had a 9.8 per cent loss after 60 cycles of

freezing and thawing A sand containing argillaceous sandstone with a 44.4 per cent magnesium sulphate loss had a 21.6 per cent loss after 60 cycles of freezing and thawing The sodium sulphate loss of the latter sample was 7.7 per cent

Another test which is comparable to the magnesium sulphate test is the absorption test on fine aggregates A novel procedure was evolved for determining the absorptive power of sands Although the scale is somewhat limited, ranging only between 0.4 and 3.0 per cent, check results within 0.05 per cent are obtainable

The procedure for conducting the absorption test on fine aggregates is based upon a well known kerosene method Special apparatus consisting of a specially designed combination of a Le Chatelier flask and Babcock bottle was developed for this purpose This bottle was designed and calibrated so that direct readings are obtained The lower portion of the bottle has a volume of 57.5 cc The upper part has a bulge holding 15 cc and a tube of 5 cc The scale on the tube is divided into 100 parts so that each division is equivalent to 0.5 cc The bottle is charged with 50 grams of thoroughly washed and dried sand, and 20 cc of water white kerosene After standing for 24 hours, a saturated brine solution (NaCl) is added, and the bottle with its contents is centrifuged for 10 minutes at a speed of 600 R P M Additional brine solution is then added so that the lower kerosene meniscus is in line with the mark on the neck of the bottle The difference between the reading on a calibrated scale and 20 multiplied by 2 gives the percentage of absorption

The absorption of a fine aggregate which had a 7.9 per cent magnesium sulphate loss was 0.9 per cent, one with 20.7 per cent magnesium sulphate loss had 1.8 per cent absorption, another with 44.1 per cent magnesium sulphate loss had 2.3 per cent absorption, and so on As a general rule I have found that low magnesium sulphate losses show a correspondingly low absorption Sands having high absorptions usually have given higher magnesium sulphate losses and greater losses on freezing and thawing This is quite an important factor when we come to consider the selection of proper aggregates for concrete structures exposed to the elements During such time as moisture in the mortar is subjected to alternate freezing and thawing, disruptive forces are doing work which causes first the symptom of scaling and then the disease of disintegration The absorption of moisture by the sand particles probably causes volume changes of the particles and hence a volume change of the concrete itself Since changes in volume tend to disintegrate the concrete, if any unsound particles are present which may be attacked by natural physical forces, they are detrimental to the mortar

The failure of concrete in concrete highways in so far as materials were concerned was due to the disintegration of the mortar rather than to the coarse aggregate This disintegration manifested itself first in

the form of surface scaling, followed by progressive scaling in layers, and *D* lines. These *D* lines are lines of disintegration usually appearing on the edge of the concrete slab, and traveling toward the center. The mortar is one of the component parts of concrete which is least resistant to the weathering action of the elements. A direct relationship was found between the quality of the mortar and the durability of the concrete. Since the mortar is composed of fine aggregate and cement it is natural to believe that if the cement is of proper quality, the fine aggregate must also be of good quality in order to get maximum durability.



Figure 1. Lines of Disintegration in Concrete Slabs. Called "*D*" Lines

In all of my observations on the scaling of concrete I found that this phenomenon was usually accompanied by disintegration. The scaled areas on the slabs varied and some slabs were free from scale. There may be a number of contributing causes such as design, workmanship and sub-soil conditions, but in so far as materials are concerned this irregularity in the number of scaled slabs has been traced to the non-uniformity of the fine aggregate derived from the same source of supply. For example, from one of the questionable sources of supply three samples of sand were submitted for test. On analysis of the results

obtained on these three samples, some very illuminating information was uncovered by the rational chemical analysis, the magnesiums sulphate test and the absorption test.

Sample no.	Percentage of kaolin	Sodium sulfate test	Magnesium sulphate test	Absorption
A	8.06	4.0	15.6	1.6
B	13.63	4.2	29.1	2.0
C	12.98	3.3	24.5	1.7

It is readily seen that there is a wide variation in the quality of the material as indicated by the difference in the percentage of kaolin, and the losses on the magnesium sulphate test. The differences are not

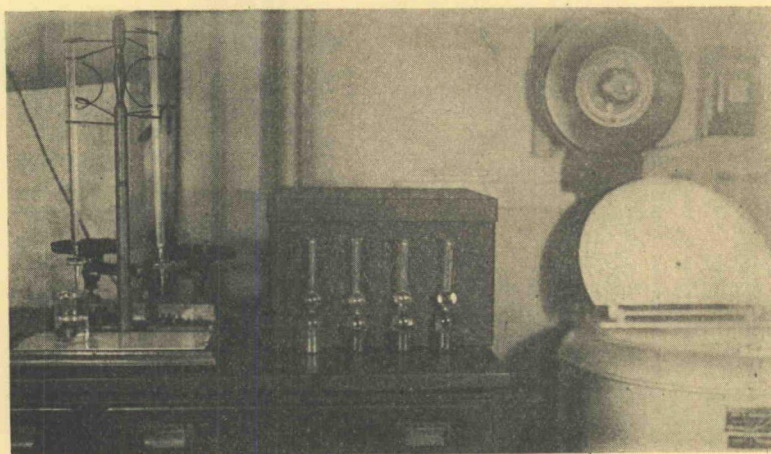


Figure 2. Apparatus for Determining Absorption of Sand

apparent with the sodium sulphate test. With the higher kaolin content we had correspondingly higher percentage losses by the magnesium sulphate test. This was in turn substantiated by the results obtained by the absorption test.

A close correlation between the results of the magnesium sulphate soundness test on aggregates and field performance was found. A poor service record was found for the great number of concrete roads built between 1921 and 1931 with fine aggregates having a magnesium sulphate loss of 20 per cent and over. For example, the fine aggregate from a very large commercial source of supply, which had a magnesium sulphate loss of 20.7 per cent and a corresponding loss of 6.8 per cent in the sodium sulphate test, had been used in the construction of over thirty concrete roads built from 1921 to 1931. Several of these roads built in 1923 had deteriorated so badly that they had to be surface treated and covered up in 1928. Others built in 1924 were completely covered in

1929, and so on. A fine aggregate from another commercial bank in a different section of the state, which had a magnesium sulphate loss of 20.2 per cent and a sodium sulphate loss of 4.7 per cent, has given a similar service record.

On the other hand, concrete highways constructed with those fine aggregates which showed magnesium sulphate losses below 17 per cent gave good service records. In New York State we have found that the character of the coarse aggregate has had very little or no effect on the durability of concrete. Where failures have occurred it has usually been traced to the quality of the mortar.

Concrete roads constructed in New York State with our present day cements and questionable aggregates have had an average life of about

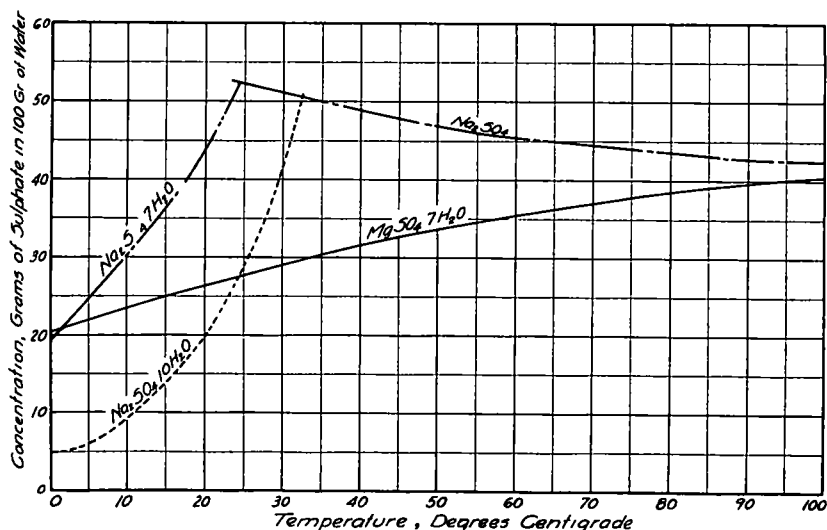


Figure 3. Solubility of Sodium and Magnesium Sulphates in Water

five or six years. With better workmanship the life of the concrete has been extended for another year or two. Those roads constructed with the better fine aggregates, having not more than 17 per cent magnesium sulphate loss, have stood up fairly well and given good service.

Figure 3 shows the solubility curves of sodium sulphate and magnesium sulphate at different temperatures. Between 68 and 86°F, the normal range of temperatures prevailing in the laboratory, the solubility of magnesium sulphate varies between 26.2 and 29 per cent, whereas, the solubility of the sodium sulphate varies between 19.4 and 40.8 per cent. These curves show why temperature control is unnecessary with the magnesium sulphate solution and how essential it is with sodium sulphate.

Figure 4 shows the sodium sulphate and magnesium sulphate losses on the separated sizes, at the end of a five cycle test. The abscissas

represent the sizes tested, and the ordinates the percentages of loss. The differences in loss on the separated sizes are not so pronounced in sample No. 1 as they are in the other samples. No. 1 is a quartz sand, No. 4 a feldspar, No. 10 a sandstone containing dolomitic limestone, No. 14 a quartz sand containing some argillaceous materials. No. 16 and No. 23 contain argillaceous sandstones. No. 27 and No. 29 contain shale and shaly sandstone. There is very little or no relationship between these two salt tests. The differences are not so pronounced with the better quality sands as with those of inferior quality.

Figure 5 illustrates graphically our laboratory results on some typical fine aggregates taken from different sections of the state. It will be noted that up to a certain point the small losses of the sodium sulphate

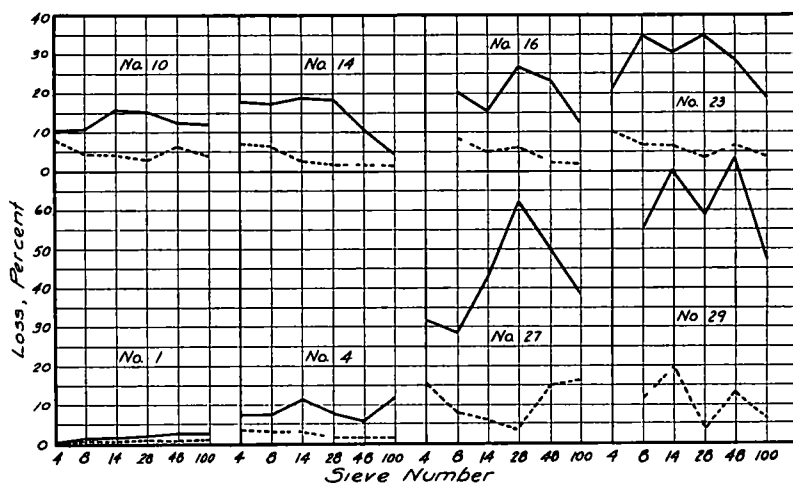


Figure 4. Comparison of Magnesium Sulphate and Sodium Sulphate Losses on Separate Sieve Sizes. Five Cycle Test

Magnesium sulphate———, sodium sulphate-----

test are about as significant as those of the magnesium sulphate test. However, as the losses become greater the results of the sodium sulphate test are not nearly so significant or pronounced as those of the magnesium sulphate test.

The horizontal line at 8 per cent on the sodium sulphate graph represents our 1932 specification limit. As the result of a thorough investigation of field service records, a tentative limit of 17 per cent has been set for the magnesium sulphate test. Out of 15 fine aggregates which failed to pass the magnesium sulphate tentative limit of 17 per cent, only 5 failed to pass the sodium sulphate test. The fine aggregates Nos. 1 to 15 inclusive used in concrete have all given good service records, while Nos. 15 to 29 have failed to render satisfactory service.

The graph showing freezing and thawing results is represented by dash

and dot lines Here again you will note the close relationship between that test and the magnesium sulphate test As intimated above, the data thus far obtained on fine aggregates show that the results obtained on the 60 cycles of freezing and thawing is equivalent to about one half the loss of the 5 cycle magnesium sulphate test Very little or no relationship exists between the freezing and thawing test and the sodium

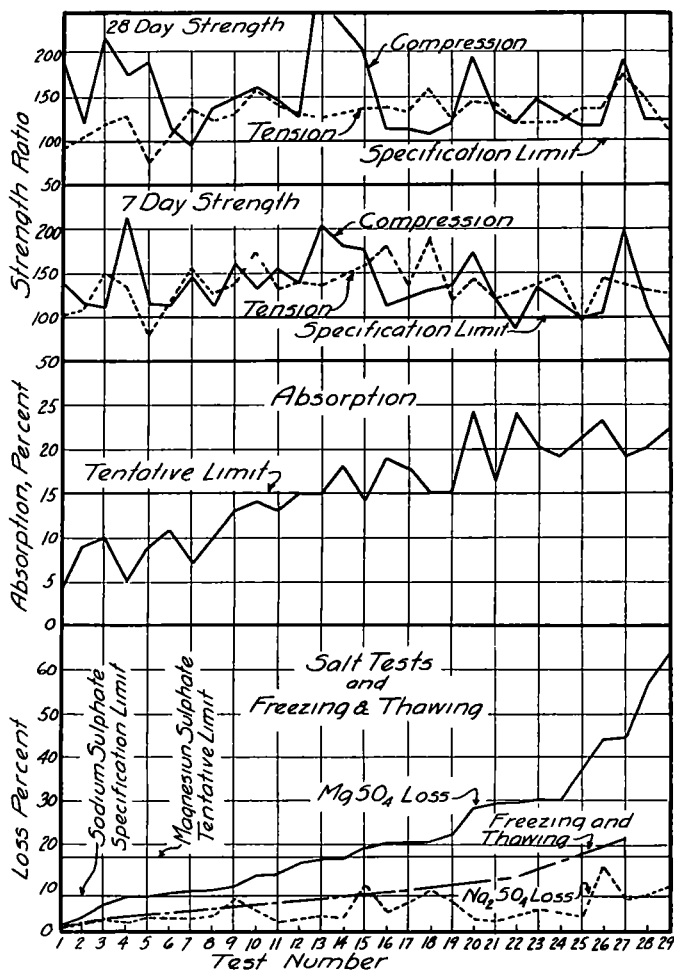


Figure 5

sulphate results Sand No 3 with a magnesium sulphate loss of 6.2 per cent had a 28.2 per cent freezing and thawing loss The sodium sulphate figure on that sand was 2.6 per cent Sand No 17 with a magnesium sulphate loss of 20.7 per cent and a corresponding sodium sulphate loss of 6.9 per cent had a freezing and thawing loss of 9.7 per cent, sand No 27 with a magnesium sulphate loss of 44.4 per cent and a

sodium sulphate loss of 7.7 per cent had 21.58 per cent loss on freezing and thawing

The graph representing the absorption test follows the same trend as the magnesium sulphate results. With an increase of loss in this salt test there is a general increase in the absorption. The tentative limit placed on the basis of this test is 1.5 per cent. This limit seems to be comparable to the 17 per cent specification limit of the magnesium sulphate test.

Above the absorption graph are plotted the strength ratio results on mortars. At first glance we see that there is no relationship between these strength tests and the accelerated durability test, freezing and thawing or absorption. The investigation disclosed the fact that we can get just as high strengths in mortars and concretes with shale sands as with quartz, feldspar and limestone sands. In fact some of our excellent quartz sands show lower strength ratios than shaly sands. Actual tests on concrete cores taken from the roadway slabs seem to corroborate this evidence. Other things being equal the strength of mortar or concrete is dependent upon the gradation of the aggregate. Any sands meeting our specification requirements for gradation, loam and silt, and organic impurities will pass the strength ratio requirement when compared with the Ottawa Standard.

CONCLUSION

I believe the future specification for fine aggregates for concrete should include the following:

- 1 Suitable and adequate requirements for gradation, silt, loam and organic impurities
- 2 A requirement that when subjected to five alternations of a saturated solution of magnesium sulphate, it shall lose not more than 17 per cent by weight
- 3 Mortar strength ratio tests need only be made when the fine aggregates has an unsatisfactory color in the test for organic material

DISCUSSION

ON

SOUNDNESS TESTS OF AGGREGATES

MR. M. H. ULMAN, *Pennsylvania Department of Highways*. We have also been unable in the Pennsylvania Department of Highways Laboratory to obtain any relationship between the results of the freezing and thawing and the sodium sulphate tests, in which there were used up to 100 cycles of freezing and thawing compared to five cycles in sodium sulphate.

Our freezing and thawing tests covered short and long alternations

of 50, 70 and 100 cycles The short cycles of freezing and thawing consisted of two hours in the cold room at a temperature of -10°F and two hours in the fog room at a temperature of 70°F and 100 per cent humidity The long cycles consisted of 24 hours in both the cold room and the fog room.

We have found in our studies that failures in concrete can also be attributed to coarse aggregates, when the latter are of unsound quality as determined by examination of the ledges and results obtained in the sodium sulphate soundness test

A concrete pavement was constructed a few years ago under the jurisdiction of the Pennsylvania Department of Highways, in which coarse aggregate was used which met the specification requirements for wear and toughness An examination of the ledge was made during the course of the construction and it was found that considerable disintegration had occurred A further study of the aggregate by the sodium sulphate soundness test demonstrated the unsound condition of the rock The use of this type stone was immediately discontinued and imported stone used The section of pavement in which the unsound coarse aggregate was used had to be replaced within a short time, while the section containing the imported stone is still in good condition In both sections the same fine aggregate, cement and water content were used

Another factor, which would lead one to be suspicious of this unsound coarse aggregate was its comparatively higher absorption compared to somewhat similar type stone This would seem to confirm Mr Paul's findings that there is a relationship between the absorption and soundness

I note with interest that Mr Paul has developed an apparatus, which he states will accurately determine the absorptive qualities of fine aggregate grains, for this has been very much needed as it is difficult to obtain accurate results with the apparatus so far recommended Check tests made with the present apparatus have also been erratic

We do not feel that at this time it would be advisable to discontinue the strength tests on fine aggregate if low colorimetric values are obtained There are sands occurring in our state, which furnish low colorimetric values, although low strengths will be obtained due to the grains not having structural strength, or in other words being of somewhat friable quality These sands would in most cases comply with the requirements of the soundness test Our present method of determining the strength of sand by compression as compared with standard Ottawa sand is not the most desirable test procedure and there is a field for considerable research covering the development of some satisfactory method Plastic or fluid mortar methods for determining the strength of sands will in many cases admit sands of fine grading, which would be unsatisfactory for use in concrete pavements

There is another factor, which should be taken into consideration for the country as a whole in interpreting the results of any soundness test on fine aggregate. The sand may fail to pass this test and would probably furnish unsatisfactory results in concrete if extremes of temperature were obtained during the early hardening period of the concrete. These same sands, however, may furnish satisfactory results in concrete and would probably be economic to use if it would be costly to import a more structurally sound sand in case where extremes of temperature would not be encountered at the time and subsequent to construction.

We have used some sands in our construction, prior to the advent of soundness determinations, which would not fully comply with the requirements of the sodium sulphate method of test, and the pavements in which they have been used are in good condition. It is probable, however, that extreme cycles of temperature changes were not encountered at the time of use or immediately following.

MR. PAUL: There is another important factor in the durability of concrete, that I would like to mention. This is the constitution of the cement. Strength has been over emphasized as a measure of the quality of concrete. With the introduction of the high lime cements, such as are now produced the strength of concrete has been increased. At the same time a greater percentage of some undesirable constituents or compounds have been incorporated in the cements. High percentages of tri-calcium silicate and tri-calcium aluminate have been the cause of some of our difficulties. It is true that a large percentage of the desirable constituents, namely, dicalcium silicate and tetra-calcium aluminate ferrite have not given us early strength, but these compounds have contributed in the long run, to the durability of the concrete structures. I suggest, that not too much emphasis be placed on the strength tests as a measure of durability of concrete, but that cement be examined through its chemical composition.