

**QUANTITY AND DISTRIBUTION OF CALCIUM CHLORIDE  
WITHIN STOCKPILES OF TREATED AGGREGATE**

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**Michigan State Highway Department  
John C. Mackie, Commissioner  
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## QUANTITY AND DISTRIBUTION OF CALCIUM CHLORIDE WITHIN STOCKPILES OF TREATED AGGREGATE

This report presents the results of field studies made to determine the amount and distribution of calcium chloride available in stockpiles of treated aggregates. More than a dozen locations were sampled in order that the effects of several variables could be studied. The more important of these variables are: climatic conditions, type of chloride used, age of stockpile, and mixing techniques. The stockpile tests were supplemented by studies of the aggregate-chloride mixture as placed on the road.

Results of this project indicate that: 1) there is considerable loss of chloride from treated stockpiles due to weather, exposure, and seepage; 2) the distribution of chloride within a stockpile is not uniform; and 3) such conditions influence the characteristics of the aggregate as placed on the road.

Research Project 59 E-19, "Concentration and Distribution of Calcium Chloride Within Stockpiles of Treated Aggregates," was initiated in July 1959 at the request of R. L. Greenman, Assistant Testing and Research Engineer. The primary purpose of this project was to determine the efficiency and effectiveness of the present methods used for treating and storing graded aggregates for highway base and shoulder construction.

MSHD Specifications require that properly prepared aggregates be treated with the equivalent of 6 lb of Type 1 calcium chloride per ton of prepared aggregate. By ASTM Standards (Designation: D98) Type 1 calcium chloride must be in flake form and contain a minimum of 77 percent absolute calcium chloride. Where other forms of calcium chloride are used the actual calcium chloride content must be converted to the Type 1 flake equivalent. Other forms of calcium chloride used in Michigan are: Type 2 (pellet or flake) containing a minimum of 94 percent calcium chloride, and calcium chloride solution of a concentration generally ranging from 32 to 38 percent chloride content depending upon temperature conditions. The form of calcium chloride used and the method of adding to the aggregate is controlled, primarily, by the individual contractor.

After treatment with chloride the aggregate is stockpiled in accordance with MSHD standard specifications. After a period of storage, aggregate is withdrawn from a stockpile as construction schedules warrant. The age of a stockpile, therefore, can vary from several weeks to possibly a year or more.

Because the primary purpose of using calcium chloride with aggregates is to provide uniform moisture control of the material during compaction on the road, any loss or non-uniformity of treatment could result in more harm than good to the finished base course. Therefore, for both engineering and economical reasons it was felt that this project should answer the following questions:

1. How uniformly is the calcium chloride added to the aggregate and how is this uniformity affected by storage?
2. How much of the chloride is available for construction purposes after prolonged storage?
3. Is the form of chloride a factor in uniformity and permanence of treatment?

In addition to testing the stockpiles directly, plans were made for observing and testing the treated aggregate during placing and after compaction on the road.

#### METHODS FOR SAMPLING AND TESTING STOCKPILES

Representative stockpiles were selected for study at various geographical locations throughout the state and included those treated with different forms of calcium chloride. All stockpiles investigated were composed of either 22A or 23A graded aggregate treated with an equivalent of 6 lb of Type 1 calcium chloride per ton of aggregate. Available background information for each stockpile was obtained.

The stockpiles studied fell into two basic shape categories, stepped or rounded, as shown in Fig. 1. Different methods of surface sampling were used for the two shapes. When layers could be clearly defined, samples were obtained at different elevations within the layer. Where no clearly cut dividing lines were visible, the stockpiles were sampled at 3 to 4 ft intervals of elevation.



Figure 1. Basic stockpile shapes: stepped (top) and rounded (bottom).

Surface sampling was accomplished by digging into the stockpile with a shovel as far as possible without caving in the excavation. Usually 2 to 3 ft was the limit of depth that could be cut into the side of a stockpile (Fig. 2).



Figure 2. Surface sampling of a stockpile.

Where loading equipment was available samples were taken at different elevations of the cut face of the stockpile. Average mixtures were obtained by sampling the mixed aggregate as loaded for hauling to the job site.

In all cases samples were taken from areas which, by their color, showed variation in chloride content, and particularly from the seepage areas usually visible along the base of stockpiles.

Several pounds of aggregate were taken at each sampling point. From these samples representative quantities were removed for individual analysis in the laboratory.

The quantity of calcium chloride in selected samples was obtained by the procedure given in ASTM Designation 0-1411-56T. Basically, this test requires: 1) removing soluble chlorides from a representative portion of the aggregate sample by agitation for 16 hours in a specified solution of ferric ammonium sulfate, 2) measuring, by titration, the quantity of soluble chlorides leached from the sample, and 3) computing the calcium chloride content and converting to flake equivalent pounds per ton of aggregate. This analysis is time consuming and quite sensitive but was generally satisfactory if carefully controlled and checked by duplicate tests. The method was checked periodically by testing control samples treated with a known quantity of calcium chloride. Very close check results were obtained.

The chemical analyses of these samples were made by J. D. Shackelford. Considerable help and direction in this work was given by W. L. Frederick and R. E. McComb of the Research Laboratory Division's Spectroscopy and Photometry Section.

#### RESULTS OF STOCKPILE TESTING

The field study of aggregate stockpiles was subject to several variables, some of which could not be controlled during this program. Weather conditions, length of storage, and method of adding the chloride were important factors affecting direct comparison of the different stockpiles.

More than a dozen stockpiles were sampled during this program, some in much more detail than others. The amount of sampling depended upon the extent of operations and equipment being used at the particular location. The more fragmentary data, obtained from "inactive" stockpiles, served to supplement the more detailed studies.

Considerable variation in chloride content was apparent in many stockpiles even by visual inspection. This was true for both the normal surface of the stockpile and for cut sections. Other stockpiles showed a uniformly dry surface but were moist an inch or so below the exposed surface. Another observable condition was the seepage of chloride around the base of many stockpiles. Such areas usually tested high in calcium chloride content.

Laboratory testing of stockpile samples amply supported the visual observations, indicating wide variations in chloride content throughout a given stockpile and showing that there was considerable overall loss of chloride.

The various stockpile tests will be discussed separately with emphasis on the more detailed studies. The stockpiles have been named for the nearest town to their location or, where there were several in one area, for the pit from which they were obtained. All calcium chloride content values presented here are expressed as pounds of Type 1 flake equivalent per ton of aggregate. Values less than 6 lb per ton of aggregate indicate the degree of chloride loss from the treated stockpile.

### Lennon Stockpiles

Two stockpiles, one a 22A, the other a 23A gravel, were studied in this area. These were the first stockpiles included in this project and the test results were included in a memorandum report dated August 24, 1959. The stockpiles were being built and hauled to the road during the testing period, so that thorough sampling could be obtained. Each had been treated with a 38 percent solution of chloride added to the aggregate on the belt by a spray bar. Considerable care had been taken to ensure uniform application of the solution. In spite of this, the stockpiles showed considerable variation in uniformity of treatment. Fig. 3 shows this condition for both the natural surface and a cut section into the 23A stockpile. Fig. 4 shows the very high seepage of chloride solution at the base of the 22A stockpile. This picture was taken during a dry period. The seepage solution had killed adjacent vegetation and tests showed it to be saturated with calcium chloride.

The laboratory tests of the various samples from the stockpiles are shown in Fig. 5. These data show a wide variation in chloride content with values ranging from practically none to a high of 18 lb per ton of aggregate. The same order of variation was obtained both near the surface and from deeply cut away sections, showing that the variation in treatment extended throughout the stockpiles rather than being merely a surface effect.

Average results obtained from mixed samples of the 22A stockpile, as loaded into trucks for haulage to the road, showed a chloride content of 4.4 lb per ton of aggregate. For this relatively young stockpile this represented a chloride loss of approximately 25 percent. This stockpile was sampled in July and August 1959.

### Kalamazoo Stockpile

This stockpile, being constructed at the time of sampling, was selected for study because of the novel method used for adding calcium chloride solution to the aggregate. The solution was applied by two spray heads



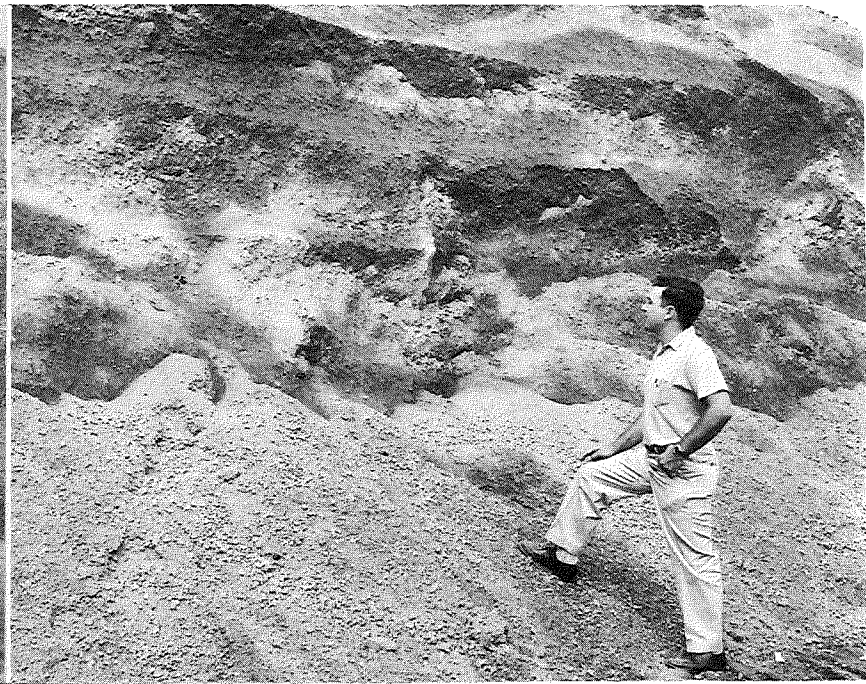


Figure 3. Variation in chloride content in natural surface (above, left) and cut face (above, right) of a Lennon stockpile.

Figure 4. Heavy seepage at a Lennon stockpile (left).



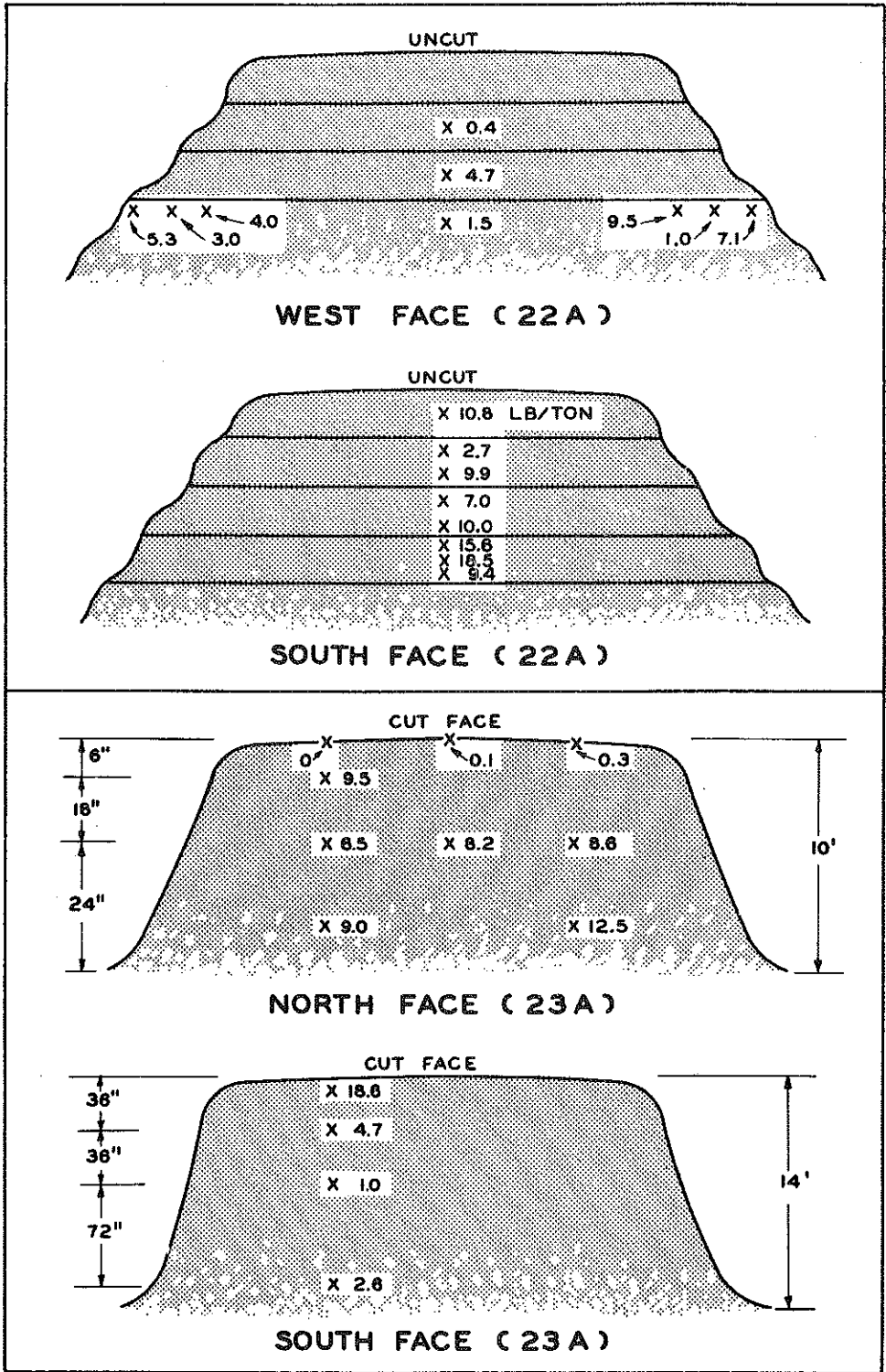


Figure 5. Lennon stockpiles of 22A and 23A material; solution-treated, being built at time of sampling.

located above and below the stream of aggregate as it dropped from the loading belt into the truck (Fig. 6). In this manner chloride was added to both sides of the stream of flowing aggregate. The method did not produce uniformity, however, because there tended to be a very high concentration of chloride in the gravel flowing to the side of the truck nearest the belt. The resulting uneven distribution of chloride in the truck and as dumped on the stockpile is also shown in Fig. 6.

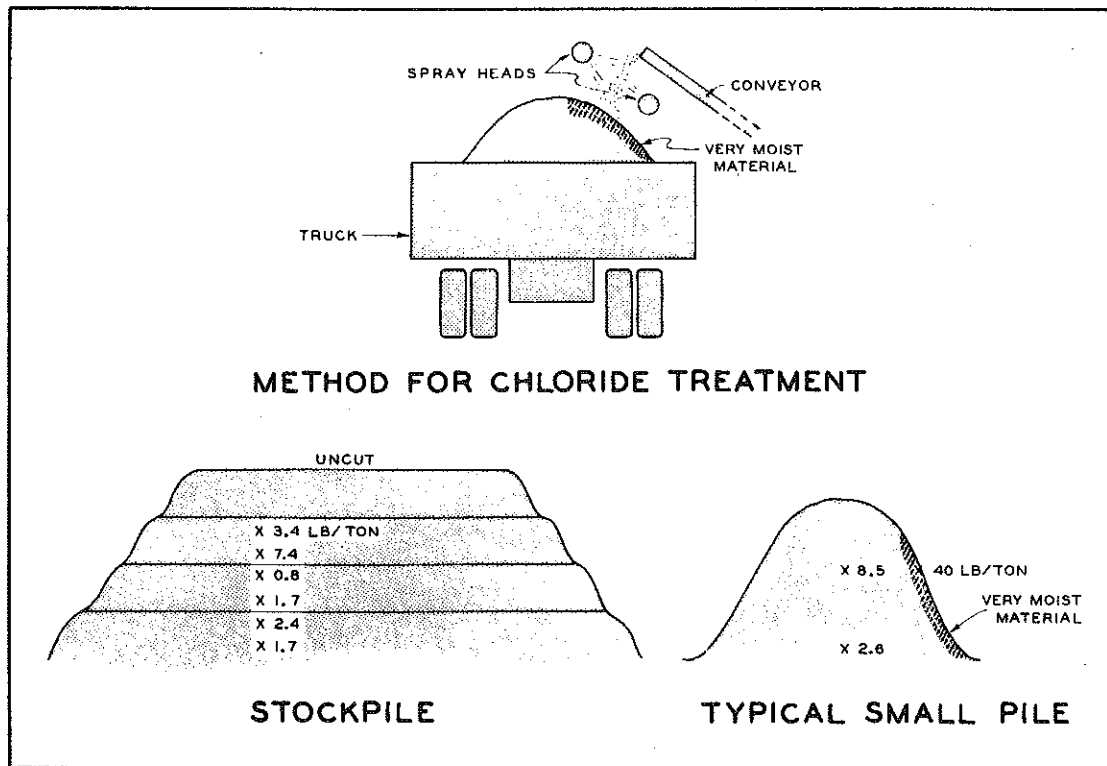


Figure 6. Kalamazoo stockpile: solution-treated, being built at time of sampling. Drawings show method of treatment and chloride concentration in a typical small pile dumped from a single truck.

The distribution of chloride at one face of the nearly completed stockpile is shown in Fig. 6. These values show that chloride had already begun to leach from the face of the stockpile although the tested areas had been in place for only a few weeks. Wide variations in amounts can also be noted. This stockpile was sampled November 6, 1959. An inch of rain had fallen two days before. Average chloride content as material was loaded for haulage to the road project was 1.9 lb per ton.

## Battle Creek Stockpile

This large stockpile had been treated with the solid form of calcium chloride; the south end with Type 1 flake, the north with Type 2 pellet. The south face had not been used but material had been hauled from the north portion. The stockpile was about one month old and heavy rain had fallen a few days prior to sampling. The stockpile was sampled August 28, 1959.

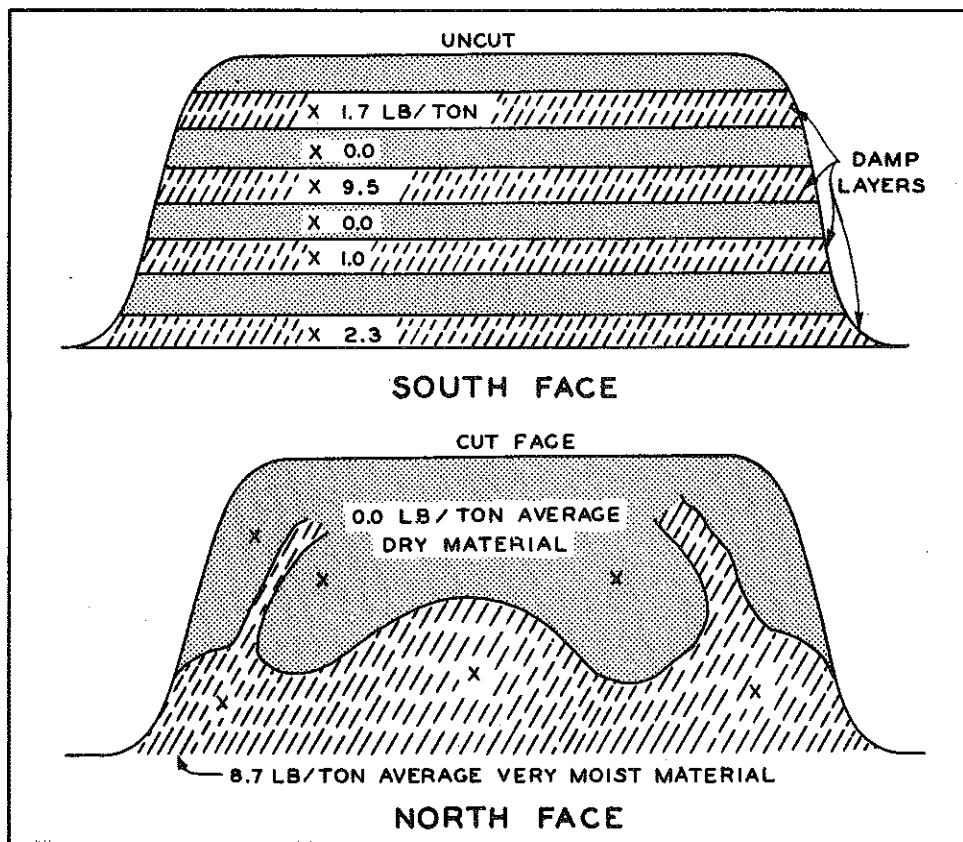


Figure 7. Battle Creek stockpile: south face Type 1 flake-treated, north face pellet-treated, one month old at time of sampling.

Fig. 7 shows the calcium chloride distribution in the north and south faces of this stockpile, including a definite layer effect visible on the south face. The light areas were dry and laboratory tests showed no chloride present in such areas. The moist areas contained chloride, but in varying amounts. This layering effect was probably due to rainfall on individual layers during construction of the stockpile. Samples were taken at a depth of 18 in. from the surface.

The north face had been cut away a few days previous to testing for haulage to the road site. Fig. 7 shows the pattern and distribution of chloride found in this area. This pattern shows a definite leaching of the chloride to the bottom portion of the stockpile, although there were streaks of chloride treated material at higher elevations. The heavy concentration of chloride, 8.7 lb per ton of aggregate, resulted in very moist material throughout the lower area. Ground around the north face had a chloride content of 5.7 lb per ton.

Although due to different weathering conditions at the time of construction the chloride distribution pattern was different for the north and south ends of this stockpile, both showed a leaching effect due to rainfall and a variable treatment. The average chloride content of the stockpile indicated a considerable loss of chloride.

#### Marshall Stockpile

This was a relatively new stockpile which had been treated with Type 1 flake calcium chloride. Samples were taken from a cut face during loading and hauling operations on August 28, 1959. In fact, activity was so great that only a few samples could be obtained without halting operations. Three samples across different elevations of the freshly cut area were obtained, mixed, and the average quantities of treatment determined (Fig. 8). The sampling values showed a large range in chloride content at the elevations sampled--up to 8 ft from the bottom. The lowest value, 2.5 lb per ton of aggregate, was taken from the mixed area near the bottom after loading. This low value was probably due to inclusion of the top portion of the stockpile which normally is quite low in chloride. However, the average mixture obtained from the face of the stockpile indicated a deficiency in chloride. About 0.5 in. of rain had fallen in the Marshall area two days prior to these tests.

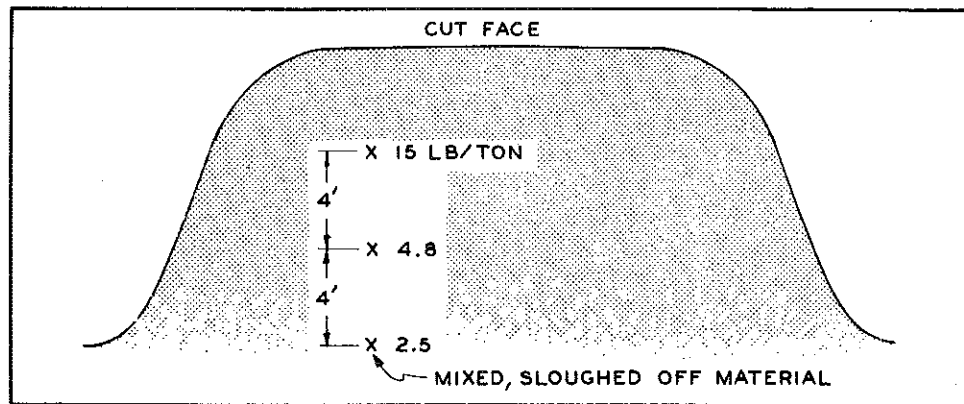


Figure 8. Marshall stockpile: Type 1 flake-treated, one month old at time of sampling.

## Devil's Lake Stockpiles

Two 22A stockpiles, treated with Type 1 calcium chloride, were tested near Devil's Lake. Both were approximately 10 months old and had been exposed through a winter.

The surface of each was quite dry, but a few inches beneath the surface the material was damp and of a generally uniform color.

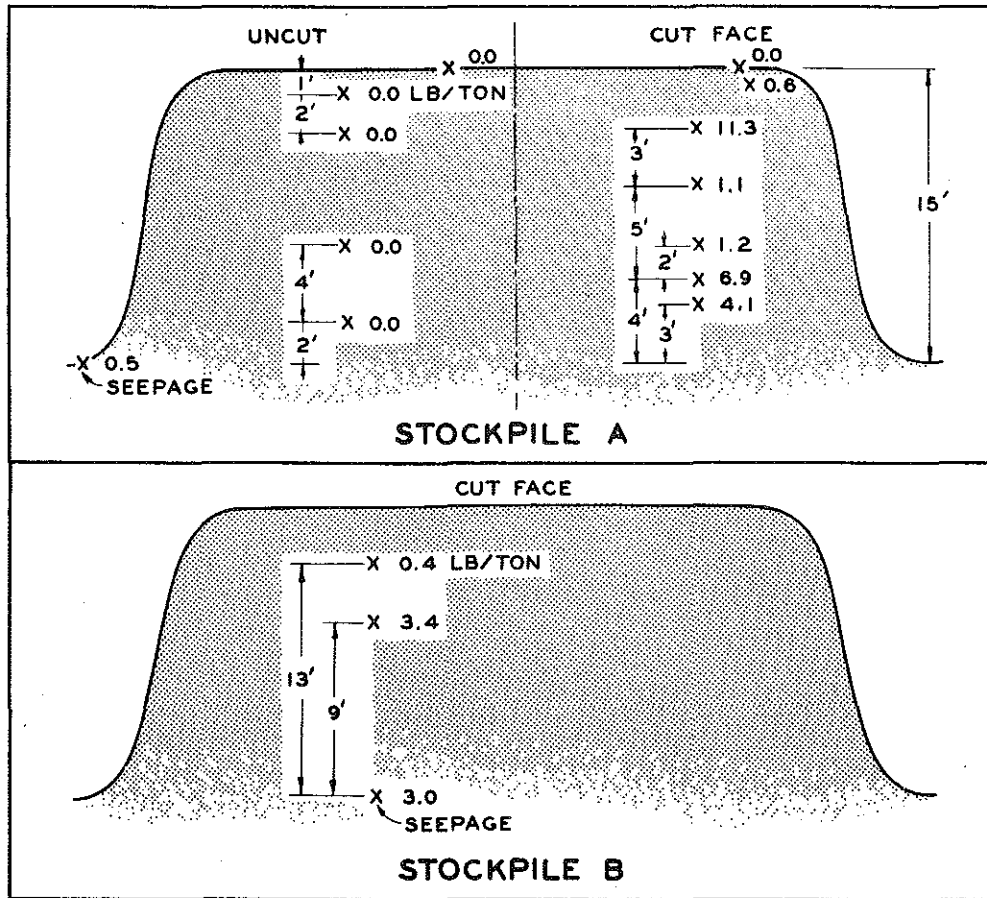


Figure 9. Devil's Lake stockpiles: Type 1 flake-treated, 10 months old at time of sampling.

Fig. 9 shows the chloride distribution and amount of treatment for each stockpile. Stockpile A showed practically no chloride in the surface excavations. Samples from the cut face, however, showed considerable chloride, but in varying amounts. The average treatment of the stockpile, as cut from the face and loaded on the truck, was 3.2 lb of chloride per ton. Stockpile B, at a cut face, showed lower chloride content at the top

than at lower elevations. The overall chloride content of this stockpile was far below specification requirements.

Howard City (Zylstra) Stockpile

This 23A gravel stockpile was treated with pellet form of Type 2 calcium chloride. It had been completed about a month at the time of first sampling, on May 31, 1960. The stockpile was sampled again on June 16.

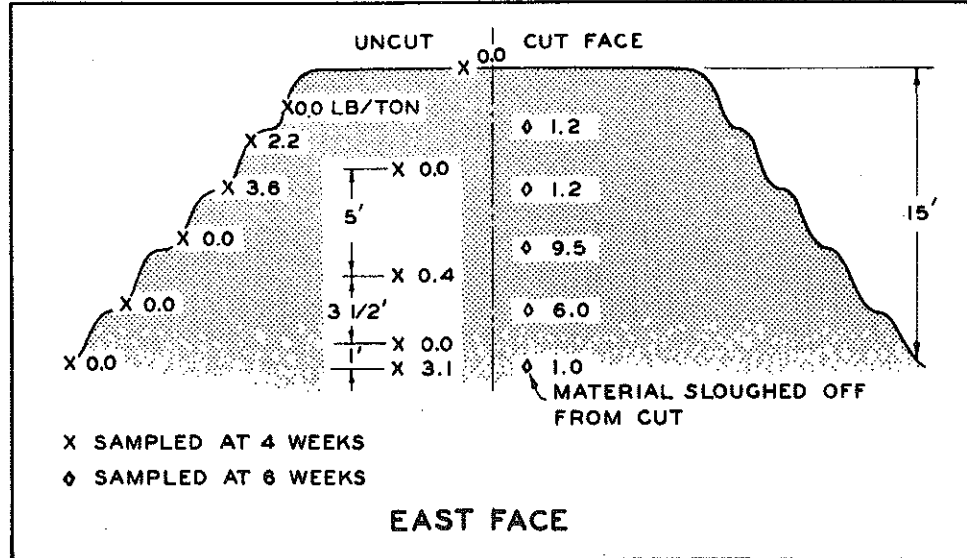


Figure 10. Howard City (Zylstra) stockpile; Type 2 pellet-treated.

As shown in Fig. 10, most of the samples taken near the surface indicated no chloride. A sample of seepage at the base, however, showed a treatment of 6.8 lb per ton of aggregate on June 31.

The cut away face showed considerable chloride present but in variable amounts with a tendency toward heavier concentration near the bottom. An average of the stockpile, taken from the haul truck, showed a chloride content of 4.6 lb per ton.

Lakeview (Robbins) Stockpile

This stockpile was composed of 22A gravel treated with the pellet form of Type 2 calcium chloride. It was about four weeks old at the time of first sampling, on April 29, 1960. The second sampling was made on May 26, and the third on June 3.





during the fall indicated wide variation in surface chloride content. Attempts to sample this stockpile during the winter were not successful due to its frozen condition.

Fig. 12 shows the results of surface sampling on June 20, 1960. Some seepage is indicated around the base, but otherwise no chloride was found within 2 ft or so of the surface. In an effort to check chloride content deeper into the stockpile, an area around the top edge was shovelled back and down about 10 ft. There was no indication of chloride at this depth.

### Woodland Stockpile

The Woodland stockpile was treated with liquid chloride in a very non-uniform manner. Fig. 13 shows how a single spray bar applied the liquid to the top side of the falling gravel as it left the conveyor belt.

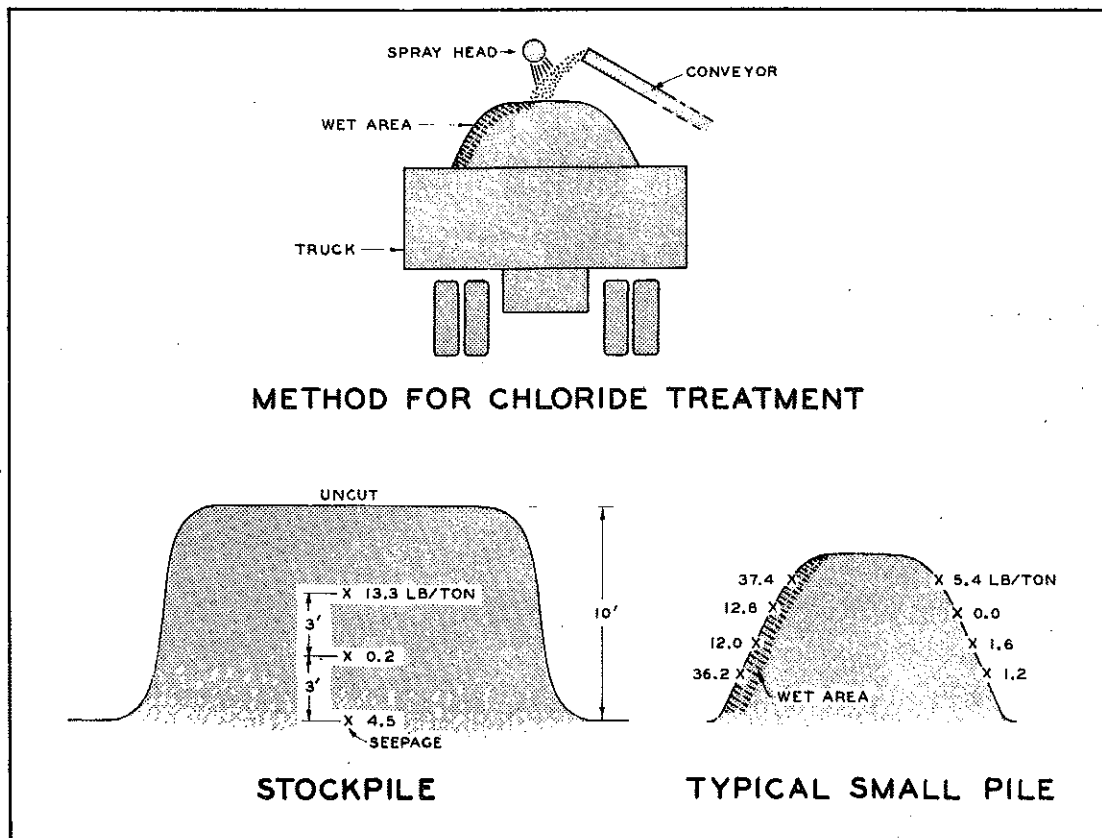


Figure 13. Woodland stockpile: solution-treated, a day to several days old at time of sampling. Drawings show method of treatment and chloride concentration in a typical small pile dumped from a single truck.

Practically all of the chloride fell into the side of the truck away from the conveyor. This resulted in the very erratic chloride contents shown in Fig. 13, with individual piles, as dumped from the truck, varying in chloride content from zero to an abnormal high of 37 lb per ton. Although the dumped piles were mixed and spread across the top of the stockpile, the surface was patched with high and low chloride contents. The main stockpile, which was only a few days old, varied in chloride content as shown in Fig. 13, on July 5, 1960.

#### St. Ignace (Pond) Stockpile

This stockpile was formed of limestone treated with liquid chloride. The uncut portion showed no chloride at or near the surface except for a few damp spots which were low in chloride. The cut away face, using the average of a large sample taken near the center of the open face, contained 4 lb of chloride per ton of aggregate. Fig. 14. shows the sampling pattern for this stockpile, which was tested on May 19, 1960.

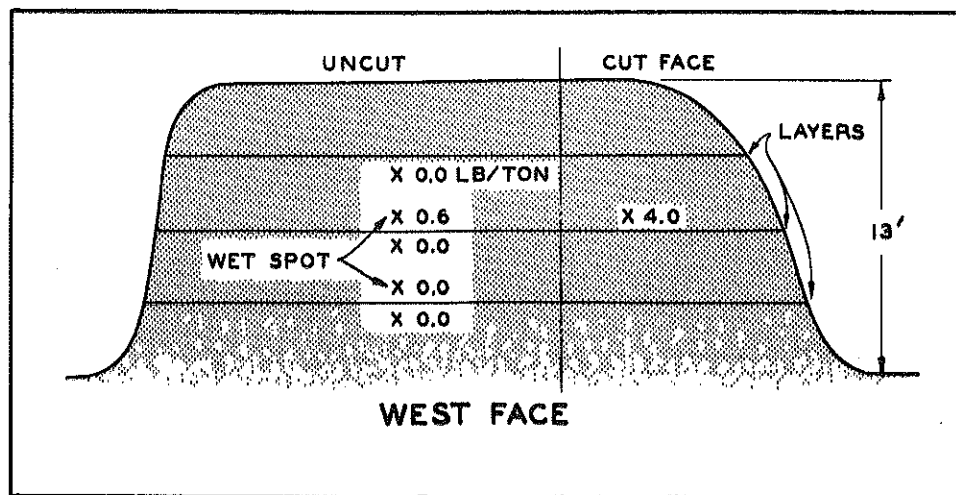


Figure 14. St. Ignace (Pond) stockpile: solution-treated, 7 months old at time of sampling.

#### Trout Lake (Furlong) Stockpile

This stockpile was composed of limestone, treated with the solution form of chloride. Samples were taken from a section that had been cut into the stockpile. Chloride values, shown in Fig. 15, were higher than normally found but showed considerable variation. A sample from the base of the cut, representing an average mixture as loaded onto the truck, averaged only 1/3 lb per ton. The stockpile was sampled on May 19, 1960.

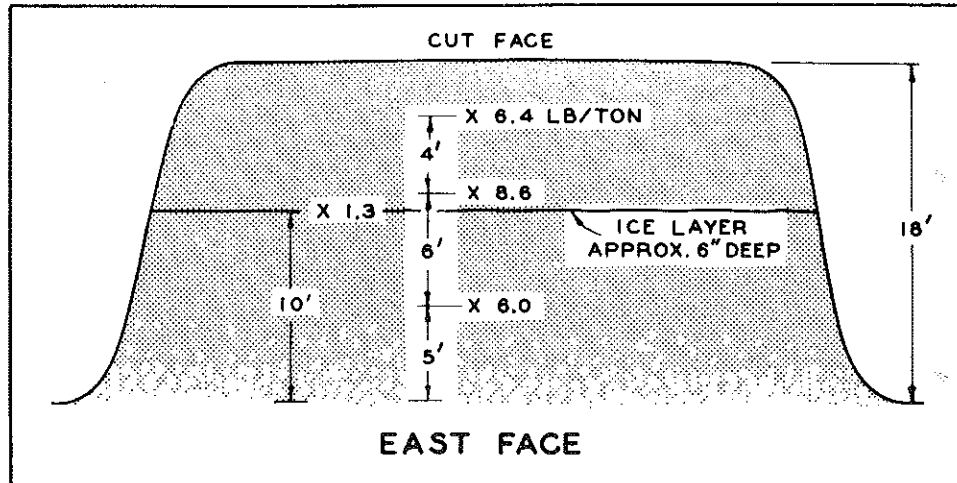


Figure 15. Trout Lake (Furlong) stockpile: solution-treated, 5 months old at time of sampling.

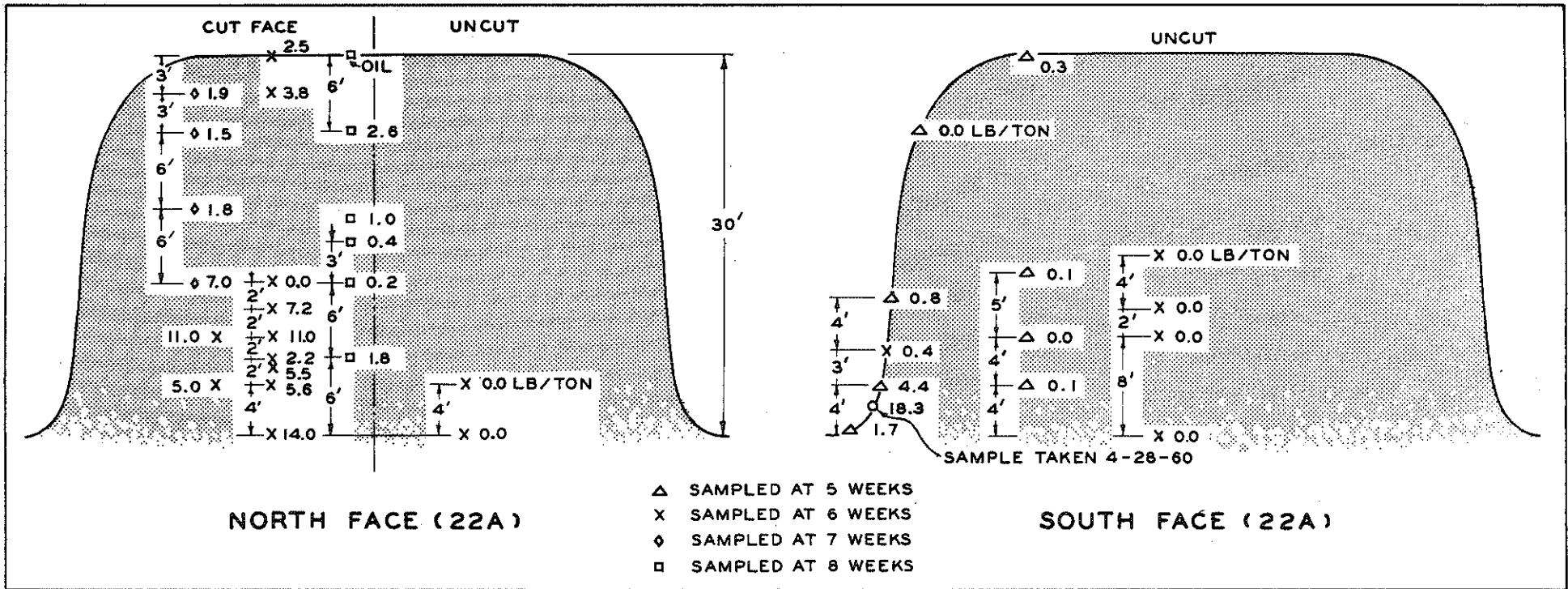
#### Howard City (Jenkins) Stockpile

This stockpile of 22A gravel was treated with Type 1 flake form of chloride. Care was exercised to add the flake chloride to the aggregate in a uniform manner. Bagged material was emptied into a small hopper from which a controlled amount of calcium chloride was fed to the aggregate on the conveyor belt prior to falling into the truck. The mixture appeared to be uniform, although light and dark spots appeared during drying. A test of one truckload of material as placed on the stockpile showed a chloride content of 7.2 lb per ton.

Detailed studies of the stockpile were made from construction through haulage for base course construction on M 46. Samples were taken from near the surface and from cut away areas. Fig. 16 presents data showing periodic variations in treatment for both cut and uncut sections. This figure shows the large differences in chloride content and the loss of chloride with exposure. On April 28, a day after construction of the 22A stockpile began, two composite samples showed contents of 9.0 and 5.7 lb per ton. Several rainfalls took place between sampling dates, the one before the eighth week samples were taken being particularly heavy. The effects of rainfall can be seen in the markedly reduced chloride quantities.

Average mixed samples from the trucks as hauled to the roadway contained about 4.7 lb of chloride per ton of aggregate.

Fig. 16 shows the chloride distribution in an adjoining 23A stockpile. Initial sampling showed some tendency for higher chloride contents at the



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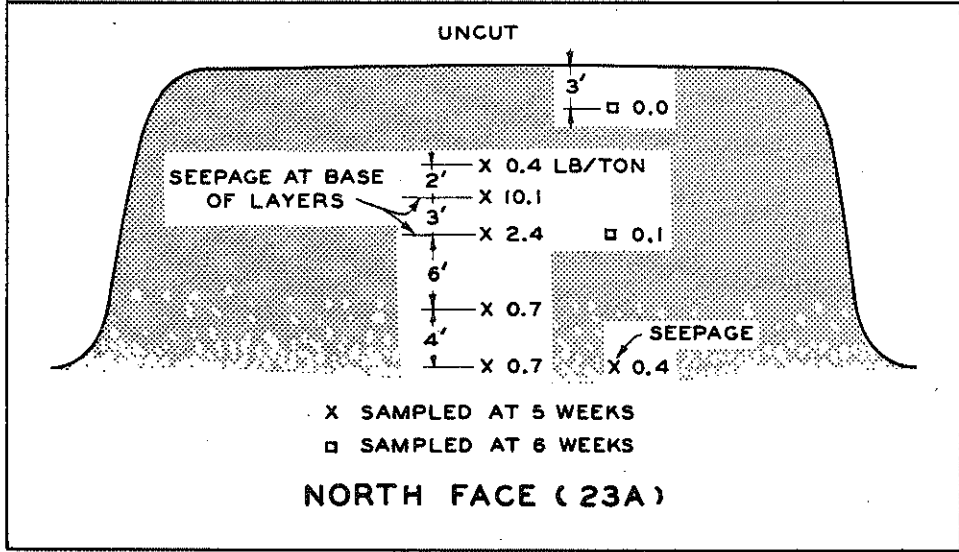


Figure 16. Howard City (Jenkins) stockpiles of 22A and 23A material; flake-treated.

bottom of individual layers. Later inspection showed a more uniform and dryer surface. Samples taken after initial testing showed considerable chloride loss.

Large samples from mixtures of the entire face of the cut section, taken at the time of loading, showed the average chloride content of the 22A stockpile to be 4.9 lb per ton of aggregate. Two weeks later this average value had dropped to 3.2 lb.

#### Vestaburg Stockpile

This stockpile had been treated with liquid chloride and had been exposed during the winter. Surface testing indicated no chloride at any elevation.

### CONDITION OF AGGREGATE AS PLACED ON THE ROAD

The primary reason for treating aggregate stockpiles with calcium chloride is to maintain a moisture content in the aggregate sufficient to allow easy and satisfactory compaction of the mixture on the road. The more uniform the moisture control, the more uniform is the compaction that can be obtained.

To determine the effectiveness of stockpile treatments under normal construction conditions, studies were made of the mixtures as applied and compacted on the road. Visual inspection of various jobs had indicated considerable spotting of compacted bases, indicating non-uniform chloride content. This condition existed where either liquid or solid forms of chloride were used.

The spotted appearance of a base in which liquid chloride was used in the aggregate is shown in Fig. 17. Although color difference is not too clear from the photograph, this area showed a wide variation in chloride content. Tests of the light and dark areas showed calcium chloride contents varying from 4.9 to 14.4 lb. This differential chloride content still remained after a surface treatment of liquid chloride had been made. The values, in this case, averaged 23.6 and 32.2 lb per ton, respectively.

Fig. 18 shows a section of the base course on M 46 after initial compaction. The aggregate (from the Howard City-Jenkins Stockpile) had been treated with Type 1 flake calcium chloride. The dry spots averaged





Figure 17. Appearance of liquid chloride treated base course (above).



Figure 18. Appearance of flake chloride treated base course (upper right).



Figure 19. Moist and dry areas of chloride treated aggregate in base course (right).

3.2 lb, while the wet spots showed 5.8 lb of chloride per ton of aggregate. The average of several samples taken at random from a section showed a chloride content of 4.1 lb per ton of aggregate.

In addition to chloride content a study was made of the moisture contents of the aggregate during loading, delivery, and after compaction on the job. As loaded at the stockpile the moisture content of the aggregate was 4.6 percent. After about a mile of haul the aggregate was delivered to the road at a moisture content of 4.1 percent. After compaction and remaining in place for about two hours the moist and dry spots showed a moisture variation of 1.4 to 4.3 percent at the surface. In areas about an inch below the surface, moisture variations ranged from 3.7 to 4.2 percent.

Fig. 19 shows the contrast in moisture content between freshly placed aggregate and material in place for an hour or more. The data concerning moisture loss all were obtained on a mild, clear day, in no way representative of an extreme weather condition.

#### LABORATORY STUDY OF CHLORIDE LEACHING

In order to study, more fully, the leaching of calcium chloride from aggregate stockpiles due to storage and rainfall, laboratory tests were devised for measuring this property under controlled conditions of moisture addition.

Fig. 20 shows the paper-lined box used in this study, the dimensions of which were approximately 16 in. square by 36 in. high. Its capacity was about 600 lb of gravel.

In this test, liquid and solid forms of calcium chloride were individually mixed with 22A gravel and the resulting mixture tamped into the box. An equivalent of 1/2-in. of rainfall was added to the surface of the gravel and the sample allowed to stand for four days. Then an additional amount of moisture, equivalent of an inch of rainfall, was added.

Samples were checked for chloride content at the start and during the progress of the test to determine variations due to simulated rainfall.

Considerable seepage was collected from the box after the last application of water and the solution tested high in chloride content for both



Figure 20. Laboratory method  
for measuring leaching  
of chloride from aggregate.

the flake and solution forms of treatment. As had been found in the field, the top layer (about one quarter of the depth) showed considerable loss of chloride due to water action. For both the liquid and solid forms of treatment, only 1 lb of chloride per ton of aggregate remained in the top layer at the conclusion of the tests. The chloride content of the other layers remained fairly constant, with the exception of the second layer from the top of the liquid treated sample which lost about half of its chloride during the leaching test.

In this test the amount of water added was small and barely enough to cause flow from the bottom of the sample. For this reason only the top layers lost chloride. In a supplemental test, using a sample treated with liquid chloride, larger amounts of water were poured on the aggregate sample. In this case all of the chloride was washed from the entire sample.

In all cases the water collected in the pan beneath the box tested high in chloride content.

#### CONCLUSIONS

The testing methods used in this study appear to be suitable for determining the distribution and concentration of calcium chloride in aggregate stockpiles. In specific cases, sampling errors could be high due to the relative sizes of the stockpile and the sample taken. However, such errors have been minimized by the large number of samples tested and by the different techniques used for sampling. From the results of this study the following conclusions may be stated:

1. Each stockpile tested showed a wide variation in chloride content at different locations within the stockpile. This was true for newly constructed stockpiles as well as for those that had undergone long term exposure to weather.

2. All stockpiles showed a significant loss of chloride averaging about 35 percent of the original treatment. Portions of some stockpiles showed 100-percent loss of treatment.

3. The primary loss of chloride was due to leaching of the stockpile by rainfall. In this case the loss was a function of frequency and rate of rainfall. However, one stockpile (Lennon) showed excessive seepage



shortly after construction, although there had been no recent rainfall. This condition was most likely due to excessive moisture in the aggregate during construction of the stockpile, which later seeped from the bottom due to consolidation of the mass.

4. Chloride variations within a stockpile also were due to poor mixing of the chloride and aggregate before adding to the stockpile. Considerable variations were found with both the liquid and dry forms of application. Variations were much more extreme, however, with the liquid form, in one case ranging from zero to 40 lb per ton of aggregate.

5. After exposure to rain there was usually no chloride remaining in the stockpile to a depth of at least 2 ft from any part of the surface (except for seepage at the base). When hauling from a stockpile, therefore, care should be taken to mix the outer layer with the rest of the stockpile before loading.

6. Variations in the chloride content of stockpiles were reflected by differential moisture conditions in the aggregate when placed on the road. Low or erratic chloride values could detrimentally affect proper compaction control.

7. There was noticeable layering of chloride in certain stockpiles. This could have been due to intermittent rainfall during construction or to accumulation of chloride at more highly compacted planes.

8. The most significant method for checking chloride content of a stockpile is to sample a fresh deeply cut area and obtain specimens representing average conditions from the bottom to the top of the cut face. This can best be done during hauling of aggregate to the road. Surface testing (to a depth of approximately 2 ft) is of value only in freshly built stockpiles.

9. The ASTM method for determining calcium chloride content of aggregate samples proved to be satisfactory. The method must be used with a great deal of care, however, and checked periodically against laboratory control samples.

10. Increasing the quantity of chloride applied does not appear to be the answer to treated stockpile deficiencies. Adding more chloride would not improve uniformity and there is no indication that more concentrated solutions of calcium chloride remain in stockpiles longer than do standard quantities.

The following recommendations are made concerning the treatment of aggregate stockpiles with calcium chloride:

1. Calcium chloride should be mixed with the aggregate just prior to placing on the roadway. This would eliminate losses due to exposure.

2. Improved methods should be developed for blending the chloride and aggregate in a more uniform manner.

3. The type of chloride should be selected only after a study of the conditions involved. When mechanical mixing is not available, more uniform mixtures can be obtained with the solid forms of chloride. Flake chloride dissolves more readily and at lower aggregate moisture content than does the pellet form. High moisture conditions in the aggregate could be aggravated by treating with the solution form of chloride, especially if such a treatment is not made uniformly.

4. A study should be made to determine how much improvement in moisture control is obtained by using calcium chloride. In particular, it should be known whether the use of chloride can be justified under all conditions throughout the construction season. In this study it was found that several untreated stockpiles had the same moisture content as did nearby treated stockpiles.