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Deicer Scaling Mechanisms In Concrete

By
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Synopsis: Three experiments were conducted to study the characteristics
of the two primary deicer scaling mechanisms in concrete: 1) a hydrau-
lic pressure mechanism, which is strongly dependent upon the degree of
saturation and the deicer gradient, and only occurs under freeze-thaw
conditions, and 2) a chemical mechanism, which operates between con-
crete and concentrated calcium chloride solutions. These two mecha-
nisms are completely independent of one another.

The experiments conducted showed: 1) the absorption of deicers
by concrete is directly related to the scaling rate under freeze-thaw
conditions; 2) the deicer content in freeze-thaw cycled mortar speci-
mens decreased with increasing depth; and 3) concentrated calcium
chloride solutions chemically react with concrete, causing deteriora-
tion. The mechanisms presented in the paper serve to explain most of
the previous observations which have been made of deicer scaling.

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INTRODUCTION

The deleterious effects of deicers on concrete has concerned concrete researchers for several decades. However, increased use of deicers on highways and especially bridges in recent years has focused attention on the problem. Between 1962 and 1969, the use of deicing chemicals on pavements and structures increased approximately 450% (1). This expansion is attributed to the "bare pavement policy" followed by most snow-belt highway maintenance forces.

There is little question that the use of deicer chemicals is linked to the surface scaling of concrete. Concretes of widely varying proportions and entrained air characteristics have been shown to be more vulnerable to scaling when exposed to deicers under freeze-thaw conditions than when exposed to water alone under these conditions (2). The deleterious effect of deicers is a major concern of the modern highway industry.

BACKGROUND

Many of the factors involved in the deicer scaling problem have been discovered through previous research and experience. Whiteside and Sweet (3) and Havens (4) demonstrated that saturation of concrete is necessary for freeze-thaw deterioration.

This factor has been supported by observations made by Verbeck and Klieger (2) and Boies and Bortz (5). They showed that specimens which were in continuous contact with moisture scaled much faster than those which were permitted to dry previous to freeze-thaw cycling in the presence of deicers. It is evident, therefore, that the amount of available freezable moisture is an important variable in the deterioration of concrete by deicers in a freeze-thaw environment.

Snyder (6) stated that a salt gradient must be present for scaling to occur. He based his statement on the observation that concrete specimens which contained salt in their mixwater were immune to deicer scaling under freeze-thaw conditions. The same type of specimens given

a drying period before testing, however, were quite vulnerable to
attack. The deicer gradient is dependent on the water-cement ratio
of the concrete (7). Much work has been done on the deicer gradient,
but no one to date has been able to establish a quantitative relation-
ship between deicer distribution and surface scaling.

Scaling under freeze-thaw conditions is dependent upon the deicer
solution concentration. Verbeck and Klieger (2) showed that in 50
cycles of freezing and thawing, solutions of 3% by weight of deicer
produced a higher rate of deterioration than either lower (to 0%) or
higher (to 16%) concentration, regardless of type of deicer (sodium
chloride, calcium chloride, urea, or ethyl alcohol).

Observations made by Neville (8) showed that, under steady state
conditions, strong calcium chloride solutions deteriorated concrete.
He also found that calcium chloride solutions removed water from the
specimens. Both wet-dry and freeze-thaw environments have been ob-
served to cause deterioration of deicer-exposed concrete (5).

PURPOSE OF THE RESEARCH

The experiments described in this paper were carried out to de-
termine the nature of the deteriorative mechanisms that result in the
diverse observations cited above with regard to deicer scaling. In
order to account for the observations of previous investigators, it
is proposed herein that two principal modes of deterioration must
exist--one physical in nature, the other chemical.

The Physical Mechanism

Previous research has shown that deicer scaling under freeze-thaw
conditions is a consequence of hydraulic pressure. The driving force
of this hydraulic pressure is, in turn, directly proportional to the
amount of water frozen. It has also been pointed out that the great-
est deterioration occurs at lower concentrations of deicers.

If we examine a phase diagram for a common deicer, calcium chlo-
ride (Fig. 1), it becomes clear why higher concentrations of deicers
are less aggressive than low concentrations. A solution containing
no calcium chloride will be entirely frozen at a temperature below
32 F, whereas a 25% calcium chloride solution will not freeze at a
temperature as low as -20 F.

The effect of solution concentration becomes even clearer when
we consider the deicer gradient in the concrete and the geometric
characteristics of scaled concrete. Scaling often occurs in the form
of small flat flakes breaking away from the mass of the pavement. The
flake itself appears to be sound, as does the mass of the concrete.
Quite likely, the reason for this phenomenon lies in the deicer grad-
ient and the degree of saturation. Above the failure plane of the
scale, the solution of deicer in the concrete pores is too concentrated
to produce sufficient freezing to cause failure.

The degree of saturation changes from that at the surface of the specimen to the background saturation of the concrete mass. The saturation at the surface is determined by the vapor pressure of the solution present. Strong calcium chloride solutions have a much lower vapor pressure than pure water. These solutions draw moisture from the concrete, lowering the surface saturation. The background saturation is determined by the original mix proportions, curing conditions, and the environment. The higher this background level, the more vulnerable the concrete is to deicer scaling. The concentration gradient and the degree of saturation together produce scaling in the form of flakes.

The basic hydraulic pressure mechanism (9) and the critical depth of saturation (10) appear to be quite adequate for explaining the geometry of the scaling process. A variable which has not been mentioned in the literature with respect to its possible contribution to the scaling process, however, is that of temperature. Every freeze-thaw cycling process inherently involves a temperature cycling process. The influence of temperature cycling on the absorption of deicer solutions has not been discussed previously.

The Chemical Mechanism

Neville (8) showed that saturated solutions of calcium chloride, without freeze-thaw cycling, were deleterious to concrete. The mode of attack varied depending on the water-cement ratio of the concrete mixture. For low w/c concretes (0.4 and 0.5 by weight), exposure to saturated calcium chloride solution produced essentially no length changes, but large weight losses and large degradations in dynamic modulus of elasticity. For a high w/c ratio concrete (0.7 by weight), the saturated calcium chloride solution produced large expansions, weight gains, and losses in dynamic E. Neville hypothesized that the degradation of the low w/c concretes resulted from leaching and resultant loss of strength. For the high w/c ratio concrete, he proposed that the degradation resulted from deposition and crystallization.

Since paving concretes will normally have w/c ratios slightly higher than Neville's low w/c concretes and much lower than his high w/c concrete, the nature of the deteriorative mechanism in the field is not clear. Also, the effect of strong solutions of sodium chloride, the other common deicing chemical, in the absence of freeze-thaw cycling has received little attention.

Scope of the Investigation

Three experiments were conducted in order to investigate the characteristics of the physical and chemical mechanisms. Two of these were designed to elaborate on changes in potential of the driving force and the location or distribution of the driving force involved in an hydraulic pressure mechanism. The third experiment was designed to directly measure the steady state chemical effects of deicers on concrete, as well as to demonstrate these effects under simulated field conditions. The experiments conducted were:

1. The absorption of deicers was monitored by measuring the weight changes of specimens subjected to thermal cycling. Control specimens, subjected to freeze-thaw cycling as well as to thermal cycling, served to demonstrate the effects of the different absorption rates.

2. The deicer distribution was determined in mortar specimens subjected to various deicer solutions under freeze-thaw conditions. The deterioration rates of these specimens were also observed.

3. Mortar specimens were soaked in strong solutions of deicers and their changes in tensile strength were measured. The evaporable water content of these specimens was also determined, to aid in interpretation of the tensile strength changes.

This study was not directed toward the use of all the deicers presently available. Only sodium chloride and calcium chloride were investigated, as they are the most popular deicers in current use. Generalization of the conclusions from this work could be extended to other deicers through the results of the work done by Verbeck and Klieger (2).

EXPERIMENTS AND RESULTS

The Specimen Mix Design

Both concrete and mortar specimens were used in the experimental work. Air entrainment was not employed in order to reduce the amount of time necessary in the laboratory to produce the desired severity of scaling.

The concrete mix was designed with proportions similar to highway-grade concrete. It was based on the cement content of 535 lb/cu yd and a water-cement ratio of 0.54 (by weight). The coarse aggregate was made up of three gradations of limestone. The size proportions were: one third 3/4 to 1/2 in., one third 1/2 to 3/8 in., and one third 3/8 to #4. Ottawa sand was used as the fine aggregate.

The concrete batches were mixed by hand in a large flat pan for a period of at least 7 min before placement. The mortar was identical to the concrete except for the absence of coarse aggregate. The mortar batches were mixed in a motor driven mortar mixer for 3 min before placement. The mixes described above were used for the preparation of all the specimens tested.

Absorption of Deicer Solutions by Concrete

Since it was not known whether the deicer scaling process involves the aggregate phase, both concrete and mortar specimens were subjected to freeze-thaw and thermal cycles in various concentrations of calcium chloride. The experimental design is presented in Table 1.

The freeze-thaw cycle ranged from 15 to 45 F (-9.4 to + 7.2 C) and the thermal cycle extended from 45 to 75 F (7.2 to 23.9 C). The saturated calcium chloride solution was used even though no freezing would be possible at the lower test temperature.

The mean weight changes of groups of five specimens exposed to different concentrations of calcium chloride solutions and subjected to thermal cycling are shown in Figs. 2 and 3. (Originally, all of the specimens weighed approximately 150 g. A 3 g weight change in these figures is equivalent to a 2% change in weight). Both the mortar and the concrete specimens demonstrated that saturated calcium chloride causes deterioration under thermal cycling in the absence of freezing and thawing. The graphs also indicate that the specimens exposed to the other concentrations absorbed solution with an increasing number of cycles.

It can be seen from the two figures that the 3% calcium chloride solutions were absorbed faster than either the tap water or the 10% calcium chloride solutions. The difference between the weights for the calcium chloride specimens was calculated to be significantly different from the others at the 99.99% level. These results are not intended to imply that 3% is a critical concentration value at which maximum absorption takes place. They do, however, indicate that there is a pessimum concentration between 0 and 10% at which maximum absorption occurs.

The effect of different environments is shown by the weight changes of the specimens, both concrete and mortar, subjected to cycles in those environments. The specimens exposed to water and those in 10% $CaCl_2$ showed no deterioration under thermal cycling and a small amount of deterioration beyond 50 cycles under freeze-thaw conditions. Extensive deterioration was observed, however, in the 3% and the saturated solutions of calcium chloride (Figs. 4 and 5). Thermal cycling had the greatest effect on the specimens in the saturated calcium chloride solution, whereas freeze-thaw cycling had the greatest effect on those in the 3% calcium chloride solution. No deterioration of specimens in the 3% solution occurred under thermal cycling, and concrete was affected more than mortar under freeze-thaw conditions. The findings related above are dramatically illustrated in the appearance of typical specimens after 77 cycles under the various test conditions as shown in Figs. 6 and 7.

Deicer Distribution in Concrete

In this experiment mortar specimens were subjected to freeze-thaw cycling, with various concentrations of deicers on their surfaces. The number of specimens, specimen type, deicer solution type, and deicer concentration are shown in Table 2. The deterioration of the specimens was determined by two rating methods, and the deicer concentration gradients in the specimens were measured periodically throughout the testing. A comparison was then made between the deicer gradient measured and the deterioration rate observed.

The specimens were cycled in an environmental chamber which al-
ternately decreased and increased the temperature at a rate of 2.5 F
(1.4 C) per hour. One freeze-thaw cycle took 24 hours to complete.
The temperature was varied from 45 F. to 15 F (+7.2 to -9.4 C). The
chloride distribution of the specimens was measured at 3, 8, 13, 18
and 23 cycles. This was done using a specific ion electrote procedure
(11). The deterioration of the specimens was determined at the same
time as the distribution was measured. A visual rating method was
used along with a weight change procedure to quantify deterioration.

Before rating, the specimens were cleaned with compressed air.
They were then visually rated for deterioration and weighed to deter-
mine the net loss of material. The visual rating was performed by a
single operator as follows. Two categories of relative deterioration
were established: (1) surface undisturbed to fine aggregate exposed,
and (2) fine aggregate displaced. The specimen was inspected, the
proportion of its surface in each category was estimated, and the per-
centage recorded. The rating number was obtained by multiplying the
percentage in category 1 by one and in category 2 by two, and adding
the result. For example, if 50% of the surface is undisturbed and
50% has fine aggregate displaced, the rating would be

(1 x 50) + (2 x 50) = 150

The visual rating system is non-linear in nature. It is sensitive
only to the percentage of area deteriorated, and not to the amount of
material lost from the surface. The method becomes completely in-
effectual once all of the surface area has deteriorated to the maximum
rating, although the specimen could continue to lose large amounts of
material.

The freeze-thaw condition caused such a high rate of deterioration
that the test was discontinued after 23 cycles. The average weight
loss at that time was 4%. Visual ratings of the concrete and mortar
specimens subjected to freeze-thaw conditions are shown in Fig. 8.
The visual rating method, because of its limited range, could not be
used to determine the deterioration of specimens after eight cycles
of freezing and thawing.

The results of the weight loss method of rating deterioration are
shown in Fig. 9. Low concentrations of sodium chloride produced signi-
ficant amounts of deterioration in the mortar specimens, whereas low
concentrations of calcium chloride did not behave in this manner.
Instead, their effect was quite similar to the deterioration rates re-
corded for the 30% calcium chloride and the 20% sodium chloride solu-
tions. The reasons for this difference in behavior of the calcium and
sodium chloride exposed mortar specimens is not known. It is suspected,
however, that these results are anomalous. There were only two speci-
mens tested at each deicer concentration. In the case of the specimens
subjected to the two lower concentrations of sodium chloride solution,
the weight loss of the duplicate specimens were in wide disagreement.
It became evident that the sample size was simply not adequate to make
a comparison of the deterioration rates.

The chloride content was measured at 1/4-in. intervals to a depth of 1 in. in the mortar specimens each time they were rated for deterioration. Powdered samples were extracted by dry drilling with a masonry bit from a single specimen exposed to each of the deicer concentrations. At the end of the experiment the second identical mortar specimen was sampled by drilling five holes at 1/16-in. intervals to a depth of 1 in.

The results of the chloride analyses at 1/4-in. intervals are shown in Fig. 10. In most cases the chloride content was greater at the surface of the specimen, decreasing with increased depth. The chloride concentration increased with time for all depths.

The results of the 1/16-in. interval analyses, made after 23 cycles, are shown in Fig. 11. These results agree very well with the 1/4-in. interval results at 23 cycles for the low concentrations of calcium chloride. They do not agree, however, for the 30% calcium chloride solution nor the sodium chloride solutions. This was probably because of higher rates of diffusion, coupled with influences from previous drill holes. The holes left in the 1/4-in. sampled specimens after sampling were filled with glazing compound, which apparently did not keep the applied solution out of the hole. After a period of time, with an increasing number of holes, a large influence on diffusion could be exerted by adjacent holes.

Chemical Attack by Calcium Chloride

The experiment was designed in two parts. The first was simply to demonstrate that the tensile strength of a specimen was influenced by its evaporable water content. The tensile strengths of two groups of specimens were compared. The one group was oven dried while the other was in a saturated state. The water contents of these specimens were measured, and the relationship between the evaporable water content and the tensile strength was determined.

The second part of the experiment was designed to measure the effect of soaking time in the two different deicer solutions. The tensile strength as well as the water content were measured periodically up to a soaking period of 84 days.

The results of these two experiments made it possible to differentiate between changes in strength due to changes in the evaporable water content and those due to the action of the calcium chloride in the concrete.

Two series of tensile specimens were prepared according to ASTM Designation C 190-70. The first series, consisting of 14 specimens, was prepared from a single batch of mortar and cured for 24 h before the molds were removed. After removal of the molds, the specimens were placed under water to cure for an additional 27 days.

The 14 specimens were then split into two groups of seven specimens each. One group was placed in a 110 (230 F) oven to dry for 24 h, and then cooled to room temperature in a dessicator. The other

seven specimens remained under water. The tensile strengths of both
groups were then measured. The evaporable water content was determined
for the group of specimens which had remained under water. The mean
tensile strengths and the evaporable water content are shown in Fig.
12.

The second series, consisting of 162 specimens, was made from
nine separate batches, prepared one day apart. The mortar and place-
ment procedures were the same as for the first series. Solutions of
sodium chloride and calcium chloride were prepared with equal* chloride
concentrations of 12.12% chloride by weight. After curing for 28 days,
54 specimens were submerged in each of these solutions, and 54 in tap
water.

Groups of nine specimens each were tested for tensile strength
at 0, 7, 14, 21, 28, 56, and 84 days of soaking. Each of these groups
contained a specimen made from each of the original nine batches of
mortar. After the specimens were broken, they were weighed and placed
in an oven to dry. Their weights were taken again, after drying, and
the evaporable water content was calculated. The variations in tensile
strength are shown in Fig. 13. The coefficient of variation of the
results indicated by any point on the graph is less than 15%, as speci-
fied by ASTM Designation C 190-70. The variation in average water
content for these specimens is shown in Fig. 14. The coefficient of
variation of the water content determinations was less than 7.5% for
all of the values obtained.

With the exception of the specimens soaked in calcium chloride,
comparison of Figs. 13 and 14 reveals a strong dependence of tensile
strength upon changes in water content brought about by soaking in the
deicer solutions. The calcium chloride specimens gained strength
initially with a decrease in water content, but then began to lose
strength with no change in the evaporable water content. This can
only be explained as a chemical attack process which effectively re-
moved material from the specimens.

The surface deterioration on the 84-day exposed calcium chloride
specimens was quite significant. The specimens were deteriorated
to such a degree that it was decided to measure their cross-sectional
areas by tracing the broken surfaces on paper. The average area was
found to be 0.785 sq in. The original area was 1 sq in. Recalcula-
tion of the average failure stress, with compensation for the change
in area of the specimens, revealed that the specimens actually broke
at an average stress of 709.4 psi rather than the 570.6 psi recorded
originally. (The data in Fig. 13 assumed that the cross-sectional
area was 1 sq in.) The recalculated 84-day strengths were comparable
to the strengths recorded early in the experiment for specimens of
the same evaporable water content. This revealed that the central
portions of the specimens were unaffected by the calcium chloride.

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*The term "equal" here should be understood to be within the
limitations of the purity of the deicer used.

DISCUSSION

Absorption of Deicer Solutions by Concrete

The major result of this experiment is that low concentrations of calcium chloride were absorbed faster than either stronger solutions or water. This fact, coupled with the dependence of the hydraulic pressure mechanism on the degree of saturation, leads directly to the other results found in the experiment: the 3% deicer solution caused the most rapid deterioration of the specimens under freeze-thaw conditions, and 10% deicer solutions and water caused much less deterioration.

A very interesting property of the chemical attack by saturated calcium chloride was demonstrated in the results of this experiment. The specimens that were cycled from 45 to 75 F deteriorated much faster than the ones cycled from 15 to 45 F. This shows that the reaction of saturated calcium chloride with concrete is dependent upon, and increases with, temperature. This result is characteristic of a chemical reaction processes and gives further evidence of the nature of the attack by concentrated calcium chloride solutions on concrete.

Deicer Distribution in Concrete

The freeze-thaw environment produced rapid deterioration of the specimens. The lower concentrations of sodium chloride (3 and 6%, by weight) were much more aggressive toward the mortar specimens than were the same concentrations of calcium chloride. These deicers appeared to be equally aggressive toward concrete specimens.

There is apparently no direct correlation between the measured chloride content and the deterioration rate of the freeze-thaw specimens. The two groups that deteriorated much faster than the others (3 and 6% sodium chloride) appeared to have an almost constant distribution of chlorides throughout their depth, as shown by the 1/4-in. gradient measurements. All of the other specimens displayed a normal diffusion-type chloride distribution: higher chloride concentration at the surface, decreasing with depth. A check on the results through the 1/16-in. gradient measurements revealed that a pattern of chloride diffusion did exist for the 3 and 6% sodium chloride specimens. Since all of the 1/4 in. samples were taken from the same specimen, it is thought that the holes from successive samplings may have influenced the results. The first hole drilled in the specimen may have permitted the sodium chloride better access to the interior of the specimen and greatly influenced the sample taken one week later, adjacent to the first hole. Since the 1/16 in. samples were taken during a single operation, the results from these are thought to be more valid.

Chemical Attack by Calcium Chloride

This experiment was designed to study the chemical attack of strong calcium chloride solutions on concrete. Tensile strength measurements were used as the indicator of the chemical reaction within the cement paste. The tensile strength, however, was shown to be highly dependent upon the evaporable water content of the specimens. Changes in the eavporable water content were brought about by the presence of strong deicer solutions. It is evident that an osmotic cell was set up between the water in the specimens and the deicer solutions. The mortar acted as the membrane and the water passed through it into the deicer solution.

One portion of the experiment showed that the tensile strength of specimens with a low evaporable water content was much higher than that of those with a high evaporable water content. The other portion of the experiment showed that both calcium chloride and sodium chloride solutions reduced the evaporable water content of the specimens. This reduction was accompanied by an increase in tensile strength.

The increase in tensile strength of the calcium chloride specimens was followed by a steady decrease. The sodium chloride specimens did not show this trend. This decrease was due to loss of effective cross section from the corrosive action of the deicer. Measurements of the sound area of broken specimens confirmed this fact.

An explanation of this corrosion can be obtained from the pH properties of the calcium chloride solution. The pH of pure calcium chloride solutions can vary from 6.0 in dilute solutions to 4.0 in saturated solutions. It is likely that these acid solutions react directly with the cement paste in the concrete to yield readily soluble salts. The dissolution of these salts would cause the deterioration noted.

CONCLUSIONS

The following conclusions appear to be warranted on the basis of the findings from the experiments described in this paper:

1. A hydraulic pressure mechanism is responsible for the scaling of concrete exposed to deicer solutions under freeze-thaw conditions. This mechanism is strongly dependent upon the degree of saturation of the concrete and the deicer gradient present.

2. A chemical reaction mechanism is responsible for deicer scaling under concentrated calcium chloride conditions.

3. The two mechanisms of deicer scaling are entirely indepen- dent of one another.

4. In freeze-thaw cycling, it is the temperature change and not the phase change which causes the concrete to absorb various concen-

trations of deicer solutions at different rates.

5. The rate of scaling under freeze-thaw conditions is directly related to the amount of absorption in concrete.

6. Under freeze-thaw conditions, deicer solutions penetrate concrete and mortar in a manner similar to diffusion—the concentration decreasing with increased depth.

7. Deicer scaling is a paste phenomenon—mortar and concrete generally deteriorated in a similar manner when exposed to deicers.

8. The chemical reaction of calcium chloride upon the concrete is dependent upon temperature—calcium chloride is more aggressive at higher temperatures.

ACKNOWLEDGMENTS

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Table 1: Design of the Deicer Absorption Experiment

| | | SOLUTION CONCENTRATION ($CaCl_2$) | | | |
		0%	3%	10%	Saturated
Temperature cycling	Concrete	5*	5	5	5
	Mortar	5	5	5	5
Freeze-thaw cycling	Concrete	5	5	5	5
	Mortar	5	5	5	5

*Number of specimens

Table 2: Design of the Deicer Distribution Experiment

| | | DEICER CONCENTRATION | | | | |
	DEICER TYPE	0%	3%	6%	20%	30%
Mortar	$CaCl_2$	2	2	2	–	2
	NaCl	–	2	2	2	–

*Number of specimens
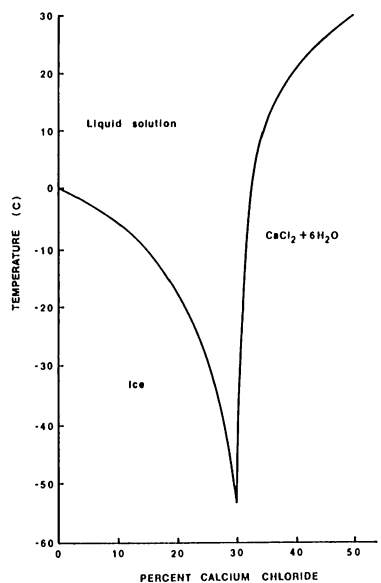
Fig. 1--Phase diagram of the system $CaCl_2$-H_2O
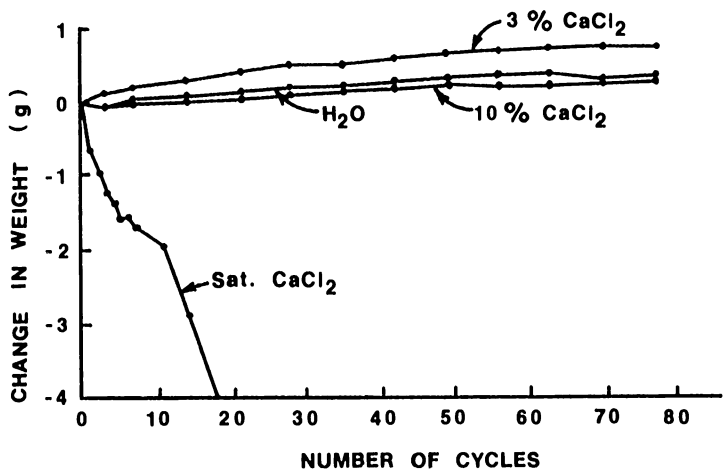
Fig. 2--Weight change of mortar specimens under thermal cycling
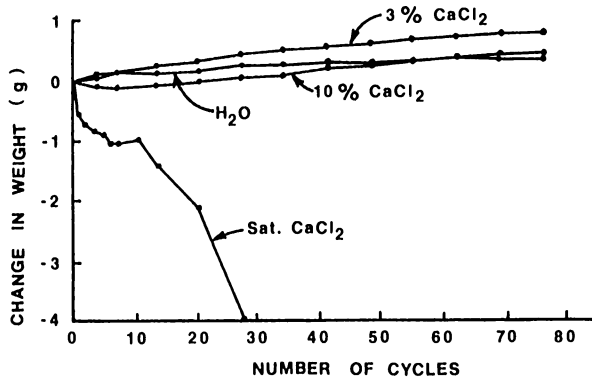
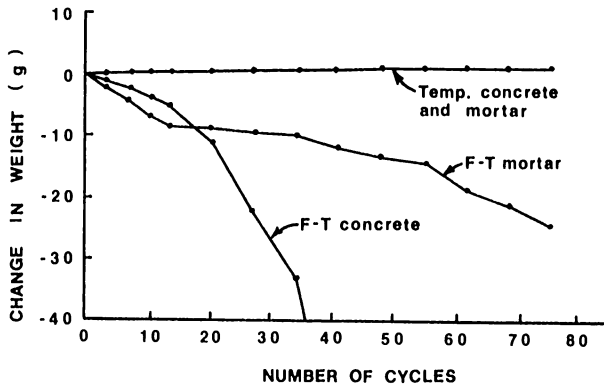Fig. 3--Weight change of concrete specimens under thermal cycling

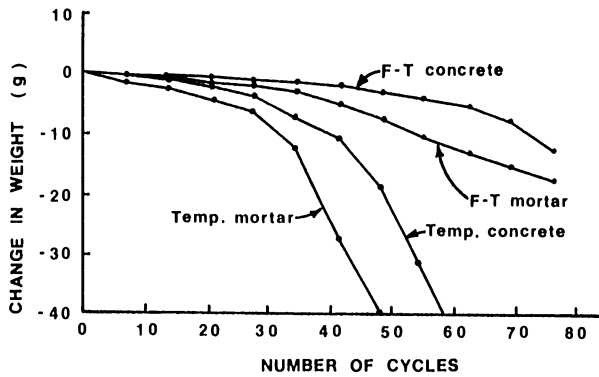Fig. 4--Change in weight of specimens in 3 percent calcium chloride solution

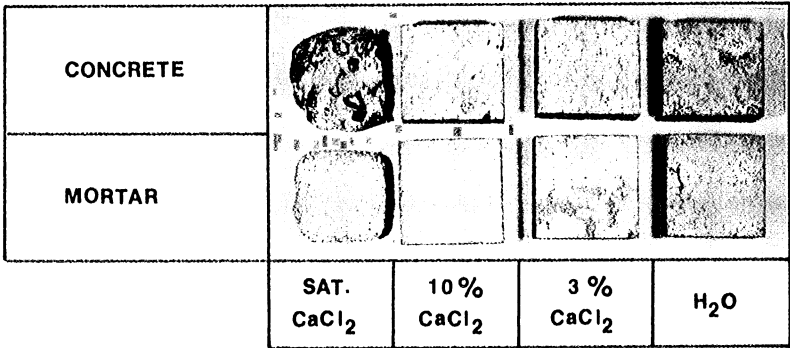Fig. 5--Changes in weight of specimens in saturated calcium chloride solution

CONCRETE				
MORTAR				
	SAT. $CaCl_2$	10 % $CaCl_2$	3 % $CaCl_2$	H_2O

Fig. 6--Typical thermally cycled specimens after 77 cycles

CONCRETE				
MORTAR				
	SAT. $CaCl_2$	10 % $CaCl_2$	3 % $CaCl_2$	H_2O
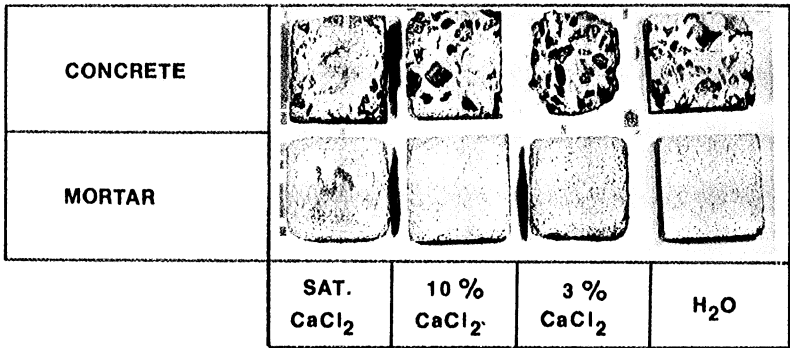
Fig. 7--Typical freeze-thaw cycled specimens after 77 cycles
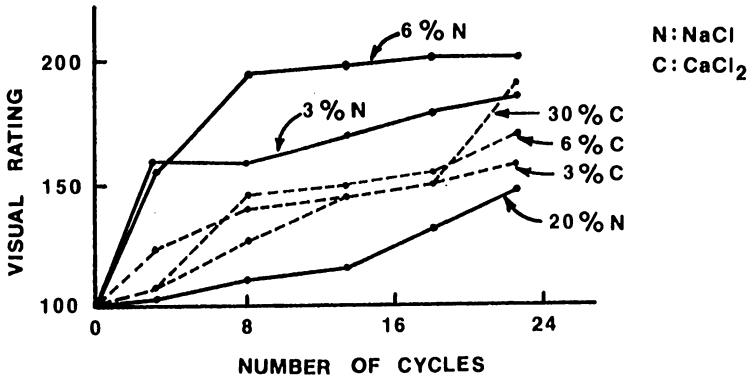
Fig. 8--Visual rating of mortar specimens exposed to freeze-thaw cycling
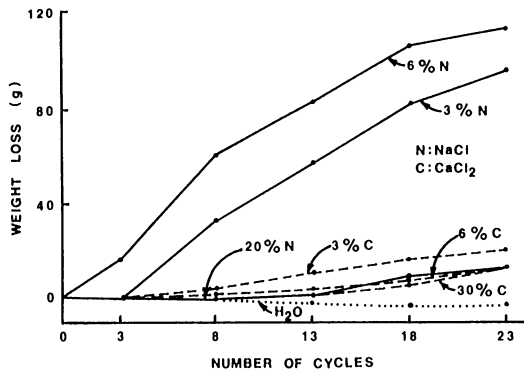
Fig. 9--Average change in weight of mortar specimens exposed to freeze-thaw cycling
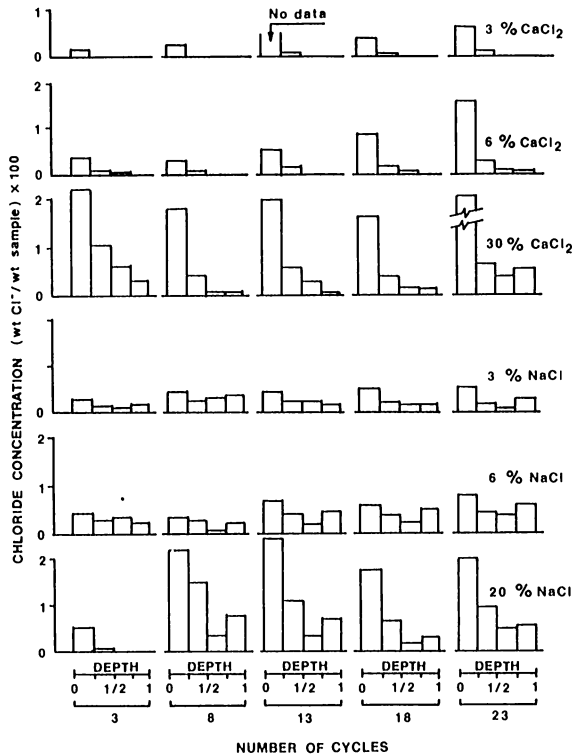
Fig. 10--Chloride contents of mortar specimens exposed to freeze-thaw cycling, measured at ¼ in. intervals

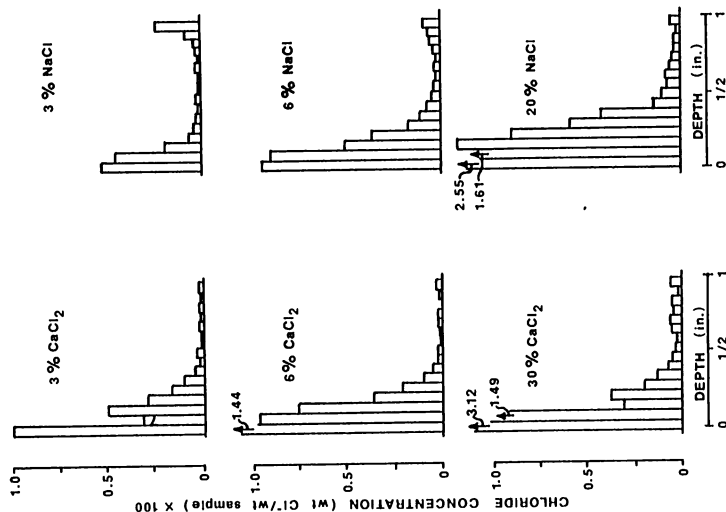Fig. 12--Tensile strength of saturated and over-
dry specimens

Fig. 11--Chloride contents of mortar specimens
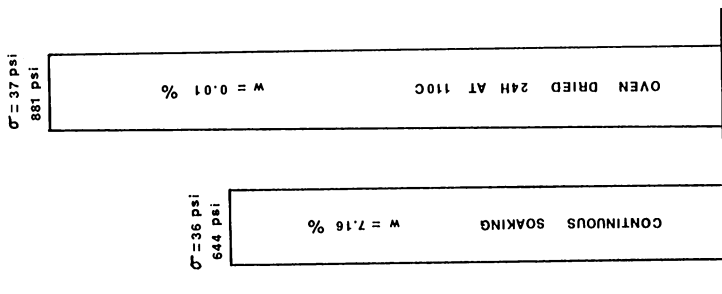exposed to freeze-thaw cycling, measured at 1/16 in.
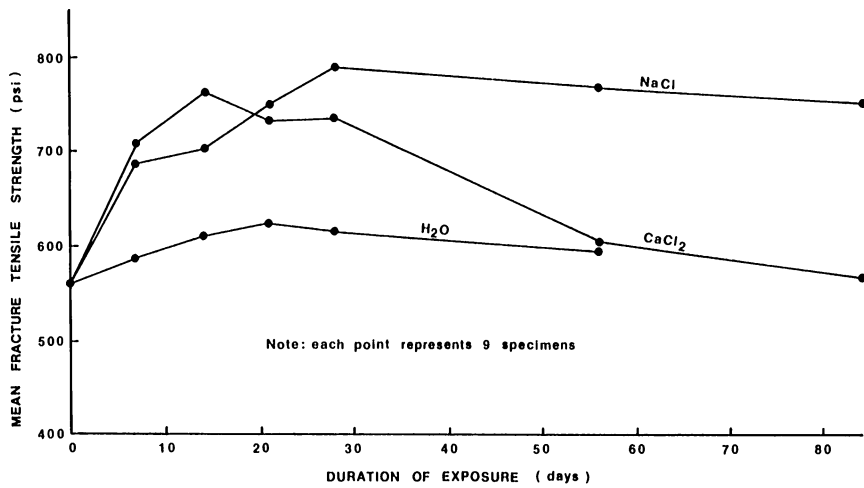intervals, after 23 cycles

Fig. 13--Tensile strength related to exposure time in solutions of equal chloride concentration
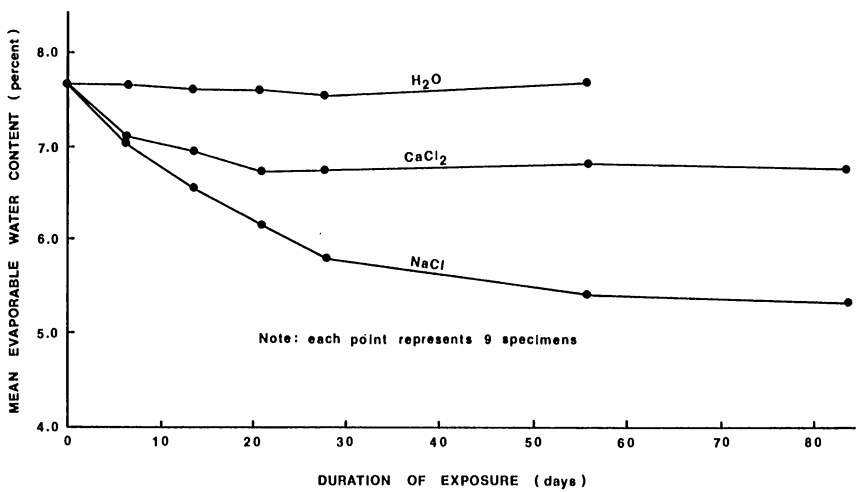

Fig. 14--Evaporable water content variation with exposure time in solutions of equal chloride concentration