# NATIONAL BUREAU OF STANDARDS REPORT

9343

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Progress Report

# INDUCED ACCELERATED DETERIORATION OF PORTLAND CEMENT CONCRETE

Semiannual ([illegible])

by

Raymond L. Blaine

Chemical Research Laboratory
RESEARCH LABORATORIES
Edgewood Arsenal, Maryland 21010

Contract D018-0-35-A6-02806

NATIONAL BUREAU OF STANDARDS
Washington, D. C. 20234

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## FOREWORD

The work described in this report was authorized under Project No. D018-0-35-A6-02806. This work was started in October 1965 and will continue for 18 months. Experimental data are contained in Vol. 1 of notebook dated October 1, 1965 assigned to R. L. Blaine.

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# DIGEST

During the first six-month period most of the mortar and concrete specimens for use in this test series have been made. Tests have indicated the salt solutions which are most aggressive on mortar specimens, and studies have been started on the feasibility of electrochemical corrosion of reinforcing steel in concrete.

Mortar specimens, 4X4X16cm, were made using a 1:3 and a 1:5 cement to sand mix with both a Type I and a Type II cement. Of these specimens, 33 sets of 10 were placed in various salt solutions and weekly measurements made for six weeks of changes in fundamental resonance frequency, of weight changes, and of electrical resistance. A number of failures were noted, —some as early as 2 weeks after being placed in the salt solutions. Ammonium salts appear to be the most aggressive, and specimens made of the Type II cement more resistant to attack than the specimens made of the Type I cement.

Concrete cylinders, both 3X6 and 6X12 inches have been made from the same cements using silicions, aggregates and mixes designed for a 5000 psi and a 2000 psi concrete. Additional small cylinders were made using a limestone aggregate. Reinforcing rods were placed in the axis of some of the cylinders. These specimens are now being cured prior to test.

Preliminary tests have indicated that reinforcing steel in concrete can be corroded by means of a direct electric current. The amount of corrosion is dependent on the electrolyte used as well as the voltage and current. Cracking of the concrete cylinders occurred in some trial experiments.

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# INDUCED ACCELERATED DETERIORATION OF PORTLAND CEMENT CONCRETE

## INTRODUCTION

There are many references in the literature to the deleterious effect on portland cement concrete caused by certain chemicals. There is, however, little information on the relative aggressiveness of the different chemicals, and to what extent the quality of the concrete or type of cement used in the concrete may affect the degree of deterioration, or, how rapidly such deterioration can be achieved. There are also references to cracking of concrete caused by corrosion of the reinforcing steel, either by the presence of salts in the concrete, or these in conjunction with stray electric currents. Very little information is available on methods for accellerating such corrosion and cracking.

In order to determine the relative aggressiveness of various chemicals which have been reported as being deleterious to concrete, a series of screening tests were first made with low pressure steam cured mortars of two qualities, of each of two types of cement, and after both moist and dry storage. Concrete cylinders have been made and cured under more normal conditions and will be exposed to the chemicals found to be the most aggressive in the mortar tests. Reinforcing bars were inserted part way into the axis of some cylinders, and these specimens will be used in studies of electrochemical corrosion as a means of cracking reinforced concrete.

This first progress report presents information on the cements used, the proportions used for the mortars and concretes and their properties, the chemical reagents used, as well as the methods of testing and the results obtained with the more aggressive chemicals on the mortar specimens. Some exploratory test results of electrochemical deterioration of concrete specimens are also presented.

## Cements

Two lots of portland cement were procured from a mill supplying the Washington, D. C. area. Standard chemical and physical tests were made to ascertain that the one lot was a Type I cement and the other a Type II cement. Both lots of cement met Federal and ASTM specifications for low alkali, non-air-entraining portland cements. The results of physical and chemical tests are presented in Appendix 1.

### Mortars

Two mortar proportions were used as follows:

| | Mortar "R" | Mortar "L" |
|---|---|---|
| 20-30 Ottawa sand, g | 600 | 600 |
| Graded Ottawa sand, g | 600 | 600 |
| #0 Gopher Silica sand, g | 300 | 300 |
| Cement, g | 500 | 300 |
| Water, ml | 250 | 225 |

Mortar "R" has a 1:3 cement to aggregate ratio with a 0.50 water cement ratio, whereas mortar "L" has a 1:5 cement to aggregate ratio with a water cement ratio of 0.75. The mortars were mixed in accordance with Federal and ASTM specifications for portland cement. Trial mixes of both mortars had a flow of 145 with 25 drops on the standard 10 inch flow table. In making the 4X4X16cm prisms the 3 gang molds were vibrated on a platform vibrator as filled in about 30 seconds. Vibration at 3600 vpm at 0.5 mm amplitude was continued an additional 90 seconds. The excess mortar was screeded off and a monel number tag placed in each specimen for later identification. The molds were stored at 95-100% relative humidity for 20 hours after which the molds were stripped and the specimens weighed in air and in water. The specimens were then placed in a container and steamed at 150°F for 24 hours after which they were stored at 95-100% relative humidity for 3-4 weeks. After this, half of the specimens of each of the groups of mortars were placed in laboratory air. (The other specimens remaining in the high humidity room.) Each specimen was placed on end with space between the specimens for circulation of air. With 2 cements, 2 mortars, and 2 conditions of storage, there were 8 variables. A total of 480 prisms were made and cured.

The results of the specific gravity determinations indicated a slightly higher specific gravity values than the theoretical:

| | Theoretical sp. gr. | Average Actual sp. gr. | S.D. |
|---|---|---|---|
| Type I cement R mortar | 2.307 | 2.317 | 0.007 |
| Type I cement L mortar | 2.285 | 2.315 | 0.006 |
| Type II cement R mortar | 2.307 | 2.356 | 0.007 |
| Type II cement L mortar | 2.285 | 2.321 | 0.004 |

S. D. is the estimated standard deviation of 12 determinations.

The compressive strength of the moist stored specimens at the time of test was determined and the following results obtained;

| | psi |
|---|---|
| Type I cement, 1:3 mortar | 8665 |
| Type I cement, 1:5 mortar | 4365 |
| Type II cement, 1:3 mortar | 10100 |
| Type II cement, 1:5 mortar | 4550 |

These compressive strength values were higher than anticipated for these mixes.

## Concrete

Three concrete mix proportions were used as follows:

| | Mix A | Mix B | Mix C |
|---|---|---|---|
| cement, lb | 24.2 | 15.95 | 24.2 |
| 3/8" aggregate, lb. | 46.2* | 46.2* | 43.0** |
| sand, lb. | 71.5 | 71.5 | 74.7 |
| water, lb. | 13.75 | 16.5 | 13.75 |

*Silicious, partially rounded.
**Crushed limestone.

Concrete mix proportions A and B were used with both the Types I and II cements and mix C only with the Type I cement. The concrete was mixed in a tilting drum mixer. The slump and weight per cubic foot was determined. The mix A and C concretes were consolidated in the 1/2 cubic foot container (for wt/ft$^3$ determination) by means of vibration because of the low slump. The 3X6 inch paper cylinder molds were filled in 3 layers and each layer consolidated by vibration on a platform vibrator. Each layer was vibrated until moisture appeared on the surface. With the lean, high slump concrete, very little vibration was required.

Reinforcing rods, 3/8 inch size, were vibrated 4 inches into the axis of some of the cylinders after filling and held in place with clamps until the molds were removed at 24 hours.

The specimens were stored at 100% relative humidity until they were 28 days old when half of each group was placed in laboratory air and the remainder (not used for compressive strength tests) remaining in the high humidity room.

The properties of the concretes are presented in Appendix B.

Using Type I cement and Mix A twelve 6X12 inch concrete cylinders were made. Half of these cylinders had 3/8 inch reinforcing rods and the others #6 reinforcing rods inserted into the axis. Twenty six 3X6 inch cylinders with 3/8 inch reinforcing rods were also made with this mix proportion. A total of 213 cylinders were made.

## Reagents

Approximately 550 grams of each of the reagents listed in Table 1 was mixed with approximately 2750 ml tap water and placed in polyethylene containers. In most instances there was complete solution, and in some there was an excess of the reagent. This quantity was adequate to cover 10 specimens placed in each container at the start of the test. The containers were not covered and tap water was added to maintain coverage of the specimens as water evaporated.

For the first series of tests, one specimen from each of the 8 variables plus 2 duplicates, ___ 1 from each of the cements, was placed in each of the containers.

The one container with water only was used as a control to determine the changes which occurred in resonance frequency with no reagent present.

Other reagents, such as DMSO, DMF, 1-2 Propanediol, DMSO with Aluminum Potassium sulfate and sugar, and DMF with the same reagents were tried but discontinued after a short time because of the difficulty of handling these toxic solvents.

Some of the 4X4X16cm mortar bars have been placed on end in 3 to 4cm of concentrated salt solutions.

## Test Procedure

The 4X4X16cm specimens were each weighed to the nearest gram, after which the fundamental resonance frequency was determined by means of a sonic apparatus. Each specimen was then pressed against contacts about 3 inches apart to determine the electrical resistance. The measurements were made prior to placing in the salt solutions and at weekly intervals thereafter. Observations were also made of any cracking, checking or warping of the specimens. After being in the solutions, the specimens were washed in tap water, brushed with a soft bristle brush, allowed to drain, and the adhering water blotted off with a paper towel before weighing and other tests.

A reduction in the fundamental resonance frequency of 1300 cycles per second was used as an arbitrary indication of failure. This corresponds to an approximate 40% reduction in the dynamic modulus of elasticity for the specimens used in this study. (A 40% reduction in the dynamic modulus of elasticity is also used in freeze-thaw durability tests on concrete.)

## Results of Test

A summary of the results obtained with the most aggressive solutions are presented in Table 2A for the Type I cement and in Table 2B for the Type II cement. The approximate time required to attain a reduction of 1300 CPS in the fundamental resonance frequency where failure occurred is presented in the one column, and in the last column is presented the reduction in fundamental resonance frequency during a 6 week test period.

It may be noted in comparing the 1:3 cement to sand and the 1:5 cement to sand mortars in these tables that the 1:3 mortars were more resistant to attack. Also the specimens made of Type II cement (Table 2B) were more resistant to chemical attack than were those of the Type I cement (Table 2A).

In addition to the effect of reagents listed in these tables, the specimens in citric acid solution sloughed off badly and at 6 weeks had lost about 10% by weight. The sodium bisulfate and potassium bisulfate solutions had a severe surface reaction with the specimens at the start, after which the specimens gained in resonance frequency. The specimens stored in solutions of the other reagents have not indicated a significant loss of strength nor loss of weight or cracking in 6 weeks of test. Specimens stored in some of the salt solutions had an apparent increase in strength.

An analysis has not been made of the nature of the failure versus the chemicals used. It was noted, however, that there was expansion in some instances, cracking in others, and softening and sloughing off of the surface with some specimens. A more complete description of the failures will be presented in a later report.

The results of the duplicate specimens were in good agreement both with respect to weight changes, and to changes in frequency.

The moist specimens stored in water for the same period as those stored in the solutions indicated very little change in strength during the test period.

Specimens placed on end in 3 to 4cm concentrated salt solutions have been observed for signs of cracking or damage. The bottom part of specimens stored in the concentrated ammonium acetate solution practically disintegrated in 24 hours and in 3 to 4 days severe sloughing occured up to 6 to 8 cm above the solution with both the Types I and II cements.

## Electrochemical corrosion of reinforcing bars in concrete

Some exploratory tests have been made of the time required to cause enough corrosion of the reinforcing bars in concrete so that the concrete specimen splits open. The 3X6 inch cylinders with reinforcing rods in the axis were placed on end in an electrolyte. The electrolyte came within 1/2 inch of the top of the cylinder. The protruding rod was connected to the anode and the electrolyte to the cathode. Various electrolytes have been used, including NaCl, $NH_4Cl$, $CaCl_2$, NaAcetate, and $Na_2SO_4$. Two voltages have been used, a 110dc and 18dc. Arrangements have been made to obtain intermediate voltages.

Small 2X4 inch cylinders could be cracked in about 1 hour with 110dc. Enough heat was generated to boil the electrolyte surrounding the specimen. With the larger specimens (3X6 inch) cracks sometimes developed in 3 to 4 hours. Using 18 volts a number of days were required to crack the cylinders. With both voltages, the corrosion product was extruded as a liquid or suspension around the top where the reinforcing rod entered the specimen. The corrosion product was then also forced out through planes of weakness or through the cracks which sometimes formed in the concrete cylinders. Although a fairly large quantity of the corrosion product came out of the concrete into the electrolyte, no further damage to the cylinder was observed. Concrete cylinders which were split open manually after the electrochemical treatment indicated severe corrosion of the reinforcing bar, and the presence of corrosion products in the cracks. The use of chlorides reduced the resistance of the concrete specimens and resulted in a greater current. However, it appeared that a soluble corrosion product was formed which, when forced out, either dried on the surface or precipitated in the electrolyte. When the acetate salt was used, the corrosion product was extruded where the steel entered the cylinder. When this cylinder was split, there was no corrosion on the steel bar in the concrete. The problem of containing the corrosion product where it will cause splitting of the concrete has not been solved.

## Plans for future work

Measurements will be continued at less frequent intervals of the changes of fundamental resonance frequency of the 4X4X16 cm mortar specimens remaining in the salt solutions.

Non reinforced 3X6 inch concrete cylinders will be placed in solutions of the more aggressive chemicals and measurements made of the changes in fundamental resonance frequency.

Experiments will be continued to determine the electrolyte and voltages best suited for the electrochemical corrosion of the steel in reinforced concrete.

# TABLE I

| No. | Reagents used |
|---|---|
| 1 | Water |
| 2 | Sodium Sulfate |
| 3 | Magnesium Sulfate |
| 4 | Stearic acid |
| 5 | Ammonium Sulfate |
| 6 | Ammonium Nitrate |
| 7 | Sodium Thiosulfate |
| 8 | Sugar |
| 9 | Sodium bisulfate |
| 10 | Potassium Sulfate |
| 11 | Ferrous Ammonium Sulfate |
| 12 | Ammonium Persulfate |
| 13 | Potassium acetate |
| 14 | Ammonium chloride |
| 15 | Sodium Sulfite |
| 16 | Ferric Ammonium Sulfate |
| 17 | Citric acid |
| 18 | Sodium Bisulfite |
| 19 | Ammonium Acetate |
| 20 | Sodium Acetate |
| 21 | Ferric Sulfate |
| 22 | Aluminum Potassium Sulfate |
| 23 | Potassium Bisulfate |
| 24 | Cupric Sulfate |
| 30 | Ammonium Sulfate, and sugar |
| 31 | Aluminum Potassium Sulfate and sugar |

TABLE 2A

| Mortar | Reagent | Time for 1300 cps reduction in resonance frequency weeks | Reduction in resonance frequency in 6 weeks cps |
|---|---|---|---|
| Type I cement, 1:3 cement to sand moist | $NH_4Cl$ | 3 | |
| | $NH_4NO_3$ | 4 | |
| | $(NH_4)_2S_2O_8$ | 5 | |
| | $NH_4Ac$ | | 750 |
| | $(NH_4)_2SO_4$ | | 350 |
| Type I cement, 1:3 cement to sand dry | $NH_4NO_3$ | 3 | |
| | $(NH_4)_2S_2O_8$ | 3 | |
| | $NH_4Cl$ | 6 | |
| | $NH_4Ac$ | | 950 |
| | $(NH_4)_2SO_4$ | | 90 |
| Type I cement, 1:5 cement to sand moist | $(NH_4)_2SO_4$ | 2 | |
| | $NH_4NO_3$ | 2 | |
| | $NH_4Ac$ | 4 | |
| | $(NH_4)_2S_2O_8$ | 3 | |
| | $NH_4Cl$ | 3 | |
| | $K_2SO_4$ | 4 | |
| | $MgSO_4$ | 6 | |
| Type I cement, 1:5 cement to sand dry | $NH_4NO_3$ | 2 | |
| | $(NH_4)_2S_2O_8$ | 3 | |
| | $NH_4Ac$ | 4 | |
| | $NH_4Cl$ | 5 | |
| | $K_2SO_4$ | 5 | |
| | $(NH_4)_2SO_4$ | 6 | |
| | $MgSO_4$ | 6 | |

TABLE 2B

| Mortar | Reagent | Time for 1300 cps reduction in resonance frequency weeks | Reduction in resonance frequency in 6 weeks cps |
|---|---|---|---|
| Type II cement, 1:3 cement to sand moist | $NH_4Cl$ | | 1210 |
| | $NH_4Ac$ | | 970 |
| | $(NH_4)_2S_2O_8$ | | 725 |
| | $NH_4NO_3$ | | 640 |
| | $(NH_4)_2SO_4$ | | 200 |
| Type II cement, 1:3 cement to sand dry | $NH_4Cl$ | | 1010 |
| | $NH_4Ac$ | | 900 |
| | $(NH_4)_2S_2O_8$ | | 780 |
| | $NH_4NO_3$ | | 770 |
| Type II cement, 1:5 cement to sand moist | $NH_4Cl$ | 2 | |
| | $NH_4Ac$ | 2 | |
| | $(NH_4)_2SO_4$ | 5 | |
| | $NH_4NO_3$ | | 1050 |
| | $(NH_4)_2S_2O_8$ | | 530 |
| | NaAc | | 110 |
| Type II cement, 1:5 cement to sand dry | $NH_4Cl$ | 3 | |
| | $NH_4Ac$ | | 1150 |
| | $NH_4NO_3$ | | 1130 |
| | $(NH_4)_2SO_4$ | | 700 |
| | $(NH_4)_2S_2O_8$ | | 120 |
| | NaAc | | 90 |

Appendix 1

## PROPERTIES OF CEMENTS
## CHEMICAL ANALYSIS

| | Type I cement | | Type II cement | |
|---|---|---|---|---|
| | 1st Test | 2nd Test | 1st Test | 2nd Test |
| Loss on ignition, % | 1.6 | 1.6 | 1.2 | 1.2 |
| Insol, residue, % | 0.14 | 0.18 | 0.18 | 0.14 |
| $SO_3$, % | 2.1 | 2.1 | 1.9 | 1.9 |
| MgO, % | 2.5 | 2.5 | 2.1 | 2.1 |
| $SiO_2$, % | 21.1 | 21.1 | 22.5 | 22.6 |
| $Al_2O_3$, % | 6.0 | 5.9 | 4.1 | 4.1 |
| $Fe_2O_3$, % | 2.4 | 2.4 | 4.1 | 4.1 |
| CaO, % | 63.7 | 63.6 | 63.6 | 63.6 |
| Total alkali as $Na_2O$, % | 0.52 | 0.54 | 0.43 | 0.42 |
| $C_3A$ | 12.0 | 11.6 | 3.9 | 3.8 |
| $C_3S$ | 48.8 | 49.3 | 48.8 | 48.4 |
| $C_2S$ | 23.8 | 23.4 | 27.8 | 28.5 |
| $C_4AF$ | 7.3 | 7.4 | 12.4 | 12.6 |

## PROPERTIES OF CEMENTS
## PHYSICAL TESTS

| | Type I cement | Type II cement |
|---|---|---|
| Autoclave expansion % | 0.11 | 0.01 |
| Initial set hrs:min | 3:35 | 4:25 |
| Final set hrs:min | 5:40 | 5:50 |
| Fineness $cm^2/g$ | 3390 | 3420 |
| Air 1:4 mortar % | 8.2 | 9.4 |
| Compressive strength 1d psi | 1260 | 1300 |
| Compressive strength 3d psi | 2801 | 2215 |
| Compressive strength 7d psi | 4095 | 3510 |

Appendix 2

## PROPERTIES OF CONCRETES

| | Slump in. | wt/ft$^3$ | bags/yd$^3$ | Comp. Strength psi | |
|---|---|---|---|---|---|
| | | | | 7d | 28d |
| Type I cement Mix A | 0.1 | 142 | 6.27 | 3590 | 5220 |
| Type I cement Mix B | 6.5 | 138 | 4.20 | 1160 | 2010 |
| Type I cement Mix C | 0.2 | 144 | | 3730 | 4760 |
| Type II cement Mix A | 0.7 | 142 | 6.35 | 3520 | 5250 |
| Type II cement Mix B | 7.5 | 137 | 4.19 | 820 | 1820 |

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13. ABSTRACT

Mortar specimens were made using a 1:3 and a 1:5, cement to sand ratio, with both a Type I and a Type II cement. The specimens were placed in various salt solutions and measurements made for 6 weeks of weight changes and of changes in fundamental resonance frequency. A number of failures were noted,- some as early as 2 weeks after being placed in the salt solutions. Ammonium salts appear to be the most aggressive, and specimens of the Type II cements more resistant to attack than the specimens of the Type I cement. Preliminary tests have indicated that reinforcing steel in concrete can be corroded by means of a direct electric current. The corrosion sometimes causes cracking of the concrete specimen.

KEY WORDS: Aggressive salts, cement properties, concrete properties, corrosion, electrochemical corrosion, mortar, reagents, and resonance frequency

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