

Resistance of Portland Cement Mortar to Chemical Attack—A Progress Report

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An extensive study to furnish information about the resistance of concrete to chemical attack by various inorganic and organic compounds has been undertaken using 1.5- by 1.5- by 10-cm mortar bars. Changes in length, weight and sonic modulus are measured periodically. The study is designed to determine relative resistance to chemical attack of various special type mortars ("microconcretes") at water-cement ratios representative of full-scale concretes. It also furnishes information which may help explain the mechanisms of attack by specific agents.

About 1,200 specimens have been placed under test; many have been exposed for more than 2 years. Most are still in satisfactory condition. Those failures which have occurred represent one of two main kinds of attack, or sometimes both. The first is removal of soluble compounds from cement by acid, sequestering agent, or exchangeable ion. The second is the deposition of new compounds within the paste. Rates of attack are compared and the nature of the attack by various groups of substances is discussed.

•MOST PORTLAND cement concrete is sufficiently resistant to its chemical environment not to suffer deterioration. Even when subjected to aggressive chemical exposure, much concrete is of sufficiently high quality to withstand some kinds of attack for substantial periods of time. The length of time that a particular quality of concrete can resist a specific chemical environment is sometimes of primary concern to the architect-engineer and to the maintenance man.

Several sources of information offer valuable guidance. Lea (1) devotes 83 pages to the discussion of resistance of concrete to organic and inorganic chemicals, natural waters, and sulfate soils. He relates performance of concrete to its physical and chemical properties. Kleinlogel (2) offers considerable information organized alphabetically for easy reference, drawing primarily on field experience. Biczok (3) gives perhaps the most extensive treatment of the relationship of attack to the properties of the concrete, citing the results of both laboratory investigations and field experience. Rabald (4) tabulates the resistance of concrete to a broad range of agents, giving resistance in four categories ranging from "practically resistant" to "unusable." Nelson (5) gives data for resistance of concrete to a similarly large group of agents. He classifies attack in terms of four categories of "corrosion rate," each representing a range of dimensional change, based on reports of many investigators. Since most of his materials are metals and other relatively nonporous materials, this change undoubtedly represents pitting or dissolving of the surface. For concrete and other relatively porous materials, however, it sometimes indicates expansion. Data for more than one concentration or for temperatures above 75 F are occasionally provided. Kleinlogel (2) gives only a limited number of references, although the original German edition gives many. Both Rabald (4) and Nelson (5) give bibliographic references but do not indicate specifically the sources of the individual values published. All of these

compilations offer extensive information in convenient form, and Lea, Kleinlogel and Biczok give helpful interpretive comment.

More detailed information can be obtained from primary sources giving detailed descriptions of field observations or laboratory tests on more limited subjects. Such reports have discussed measurements and results of attack by acids (6-14), by inorganic chemicals (6, 7, 9), by organic chemicals (8, 13-16), by sulfates, sulfate soils and sulfate waters (6, 7, 9, 17, 18, 19), by seawater (20-22), by river and groundwaters (20, 23-25), and by atmospheric gases (26-28).

When these and similar references have been searched, there may be unanswered questions. These questions may pertain to the severity with which a material attacks concrete or the existence of a limiting concentration below which, or even above which, attack will not occur. (Such information may be available for some materials; for example Locher and Pisters (25) have reported on the problem of safe concentrations.) There may be questions whether there is a type of cement which will not be attacked or if there is a water-cement ratio which will sufficiently reduce permeability and increase strength to make a concrete adequately resistant to a given agent. The effect of curing time may be of interest. There may be a need to select a less deleterious compound that can be substituted for the aggressive agent.

For a given chemical agent, some of these questions may be adequately answered, but this is likely to be the exception. Comparison of the rate or extent of chemical attack as a function of the identity of the chemical, the quality of the concrete, the type of cement, or some other variable may be adequate within a single published investigation. However, such comparisons are difficult among separate investigations because of the effects of different sizes of specimens, different proportions of concrete or mortar, different exposures, and different methods of evaluation.

CONCEPT AND PLAN

Nature of Present Study

There is, therefore, a need for a comprehensive study. Such a study should show not only the resistance of concrete to inorganic chemicals, organic chemicals and complex materials, but also broader comparisons of the effects of solution concentrations, cement type, water-cement ratio and cure time. The study could also help to form a new basis of comparison of the many existing reports at present only vaguely related to one another.

To fulfill this need for a comprehensive investigation, a long-time study of the resistance to chemical attack of portland cement mortar (proportioned as microconcrete) was undertaken. Chemicals, most of them inorganic, are added to the program as time and space allow. The program is intended to be open-end and to continue for an indefinite time. Some specimens have been under test for more than 2 years, with no definite termination yet scheduled, and some may be left under test for 5, 10 or more years.

The test method permits exposure of the same kind of specimen to all kinds of aggressive agents, regardless of the kind of attack anticipated. To provide adequate information, the study requires measurement of more than one physical property on the same specimen. If the reaction mechanism is not known, the variety of diagnostic evidence obtained from the several kinds of measurement is more likely to help explain the mechanism than the evidence from a single kind of measurement.

In addition, the test method is considered to be applicable to (a) conditions of high or low temperature, (b) cycling between wet and dry exposures, (c) cycling between two temperatures, or (d) exposure to gaseous agents. It may also be as readily adaptable to evaluation of protective coatings or other protective treatments as to a study of the substrate. (A limited number of tests at temperatures up to 200 F are reported here. Other tests not reported are in progress to evaluate the effect of surface treatment by silicon tetrafluoride gas to improve resistance to chemical attack.)

Substances to Be Studied

The study of resistance of portland cement concrete to chemical attack is potentially

the study of its resistance to attack by all other forms of matter, yet the existing knowledge of the properties of these forms permits elimination of large groups which are not aggressive. It would be impossible to study all of those remaining substances of possibly deleterious nature which might at some time come into contact with concrete. A judicious choice must be made so that the number of substances studied is small enough to fall within the scope of one investigation, but large enough to represent many broad classes of aggressive agents.

Accordingly, many of the substances in this study were chosen to be representative of a class of chemicals, one or more of which might be expected to come into contact with concrete; for example, sodium chromate was chosen to represent all chromate salts. Other compounds were chosen simply because they are known to come into frequent contact with concrete, even though they may belong to a class of compounds already represented. Some anions, such as chlorides, nitrates and sulfates, are represented in the salts of several metals. Most of the inorganic compounds were salts, but acids were also included and alkalis will be included later. When the intent was to determine the effect of the anion, the sodium salt was usually, but not always, the most convenient or appropriate salt for investigation. Substances known to be deleterious to concrete have been included whether they are complex materials or have simple and known chemical identities. Such complex materials as molasses, urine, blood, paper mill waste, and other naturally occurring, refined, or waste products will eventually be included. Two such materials already under test are linseed oil and seawater.

Specimen Size

Small mortar specimens were used to economize on storage space (Fig. 1), minimize the cost of chemicals (frequently replaced), simplify handling and measuring, and reduce the time required to attain significant effects. Prisms, 1.5- by 1.5- by 10-cm, which contain no reference points, similar to those used to determine sulfate resistance in Canadian Standard A5-1961 (29), were used. Measurements were made

of changes in length, weight, or Young's modulus of elasticity, all useful in quantitative evaluation of specimen exposure to the test solutions (6, 7, 8, 27, 28, 30).

TABLE I
CHARACTERISTICS OF CEMENTS USED

Composition	ASTM Type I	ASTM Type V	0-C ₃ A
SiO ₂ , %	20.9	24.4	20.9
Al ₂ O ₃ , %	5.7	3.1	2.8
Fe ₂ O ₃ , %	3.0	3.3	4.5
CaO, %	63.8	63.8	66.1
MgO, %	2.4	1.6	2.0
SO ₃ , %	2.6	1.4	3.0
Na ₂ O, %	0.25	0.08	0.08
K ₂ O, %	0.46	0.22	0.16
Loss on ignition, %	0.67	0.8	0.68
Insol. residue, %	0.09	0.09	0.03
Free CaO	0.18	0.5	0.85
Mn ₂ O ₃	0.28	0.09	0.12
Sp. gr. (kerosene)	3.20	3.210	3.179
Passing No. 325 sieve, %	91.04	98.0	-
Fineness, sq cm/gm ²	3,070	3,483	4,620
Compound composition:			
C ₂ S, %:			
Potential, calc.	51	41 ^b	76
Quantitative ^c	51.0	45.5	67.9
C ₃ S, %:			
Potential, calc.	21	39 ^b	2
Quantitative ^c	24.9	39.6	13.4
C ₃ A, %:			
Potential, calc.	9.9	3.7 ^b	0.0
Quantitative ^c	5.7	0.2	0.0
C ₄ AF, %:			
Potential, calc.	9.2	10.0 ^b	-
Quantitative ^c	10.3	9.0	10.0
Pot. ss C ₄ AF + C ₂ F,			
Calc. ^d %	-	-	13.5

^aAir permeability.

^bLTS-51, composition as reported by Lerch and Ford (34).

^cCalculated from x-ray diffraction data as described by Kantroet al. (35).

^dSolid solution of calcium aluminoferrite and calcium ferrite.

Qualities of Concrete to Be Studied by Means of Mortar

The choice of specimen size required the use of mortar instead of concrete. While the rate of attack on small specimens is high, because of their high surface-to-volume ratio, mortar specimens are nevertheless acceptable alternatives to concrete if they can be successfully made as microconcrete at water-cement ratios representative of full-scale concrete. Two water-cement ratios were chosen, 0.60 to represent paste in acceptable concrete of ordinary quality, and 0.40 to represent the paste in a high-quality concrete.

Since the primary concern was the resistance of the hydrated paste to chemical attack, silica sand, an aggregate immune to attack by most agents, was selected. The sand had the following

grading: passing No. 20 sieve, 97 to 100 percent; passing No. 30 sieve, 78 to 81 percent; passing No. 50 sieve, 34 to 40 percent; passing No. 100 sieve, 20 to 22 percent; and passing No. 200 sieve, 4 to 5 percent.

Three portland cements were used. The first was a Type I portland cement, the only one used at both water-cement ratios. Mortars of Type V portland cement were included in the study because of their low tricalcium aluminate (C_3A) content. Portland cements of low C_3A content are commonly used to reduce or prevent attack by some chemicals, notably sulfates, which react with C_3A . A portland cement containing no C_3A was also used. Mortars made with Type V and zero- C_3A cements were prepared at a water-cement ratio of 0.40. The chemical compositions of the three cements are given in Table 1, including both the potential compound compositions and the quantitative X-ray diffraction compositions.*

Specimens of each kind of mortar were cured for either 3 or 28 days before they were exposed to chemical attack. Mortar compositions and curing times of the eight different qualities of mortar tested are given in Table 2. For agents which attack C_3A in hydrated cement, the resistance of these mortars was expected to increase progressively from top to bottom of the table. Within each of the four major classifications, the mortars cured 28 days were expected to have better resistance than those cured 3 days.

Solution Concentrations

The more concentrated solutions can usually be expected to be more aggressive than dilute solutions. It was desirable to design the tests to be representative of the possible range of concentration for any given chemical. This range varies widely from one chemical to another. The solubility of sodium fluoride, for example, is

TABLE 2
COMPOSITION AND QUALITY OF MORTARS USED

Cement Type	C_3A (%)	W/C (by wt)	Cement/Agg. (by wt)	Moist Cure, 73 F (days)	Typical Dynamic Mod. ^a (psi $\times 10^6$)	Avg. Unit Wt. at End of Cure, (lb per sq ft)
I	9.9	0.60	0.25	3	1.6×10^6	140
	9.9	0.60	0.25	28	2.1	140
	9.9	0.40	0.40	3	2.2	144
	9.9	0.40	0.40	28	2.5	146
V	3.7	0.40	0.40	3	1.9	144
	3.7	0.40	0.40	28	2.5	145
0- C_3A	0	0.40	0.40	3	2.5	145
	0	0.40	0.40	28	2.7	146

^aAt end of cure.

*For the purposes of this report interpretations are based primarily on the potential compound compositions. This approach is justified by the existence of a large body of experience correlating potential compound composition with field performance. The quantitative X-ray diffraction data are presented as additional information. Interpretation of chemical resistance on the basis of these data is beyond the scope of this paper. As field experience develops, and supplements the data on resistance to chemical attack presented here, the quantitative X-ray data will be useful in making new correlations.

TABLE 3
COMPOSITION OF SYNTHETIC
SEAWATER SOLUTIONS

Ion	Weight (gm/1 solution)	
	Conc. Syn. Solution ^a	Syn. Solution ^b
Na ⁺	54.64	10.93
K ⁺	1.98	0.40
Mg ⁺⁺	6.72	1.34
Ca ⁺⁺	2.14	0.43
CO ₃ ⁻⁻	0.36	0.07
SO ₄ ⁻⁻	13.72	2.74
Cl ⁻	98.74	19.75
Br ⁻	<u>0.34</u>	<u>0.07</u>
Total	178.64	35.73

^aMade with following quantities of salt per liter of solution: NaCl, 138.9 gm; MgCl₂ · 6 H₂O, 41.50 gm; MgSO₄, 8.75 gm; CaSO₄, 6.45 gm; K₂SO₄, 4.00 gm; KBr, 0.50 gm; and CaCO₃, 0.60 gm.

^bEquivalent to the analysis given by Challenger Expedition (31), which also reported mean density to be 1.027 gm/ml.

discrepancies may result from differences in analytical techniques or may indicate differences in disproportionation in compounds which are not entirely stable.

Dilute solutions were prepared for the twofold purpose of (a) comparing low concentrations and (b) making this comparison on the basis of chemically equivalent concentration rather than equal weight. Solutions were prepared at 0.1 normal concentration, and normalities were calculated from potential ion exchange with respect to the anion, assuming no oxidation or reduction. The weights of solute used to make these solutions are given in Tables 9 through 11. Because some of the chemicals used were not of the highest purity, and because the solutions were not assayed nor adjusted in concentration, the "0.1 N" concentrations are only closely approximated.

Mineral acids, purchased as solutions, were tested at the concentrations purchased as well as at nominally 0.1 N.

Synthetic seawater was made to the composition (Table 3) reported by the Challenger Expedition (31), except for dissolved gases. The synthetic seawater was also used at 5 times normal concentration in exploratory tests at temperatures of 160 and 200 F, using 28-day-cured Type V mortar. Such results would help to evaluate the potential performance of concrete in evaporators for desalination of seawater.

The various organic chemicals to which mortar prisms are exposed are reported in Table 13 (Appendix). The concentrations chosen for study depend largely on the agent. Some are used on a 0.1 M basis; others are made at somewhat arbitrary concentrations, thought to be of interest, in water or in benzene. Some are liquids used without dilution or solids which can be made into solutions with negligible amounts of water.

Physical Conditions of Tests

Complete continuous immersion of the specimen, the easiest exposure to maintain,

about 42 gm/l of water; that of ammonium bisulfate is about 3,350. Therefore, it was desirable to study the upper end of the possible range of concentration for each chemical agent.

Saturated solutions were not used because of possible adherence of the crystals of the solute, which might have obscured observation of any new deposits formed on the prisms or the container walls during test and measurement. Solutions at 90 percent of saturation were used for the inorganic solids. These were conveniently prepared by dilution of 360 ml of stock saturated solutions with 40 ml of water before introduction into test containers. The amounts of solute in these solutions, calculated from published solubilities of the test chemicals, are listed to the right of the chemical names in Tables 9 through 11 of the Appendix. There is some uncertainty in such calculated values for two reasons:

1. Not all of the chemicals used were pure substances, and the presence of other dissolved substances affects solubility; and

2. Published solubility data are not always reliable and are sometimes in disagreement from one source to another;

was used in these tests. To date most tests have been conducted at room temperature. However, other conditions or combinations, such as partial immersion, intermittent immersion, or temperature cycles, may be included in subsequent tests.

SPECIMEN PREPARATION AND TESTING

Preparation and Exposure

Mortars were made in a room at 73 F and 50 percent R. H., following the mixing procedure specified in ASTM Method C 305-59T, but using a modified paddle with rubber wipers. Specimens were cast in two layers, tamped with a hard rubber tamper of $\frac{1}{2}$ - by 1-in. cross-section, cut off level, and lightly steel troweled. Although the zero-C₃A mortar had greater flow than the other two mortars at the same water-cement ratio, the unit weights determined on hardened prisms were about the same (Table 2). After being cured 1 day in the moist room, the prisms were stripped from the molds and capped on the ends with a neat paste of the same cement used in making the mortar. The caps served as the surfaces on which the anvil and the spindle of the length comparator bore when making measurements. Metal gage plugs were not used because of the possibility of corrosion. The capped specimens were returned to the moist room until the 3-day or 28-day curing was complete. At the end of the curing period, measurements were made of length, weight, and fundamental transverse frequency. The specimens were then placed in the test liquids.

The capping of prisms was abandoned in January 1965 because a significant number of caps had been lost during the course of the test, destroying the continuity of length change measurements. It was found that good measurements could be made on uncapped specimens. However, no data on uncapped specimens are included in this report.

Prisms were exposed to various chemical agents by storage of duplicate specimens from the same batch in 400 ml of liquid in a covered container. Specimens in aqueous inorganic solutions were stored in polystyrene boxes with internal polystyrene supports for the specimens (Fig. 1). A bead of vaseline was placed around the top edge of each plastic container to minimize evaporation and prevent creep of crystals onto the exterior surface. Specimens in organic liquids or hot liquids were stored in 16-oz wide-mouth clear flint glass bottles with plastic screw caps and paper or metal foil gaskets (Fig. 1).

The pH was determined for each inorganic solution at the start of test and occasionally at later ages. A glass electrode was used for most measurements, but an antimony electrode was used for sodium salts. Additional determinations were made with Hydrion paper; these did not always agree closely with the electrometric values. The initial pH for each solution is given in Tables 9 through 11 of the Appendix.

The 0.1N solutions were changed every 3 mo* and 90 percent saturated solutions every 6 mo, except as noted for acids. Organic liquids (other than acids) at concentrations greater than 100 gm/1,000 ml solvent were changed every 6 mo. Those at lower concentrations were changed every 3 mo.

Testing

After the initial measurements, periodic measurements of length, weight and fundamental transverse frequency were made at ages measured from time of immersion of 1, 3, 7, 14, 21, and 28 days. Measurements were then made monthly until age 6 mo and every 3 mo thereafter.

Length measurements were made in the comparator, readable to 0.01 mm, which conforms to Canadian Standard A5-1961 (29) (Fig. 2). Specimens were always placed in the comparator in the same way to avoid error. Weights were measured to the nearest 0.01 gm on the 120-gm capacity torsion balance also shown. Fundamental transverse frequency was determined by a procedure similar to that of ASTM Method

*For some solutions this was longer than required to exhaust the solution, but the period was adopted in the interest of uniformity of procedure.



Figure 1. Storage of specimens in limited space, using polystyrene boxes and glass bottles.

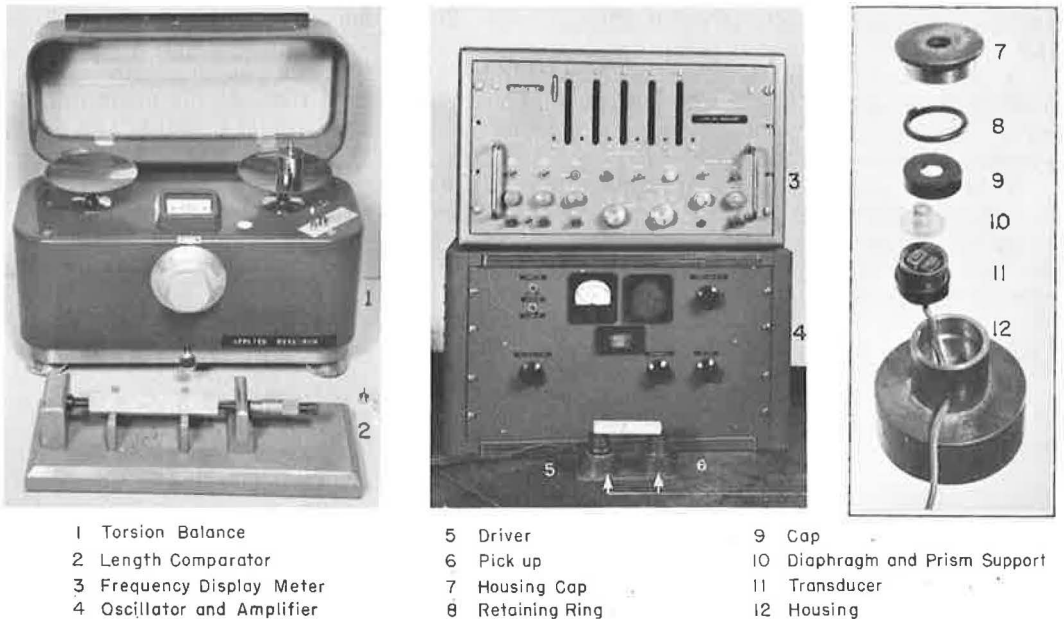


Figure 2. Equipment used for physical measurements.

C 215-60, except that the specimen is supported by the driver and pickup, positioned just below the two ends (Fig. 2).

The main body of test is given in the Appendix—for inorganic compounds in Tables 8 through 11, for seawater in Table 12, and for organic compounds in Tables 13 and 14.

NATURE OF TEST RESULTS

The test results may be used for at least partial diagnosis of the mechanism of the attack observed on the specimens. They may also serve to evaluate resistance to attack relative to other mortar compositions and qualities. In addition, they may be used to evaluate resistance to other chemicals and to another concentration of the same chemical.

Use in Diagnosis

Diagnosis depends on inferences which may be drawn from each of the measurements. The evidence from several measurements may provide a set of inferences which together are of considerable help in picturing the nature of the attack.

Increase in weight may mean that water is being absorbed by a specimen that is less than saturated. A larger increase may mean that the specimen is being penetrated by a liquid which is more dense than water and is being absorbed. It may mean that crystals are being formed within the specimen. These are usually more dense than the liquid and add more weight. If the crystals cause expansion they may open cracks, creating new paths for ingress of liquid and new space in which additional crystals can deposit and increase the weight further. Crystals sometimes also deposit on the surface and add to the weight but do not penetrate significantly. It is not always possible, however, to determine visually whether the crystals are on the surface only.

A decrease in weight may mean that water is being replaced by a less dense liquid. It usually means that hydrated cement compounds are being leached from the mortar and the binder material is dissolving. As this process progresses, sand grains, insoluble reaction products, or other debris may deposit on the bottom of the container.

An increase in length is likely to indicate deposition of crystals either on the surface of the prism or in the interior. When small cracks begin to open within the specimen, the process is likely to be accelerated.

A decrease in length is likely to mean that compounds are being leached from the cement. However, in the present study a large, abrupt decrease may have meant that one or both caps had broken free of the specimen.

An early increase in fundamental harmonic frequency usually means continued hydration of portland cement, and continued slow increase may indicate the same thing. An increase in fundamental harmonic frequency may also mean that crystals are being deposited within the specimen.

If continuing deposition of crystals within the specimen causes microcracking, the fundamental harmonic frequency will eventually decrease. When the rate of microcracking becomes high, the fundamental harmonic frequency will decrease rapidly. Another cause of decrease in fundamental harmonic frequency is the loss of binder or mortar by solution.

Use of Data for Evaluation

In many aqueous solutions, the specimens increased in strength during the early part of the test, as indicated by increase in dynamic modulus of elasticity. The dynamic modulus of elasticity is given by

$$E = CWN^2 \quad (1)$$

where E is the dynamic Young's modulus of elasticity, W is the weight of the specimen, N is the fundamental transverse frequency, and C is a constant which depends on the dimensions of the specimen and Poisson's ratio. It has the dimensions of strength and is related to it. Independently it is not a reliable index of strength, but it can indicate whether the strength of a given specimen is increasing or decreasing.

TABLE 4

CHANGE IN LENGTH AND WEIGHT OF PRISMS DURING STORAGE IN MAGNESIUM NITRATE SOLUTIONS COMPARED TO SATURATED LIMEWATER^a

Cement Type	W/C	Cure (days)	Length Change ^b (%)								Weight Change ^c (%)							
			3 Mo	6 Mo	9 Mo	12 Mo	15 Mo	18 Mo	21 Mo	24 Mo	3 Mo	6 Mo	9 Mo	12 Mo	15 Mo	18 Mo	21 Mo	24 Mo
(a) Immersion in 0.1 N Magnesium Nitrate																		
I	0.6	3	0.24	0.28	0.29	0.32	0.31	0.34	0.35	0.34	2.74	2.95	2.99	3.33	3.41	3.52	3.60	3.52
	0.6	28	-0.08	-0.08	-0.08	-0.10	-0.08	-0.06	-0.05	-0.06	0.65	1.27	1.46	1.54	1.77	1.92	2.04	2.23
	0.4	3	0.24	0.38	0.42	0.42	0.42	0.48	0.46	0.53	2.99	3.40	3.62	3.62	3.58	3.70	3.64	3.77
V	0.4	28	0.00	0.00	0.00	0.02	0.04	0.04	0.04	0.07	0.37	0.56	0.59	0.82	1.09	1.37	1.48	1.56
	0.4	3	0.30	0.34	0.41	0.46	0.50	0.50	0.51	0.50	4.26	4.79	5.15	5.13	5.17	5.34	5.39	—
	0.4	28	0.20	0.00	-0.01	-0.02	0.00	0.03	0.04	0.10	0.34	0.74	1.13	1.38	1.49	1.60	1.88	2.26
0-C ₃ A	0.4	3	0.12	0.22	0.24	0.27	0.26	0.40	0.29	0.26	2.41	3.28	3.83	3.76	4.01	—	4.12	4.16
	0.4	28	0.00	0.04	0.06	0.07	0.05	0.06	0.07	0.08	1.22	1.98	2.22	2.07	2.19	2.30	2.40	2.56
(b) Immersion in 90% Saturated Magnesium Nitrate																		
I	0.6	3	0.75	1.74	—	—	—	—	—	—	4.43	5.28	—	—	—	—	—	—
	0.6	28	-0.02	0.22	0.72	1.48	1.86	2.24	2.42	—	-0.04	2.85	3.54	4.25	4.67	4.75	4.17	—
	0.4	3	0.04	0.23	0.60	0.92	1.27	1.62	1.94	—	0.15	1.59	2.31	3.15	3.66	4.21	3.71	—
V	0.4	28	0.02	0.10	0.45	1.02	1.37	1.67	2.16	2.84	0.56	0.74	1.64	2.53	3.67	4.09	4.39	4.84
	0.4	3	0.05	0.10	0.18	0.28	0.37	0.42	0.48	0.45	0.57	2.01	2.99	4.43	4.88	5.22	5.42	4.75
	0.4	28	0.00	0.10	0.22	0.34	0.40	0.48	0.11	0.16	0.42	2.08	2.59	3.09	3.25	3.13	10.66	7.42
0-C ₃ A	0.4	3	0.09	0.40	0.62	1.02	1.57	2.08	2.56	—	0.61	2.12	2.12	2.86	3.92	—	5.56	8.40
	0.4	28	0.09	0.32	0.46	0.84	1.21	1.95	2.88	—	1.73	2.18	2.13	2.73	3.20	3.20	3.18	—
(c) Immersion in Saturated Limewater																		
I	0.6	3	0.02	0.00	0.00	0.00	0.02	-0.02	-0.01	-0.01	1.16	1.36	1.56	1.80	1.92	1.98	2.04	2.18
	0.6	28	0.00	-0.01	-0.02	-0.02	—	-0.02	-0.02	-0.04	0.88	1.18	1.45	1.36	—	1.89	1.96	1.91
	0.4	3	0.02	0.00	0.00	-0.02	-0.04	-0.06	-0.03	-0.03	1.44	1.52	1.56	1.64	1.74	1.84	1.76	1.97
V	0.4	28	0.01	0.00	0.00	0.00	—	-0.01	0.00	0.00	0.78	0.96	1.30	1.16	—	1.31	1.36	1.35
	0.4	3	0.00	0.00	0.00	0.00	-0.02	-0.01	0.00	-0.01	0.84	1.06	1.16	1.21	1.30	1.30	1.33	1.36
	0.4	28	0.00	-0.01	-0.01	—	-0.03	-0.03	-0.03	-0.03	0.20	0.44	0.56	—	0.67	0.73	0.72	0.71
0-C ₃ A	0.4	3	0.02	0.02	-0.01	0.00	-0.02	-0.02	0.00	0.00	0.47	0.56	0.48	0.42	0.16	0.40	0.54	0.62
	0.4	28	0.02	0.02	0.02	0.05	—	0.02	0.02	0.02	0.24	0.10	0.34	0.51	—	0.55	0.54	0.55

^aEach value average of two prisms.^bAll values positive (expansion) unless otherwise indicated.^cAll values positive (increase in weight) unless otherwise indicated.

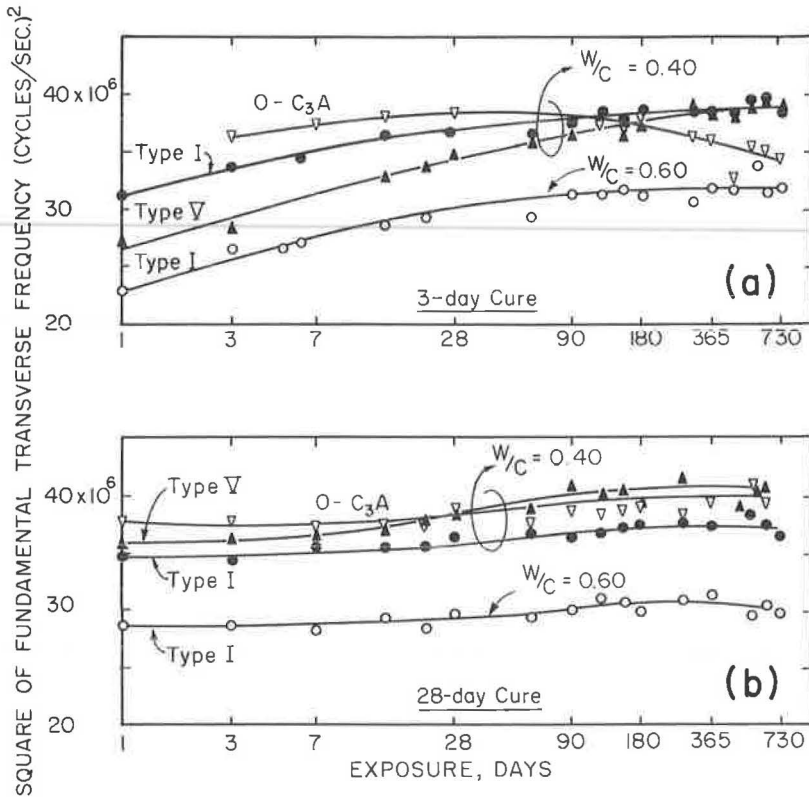


Figure 3. Effect on dynamic modulus of prisms of 2-yr immersion in saturated limewater (modulus expressed as square of fundamental transverse frequency).

TABLE 5
COMPARISON OF POSSIBLE CRITERIA OF FAILURE BASED
ON LOSS OF SONIC MODULUS^a

Criterion	Time to Failure (days)					
	Type I, W/C 0.60		Type I, W/C 0.40		O-C ₃ A, W/C 0.40	
	3 Days	28 Days	3 Days	28 Days	3 Days	28 Days
Loss of 50% of initial modulus	100	260	620	550	560	320
Decrease in modulus						
To 20×10^6 cycles ² /sec ²	18	190	450	500	280	350
To 18×10^6 cycles ² /sec ²	26	220	520	520	350	420
To 15×10^6 cycles ² /sec ²	42	270	660	610	500	540
To 10×10^6 cycles ² /sec ²	92	375	—	—	—	—

^aExpressed in terms of square of fundamental frequency; specimens stored in 90% saturated magnesium nitrate solution.

In these tests, the increase was attributed to continuing cement hydration. Specimens cured only 3 days before storage in the test solutions showed greater increase in strength, indicating more continuing hydration than those cured 28 days. The rate of change in saturated limewater is shown in Figure 3. Most of the mortars stored in saturated limewater increased considerably in modulus and, hence, in strength. Specimens cured 28 days and then stored in saturated limewater gained strength more slowly (Fig. 3b) Table 2 gives typical values of initial dynamic modulus of each of these mortars.

Changes in length and weight of these same specimens were not large during the first 24 mo. Data for 15, 21 and 24 mo are given at the ends of Tables 11, 10, and 9, respectively.

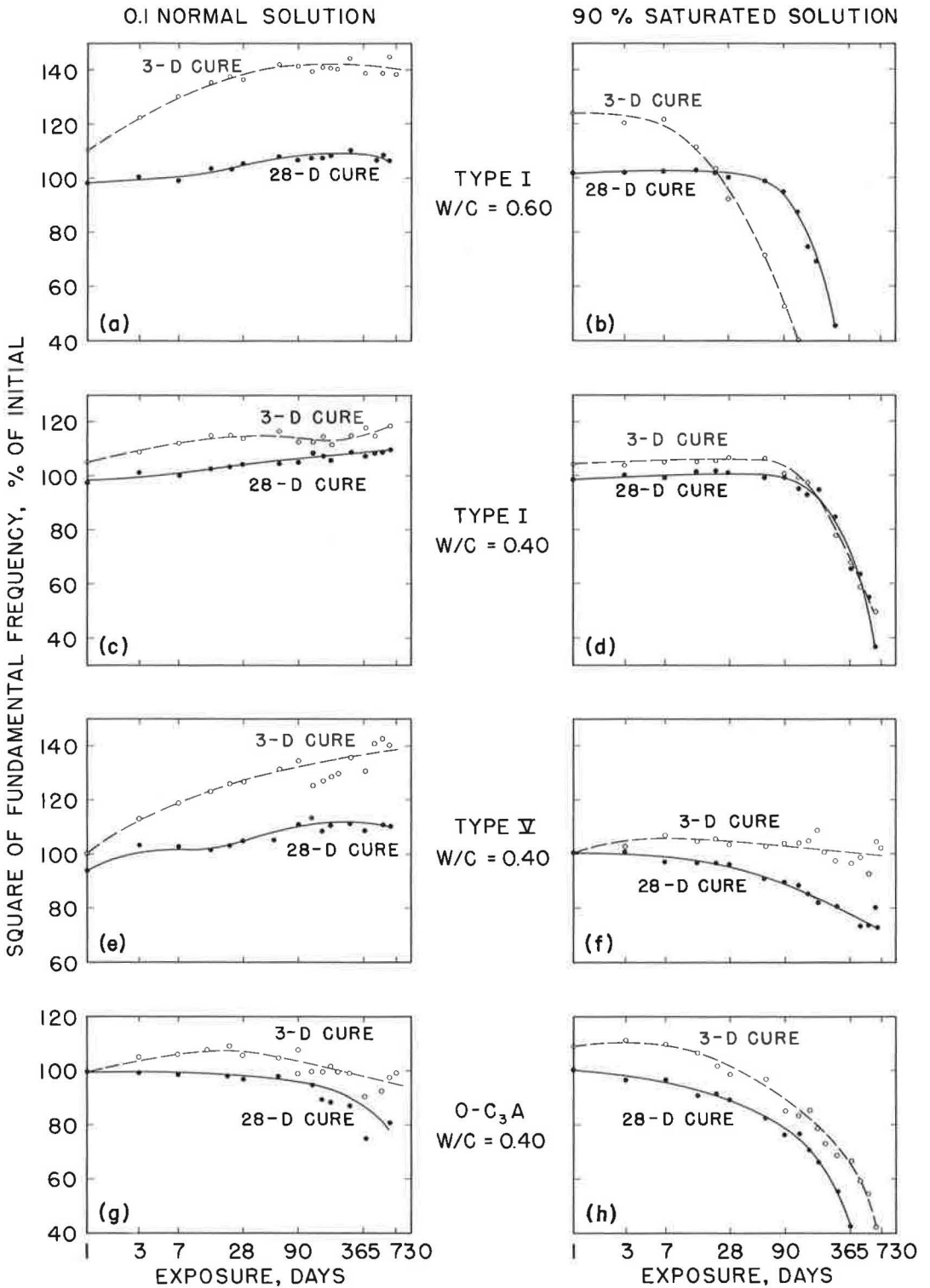


Figure 4. Change in modulus of prisms used to determine rate of deterioration (specimens in magnesium nitrate solutions).

Criteria of Failure.—The criteria of failure selected were a change in length of ± 0.20 percent, a change in weight of ± 10 percent, or a loss in sonic modulus of 50 percent. The change in sonic modulus may usually be calculated with sufficient accuracy by comparing the squares of the fundamental transverse frequency. For additional information, the tests were continued beyond the point of failure indicated by any single criterion.

Change in Length and Weight.—For some chemicals, measurements of change in length are demonstrably useful in revealing considerable differences among the various mortars in the two-solution concentrations. For example, data for specimens immersed in magnesium nitrate solutions for periods up to 24 mo are given in Table 4. Several reached the arbitrarily chosen criterion of failure at early ages, and the magnitudes vary considerably at any age. Changes in weight of the mortars in the same solutions during the same period also differentiated among them (Table 4).

Change in Dynamic Modulus.—The criterion of failure in terms of dynamic modulus was chosen to be a reduction of modulus to 50 percent of initial (expressed as the square of the fundamental frequency, as previously noted). This criterion, commonly used in durability studies, gives an indication of how far microcracking has progressed.

An alternative criterion could have been adopted, setting failure in terms of some absolute value of modulus. Any of the various possible end points, such as those given in the last four lines of Table 5, might have been chosen. Of these, a decrease in the square of the fundamental frequency to 15×10^6 (or a decrease in the frequency to 3,800, rounding to the next lower value) might seem to be the most suitable. However, this criterion could easily result in equating the strengths of two mortars, one having high initial strength and much ultimate microcracking and the other having low initial strength but also little microcracking. The criterion would put more emphasis on residual strength than on integrity of the specimen. Consequently, this criterion was not chosen for this program.

In using the criterion which was chosen, however, the reader should be aware of its limitations. He could be misled while trying to draw conclusions about the relative strengths of mortars of different initial quality. For example, in looking at Figure 4h, he would note that the mortar cured 3 days did not reach the failure criterion until about 560 days, compared to only 320 days for 28-day-cured mortar, as also recorded in Table 5. He would next observe that the curve for the mortar cured 3 days was higher; in the absence of additional information he might conclude that this mortar had higher strength throughout the test.

A different impression is obtained when the moduli are compared as absolute values, rather than as percentages of initial. Such a comparison has been made for the same specimens in Figure 5d. Here the reader would draw a different impression; i. e., that the mortar cured 28 days was stronger at all ages shown. Figures 5a, b, and c also convey impressions somewhat different from Figures 4b, d, and f, which are based, respectively, on the same original data.

Thus, the reader must be careful not to infer too much about the strengths of prisms from the squares of fundamental frequency, N^2 , which are given in terms of percent of initial. These are recorded in Tables 9 through 12 and 14. He may find it possible, however, to make very rough comparisons between mortars by using the absolute values of N , in cycles per second, which are recorded in the same tables.

There are 16 sets of test data for any given inorganic chemical, representing 8 mortars (Table 2), each immersed in two different concentrations of solution. Examples of the manner in which these sonic data were utilized are the 16 typical curves for solutions of magnesium nitrate shown in Figure 4. These curves should be studied in conjunction with Figure 3, which shows the effect of control specimens immersed in limewater where chemical attack is presumably not involved.

Most of the specimens immersed in 0.1N solutions of magnesium nitrate gained in strength; those cured 28 days gained at a rate about equivalent to that of similar specimens in saturated limewater. The specimens in 90 percent saturated solutions of magnesium nitrate gained little or no strength and eventually weakened.

The test was effective in sorting out the relative resistances of eight mortars in magnesium nitrate solutions of two different concentrations. The poorest quality of

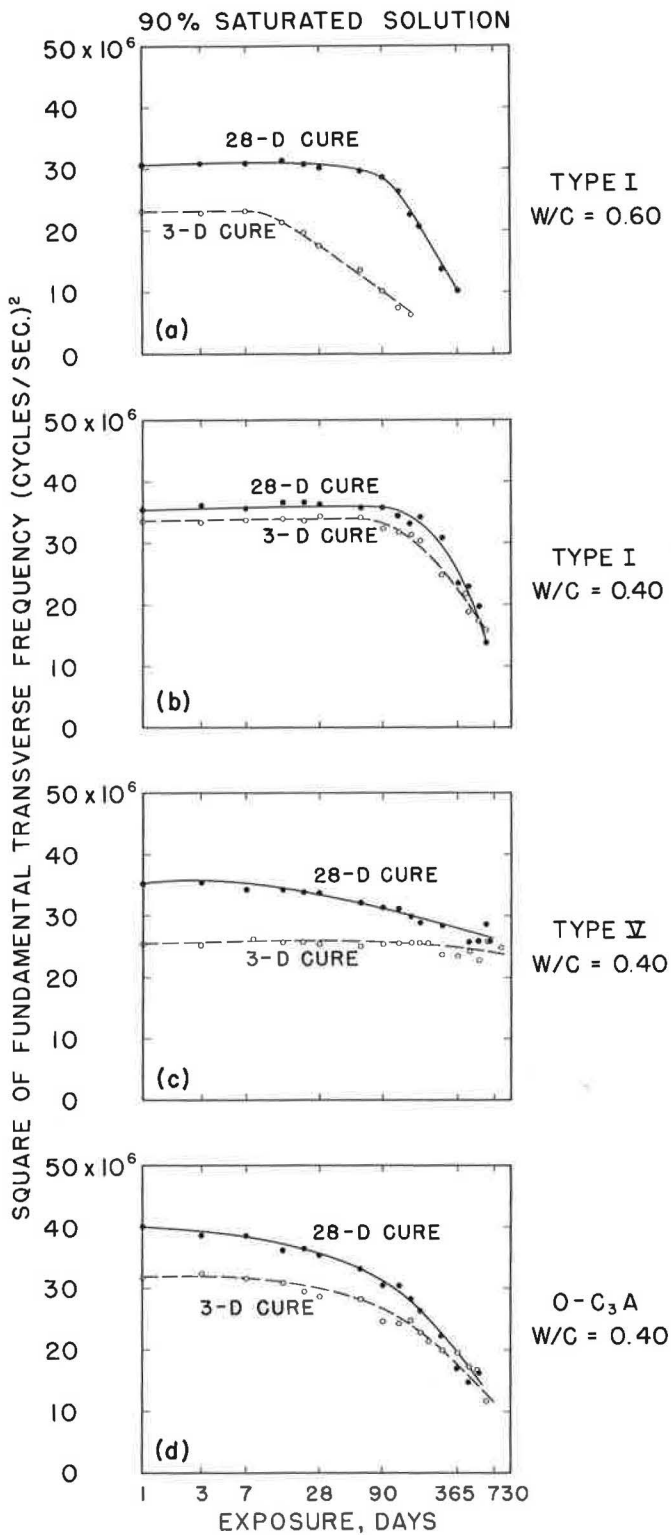


Figure 5. Modulus of prisms compared to that of other specimens forms measure of relative quality (specimens in magnesium nitrate).

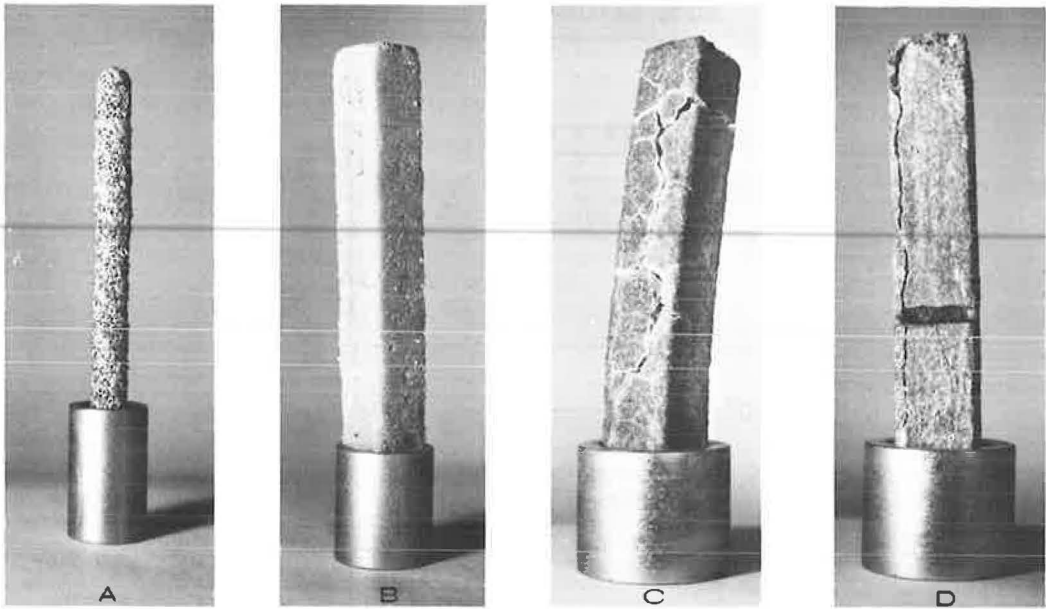


Figure 6. Examples of various kinds of deterioration.

TABLE 6
COMPARISON OF RATE OF pH CHANGE
WITH CONCENTRATION

Solution	Concentration (gm/l)	pH		Elapsed Time (mo)
		Init.	Later	
Sodium bisulfate:				
0.1 N	6.00	1.4	9.6	2.6
90% sat.	387	0.0	0.5	2.8
Potassium persulfate:				
0.1 N	13.57	1.8	9.0	2.6
90% sat.	180	1.6	8.9	2.7
Sodium dichromate:				
0.1 N	14.90	3.9	9.4	2.8
90% sat.	1,845	3.2	4.9	2.8

mortar had failed in 90 percent saturated solution after 3 mo (Table 10). At the other extreme, neither of the Type V mortars had failed by this criterion even after 2 yr (3 mo later than the comparative data given in Table 10), although they had weakened. There were indications of strength loss, or lack of strength gain, among mortars in 0.1 N solutions, and some at least are expected to fail eventually.

Degree of Concordance Between Specimens

For each exposure the two companion specimens were made from the same batch of mortar. Individual specimens of each pair were in fair agreement with each other initially. Of all the specimens used in the program, 98 percent of the pairs had differences in initial fundamental frequency of 300 or less. At failure the agreement in

fundamental frequency between specimens tended to spread out greatly, so that only 75 percent of them had differences of 300 or less. This is understandable, since the values were decreasing rapidly in the period just preceding failure. When the age at failure by the criterion of dynamic modulus was calculated, 80 percent of those which failed by dissolving had differences in age at failure of 30 days or less. Among those which failed by expansion the differences in age at failure were considerably greater.

In general, on a percentage basis, agreement between specimens with respect to change in length or weight was as good as with respect to change in fundamental frequency, or better.

Visual Evidence of Deterioration

In badly deteriorated specimens, several kinds of change in outward appearance may be seen. Several typical examples are shown in Figure 6. Specimen A shows the kind of attack that may be observed with acids. In specimen B, much hydrated portland cement has been dissolved away by acid, leaving loosely bound silica sand in almost the original shape and size of the specimen. The expansion cracking and warping seen in specimen C are typical of sulfate attack. Expansion sometimes causes cracking parallel to each of the 12 edges of the prism, as shown in specimen D, which has been cut in two for examination.

RESISTANCE TO ATTACK BY INORGANIC SUBSTANCES

The following descriptions of resistance to attack are based on chemical classification; discussions of acids, hydroxides, various cations and anions, and seawater are taken up in alphabetic sequence. Quantitative comparisons, if significant, are made between types of cement, water-cement ratios, amount of cure of the mortar, concentration of the solution, or kinds of ions. Where possible, comments are made on the mechanism of attack as understood from the literature or as inferred from the tests and supplementary observations described here.

Acids and Acidity

Acids attack concrete by dissolving both hydrated and unhydrated cement compounds, as well as carbonate aggregate. Such aggregate was not used in these tests. Many of the salts tested are also acidic through either ionization or hydrolysis, or both, and have pH below 7.0, as indicated in Tables 8-10. Many such salts thus tend to react like acids and dissolve concrete.

Acidity is commonly expressed in terms of pH; values progressively lower than 7 represent progressively higher acidity, and progressively higher potential for reaction with concrete. Yet pH values by themselves must be interpreted with caution. The extent of reaction will depend upon the total amount of acid present, and not simply on pH. For example, a dilute, strong acid might have the same pH as a concentrated, weak acid, and an equal volume of the two might consequently attack concrete at the same initial rate. However, since there is less of the strong acid present, it would not react to the same extent, and would not finally dissolve as much concrete.

During the reaction, the change in pH may be large. In the tests reported here, two test specimens totaling either about 18 or 28 gm of cement before hydration would, when even partially hydrated, impart a pH of 12 or more to 400 ml of water containing no solute, or react ultimately with roughly 0.4 or 0.7 gm-eq. wt of acid. Consequently, for dilute solutions, including some 90 percent saturated solutions of low concentration, the pH rose rapidly, in some instances above 7.0. For example, a Type I cement mortar at 0.4 water-cement ratio cured 3 days was immersed in 0.1 N ammonium chloride solution. After 1.3 mo immersion, the pH had changed from 5.6 to 10.2. The concentration was initially 5.35 gm/l.

Solutions of higher concentration usually changed pH more slowly than those of low concentration, as indicated by Table 6. The test was performed in contact with static liquids; flowing liquids in the same concentrations would be expected to maintain lower pH, as though the test solution were being continually renewed. In these tests the

effect of pH is obscured to varying degrees by the changing concentration of the solution.

However, no simple correlation was found, as indicated by Figure 7, between the rate of change in weight of the mortar and the initial pH of the solution. In this figure the change in weight between 3 and 28 days is plotted against the initial pH for a Type V cement mortar made at a water-cement ratio of 0.4 and cured 3 days. The distribution was similarly diffuse for the remaining mortars. The rate of weight loss was not predictable on the basis of pH alone, no matter whether any particular group of solutions is considered or all are considered together. Yet it may be noted that, among these compounds, when very large losses did occur it was when the initial pH was 5.3 or lower.

A limited number of tests have been completed using mineral acids; results are reported in Table 8 (Appendix) on specimens made with Type I cement. The rapid rate of reaction quickly depleted the acid in 0.1N solutions, and for the first 3 wk, these solutions were renewed daily except on weekends. They were renewed weekly for 2 wk and less frequently thereafter. The data indicated a fairly constant loss of weight per change of 0.1N solution.

Specimens failed in 0.1N hydrochloric acid and 0.1N nitric acid after about the same amount of exposure, but attack by 0.1N sulfuric or phosphoric acid was considerably slower.

The concentrated mineral acid solutions were not renewed during the period of test, which was usually brief. The first measurements of the specimens after immersion were made at 6 hr; failures earlier than 6 hr were estimated by interpolation. Failures came early in concentrated hydrochloric and nitric acids. Concentrated phosphoric acid caused early failure as judged by expansion, but did not cause so rapid decline in strength as hydrochloric and nitric acids. Concentrated sulfuric acid was least aggressive by all criteria, probably because of the very small amount of water present. When an amount of water was present equivalent to that in solutions of other acids (note the 0.1N solutions), the sulfuric acid was slightly more aggressive than the phosphoric acid. Calcium sulfate and calcium orthophosphate, reaction products, have such limited solubilities that they undoubtedly deposit on the surface and reduce the rate of attack of sulfuric and phosphoric acids.

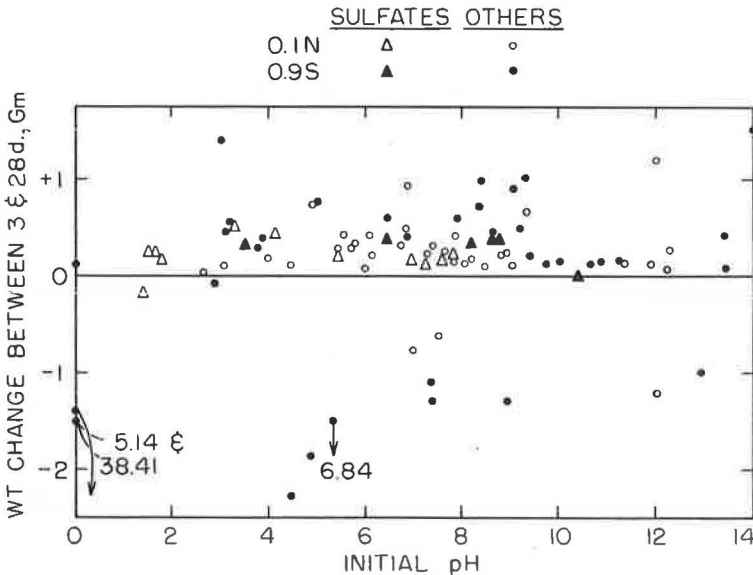


Figure 7. No correlation found between initial pH of solutions and weight loss of mortar bars in test used (Type V cement, W/C = 0.40, 28-day cure).

Aluminates

Sodium aluminate is a highly soluble aluminate of high alkalinity. The pH of the 0.1 N solution before immersing specimens in it was 12.3. Changes in specimens immersed in 0.1 N solutions were not large after 24 mo (Table 9). In the saturated solution, specimens made with Type I cement at a water-cement ratio of 0.60 and cured 28 days increased in length and considerably in weight, presumably because of crystal buildup on the prisms. The crystals were identified by X-ray analysis as gibbsite, a form of hydrated alumina.

Ammonium Salts

Many ammonium salts attack concrete. Water dissolves calcium, sodium and potassium hydroxides from hydrated portland cement. In the presence of these hydroxyl ions, ammonia gas is released. The calcium, sodium and potassium then replace it in solution, allowing more of the hydroxides to dissolve (1, 3).

The effects of specific ammonium salts are discussed in the sections dealing with the anions.

Borates

Sodium perborate has a relatively low solubility and produces alkaline solutions. No serious changes in length or weight were measured during a period of 24 mo, and all mortars gained strength during immersion (Table 9). Sodium tetraborate (borax) also has a relatively low solubility and produces slightly less alkaline solutions. All of the mortars gained in length and weight, some of them substantially, during 24 mo of immersion. However, most of the change was perhaps caused by the deposition of crystals on the ends and sides of the prisms. Almost all gained in strength during 24 mo.

Carbonates and Bicarbonates

Neither the so-called ammonium carbonate solution nor the ammonium bicarbonate had caused distress in any of the specimens up to age 21 mo. This result may be related to the very limited solubilities of calcium carbonate and bicarbonate. Not all mortars are yet under test (Table 10). The changes in length, weight, and sonic modulus of any given mortar were roughly comparable, whether the mortar was immersed in ammonium carbonate, ammonium bicarbonate, sodium carbonate, or sodium bicarbonate.

Ammonium carbonate solutions have an ambiguous nature (thought to be an equimolar mixture of NH_4HCO_3 and $\text{NH}_4\text{CO}_2\text{NH}_2$) and are known to change composition with age. The solution used in these tests was made from "ammonium carbonate" with a minimum ammonia content of 33 percent. After being stirred for several hours, the saturated solution contained only about 360 gm of salt per liter, though the salt is said to have a solubility of 1,000 gm/l at 59 F. However, ammonia volatilized throughout the stirring operation, so the solution was deficient in ammonia (molar ratio of ammonia to carbon dioxide analyzed at 1.93) and higher in ammonium bicarbonate content. Ammonium bicarbonate is only soluble to the extent of 212 gm/l, so the production of this salt would tend to drive salt out of solution.

It seems likely that the so-called 90 percent saturated ammonium carbonate solution used in these tests began at a maximum concentration of about 0.90×360 gm/l or less. During the test the solution would lose more ammonia and partially convert further to the bicarbonate, reducing the solubility further toward the limiting value of 212 gm/l, the estimated solubility of ammonium bicarbonate.

Chlorates

Several mortars failed by the length criterion in 90 percent saturated sodium chlorate (Table 9) at relatively early ages. Two failed in 0.1 N solution also. No failures were observed in the perchlorate, which was considerably more concentrated in the

90 percent saturated solution. The specimens, left under test, still presented a good visual appearance after 24 mo. The changes in length, weight, and sonic modulus during 24 mo are given in Table 9. Some specimens suffered loss of modulus in 90 percent sodium chlorate; the losses were smaller in sodium perchlorate. The Type V specimens cured 3 days were in the best condition with respect to sonic modulus in 90 percent saturated solutions of both salts.

Chlorides and Other Halides

Data are available only on specimens cured 3 days. After 15 mo of immersion in ammonium chloride, all specimens had lost weight significantly and had undergone large changes in sonic modulus (Table 11). Specimens of all four kinds of mortar failed by the modulus criterion in 3 to 12 days when immersed in 90 percent saturated solution. Failures in some of the same specimens were indicated at later ages by other criteria. In 0.1 N solution, the zero-C₃A mortar had failed by the modulus criterion after 6.5 mo. Others were approaching failure at 15 mo.

Specimens were in generally good condition after 15 mo in sodium chloride solutions (Table 11), although failure by increase in length was indicated after 12.5 mo for the Type I cement at 0.4 water-cement ratio in 90 percent saturated solution. The test is continuing.

Sodium bromide can be more deleterious. Its solubility is higher than that of sodium chloride. In 90 percent saturated solutions, the Type I portland cement mortars suffered severe loss in modulus, large increase in length, and considerable increase in weight; unexpectedly, the Type I mortar with the lower water-cement ratio was the more severely affected of the two, and failed earlier (Table 11). The zero-C₃A cement mortar had suffered considerable loss in strength by the end of 15 mo. The Type V cement was still in good condition, though the square of the frequency had decreased to 106 percent of initial from the maximum of 120 percent reached at 3 mo. Specimen D of Figure 6 was disrupted by sodium bromide.

The 90 percent saturated solution of sodium fluoride was much less aggressive than that of sodium bromide, as one might expect from its much higher pH and lower concentration and the precipitation of calcium fluoride. Biczok (3) says that sodium fluoride hardens concrete by changing lime to the hard calcium fluoride. It nevertheless caused some reduction in modulus in the Type I mortar made at 0.6 water-cement ratio and in the zero-C₃A mortar (Table 11).

Of the cements tested, Type V appeared to be the most resistant to 90 percent saturated solutions of sodium halides. All cements withstood 0.1 N solutions quite well during the first 15 mo of test.

Chromates and Dichromates

All specimens tested were in good condition after 15 mo of immersion in the sodium chromate solutions. However, most of those in sodium dichromate were not (Table 11). In 0.1 N solutions, all specimens suffered severe loss in modulus, and most of them failed before the 15 mo had elapsed. The initial pH of the solution had been 3.9. Although this increased to between 8 and 10 during continued immersion, the losses in weight recorded for most specimens indicated that there had been a considerable amount of cement dissolved.

In 90 percent saturated solutions of sodium dichromate, the losses in sonic modulus and in weight were less than in the dilute solutions, even though the pH rose only to 4 or 5 during test. However, the high concentration of the solution, 1,845 gm/l, may have reduced the solubility of the reaction products. At intermediate concentrations the solution might be considerably more destructive. Biczok (3) notes that potassium dichromate is highly aggressive.

Ferrocyanides

Among the specimens tested for a period of 21 mo, none was significantly damaged by the sodium ferrocyanide solutions. Data are given in Table 10.

Fluosilicates

All specimens immersed in magnesium fluosilicate had good strength after a period of 15 mo, even though most of those in 90 percent saturated solutions had undergone a slight loss in weight. The small weight loss perhaps represents some dissolving of cement hydration products, since the 90 percent saturated solution is strongly acidic. The reaction products, however, are compounds of very low solubility—calcium fluoride, magnesium fluoride and silica. These are generally believed to deposit in the pores, making the hydrated cement harder and less permeable. Magnesium and zinc fluosilicates have long been used for this purpose.

Hydroxides

Any limited volume of water in contact with hydrated portland cement soon becomes saturated with calcium hydroxide, because there is more than enough calcium hydroxide present to exceed its very low solubility. When solutions of distilled water in contact with the specimens were not disturbed, the calcium hydroxide diffused slowly enough so that the total solution was less than half saturated after a week. However, measurements of pH indicate that in the vicinity of the specimens the solutions probably became saturated with calcium hydroxide within about a day.

Saturated calcium hydroxide (saturated limewater) was accordingly used as the control for the tests in this program; the data are given in Table 4, and in Tables 9, 10, and 11 of the Appendix, and are plotted in Figure 3. This is the only hydroxide which has yet been tested in this program.

Solutions of sodium and potassium hydroxide at high concentrations are reported to be deleterious to concrete (1, 3), depending on its quality. These will be investigated as the program continues.

Magnesium Salts

Magnesium salts tested included the nitrate (Tables 4 and 10, and Figures 4 and 5), the fluosilicates and the sulfate (Table 11). Most magnesium salts are destructive to concrete. They leach out calcium hydroxide to form the soluble calcium salt and deposit magnesium hydroxide, which is far less soluble than calcium hydroxide. They are also said to react with the other cement hydration products to form such compounds as magnesium hydroxide, soluble calcium salts, silica and alumina; the latter two possibly react with the magnesium hydroxide to form hydrated magnesium silicates and magnesium aluminate (1, 25, 36, 37).

The 90 percent saturated magnesium nitrate solution had the highest magnesium concentration of the three magnesium salts tested:

Mg in $\text{Mg}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O} = 109 \text{ gm/l}$;
 Mg in $\text{MgSO}_4 = 63 \text{ gm/l}$; and
 Mg in $\text{MgSiF}_6 = 53 \text{ gm/l}$.

This may be one reason that magnesium nitrate was the most destructive. Length changes at 15 mo caused by magnesium nitrate solutions may be found in Table 4 and compared with those caused by fluosilicate and sulfate and recorded in Table 11. Type V cement was more resistant to increase in length when immersed in magnesium nitrate than the other cements. Magnesium fluosilicate had not caused any distress by the end of 15 mo; its hardening effect has been previously discussed under fluosilicates.

When magnesium nitrate and magnesium sulfate are compared on the basis of equivalent concentrations, in 0.1 N solutions, the differences are not nearly as great. (The 15-mo data from Table 4 may again be compared with the data of Table 11.) However, the length changes of all specimens cured only 3 days are excessive when immersed in magnesium nitrate for 15 mo, but the Type V cement and zero-C₃A cement specimens cured 3 days were still in good condition after 15 mo in magnesium sulfate.

Manganates

Potassium permanganate (Table 11) was the only salt studied in this group. No

distress has been shown during 15 mo of immersion by any of the specimens tested, although not all specimens have yet been put under test.

Molybdates

The specimens made with Type I cement at a water-cement ratio of 0.60 and cured 28 days had gained 10.60 percent in weight after 15 mo of immersion in 90 percent saturated sodium molybdate of specific gravity 1.395 (Table 11). It seems likely that some kind of solid has been deposited within the prisms. For these particular prisms, the increase was 5.48 gm per prism. If there were no solids deposited and no change in volume of the prism, this weight change would have required replacement of 13.9 ml of water in the prism by an equal amount of the denser solution. However, 13.9 ml is far more than the total amount of liquid which can be accommodated by the prism, as approximately equal to the volume of mixing water (5.6 ml). The specific gravity of sodium molybdate salt, however, is 2.59. The observed weight increase would require replacement of only 3.44 ml of water per prism by salt crystals. The deposition of solids thus comes much closer to satisfying the volume limitations of the material which has caused the weight increase. All of the specimens immersed in 90 percent saturated sodium molybdate had considerably increased in weight. The zero-C₃A specimens increased the least.

None of the specimens in any of the solutions underwent excessive length change in 15 mo. All had fundamental sonic frequencies higher than the original. The Type I specimens with water-cement ratio of 0.60, cured 28 days, began to decline in sonic frequency after this time, however, whether they were immersed in 0.1N or 90 percent saturated solutions.

Nitrates and Nitrites

Of the nitrates, ammonium nitrate was by far the most destructive. No specimens withstood the attack of 90 percent saturated ammonium nitrate for more than about 2 wk, and many of them failed much earlier (Table 10). Even a 0.1N solution was destructive to most specimens; only the Type V specimens cured 3 days survived after 21 mo. The rapidity of the attack by ammonium nitrate can be attributed to the ammonium cation.

Magnesium nitrate is next to ammonium nitrate in severity of attack. Here again the cation (magnesium) is responsible. Although the pH of the 90 percent saturated solution was initially only 3.8, and although a large amount of salt was present (1,152 gm/l of water), the pH did not remain low. When measured after one set of Type I specimens had been immersed for 3 wk, the pH had risen to 7.5; similar measurements were obtained on solutions containing other mortars. Specimens suffered from changes in weight, length and sonic modulus. The weight changes of all specimens were positive (Table 10), indicating that if there was any acid attack it was less significant than deposition or absorption of additional substances in the mortar.

The Type V cement was the most resistant to attack by 90 percent saturated magnesium nitrate, although it does not sustain continuous attack indefinitely (Table 10, Fig. 4f). The zero-C₃A cement mortar was less resistant to attack in this solution than Type I cement made at the same water-cement ratio. In 0.1N solutions, all specimens cured only 3 days had excessive increases in length during 21 mo of immersion, but all specimens cured 28 days still had acceptable length changes after 21 mo. Figures 4a and g indicate that two of the mortars may have been starting to deteriorate in 0.1N solution, although they were still in good condition at that time.

In some specimens immersed in 90 percent saturated solutions of potassium or sodium nitrates, excessive expansions were measured; one was 0.86 percent at 21 mo (Table 10). In potassium nitrate, acceptable expansions were measured only with mortars made of Type V cement; in sodium nitrate, expansions were acceptably low in mortars made with either Type V or zero-C₃A cement. The reasons for the excessive expansions are not yet known. A qualitative test of the stock saturated solution of sodium nitrate showed that the solution contained a small amount of sulfate. However, 90 percent saturated potassium nitrate, which produced more expansion in every instance than sodium nitrate, contained essentially no sulfate.

The apparent expansion and increase in weight of specimens immersed in lead nitrate may have been only the heavy crystal buildup on the surfaces of the specimens. The X-ray diffraction pattern of this precipitate indicated the possible presence of lead silicates, lead aluminosilicate, lead phosphates, and lead sulfates. Qualitative analysis shows that there has been some ion exchange, which can only mean that the hardened cement paste has been altered.

Most specimens in sodium nitrite appeared to be in good condition after 21 mo immersion, although the sonic modulus was beginning to decrease in some (Table 10). Two specimens indicated excessive expansion; this was probably because the caps had begun to crack and were perhaps coming loose.

Phosphates

Monobasic sodium orthophosphate was the only one of the three orthophosphates which had damaged specimens seriously by the end of 15 mo. The deterioration occurred only in specimens immersed in 90 percent saturated solutions. The acidity of these solutions remained strong, and the pH typically rose no higher than 3.6 during 6 mo of immersion. Visual examination showed that all specimens in 90 percent saturated solutions had become white from deposition of insoluble reaction products and had been eroded along the edges. Caps also became whitened, eroded along the edges, and expanded at the center. Measured length changes, usually large (Table 11), may have been partially the result of these changes in the caps. At later ages, longitudinal cracks formed along the edges and caused failure. No failures had occurred by age 15 mo, but sonic moduli were decreasing significantly, as indicated by the value of N^2 in Table 11. Specimens made of zero- C_3A cement had larger increases in weight than other cements.

Specimens in sodium hexametaphosphate gave evidence of slight surface erosion, although they were in good condition as far as changes in length, weight and sonic modulus indicated (Table 11). Specimens in sodium pyrophosphate, however, were in good condition in all respects.

Seawater

Mather (32) has discussed the mechanism of deterioration of concrete by seawater, which may involve attack by sulfate, chloride, carbonate, bicarbonate, alkali metal and magnesium ions. The actions of some of these ions are discussed individually elsewhere in this paper. The condition of specimens of the various mortars after immersion for 6 mo in synthetic seawater is given in Table 12 of the Appendix. Type I specimens and zero- C_3A specimens cured 3 days had already exceeded the expansion limit of 0.20 percent before 6 mo had elapsed, and the time required for failure is given in the table. The mortar made with Type V cement was superior to the others in resisting seawater attack.

The Type V mortar cured 3 days was also tested at temperatures of 160 and 200 F in solutions of synthetic seawater and of seawater at 5 times this concentration (Table 12). All such specimens were in good condition after 6 mo.

Stannates

Only one stannate, sodium stannate, was tested; this produced highly alkaline solutions (Table 11) and did not harm the mortars during 15 mo of immersion. Some gained a considerable amount of weight.

Sulfates and Bisulfates

Sulfate Attack.—The destructive action of sulfates on concrete is primarily the result of their reaction with either C_3A or the C_3A hydration products to form the high-sulfate form of calcium sulfoaluminate (ettringite, $C_3A \cdot 3CaSO_4 \cdot 32H_2O$). The crystalline reaction product is of larger volume than the original aluminate constituent, and expansion results. The concrete or mortar increases in compressive strength at first, because of the increase in solid matter, even though it is changing chemically. As the process continues the concrete or mortar expands, cracks, becomes progres-

TABLE 7
ADDITIONAL IONS PRESENT IN
SULFATE SOLUTIONS

Solution	Addition Ion	
	0.1 N	90 Percent Sat.
Sodium bisulfate	H ⁺ ^a	H ⁺ ^a
Ammonium sulfate	NH ₄ ⁺ , H ⁺ ^b	NH ₄ ⁺ , H ⁺ ^b
Ammonium bisulfate	NH ₄ ⁺ , H ⁺ ^a	NH ₄ ⁺ ^c , H ⁺
Copper sulfate	H ⁺	H ⁺
Manganese sulfate	H ⁺ ^d	H ⁺
Magnesium sulfate	Mg ⁺⁺	Mg ⁺⁺

^aAcidity did not persist, since pH rose to more than 9 during test.

^bAcidity did not persist, since pH quickly rose to more than 7.

^cAmmonium ion was probably not aggressive at low pH of solution, which remained at a measured value of 0.

^dAcidity did not persist, since pH rose above 7 in about 1 mo.

in Table 7. Hydrogen ion was present in copper and manganese sulfates because of hydrolysis. Both the 0.1 N and 90 percent saturated solutions of copper sulfate remained distinctly acid throughout the test, but only the 90 percent saturated manganese sulfate remained acid, as indicated in Table 7. The 90 percent saturated ammonium bisulfate, which had the unusually high concentration of 3,015 gm/l (Table 11), remained at a measured pH of 0 throughout the test, whereas the 0.1 N solutions rose to pH values between 9 and 10. Ammonium sulfate solutions, both 0.1 N and 90 percent saturated, were initially acidic (Table 11) but quickly rose in pH.

Sodium bisulfate had an unusually low initial pH (Table 11) which remained persistently low in the 90 percent saturated solutions throughout the test, never rising above 1. The 0.1 N solutions, however, reached pH values of about 10. In the 90 percent saturated solutions one would naturally expect the dissolving action of the acid to open the structure to rapid penetration and attack by sulfate. The dissolving action was the predominant mode of attack. Deterioration by sodium bisulfate was faster than by any other sulfate salt tested. All mortars tested failed in a very short time in the 90 percent saturated solution, and none lasted longer than 7 mo in 0.1 N solution.

Ammonium Sulfates.—Ammonium sulfate and bisulfate solutions were highly aggressive, causing failures in all mortars tested whether immersed in 0.1 N or 90 percent saturated solutions (Table 11). In 90 percent saturated solutions of ammonium bisulfate, acid attack predominated over sulfate attack much like sodium bisulfate, and caused failures almost as rapidly with all of the mortars tested. Specimen A of Figure 6 shows a specimen which failed in less than a month in ammonium bisulfate but which was photographed after 15 months storage in the original solution. In 0.1 N solution the nature of the attack differed somewhat because the rise in pH from 1.6 to more than 9 had the effect of replacing acid attack by ammonium ion attack. The passage of calcium ions from the hydrated cement into solution to replace ammonium ions which were breaking down by volatilization of ammonia (see Ammonium Salts) would make the cement more porous and more subject to attack by sulfate ions. All of the mortars tested succumbed to this attack by 0.1 N solution (Table 11). Type V cement and zero-C₃A cement were not immune.

The action of ammonium sulfate was similar, except that the hydrogen ion concentration in the 90 percent saturated solution was initially lower and decreased more quickly. Thus, ammonium ion was the effective agent, with sulfate ion, in both the 90 percent saturated solution and the 0.1 N solution and produced more solid matter of a different kind in the bottom of the container. The rate of attack by ammonium sulfate was not greatly different from that of the bisulfate; apparently it was faster for some mortars and slower for others.

sively weaker, and finally disintegrates. Specimen C of Figure 6 has undergone sulfate attack.

Sodium Sulfate.—Sulfate attack is well illustrated by the effect of 90 percent saturated sodium sulfate, which caused early failure of Type I cement mortars by one or more of the three criteria (Table 11). Of all the mortars tested, Type V cement mortar was most resistant to attack. None of the mortars yet tested in 0.1 N solutions had failed by the end of 15 mo, but even in such a dilute solution the Type I cement mortar made at 0.40 water-cement ratio and cured 3 days had undergone a serious loss in dynamic modulus.

Association of Other Aggressive Ions with Sulfate.—In each of the other sulfate solutions tested, there was an additional aggressive ion or two present, as indicated

Copper Sulfate.—Copper sulfate remained acidic even in the 0.1 N solutions, where the pH rose to less than 5. Here the deposition of calcium sulfate on the surface perhaps slowed the rate of attack by acid and prevented the depletion of a small amount of acid present. Prisms had relatively uneroded surfaces with sharp edges after 15 mo. Sulfate attack proceeded, however, as indicated by the increases in weight and length and, in one set of specimens (Table 11), serious decrease in dynamic modulus of elasticity.

Acid attack is obvious in the weight losses and erosion of specimens in the 90 percent saturated solutions. However, these changes were accompanied by internal deposits formed by sulfate action, as evidenced by the expansions of all specimens and the decrease in dynamic modulus of many of them.

Type V cement mortars appeared to be the most resistant of all the mortars tested in both 0.1 N and 90 percent saturated solutions of copper sulfate, but they also failed.

Manganese Sulfate.—Manganese sulfate is more acidic than copper sulfate, as indicated by the initial pH (Table 11), but during a $\frac{1}{2}$ -mo immersion of specimens in 0.1 N solutions, the pH rose above 7. The action seems to be much like that of copper sulfate, with some differences in magnitude of changes in length, weight and dynamic modulus of elasticity. Specimens made with Type V cement were still resistant after 15 mo, when all others had failed.

In the 90 percent saturated solutions of manganese sulfate, however, acid attack was far more pronounced than in copper sulfate, as shown by the considerably larger weight losses. The initial pH was lower than that of copper sulfate (Table 11), but the final pH was higher (about 6 for manganese sulfate compared to about 3.5 for copper sulfate). This indicates more depletion of hydrogen ion, perhaps through more extensive chemical reaction. The changes in specimen length were always negative, showing the predominance of acid attack over sulfate attack at this concentration.

Magnesium Sulfate.—At the equivalent concentrations represented by the 0.1 N solutions, magnesium sulfate was more severe than sodium sulfate in its attack on specimens of all kinds (Table 11). The difference is perhaps in the aggressive effect of magnesium ion (see Magnesium Salts). Magnesium sulfate was less aggressive than the salts of ammonia, copper and manganese and the acid salts of ammonia and sodium, discussed previously. In 0.1 N solutions, only the Type I cement mortar made at a water-cement ratio of 0.60 and cured 3 days suffered changes in length and dynamic modulus at all comparable to those in solutions of the other cations. The Type I mortar at a water-cement ratio of 0.40 cured 3 days suffered failure by the expansion criterion in 14.5 mo. All other specimens were in satisfactory condition after 15 mo.

In 90 percent saturated solutions, magnesium sulfate was less aggressive than sodium sulfate, although the concentration was higher in the magnesium sulfate solution and although two kinds of aggressive ions were present in the solution. Some specimens in magnesium sulfate increased in weight because of the deposition of new crystalline material within them. As the process proceeded, however, debris began to slough off and deposit on the bottom of the container, causing losses in weight to the prisms (Table 11). Type V cement did not appear to be noticeably more resistant than Type I in this solution.

Other Sulfur-Containing Anions

Sodium sulfide (Table 11) was the only sulfide tested. The solutions were highly alkaline in both concentrations, and no deterioration of the mortar was measured. For a period of 15 mo, in the absence of oxidizing agents, sodium sulfide appears to have been harmless.

In 0.1 N solution, sodium sulfite was at least as aggressive to the mortars tested as sodium sulfate of the same concentration, or more so (Table 11). Sodium bisulfite appeared to be more aggressive to some mortars than sodium bisulfate but not to others. All mortars withstood 0.1 N sodium bisulfite for 15 mo except Type I cement mortar made at a water-cement ratio of 0.60 and cured 3 days; this failed in expansion in 9.5 mo. Of those tested in sodium bisulfite, only Type V cement mortar cured 3 days survived 15 mo.

Both Type V and zero-C₃A cement mortars were resistant for 15 mo to attack by 90 percent saturated sodium sulfite (Table 11). Failures occurred in all Type I cement mortars except the one made at 0.40 water-cement ratio cured 3 days, which, for unknown reasons, far outlasted the mortar cured 28 days.

Sodium bisulfite in 90 percent saturated solution was less concentrated, but more acidic and more destructive than sodium sulfite, and failures occurred earlier.

Sodium thiosulfate was about as aggressive in 0.1 N solution as in 90 percent saturated (Table 11). Failures were indicated by excessive changes in length. Type V cement mortar and zero-C₃A cement mortar were resistant to expansion, but in the 0.1 N solution, the zero-C₃A cement mortar cured 3 days failed by expansion in 10 mo. The mortar of ostensibly lowest quality, on the other hand, with Type I cement at 0.60 water-cement ratio cured 3 days did not fail in 15 mo in 0.1 N solution in these tests.

In 0.1 N solution potassium persulfate caused large expansions and failures in those specimens made of Type I cement mortar (Specimen C, Fig. 6), but not of either the Type V or zero-C₃A (Table 11). It was highly destructive in 90 percent saturated solutions, causing failures in Type V and zero-C₃A mortars as well as Type I, though at later ages. The solutions were strongly acid initially, but the pH rose to about 9 or 10 in both the 0.1 N and 90 percent saturated solutions during test.

RESISTANCE TO ATTACK BY ORGANIC SUBSTANCES

The tests conducted so far with organic substances have been limited to mortars made with Type I cement, at water-cement ratios of 0.60 and 0.40, moist cured 28 days. The other mortars will be tested as the program continues. Data are available only up to 6 mo of immersion.

Acids

One schedule for renewal of organic acid solutions was planned for those less concentrated than 100 gm/l, and another for more concentrated solutions. Both schedules were interrupted, but the history of each pair of specimens may be seen in a general way from the number of solution changes and the length of exposure recorded in Table 13 in the Appendix.

The four acids tested were concentrated lactic acid, 0.1 M lactic acid, a mixture containing 2 percent lactic plus 2 percent acetic acid in a single solution and a solution of 200 gm stearic acid per liter of benzene. Specimen B, of Figure 6 is shown after 3 mo of immersion in the lactic-acetic acid mixture, which had been renewed twice during that time. All four solutions were highly destructive (Table 13), including the two in contact with very little water. One of these was concentrated lactic acid which contained only sufficient water to liquefy, and the other was stearic acid dissolved in benzene with no water present. The prisms themselves, however, contained some absorbed water initially.

Alcohols

Specimens were immersed in methyl alcohol, tertiary butyl alcohol, ethylene glycol and glycerol made to the concentrations given in Table 14. The changes in weight and length which occurred in 6 mo were acceptably low at all concentrations tested. Strength appeared to be diminishing in the concentrated liquids at 6 mo, but the specimens were still in good condition at that time.

Amino Acid Salts

The disodium salt of ethylenediamine tetraacetic acid (EDTA) was dissolved in water to concentrations of 34.42 gm/l of solution (0.1 M), and 125 gm/l of water. EDTA is a sequestering agent for calcium, and the solutions used had initial pH values of about 6.

As with the organic acids, the schedule for renewal of solutions was interrupted, but the number of exposures to fresh solution and the total exposure time are given in Table 13. As indicated in the table, EDTA is as aggressive as a weak acid.

The more concentrated solution was changed monthly, like the more concentrated acid solutions, and the total elapsed time before failure was recorded. The time to failure recorded by this method was somewhat longer.

Drying Oils

Specimens were immersed in a pure grade of boiled linseed oil, undiluted. After 6 mo of immersion, the specimens were still in good condition (Table 4).

Esters

Specimens immersed in undiluted butyl stearate were in good condition after 6 mo (Table 4), as were specimens immersed in a solution of 30 gm technical grade glyceryl tristearate per liter of benzene (Table 14).

Hydrocarbons

Benzene caused no great change in the specimens in 6 mo (Table 14).

Sugars

Sucrose was made to two concentrations (Table 14), neither of which damaged the specimens in 6 mo.

RELATIVE RESISTANCE OF THE MORTARS

Type V cement mortar was more resistant to sulfate attack than either Type I or zero-C₃A, as indicated by its greater durability in sodium sulfate, sodium sulfite, and sodium thiosulfate. However, in acid salts or other salts with low pH, such as ammonium bisulfate, sodium bisulfate, copper sulfate, manganous sulfate, and potassium persulfate, Type V was not significantly more resistant, if at all. Nor was it very resistant in ammonium sulfate or magnesium sulfate, salts in which the cation is also aggressive.

The resistance of zero-C₃A cement mortar was inferior to that of Type V with respect to many of the chemicals studied so far. Insofar as this observation is dependent on the change in dynamic modulus, it may partly reflect the lower modulus of the zero-C₃A cement as measured at later ages (Fig. 3), particularly for specimens cured 3 days. In addition to the absence of potential C₃A, the zero-C₃A cement also differed from the Type V cement in that it contained considerably more C₃S, a factor which may influence chemical resistance.

Type I cement mortar made with a water-cement ratio of 0.40 was usually more resistant than that made with a water-cement ratio of 0.60. Longer cure time usually increased resistance. Exceptions to either of these observations may have been the result of experimental error.

PRODUCTS OF ATTACK

Efforts have been made to detect and identify products of reaction between hydrated portland cement and some of the chemicals studied in this investigation. Petrographic examination showed that various foreign crystalline materials were present in the interior of some mortar bars. These were usually so finely crystallized, so small in quantity, or so embedded in other materials that they could not be positively identified. Amorphous materials were sometimes seen. X-ray diffraction measurements on powders or on polished surfaces from the mortar bars also did not lead to positive identification.

However, some auxiliary tests were performed which produced some helpful information. The Type I cement was hydrated at a water-cement ratio of 2.0 in a polyethylene bottle while agitating for 7 days by keeping the bottle clamped to a rotating wheel. The agitation continually exposes fresh surface and permits the hydration to approach completion in this period of time without binding the products into a mass. After 7 days, the excess water was filtered off and the solids were vacuum dried and bottled.

Samples of this powder were then weighed and exposed to solutions in plastic boxes in approximately the same proportions involved when mortar bars were immersed. Additional samples were exposed to solutions in polyethylene bottles stoppered to exclude carbon dioxide. The purpose was to increase the amount of surface area immediately accessible to the solution, in the hope that if any reaction occurred it might go more nearly to completion than in the prisms, and produce larger identifiable crystals. Solutions used were 90 percent saturated sodium chlorate, lead nitrate, potassium nitrate, sodium nitrate, sodium nitrite, sodium bromide, sodium chloride, and sodium fluoride. After a month or more of exposure, samples withdrawn from the top surface of the solid layer were examined by X-ray diffraction. At the present time only some general conclusions can be drawn.

After exposure to the solutions, the hydrated pastes no longer showed the presence of calcium sulfate, but sulfate was present in each of the solutions, showing that calcium sulfate had come out of the solid into solution. However, there was evidence of new compounds formed by reaction with the various solutions. These were deposited in the paste and appeared to be bromoaluminates, chloroaluminates, or nitroaluminates, depending on the solution used. Several kinds of anions are known to combine with calcium aluminate (1), possibly producing compounds which cause expansion.

Calcium fluoroaluminate did not form by reaction with the sodium fluoride solution; calcium fluoride, which was identified, has a low solubility and forms preferentially. The reaction of lead nitrate with paste is perhaps also more complex than reaction with other nitrates; the sample had many diffraction peaks.

There was no X-ray evidence of addition compounds with calcium silicate hydrates, but evidence from other work indicates that these hydrates can take up quantities of other anions without noticeable modification of the broad peaks of their diffraction patterns. Hence, it is uncertain whether compounds other than calcium aluminate hydrate also react with aggressive solutions to form compounds which may cause expansion. It should be noted that the zero-C₃A mortar underwent some expansion when in contact with sodium chlorate, lead nitrate, and potassium nitrate (Tables 9 and 10), but it is not known whether this expansion was caused by formation and deposition of new compounds.

SUMMARY

The resistance of portland cement concrete to chemical attack is being studied by using small mortar (microconcrete) specimens immersed in aggressive liquids and measuring changes in length, weight and dynamic modulus of elasticity. The specimens are 1.5- by 1.5- by 10-cm mortar prisms. The mortar is made with well-graded fine silica sand, which is (for all purposes of the study) inert to the chemicals to be studied. Mortars were made to water-cement ratios such as those which are attainable in concrete so that the hydrated paste is representative of the paste fraction of concrete in permeability and strength.

Because the method uses three measurements—length, weight and fundamental harmonic frequency—it provides more information for explaining the mechanism of attack than if only one or two measurements were used. When it is necessary to draw additional conclusions about the mechanism of attack, these measurements can be supplemented by chemical, X-ray, and petrographic studies of the deteriorated mortars, solutions, precipitates, and residues.

The study confirms the observations of other investigators about some liquids that are detrimental or may be detrimental to concrete. Among these are seawater and solutions of ammonium salts, magnesium salts, organic and inorganic acids, sulfates, sulfites, thiosulfate, and salts producing low pH. Some other chemicals have also been found to be detrimental to some concretes at rates of attack that depend on exposure conditions. These include solutions of lead nitrate, potassium nitrate, sodium bromide, sodium dichromate, sodium chlorate, and EDTA (ethylenediamine tetraacetic acid, disodium salt—a sequestering agent for calcium). During the reported times of exposure, other chemicals have appeared to be less aggressive. More information about these will be obtained as the tests continue.

Resistance of mortar was increased by longer cure time and by decrease in water-cement ratio. The Type V cement mortar was more resistant to sulfate attack than the other mortars, but not to acidic sulfates or those which contained ammonium or magnesium. The zero-C₃A cement mortar was generally lower in resistance to chemical attack than Type V.

The rate of attack may be directly related to the activity of the aggressive ion. Solutions of high concentration were generally more aggressive than those of low concentration. In sulfuric acid, however, a Type I cement mortar had only about the same resistance in a solution of 9.81 gm/l, replenished 5 times a week, as in a solution of 960 gm/kg which was not replenished.

Another striking exception to the increase in rate of attack with increase in concentration of the aggressive chemical was shown consistently in the data for sodium dichromate solutions. Solutions containing 14.90 gm/l of solution were more deleterious than those containing 1,845 gm/l of solvent.

The rate of attack may sometimes be affected by the solubility of the reaction products in the particular concentration of the aggressive solution. For example, the weight loss from the mortar after each change of 0.1 N sulfuric acid was not sufficient to provide enough calcium sulfate to saturate the solution. This would prevent calcium sulfate from depositing on the prisms. The solubility of calcium sulfate in the concentrated acid solution is much lower, however, and was exceeded within a few hours. Furthermore, this solution was not renewed and for almost the entire period of immersion in concentrated H₂SO₄, calcium sulfate was being deposited on the surfaces of the prisms, impeding the reactions.

Mortars of Type V cement immersed in hot seawater for 6 mo appeared to be more resistant to attack than those immersed in seawater at 73 F. The same may be true for mortars immersed in seawater at 5 times its natural concentration. Ettringite crystals apparently do not form at the temperatures utilized, 160 and 200 F; therefore, in this exposure the increase in temperature appears to have been beneficial. Freedom from formation of ettringite and consequent expansion may be of considerable importance to the future use of concrete in this kind of application.

It is well known that lowering of pH decreases the resistance of mortar by increasing the rate of solution. However, the chemical composition of an aggressive liquid was at least as important as pH in influencing the rate at which a mortar prism lost weight. When the pH of many chemicals was compared with weight loss of mortar bars, no correlation was found.

ACKNOWLEDGMENT

The author wishes to acknowledge the assistance of Borje Ost, of these laboratories, in calculating and compiling most of the data in Tables 9 through 14.

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Appendix

DATA FROM EXPOSURE TESTS

Explanatory Notes

Following each name and chemical formula in the tables are letters in parentheses to indicate the grade (purity) of chemical used according to the following code:

ACS = American Chemical Society,

NF = National Formulary,

P = Purified,

PI = Pure Industrial,

Pr = Practical,

R = Reagent,

T = Technical, and

USP = U. S. Pharmacopoeia.

Data indicating concentrations of solutions are indicated as shown below. The initial pH, where applicable, follows immediately after the solution concentration.

Table	Kind of Solution	Units Used to Express Concentration	Location of Concentration Data with Respect to Chemical Name
8	0.1 N	Grams per liter of solution	Below, left
8	Concentrated	Grams per kilogram of solution	Below, right
9-11	0.1 N	Grams per liter of solution	Left
9-11	90% saturated	Grams per liter of water	Right
13, 14	0.1 M	Grams per liter of solution	Below, left
13, 14	Intermediate concentrations	Grams per liter of solvent	Below, center
13, 14	Concentrated liquid	Grams per liter of solvent (except as noted)	Below, right

TABLE 8
AGES AT FAILURE IN MINERAL ACIDS

All specimens were made with Type I cement, with W/C = 0.60, cured 3 days. The 0.1-normal solutions were renewed 5 times per week for three weeks, weekly for two weeks, and less frequently thereafter.

The concentrated solutions were used as received and the concentration recorded below is the average given on the label of the bottle. These were not renewed during the period of test.

Three criteria of failure are used:

$$\Delta L = \pm 0.20\%$$

$$\Delta W = \pm 10.0\%$$

$$N^2 = 50\% \text{ of initial}$$

Criterion	Time to Failure by Various Criteria		
	0.1 N Solutions		Concentrated Solutions
	No. of Fresh Solutions	Total Exposure Time	Total Exposure Time
	3.65 g./l.	Hydrochloric Acid, HCl (ACS)	370 g./kg.
ΔL	2	3d.	2h.
ΔW	9	14d.	- ^a
N^2	10	15d.	5h.
	6.30 g./l.	Nitric Acid, HNO ₃ (ACS)	700 g./kg.
ΔL	3	4d.	2h. ^a
ΔW	9	14d.	- ^a
N^2	11	16d.	5h.
	9.80 g./l.	Phosphoric Acid, H ₃ PO ₄ (ACS)	850 g./kg.
ΔL	22	127d.	6h.
ΔW	20	106d.	1.5d.
N^2	- ^a	- ^a	15d.
	9.81 g./l.	Sulfuric Acid, H ₂ SO ₄ (ACS)	960 g./kg.
ΔL	10 ^b	15d. ^b	24h. ^c
ΔW	15	39d.	18d.
N^2	16	56d.	- ^a

^aDiscontinued before failure was indicated by this criterion.

^bOne specimen only. Increase in length.

^cDecrease in length.

TABLE 9
CONDITION OF SPECIMENS STORED IN INORGANIC AQUEOUS SOLUTIONS FOR
24 MONTHS

The change in length (ΔL), the change in weight (ΔW), and the square of the frequency (N^2) are all expressed as percent of initial readings.

Criteria of failure are:

$$\begin{aligned}\Delta L &= + 0.20\% \\ \Delta W &= + 10.0\% \\ N^2 &= 50\% \text{ of initial}\end{aligned}$$

Ages at failure are interpolated to nearest 0.1 month up to 3 months and nearest 0.5 month above 3 months.

Note: Where no failure has occurred for a particular agent, the "age at failure" lines have been omitted.

Cement	In 0.1 Normal Solutions								In 90% Saturated Solutions											
	W/C=0.6		Type I		W/C = 0.4		Type V		O-C ₃ A		W/C=0.6		Type I		W/C = 0.4		Type V		O-C ₃ A	
	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.
ALUMINATE:	8.20 g./l., pH = 12.30								Sodium Aluminate NaAlO ₂ , (T)								1156 g./l., pH = 14.15			
ΔL	+0.8	-.06	+0.3	-.06	-.03	-.12	+0.4	-.03		+0.96 _a									+0.34 _a	
ΔW	+2.3	+0.9	+1.6	+1.6	+3.7 ^u	+0.7	+1.6	+1.1												
N^2	154	115	136	112	151	112	112	104		63										129
N, cycles/sec.	5750	6010	6330	6310	6240	6280	6380	6480		4190										6890
Age at Failure, Mo.	ΔL	-	-	-	-	-	-	-		4.5 _a										11.0 _a
	ΔW	-	-	-	-	-	-	-												
	N^2	-	-	-	-	-	-	-												
BORATE:	9.98 g./l., pH = 10.43								Sodium Perborate, NaBO ₃ .H ₂ O, (T)								40 g./l., pH = 10.78			
ΔL	+0.6	-.09	+0.8	+0.4	+0.2	-.06	+0.2	.00	+0.8	-.05	.00	-.04	.00	-.04	-.02	-.02				
ΔW	+1.1	+0.1	+1.1	+0.4	+0.8	+0.5	+0.8	+0.5	+1.5	+0.2	+1.2	+0.4	+1.2	+0.7	+1.0	+0.5				
N^2	150	110	140	106	150	115	116	106	164	130	136	108	150	116	156	112				
N, cycles/sec.	5790	5780	6370	6330	6320	6340	6460	6640	5950	5920	6380	6230	6420	6340	6550	6100				

19.07 g./l., pH = 9.22					Sodium Tetraborate, Na ₂ B ₄ O ₇ ·10H ₂ O, (NF)							44 g./l., pH = 9.39					
ΔL	+4.2 ^b	+3.4 ^b	+3.3 ^{b,c}	+4.8 ^b	+3.6 ^b	+1.4 ^b	+1.6 ^b	+0.9 ^b	+0.8 ^b	+1.0 ^b	+1.0 ^b	+1.1 ^b	+0.8 ^b	+1.16 ^b	+1.0 ^b	+1.2 ^b	
ΔW	+8.2	+10.2 ^a	+6.6 ^c	+5.2	+6.6	+8.1	+5.5	+7	+6.7	+10.2	+4.2	+4.5	+4.6	+4.5	+4.4	+4.8	
N ²	155	97	124 ^c	106	148	107	105	98	171	112	135	113	157	113	115	118	
N, cycles/sec.	5720	5280	5960 ^c	6140	6100	5760	6220	6080	6140	5620	6460	6410	6470	6080	6420	6450	
Age at	ΔL	1.6 ^b	4.5 ^b	-	4.5 ^b	8 ^b	7.5 ^b	-	-	-	-	-	-	-	-	-	
Failure	ΔW	-	22.5 ^a	-	-	-	-	-	-	-	-	-	-	-	-	-	
Mo.	N ²	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
CHLORATE:		10.64 g./l., pH = 7.35					Sodium Chlorate, NaClO ₃ , (T)						891 g./l., pH = 7.55				
ΔL	+0.3	-0.2	+1.7	+2.7	+0.4	+0.4	+2.0	+1.0	+2.8	+6.1	+8.2	+1.22	+1.16	+1.14	+4.6	+2.4	
ΔW	+3.1	+2.0	+1.9	+1.8	+2.3	+9	+2.0	+1.1	+4.6	+3.6	+5.2	+4.8	+4.6	+4.3	+2.8	+3.4	
N ²	146	112	129	101	151	117	111	96	108	75	92	62	117	98	86	88	
N, cycles/sec.	5720	5820	6340	6160	6150	6280	6120	6030	4860	4860	5400	4880	5400	5760	5400	5930	
Aged at	ΔL	-	-	22 ^a	-	-	20 ^a	-	4	0.9	1.5	1.5	-	-	2.5	22.5	
Failure,	ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Mo.	N ²	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
		12.24 g./l., pH = 7.70					Sodium Perchlorate, NaClO ₄ , (R)						1724 g./l., pH = 5.95				
ΔL	-0.2	+1.2	+1.8	-1.1	+0.6	+0.7	+0.6	-0.2	+0.4	+0.1	+0.4	+0.1	.00	-0.4	-0.1	-0.1	
ΔW	+3.4	+1.9	+2.0	+1.6	+2.1	+2.2	+2.2	+1.4	+5.5	+4.8	+2.8	+3.9	+2.3	+3.4	+3.3	+2.3	
N ²	143	104	128	105	154	108	105	100	105	102	99	89	110	93	105	94	
N, cycles/sec.	5570	5560	6290	6230	6200	6080	6000	6280	4900	5410	5610	5800	5360	5810	6070	5990	
CONTROL:		Saturated Limewater, Ca(OH) ₂ , (R)										1.5 g./l., pH = 12.20					
ΔL	-	-	-	-	-	-	-	-	-	-0.1	-0.4	-0.3	.00	-0.1	-0.3	.00	
ΔW	-	-	-	-	-	-	-	-	-	+2.2	+1.9	+2.0	+1.4	+1.4	+7	+6	
N ²	-	-	-	-	-	-	-	-	-	140	103	128	104	156	109	98	
N, cycles/sec.	-	-	-	-	-	-	-	-	-	5660	5410	6240	6020	6240	6230	5870	

- a/ Some uncertainty in wt., or time of failure based on change in wt., because of dense crust of crystals on exterior.
- b/ Some uncertainty in length, or time of failure based on change in length, because of crust of crystals on ends.
- c/ Measurement on one specimen only.
- d/ Failure indicated by only one of two specimens under test.

CONDITION OF SPECIMENS STORED IN INORGANIC AQUEOUS SOLUTIONS FOR
21 MONTHS

The change in length (ΔL), the change in weight (ΔW), and the square of the frequency (N^2) are all expressed as percent of initial readings.

Criteria of failure are:

$$\begin{aligned}\Delta L &= \pm 0.20\% \\ \Delta W &= \pm 10.0\% \\ N^2 &= 50\% \text{ of initial}\end{aligned}$$

Ages at failure are interpolated to nearest 0.1 month up to 3 months and nearest 0.5 month above 3 months.

See also "Explanatory Notes for Tables 7-10, 12 and 13."

Note: Where no failure has occurred for a particular agent, the "age at failure" lines have been omitted.

Cement Curing Period	In 0.1 Normal Solutions								In 90% Saturated Solutions								
	W/C=0.6		W/C = 0.4				W/C=0.6		W/C = 0.4								
	Type I	Type V	Type I	Type V	O-C ₂ A	Type I	Type V	Type I	Type V	O-C ₂ A	Type I	Type V	O-C ₂ A				
	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	
CARBONATES:	4.80 g./l., pH = 9.04 Ammonium Carbonate, (NH ₄) ₂ CO ₃ , (ACS)								< 325 g./l. ^a , pH = 9.09								
ΔL	-0.2	-0.3	-0.4	-0.5	-0.5	-0.5	-0.5	-0.5	-0.4	-0.6	-0.8	-0.8	-0.8	-0.8	-0.8	-0.8	
ΔW	-2	+2	+1	+1	+2	+2	+2	+2	+4.5	+2.2	+2.5	+2.5	+2.5	+2.5	+2.5	+2.5	
N^2	120	106	113	103	103	103	103	104	107	107	124	124	124	124	124	124	
N,cycles/sec.	5650	6400	6380	6380	6380	6380	6380	6380	5400	6400	6760	6760	6760	6760	6760	6760	
	3.95 g./l., pH = 8.06 Ammonium Bicarbonate, NH ₄ HCO ₃ , (R)								191 g./l., pH = 8.38								
ΔL	+0.2	-0.4	+0.2	-0.7	-0.5	-0.2	-0.1	-0.6	-0.4	-0.4	-0.2	-0.4	-0.2	-0.6	-0.6	-0.2	
ΔW	+1.1	-2	+1.1	+2	+1.3	+9	+1.2	+8	+5	+2.4	+2.3	+3	+1.5	+1.2	+5	+5	
N^2	145	111	125	106	153	114	112	101	134	107	136	107	120	112	106	106	
N,cycles/sec.	5490	5850	6200	6350	6350	6230	6450	6260	5310	5750	6360	6340	6300	6380	6400	6400	
	5.30 g./l., pH = 11.30 Sodium Carbonate, Na ₂ CO ₃ , (R)								200 g./l., pH = 11.63								
ΔL	.00	-0.6	-0.2	-0.2	-0.4	-0.2	-0.2	-0.5	+1.6	+0.6	+1.0	+1.2	+0.2	.00	+0.6	+0.2	
ΔW	+5	-2	+6	.0	+6	+3	.4	+1	+6.1	+5.4	+4.2	+4.4	+3.4	+2.5	+2.6	+2.4	
N^2	150	113	132	112	140	114	114	102	121	95	128	107	147	111	105	99	
N,cycles/sec.	5610	5650	6250	6350	6090	6370	6370	6400	5020	5120	6240	6220	6250	6300	6200	6160	
	4.20 g./l., pH = 8.15 Sodium Bicarbonate, NaHCO ₃ , (R)								86 g./l., pH = 8.35								
ΔL	-0.2	-0.4	.00	-0.8	-0.1 ^b	-0.5	+0.1	-0.6	-0.2	-0.6	-0.2	-0.5	-0.4	-0.6	-0.5	-0.6	
ΔW	+6	+2	+8	+1	+6 ^b	+3	+5	+2	+5.5	+2.3	+2.6	+1.1	+2.2	+7	+8	+4	
N^2	156	111	130	110	155 ^b	114	116	102	108	103	130	105	149	115	113	102	
N,cycles/sec.	5570	5630	6350	6320	6370 ^b	6310	6440	6260	4770	5720	6180	6160	6300	6310	6420	6330	
FERROCYANIDE:	12.10 g./l., pH = 8.23 Sodium Ferrocyanide, Na ₄ Fe(CN) ₆ ·10H ₂ O, (T)								286 g./l., pH = 9.60								
ΔL	+0.6	+0.6	+1.0		+1.4	+1.5		+1.8	+1.1	+1.4		+1.6	-0.1				
ΔW	+9	+2	+1.1		+6	+2		+1.4	+5	+1.2		+1.2	+2				
N^2	143	102	123		149	110		142	97	124		142	110				
N,cycles/sec.	5580	5460	5840		6210	6250		5500	5430	5720		6030	6340				
NITRATES:	8.00 g./l., pH = 6.03 Ammonium Nitrate, NH ₄ NO ₃ , (ACS)								1692 g./l., pH = 4.88								
ΔL	+0.4	+0.1	+2.4	+4.5	-1.6	+1.2	+1.3	+0.7	c	-1.9	-1.6	-1.7	c	-1.6	c	c	
ΔW	-4.8	-4.1	-5.3	-5.2	-3.0	-4.00	-5.9	-4.8	c	-5.9	-8.1	-9.1	c	-9.0	c	c	
N^2	23	22	20	14	57	44	26	43	c	d	9	d	c	d	c	c	
N,cycles/sec.	2160	2640	2520	2290	3220	3960	2990	4960	c	d	1770	c	c	d	c	c	
Age at Failure	-	-	18	8	-	-	-	-	-	-	-	-	-	-	-	-	
Mo.	N ²	11	8	11.5	9.5	-	15	11.5	14	0.07	0.2	0.2	0.4	0.2	0.5	0.1	
	15.56 g./l., pH = 4.96	Lead Nitrate, Pb(NO ₃) ₂ , (P)								495 g./l., pH = 2.98							

ΔL	+4.1	+3.0	+3.9	+3.6	+4.2	+3.4	+0.6	+1.6	+1.9	+1.1 ^b	+2.5	+5.8	+2.0	+1.5	+2.3	
ΔW	+5.3	+6.9	+4.6	+6.6	+4.6	+3.7	+5.9	+6.8	+17.7	+11.9 ^b	+18.2	+15.1	+21.1	+7.0	+16.0	
N^2	121	90	100	75	123	87	79	69	113	80 ^b	87	72	104	70	80	
N, cycles/sec.	4920	5230	5660	5800	5470	5590	5270	5360	4710	5020 ^a	5320	5130	5090	4970	5180	
Age at ΔL	0.5	1.0	5	1.4	-	7	-	-	3	11.5	2.7	2.2	5.5	-	2.8	
Failure, ΔW	-	-	-	-	-	-	-	-	5.5	10	8	7	5	-	4	
Mo. N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	3.5	
12.82 g./l., pH = 6.90			Magnesium Nitrate, $Mg(NO_3)_2 \cdot 6H_2O$, (T)								1152 g./l., pH = 3.75					
ΔL	+3.5	-0.5	+4.6	+0.4	+5.1	+0.4	+2.9	+0.7	c	+2.42	+1.94	+2.16	+4.8	+4.5 ^b	+2.56	+2.88
ΔW	+3.6	+2.0	+3.6	+1.5	+5.4	+1.9	+4.1	+2.4	c	+4.2	+3.7	+4.4	+5.4	+10.7	+5.6	+3.2
N^2	145	107	119	110	140	110	98	81	c	21	50	37	106	81	42	d
N, cycles/sec.	5520	5710	6140	6290	5960	6280	5810	5810	c	2490	4000	3660	5080	5350	3410	d
Age at ΔL	2.1	-	2.2	-	0.4	-	5.5	-	0.8	6	5.5	7	9.5	8.5 ^b	3.5	4.5
Failure, ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	20.5 ^b	-	-
Mo. N^2	-	-	-	-	-	-	-	-	3	9.0	-	19	-	-	18	12
10.11 g./l., pH = 6.81			Potassium Nitrate, KNO_3 , (T)								284 g./l., pH = 6.89					
ΔL	+0.5	+1.0	+1.1	+1.0	+1.1	-0.6	+0.6	+0.5	+3.4	+3.3	+6.2	+8.6	+2.9	+1.9	+2.8	+2.6
ΔW	+2.8	+1.8	+2.4	+1.0	+3.6	+1.5	+2.6	+1.4	+3.2	+3.5	+4.8	+3.8	+6.2	+2.7	+3.6	+2.4
N^2	161	107	129	107	123	117	122	98	126	98	106	89	138	103	95	85
N, cycles/sec.	5700	5790	6470	6220	6160	6380	6270	6280	5140	5430	5900	5700	5940	6090	5630	5900
Age at ΔL	-	-	-	-	-	-	-	-	3.5	6	3	2.5	7.5	-	8	15.5
Failure, ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mo. N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
8.50 g./l., pH = 7.70			Sodium Nitrate, $NaNO_3$, (T)								788 g./l., pH = 7.52					
ΔL	+0.2	+0.8	+1.9	.00	.00	-0.2	+0.7	.00	+2.0	+2.8	+5.7	+2.4	+1.2	.00	+1.9	+0.3
ΔW	+2.8	+1.8	+2.6	+1.5	+2.0	+1.5	+1.8	+1.2	+4.5	+3.5	+4.1	+2.9	+4.2	+2.5	+3.2	+2
N^2	152	117	128	110	153	114	108	93	124	93	98	91	127	95	85	96
N, cycles/sec.	5940	5740	6290	6380	6340	6270	6290	6260	5310	5300	5560	5800	5780	5830	5600	6080
Age at ΔL	-	-	4	-	-	-	-	-	21	16.5	3	21	-	-	-	-
Failure, ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mo. N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
NITRITE:			6.90 g./l., pH = 7.70				Sodium Nitrite, $NaNO_2$, (ACS)				765 g./l., pH = 8.00					
ΔL	+0.2	+0.4	+0.9	.00	+0.2	-0.2	+1.0	-0.4	+1.8	+5.6 ^b	.00	+2.8	+0.5	e	f	-1.4
ΔW	+2.6	+1.9	+2.1	+1.8	+1.8	+1.2	+1.5	+1.0	+3.1	+2.3	+3.5	+1.9	+1.9	+2.0	+1.2	-1.6
N^2	145	105	126	103	151	115	107	97	119	96	109	92	126	84	103	78
N, cycles/sec.	5850	5740	6220	6260	6240	6350	6350	6320	5210	5480	5800	5840	5870	5450	6150	5560
Age at ΔL	-	-	-	-	-	-	-	-	-	7.5 ^b	-	16	-	-	-	-
Failure, ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mo. N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CONTROL:			Saturated Lime water, $Ca(OH)_2$, (R)								1.5 g./l., pH = 12.20					
ΔL	-	-	-	-	-	-	-	-	-	-0.1	-0.2	-0.3	.00	.00	-0.3	.00
ΔW	-	-	-	-	-	-	-	-	-	+2.0	+2.0	+1.8	+1.4	+1.3	+7	+5
N^2	-	-	-	-	-	-	-	-	-	138	106	132	108	160	115	99
N, cycles/sec.	-	-	-	-	-	-	-	-	-	5610	5490	6310	6120	6300	6380	5930

a/ See text.
b/ Measurement on one specimen only.
c/ Test terminated at earlier age.

d/ Accurate reading not obtainable.
e/ Caps of prisms are loose.
f/ Caps of prisms are gone.

TABLE 11

CONDITION OF SPECIMENS STORED IN INORGANIC AQUEOUS SOLUTIONS FOR
15 MONTHS

78

The change in length (ΔL), the change in weight (ΔW), and the square of the frequency (N^2) are all expressed as percent of initial readings.

Criteria of failure are:

$$\begin{aligned}\Delta L &= + 0.20\% \\ \Delta W &= \mp 10.0\% \\ N^2 &= 50\% \text{ of initial}\end{aligned}$$

Ages at failure are interpolated to nearest 0.1 month up to 3 months and nearest 0.5 month above 3 months.

Note: Where no failure has occurred for a particular agent, the "age at failure" lines have been omitted.

Cement Curing Period	In 0.1 Normal Solutions								In 90% Saturated Solutions											
	W/C=0.6		Type I		W/C = 0.4		Type V		O-C ₂ A		W/C=0.6		Type I		W/C = 0.4		Type V		O-C ₂ A	
	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.
CHLORIDES, HALIDES:	5.35 g./l., pH = 5.56				Ammonium Chloride, NH ₄ Cl, (ACS)								335 g./l., pH = 4.47							
ΔL	.00	+0.02		+0.06		+0.06		-0.14		-0.10		-0.10		-0.10		-11.9				
ΔW	-4.6	-3.3		-2.8		-5.8		-8.0		-9.8		-11.0		-11.9						
N^2	55	56		77		36		a		a		a		a						
N, cycles/sec.	3520	4200		4660		3600		a		a		a		a						
Age at Failure, Mo.	ΔL	-	-	-	-	-	-	-	-	16.5	-	2.9	-	1.8	-	-	-	-	-	4
	ΔW	-	-	-	-	-	-	-	-	0.2	-	0.4	-	0.1	-	-	-	-	-	-
	N^2	-	-	-	-	6.5	-	0.1	-	0.2	-	0.4	-	0.1	-	-	-	-	-	-
	5.84 g./l., pH = 6.65				Sodium Chloride, NaCl, (ACS)								324 g./l., pH = 8.39							
ΔL	+0.04	+0.03		+0.02		-0.03		+0.14		+0.23		+0.15		+0.13		+2.8				
ΔW	+2.1	+2.6		+1.9		+1.7		+2.4		+3.5		+3.2		+2.8						
N^2	137	118		145		104		119		97		120		95						
N, cycles/sec.	5580	6260		6280		6080		5210		5600		5650		5880						
Age at Failure, Mo.	ΔL	-	-	-	-	-	-	-	-	12.5	-	-	-	-	-	-	-	-	-	-
	ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	10.29 g./l., pH = 7.50				Sodium Bromide, NaBr, (R)								826 g./l., pH = 7.45							
ΔL	+0.02	+0.04		+0.10		+0.02		+1.33		+1.74		+0.31		-0.26		+3.0				
ΔW	+2.7	+2.6		+2.4		+2.2		+7.5		+14.4		+5.1		+3.0						
N^2	137	123		146		105		33		19		106		75						
N, cycles/sec.	5670	6340		6340		6240		2660		2530		5250		5190						
Age at Failure, Mo.	ΔL	-	-	-	-	-	-	4.5	-	3.5 ^b	-	3	-	2.0	-	-	-	-	-	-
	ΔW	-	-	-	-	-	-	-	-	7.5 ^b	-	-	-	-	-	-	-	-	-	-
	N^2	-	-	-	-	-	-	9.5	-	9 ^b	-	-	-	8.0 ^b	-	-	-	-	-	-
	4.20 g./l., pH = 7.82				Sodium Fluoride, NaF, (T)								38 g./l., pH = 8.51							
ΔL	-0.02	-0.03		-0.02		-0.06		-0.04		-0.02		-0.02		-0.02		+2.8				
ΔW	+1.4	+2.6		+2.2		+1.0		+2.5		+2.5		+2.0		+2.8						
N^2	116	110		139		99		74		103		129		88						
N, cycles/sec.	5170	5810		6000		6030		3990		5690		5830		5740						
CHROMATES:	8.10 g./l., pH = 8.83				Sodium Chromate, Na ₂ CrO ₄ , (T)								644 g./l., pH = 8.92							

ΔL	.00	+0.04	+0.02	.00	+0.02	+0.02	+0.03		+0.4	+0.5	-0.02	+0.6	-0.07	-0.07	-0.04	-0.08
ΔW	+3	+1.0	+3	+1.2 ^c	+3	+9	+3		+3.1	+3.3	+1.0	+4.0	+1.8	+2.8	+1.7	
N^2	110	128	115	143 ^c	114	104	104		115	121	103	154	110	104	105	
N , cycles/sec.	5840	6220	6640	5960 ^c	6260	6140	6430		5880	6180	6420	6180	6340	6090	6440	
14.90 g./l., pH = 5.09				Sodium Dichromate, $Na_2Cr_2O_7$, (T)								1845 g./l., pH = 4.10				
ΔL	+0.03	+0.07	+0.14	.00	+0.04	+0.02	+0.03	.00	+0.12	+0.24	-0.03	+0.24	-0.04	-0.17 ^c	-0.11	-0.10
ΔW	-4.8	-4.6	-4.0	-2.6	-2.4	-5.2	-3.4	-6.4	+7.8	+4.6	+3.8	-1.5	+2.4	-0.5 ^c	-0.6	-0.5
N^2	41	33	49	54	76	46	57	46	158	100	117	93	132	96 ^c	103	99
N , cycles/sec.	2900	3080	3680	4580	4370	4170	4640	4320	5870	5370	5810	5880	5800	5970 ^c	6050	6170
Age at ΔL	-	-	17	-	-	-	-	-	-	10.5 ^b	-	9.5 ^b	-	-	-	-
Failure, ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mo. N^2	12	10	15.5	-	-	13.5	-	11	-	-	-	-	-	-	-	-
FLUOSILICATE: 8.32 g./l., pH = 3.09				Magnesium Fluosilicate, $MgSiF_6$, (P)								360 g./l., pH = 0 ¹				
ΔL	-0.02	.00	-0.02	+0.05 ^c	-0.04	-0.03			-0.08	-0.02	-0.07	-0.05	-0.06	-0.06		
ΔW	+1	+1.2	+6	+1.3 ^c	+5	+1.2			-3	-4	-6	-2	-7	+4		
N^2	113	130	111	157 ^c	115	110			104	120	106	146	103	120		
N , cycles/sec.	5650	6220	6320	6290 ^c	6370	6290			5520	6190	6130	6110	5150	6450		
MANGANATE: 15.80 g./l., pH = 7.87				Potassium Permanganate, $KMnO_4$, (R)								76 g./l., pH = 9.45				
ΔL	+0.2	+0.4	-0.03			-0.14			-0.01	+0.05	-0.02	-0.02		+0.08 ^c		
ΔW	+4	+1.4	+0			+1.3			+5	+2.1	+1	+1.8		+1.6		
N^2	116	136	105			111			115	128	105	153		113		
N , cycles/sec.	5680	6240	6360			6290			5710	6250	6340	6290		6340		
MOLYBDATE: 12.10 g./l., pH = 7.88				Sodium Molybdate, Na_2MoO_4 , (R)								688 g./l., pH = 9.00				
ΔL	.00	+0.02	-0.02	.00		.00			+0.02	-0.02	-0.06	-0.04		-0.12		
ΔW	+3	+1.1	+2	+1.6		+1.4			+10.6	+6.8	+4.7	+7.0		+3.4		
N^2	116	127	108	148		110			124	138	125	161		113		
N , cycles/sec.	5720	6280	6360	6250		6330			6100	6530	6560	6380		6320		
Age at ΔL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Failure, ΔW	-	-	-	-	-	-	-	-	11	-	-	-	-	-	-	-
Mo. N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
PHOSPHATES: 10.27 g./l., pH = 11.87				Sodium Orthophosphate, $Na_3PO_4 \cdot 8H_2O$, (R)								188 g./l., pH = 12.64				
ΔL	-0.13	-0.05	-0.04	-0.02	-0.02	-0.02	-0.04	.00	-0.04	-0.04	.00	-0.02	-0.04	.00	-0.04	.00
ΔW	+2	-1	+4	-1	+4	+1	+4	.0	+1.9	+1.6	+0.7	.0	+1.2	+2	+5	-1
N^2	145	117	128	106	152	119	111	109	139	102	125	108	145	116	117	108
N , cycles/sec.	5860	5830	6220	6380	6310	6540	6360	6560	5610	5720	6210	6450	6260	6350	6450	6480
4.73 g./l., pH = 8.50				Sodium Orthophosphate, Dibasic, Na_2HPO_4 , (ACS)								79 g./l., pH = 8.98				
ΔL	+0.02	-0.02	-0.06	.00	-0.04	+0.07	-0.03	+0.02	-0.04	-0.03	+0.01	+0.03	-0.03	.00	-0.06	.00
ΔW	+5	-1	+5	-2	+7	+3	+6	+4	+1.7	-6	+3	-2	+6	+2	+4	.0
N^2	147	105	128	113	155	117	116	105	155	115	129	117	153	118	113	102
N , cycles/sec.	5890	5660	6260	6380	6370	6420	6360	6540	5870	5790	6350	6480	6380	6430	6370	6480
4.60 g./l., pH = 5.40				Sodium Orthophosphate, Monobasic, $NaH_2PO_4 \cdot H_2O$, (R)								990 g./l., pH = 3.18				
ΔL	+0.16	+0.13	+0.03	+0.14	+0.18	+0.10	d	.00	+0.86 ^d	+0.06	+0.48	+0.27	+0.63	+0.89 ^d	e	+0.86 ^d
ΔW	+2.4	+1.2	+1.3	+1.2	+2.2	+1.6	+1.8	+1.3	-1.1	+2.6	+1	-1.2	+9	+4.5	+2.4 ^e	+7.0
N^2	139	102	119	102	145	105	109	100	81	68	83	81	83	79	73 ^e	71
N , cycles/sec.	5480	5540	6090	6200	6200	6200	6360	6340	4280	4440	5150	5590	4760	5390	5140 ^e	5340
Age at ΔL	-	-	-	-	-	-	-	-	6.5	-	8.5	14.5	3.5	1.2	2.2	1.4
Failure, ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mo. N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

TABLE 11 (Cont.)

Cement	In 0.1 Normal Solutions								In 90% Saturated Solutions															
	W/C=0.6				W/C = 0.4				W/C=0.6				W/C = 0.4											
	Type I		Type I		Type V		O-C ₃ A		Type I		Type I		Type V		O-C ₃ A									
Curing Period	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.								
	10.20 g./l., pH = 6.25								Sodium Hexametaphosphate, Na ₃ (PO ₃) ₆ , (T)								297 g./l., pH = 5.00							
ΔL	-0.12	+0.03	-0.02	-0.04	+0.04	-0.07	+0.06	.00	+0.02	-0.12	+0.08	+0.07	+0.12	+0.20	+0.12	+0.08								
ΔW	-2.2	-1.5	-1.7	-.9	+6	+7	-.5	-.3	-3.1	+1.8	-.5	+1.5	-.3	+1.6	+1.2	-.1								
N ²	125	109	114	99	139	104	97	94	96	109	104	106	128	111	97	82								
N, cycles/sec.	5420	5670	6030	6150	6200	6200	5850	6290	4850	5660	5760	6330	5900	6360	5920	5850								
	6.65 g./l., pH = 9.80								Sodium Pyrophosphate, Na ₄ P ₂ O ₇ , (T)								58 g./l., pH = 10.20							
ΔL	+0.04	+0.04	+0.08	+0.04	+0.09	+0.05	+0.08	+0.06	+0.05	+0.04	+0.05	+0.08	+0.10	+0.06	+0.03	+0.02								
ΔW	+2.1	+1.2	+1.5	+1.2	+2.0	+1.4	+2.1	+1.4	+1.9	+1.0	+1.8	+1.4	+1.6	+1.6	+1.4	+1.2								
N ²	158	118	119	107	160	110	110	106	128	117	126	114	149	120	114	103								
N, cycles/sec.	5860	5750	6180	6370	6460	6420	6390	6460	5620	5870	6190	6430	6580	6360	6470	6420								
STANNATE:	13.33 g./l., pH = 11.92								Sodium Stannate, Na ₂ SnO ₃ ·3H ₂ O, (T)								556 g./l., pH = 12.90							
ΔL	.00	-.03	+0.08	-.02	.00	+0.02	+0.01	-.02	-.04	-.04	-.03	-.02	-.02	-.03	+0.01	-.02								
ΔW	+1.8	+6	+2.6	+3	+1.5	+9	+1.0	+4	+6.2	+3.3	+1.6	+7	+3.0	+4	+5	+2								
N ²	149	109	116	105	152	112	114	101	167	127	128	111	155	111	116	103								
N, cycles/sec.	5920	5630	6050	6400	6420	6320	6490	6470	6170	6090	6300	6410	6580	6400	6520	6420								
SULFATES:	6.61 g./l., pH = 5.43								Ammonium Sulfate (NH ₄) ₂ SO ₄ , (ACS)								681 g./l., pH = 5.32							
ΔL	f	+1.63	+3.08	+3.26	+7.4		+6.4		f	f	f	f	f	f	f	f								
ΔW	f	+4.8	+5.2	+3.5	+2.4		+3.5		f	f	f	f	f	f	f	f								
N ²	f	68	22	20	90		64		f	f	f	f	f	f	f	f								
N, cycles/sec.	f	4470	2680	2720	4850		4780		f	f	f	f	f	f	f	f								
Age at	ΔL	2.5	2.8	4	3	7	4.5		0.1	.06	0.1	0.4	0.3			0.1								
Failure, ΔW	-	-	-	-	-	-	-		(0.2) ^g	(0.2) ^g	0.6 ^c	0.5	0.7			1.1								
Mo.	N ²	6	-	7	4.5	-	-		0.2	(0.2) ^g	(3) ^g	2.6 ^c	(3) ^g			3.5								
	5.76 g./l., pH = 1.55								Ammonium Bisulfate, NH ₄ HSO ₄ , (R)								3015 g./l., pH = 0 ¹							
ΔL	f		+3.08	+2.28	+1.19		+1.62		f	f	f	f	f	f	f	f								
ΔW	f		+3.9	+2.9	+1.0		+3.0		f	f	f	f	f	f	f	f								
N ²	f		37	28	81		54		f	f	f	f	f	f	f	f								
N, cycles/sec.	f		3480	3270	4820		4430		f	f	f	f	f	f	f	f								
Age at	ΔL	2.6		4	4	4.5	4.5		0.3		0.2	0.1	0.2			0.2								
Failure, ΔW	-	-	-	-	-	-	-		0.1		0.5	0.5	0.3			0.7								
Mo.	N ²	-		6	7	-	-		(2) ^g		5.5	8	6.5			6								
	7.98 g./l., pH = 4.17								Copper Sulfate, CuSO ₄ , (ACS)								718 g./l., pH = 3.50							

	ΔL	+1.05	+1.31	+1.57	+1.42	+1.26	+1.22	+1.29	+1.45	+1.84	+1.46	+1.64	+1.54	+1.31	+1.30	+1.37
	ΔW	+6.0	+5.0	+4.1	+2.9	+4.4	+3.2	+4.9	+4.0	-4.9	-4.3	-6.0	-7.9	-7.5	-8.6	-13.4
	N^2	110	58	116	100	153	115	110	105	83	71	93	80	123	90	82
	N, cycles/sec.	5000	4180	6000	6100	6400	6380	6460	6540	4340	4680	5290	5500	5630	5750	5820
	Age at ΔL	5.5	7.5	7	11	10	13.5	11	4.5	4.5	6.5	8	10	11.5	5	4
	Failure, ΔW	-	-	-	-	-	-	-	-	-	-	-	-	-	-	12.5
	Mo. N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
6.02 g./l., pH = 6.95																
Magnesium Sulfate, $MgSO_4$, (ACS)																
311 g./l., pH = 8.16																
	ΔL	+1.07	+1.13	+2.22	+1.02	+1.03	+1.02	+1.02	.00	f	+2.3	e	+1.40	+1.08	+1.30	e
	ΔW	+3.3	+1.8	+2.1	+4	+1.2	+1.3	+1.3	+4	f	+5	-4.0 ^e	-2	-2.6	+1.5	-3.4
	N^2	50	102	112	105	147	119	112	103	f	118	129 ^e	107	130	112	106 ^e
	N, cycles/sec.	3470	5600	5880	6260	6260	6370	6360	6470	f	5900		6250	5910	6290	6270 ^e
	Age at ΔL	7.45	-	14.5	-	-	-	-	-	7	13	-	8.5	-	11	8 ^e
	Failure, ΔW	-	-	-	-	-	-	-	-	(9) ^g	-	-	-	-	-	14 ^a
	Mo. N^2	16.5	-	-	-	-	-	-	-	(9) ^g	-	-	-	-	-	-
8.45 g./l., pH = 3.22																
Manganese Sulfate, $MnSO_4 \cdot H_2O$, (ACS)																
646 g./l., pH = 1.26																
	ΔL	+2.31	+1.64	+1.87	+1.03	+1.19	-	+1.40	-	-3.8	-2.40	-3.2	-1.02	-1.93	-	-3.6
	ΔW	+6.1	+2.6	+4.7	+1.6	+3.2	-	+3.8	-	-5.4	-38.1	-3.0	-9.8	-17.9	-	-5.1
	N^2	111 ^f	87	52	105	190	-	120	-	141	86	62	101	156	-	108
	N, cycles/sec.	4790	4970	4080	6380	6990	-	6530	-	5660	5220	4570	6140	6360	-	6360
	Age at ΔL	4	8	5.5	-	-	-	7.5	-	10.5	2.0	10.5	4	7	-	6
	Failure, ΔW	-	-	-	-	-	-	-	-	-	0.8	-	-	8	-	-
	Mo. N^2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
7.10 g./l., pH = 8.15																
Sodium Sulfate, Na_2SO_4 , (R)																
175 g./l., pH = 7.00																
	ΔL	+1.06	+1.05	+1.02	.00	+1.02	-	.00	>+3.46	+5.55	+5.04	f	+2.8	-	-	+4.8
	ΔW	+1.1	+1.5	+1.9	-1	+1.9	-	+1.8	+11.8	+12.6	+13.4	f	+4.0	-	-	+5.6
	N^2	138	105	64	108	193	-	118	73	26	39	f	194	-	-	80
	N, cycles/sec.	5520	5560	4620	6470	7090	-	6480	4040	2790	3540	f	6780	-	-	5460
	Age at ΔL	-	-	-	-	-	-	-	0.3	1.0	2.4	3.5	12 ^b	-	-	8.5
	Failure, ΔW	-	-	-	-	-	-	-	10.5	5.5	11	(6) ^g	-	-	-	-
	Mo. N^2	-	-	-	-	-	-	-	-	2.1	12	(6) ^g	-	-	-	-
6.00 g./l., pH = 1.42																
Sodium Bisulfate, $NaHSO_4$, (T)																
387 g./l., pH = 0 ^l																
	ΔL	e	-	+1.76	+1.84	+1.69	-	+1.74	f	-	f	f	f	f	-	f
	ΔW	e	-	-0.5	+1.9	-2.2	-	-1.8	f	-	f	f	f	f	-	f
	N^2	e	-	47	72	117	-	76	f	-	f	f	f	f	-	f
	N, cycles/sec.	e	-	3910	5220	5560	-	5260	f	-	f	f	f	f	-	f
	Age at ΔL	2.5	-	3.5	7	4.5	-	4.5	0.07	0.07	0.07	0.03	.07	-	0.07	
	Failure, ΔW	-	-	-	-	-	-	-	0.03	0.07	0.07	0.07	.07	-	0.1	
	Mo. N^2	-	-	14.5	-	-	-	-	(0.1) ^g	0.6 ^c	0.6	.6	.6	-	0.6	
SULFIDE:																
12.01 g./l., pH = 12 to 12.5 ^h																
Sodium Sulfide, $Na_2S \cdot 9H_2O$, (T)																
507 g./l., pH = 13 to 14 ^h																
	ΔL	+1.02	+1.02	+1.05 ^c	+1.01	-0.01	.00	.00	-0.01	+1.16	+1.07	+1.07	+1.03 ^c	+1.06	+1.04	+1.04
	ΔW	+1.6	-1.2	+1.4 ^c	.0	+1.5	+1.1	+1.5	.0	+1.2	+1.1	+1.0	+1.6 ^c	+1.2	+1.0	+1.4
	N^2	149	116	118 ^c	104	148	110	115	102	141	111	114	104 ^c	132	113	111
	N, cycles/sec.	5730	5800	5980 ^c	6370	6450	6390	6450	6470	5520	5590	5960	6320 ^c	6000	6430	6260

TABLE 11 (Cont.)

Cement Curing Period	In 0.1 Normal Solutions								In 90% Saturated Solutions							
	W/C=0.6		W/C = 0.4						W/C=0.6		W/C = 0.4					
	Type I		Type I	Type V		O-C ₃ A		Type I	Type I		Type V	O-C ₃ A				
	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.
SULFITES:	12.61 g./l., pH = 9.00				Sodium Sulfite, Na ₂ SO ₃ ·7H ₂ O, (ACS)				472 g./l., pH = 9.48							
ΔL	+0.34	+0.07	+0.15	+0.02	+0.10	+0.02	+0.06	-0.02	f	-	+0.02	+6.46	-0.04	+0.10	-0.03	-0.04
ΔW	+1.2	+0.1	+1.0	.0	+1.2	+0.5	+1.4	-0.1	f	-	+1.6	+12.5	+0.8	+2.8	+1.4	+1.0
N ²	122	115	126	108	154	114	113	104	f	-	112	14	150	106	110	98
N, cycles/sec.	5280	5910	6320	6390	6540	6550	6430	6640	f	-	5940	2300	6310	6100	6360	6350
Age at Failure, Mo.	ΔL	9.5	-	-	-	-	-	-	7.5	-	-	8.5	-	-	-	-
	ΔW	-	-	-	-	-	-	-	-	-	-	13.5	-	-	-	-
	N ²	-	-	-	-	-	-	-	<15 ⁱ	<12 ⁱ	-	12	-	-	-	-
	5.20 g./l., pH = 2.20				Sodium Bisulfite, NaHSO ₃ , (ACS)				90 g./l., pH = 2.45							
ΔL	e	+4.48 ^c	+0.76	+1.38	+0.24		+0.90		f	+0.08 ^c	+3.66	.00	e		e	-26.5 ^e
ΔW	e	+0.3 ^c	+2.6	+2.0	-2.3		-5.9		f	-1.4 ^c	-10.9	-11.0	-27.0 ^e			
N ²	e	12 ^c	84	47	133		48		f	92 ^c	23	89	127 ^e			73 ^e
N, cycles/sec.	e	1880 ^c	5170	4240	5810		4080		f	5290 ^c	2260	5890	5710 ^e			5200 ^e
Age at Failure, Mo.	ΔL	3	3	7	5.5	13	6	2.0	2.7 ^c	2.8	-	5 ^c				2.7 ^c
	ΔW	(6) ^g	-	-	-	-	-	4.5	-	5.5	4.5	5				4.5
	N ²	6	5	-	-	-	-	2.8	-	5.5	-	-				-
THIOSULFATE:	12.41 g./l., pH = 7.28				Sodium Thiosulfate, Na ₂ S ₂ O ₃ ·5H ₂ O, (T)				958 g./l., pH = 8.70							
ΔL	+0.12		+0.28	+0.40	+0.10		+0.36		+0.14		+0.38	+0.13	+0.12			+0.12
ΔW	+1.6		+2.3	+2.0	+1.8		+1.8		+3.6		+4.2	+1.2	+3.8			+2.3
N ²	129		110	84	145		93		125		107	92	128			101

N, cycles/sec.	5400	6010	5630	6240	5810	5430	5860	5850	5920	6150			
Age at ΔL	-	13	11.5	-	10	-	9	-	-	-			
Failure, ΔW	-	-	-	-	-	-	-	-	-	-			
Mo. N^2	-	-	-	-	-	-	-	-	-	-			
PERSULFATE:		13.57 g./l., pH = 1.79				Potassium Persulfate, $K_2S_2O_8$, (ACS)			180 g./l., pH = 1.55				
ΔL	k	+4.1	+3.4	+0.8	+1.3	+2.61	+2.63	+4.44	+3.2	+1.86			
ΔW	+6.3	+1.4	+9	+9	+1.3	+6.1	+4.7	+6.7	+2.0	+6.8			
N^2	52	117	92	154	105	124	69	34	137	67			
N, cycles/sec.	3500	6120	5980	6270	6350	5400	4690	3550	5980	5050			
Age at ΔL	4	9.5	13	-	-	0.4	6	0.9	9.5	6.5			
Failure, ΔW	-	-	-	-	-	-	-	-	-	-			
Mo. N^2	-	-	-	-	-	-	-	13	-	-			
CONTROL:		Saturated Limewater, $Ca(OH)_2$, (R)					1.5 g./l., pH = 12.20						
ΔL						-0.02	-0.02	-0.03	.00	-0.02	-0.03	+0.04	+0.04
ΔW						+1.9	+1.6	+1.8	+1.2	+1.3	+7	+2	+5
N^2						140	106	132	108	153	116	92	100
N, cycles/sec.						5650	5500	6310	6130	6170	6410	5700	6110

- a/ Accurate reading not obtainable.
- b/ Failure indicated by only one of two specimens under test.
- c/ Measurement on one specimen only.
- d/ Some uncertainty in weight, because of crystals on exterior.
- e/ Caps of prisms are gone.
- f/ Test terminated earlier than 15 months.
- g/ Failure not indicated by this criterion, but test terminated at age shown.
- h/ By Hydrion paper. Not readily measurable electrometrically.
- i/ Age of failure unknown (incomplete data), but specimen too weak to handle at age indicated.
- k/ Too warped to measure .
- l/ As read on pH meter, which has lower limit of 0.

TABLE 12
CONDITION OF SPECIMENS STORED IN SYNTHETIC
SEAWATER FOR 6 MONTHS

The change in length (ΔL), the change in weight (ΔW), and the square of the frequency (N^2) are all expressed as percent of initial readings.

Composition of synthetic sea water is given in Table 3.

Criteria of failure are:

$$\Delta L = \pm 0.20\%$$

$$\Delta W = \pm 10.0\%$$

$$N^2 = 50\% \text{ of initial}$$

However, no failures had occurred in the first six months, with the possible exception noted in the footnote for specimens stored at 73 F.

Cement Type	Synthetic Seawater									Concentrated Synthetic Sea-water ^a
	W/C = 0.6		W/C = 0.4				W/C = 0.4		Type V	
	Type I		Type I		Type V		0-C ₃ A			
Curing Period	3d.	28d.	3d.	28d.	3d.	28d.	3d.	28d.	3d.	
Stored at 73 F ^b										
ΔW	% of	+3.0	1.2	+3.0	+ .7	+3.2 ^c	+ .7	+1.8	+1.0	+4.6
N^2	Initial	118	111	111	109	+126 ^c	109	91	98	139
N		5190	5580	5980	6110	5540 ^c	6240	5640	6200	5650
Stored at 160 F										
ΔL						+ .19				+ .18
ΔW	% of					+2.2				+3.8
N^2	Initial					149				149
N						5720				5740
Stored at 200 F										
ΔL						+ .16				+ .13
ΔW	% of					+2.3				+3.7
N^2	Initial					153				147
N						5860				5900

^aConcentration is 5 times that of synthetic seawater.

^bSome uncertainty in length, or time of possible failure based on length, because of crust of crystals on ends. When such crystals were removed from crystals by gentle scraping, no increase in length was any longer measured.

^cAverage of 2 sets of two specimens each.

TABLE 13
AGES AT FAILURE IN ORGANIC ACIDS AND "EDTA"

All specimens were made with Type I cement and moist cured 28 days.

Solutions of less than 100 g./1000 ml. were renewed more frequently than those of higher concentrations, but both schedules were interrupted and were somewhat irregular. The number of fresh solutions used for each pair of specimens is recorded below.

The change in length (ΔL), the change in weight (ΔW), and the square of the frequency (N^2) are all expressed as percent of initial readings.

Criteria of failure are:

$$\begin{aligned}\Delta L &= \pm 0.20\% \\ \Delta W &= \pm 10.0\% \\ N^2 &= 50\% \text{ of initial}\end{aligned}$$

Age at Failure by Various Criteria, Months											
In 0.1 Molar Solutions				In Intermediate Concentrations				In Concentrated Liquids			
W/C = 0.6		W/C = 0.4		W/C = 0.6		W/C = 0.4		W/C = 0.6		W/C = 0.4	
No. of Fresh Solutions	Total Exposure Time	No. of Fresh Solutions	Total Exposure Time	No. of Fresh Solutions	Total Exposure Time	No. of Fresh Solutions	Total Exposure Time	No. of Fresh Solutions	Total Exposure Time	No. of Fresh Solutions	Total Exposure Time
ACIDS:											
Lactic Acid, $\text{CH}_3\text{CHOHCOOH}$, (USP)											
9.01 g./l. solution											
a											
ΔL	5	61d.	9	105d.				0	12d.	0	10d.
ΔW	9	104d.	7	100d.				0	24d.	1	79d.
N^2	11	110d.	14	124d.				-	-	-	-
Stearic Acid, $\text{CH}_3(\text{CH}_2)_{18}\text{COOH}$, (USP)											
200 g./l. benzene											
ΔL				0	4d.	0	4d.				
ΔW				0	2d.	0	4d.				
N^2				-	-	-	-				
Lactic Acid, $\text{CH}_3\text{CHOHCOOH}$, (USP) Acetic Acid, CH_3COOH , (Pr)											
20 g. lactic + 20 g. acetic/0.960 l. water											
ΔL				0	5h.	0	12d.				
ΔW				4	7d.	2	84d.				
N^2				1	4d.	0	72d.				
AMINO ACID SALT: "EDTA" - Ethylenediamine tetraacetic acid (disodium salt), $\text{NH}_2\text{CH}_2\text{N}(\text{CH}_2\text{COOH})_2(\text{CH}_2\text{COONa})_2$, (R)											
34.42 g./l. solution											
125 g./l. water											
ΔL	0	5h.	0	66d.b/	0	62d.	1	72d.			
ΔW	4	104d.	5	94d.	1	106d.	2	120d.			
N^2	5	109d.	10	112d.	0	12d.	1	94d.			

^a100% of grade received, used without dilution.

^bOne specimen only.

TABLE 14

CONDITION OF SPECIMENS STORED IN ORGANIC CHEMICALS FOR 6 MONTHS

All specimens made with Type I cement and moist cured 28 days.

The change in length (ΔL), the change in weight (ΔW), and the square of the frequency (N^2) are all expressed as percent of initial readings.

		In 0.1 M Solutions		In Intermediate Concentrations		In Concentrated Solutions or Liquids	
		W/C = 0.6	W/C = 0.4	W/C = 0.6	W/C = 0.4	W/C = 0.6	W/C = 0.4
ALCOHOLS:							
Methyl Alcohol, Absolute, CH_3OH , (Pr)							
		3.20 g./l. solution		500 g./500 ml. water		a	
ΔL		-.02	-.04	-.02	-.04	-.02	-.01
ΔW	% of	+6	+2	-.2	-.5	-.5	-1.0
N^2	Initial	101	106	98	98	86	94
N		5270	6120	5090	5770	4760	5700
t-Butyl Alcohol, $(\text{CH}_3)_3\text{COH}$, (R)							
		7.41 g./l. solution		500 g./500 ml. water		a	
ΔL		.00	.00	+.02	+.02	-.04	-.05
ΔW	% of	+8	+6	-.4	-.8	-1.4	-1.5
N^2	Initial	107	100	102	100	97	90
N		5280	5820	5160	5800	4980	5600
Ethylene Glycol, $(\text{CH}_2\text{OH})_2$, (T)							
		6.21 g./l. solution		500 g./500 ml. water		a	
ΔL		-.01	.00	.00	.00	-.15	-.03
ΔW	% of	-.0	+6	+.8	+.1	+.5	-1.2
N^2	Initial	108	100	94	96	85	87
N		5420	5780	5090	5670	4700	5440
Glycerol, $\text{CH}_2\text{OHCHOHCH}_2\text{OH}$, (ACS)							
		9.21 g./l. solution		500 g./500 ml. water		a	
ΔL		.00	.00	.00	.00	.09	-.10
ΔW	% of	+9	+6	+.8	-.4	-1.1	-3.5
N^2	Initial	105	101	95	102	97	92
N		5260	6020	5080	5850	5070	5700
SUGAR:							
Sucrose, $\text{C}_{12}\text{H}_{22}\text{O}_{11}$, (USP)							
		34.23 g./l. solution		500 g./500 ml. water			
ΔL		-.10	.00	-.01	.00		
ΔW	% of	+1.8	-.2	-.5	-.5		
N^2	Initial	101	101	102	99		
N		5230	5820	5080	5780		

TABLE 14 (Cont.)

		In 0.1 M Solutions		In Intermediate Concentrations		In Concentrated Solutions or Liquids	
		W/C = 0.6	W/C = 0.4	W/C = 0.6	W/C = 0.4	W/C = 0.6	W/C = 0.4
ESTERS:		Butyl Stearate, $C_{17}H_{35}COOC_4H_9$, (P)					
		a					
ΔL						- .02	+ .02
ΔW	% of					-1.1	-.9
N^2	Initial					107	102
N						5730	5910
		Glyceryl Tristearate, $(C_{17}H_{35}COO)_3C_3H_5$, (T)					
		30 g./l. benzene					
ΔL				-.09	-.09		
ΔW	% of			-.9	-1.0b		
N^2	Initial			101	97		
N				5170	5730		
HYDROCARBON:		Benzene, C_6H_6, (PI)					
		a					
ΔL						-.08	-.06
ΔW	% of					-.8	-.8
N^2	Initial					105	97
N						5060	5770
DRYING OIL:		Linseed Oil, (P)					
		a					
ΔL						+ .01	+ .02
ΔW	% of					-1.3	-.8
N^2	Initial					100	100
N						5060	5920

^a100% of grade received, used without dilution.

^bOne specimen only.