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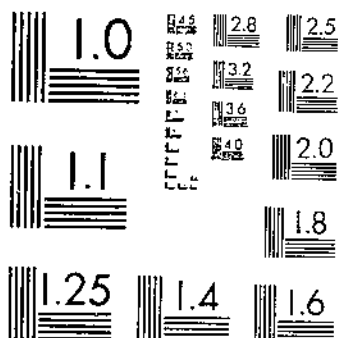
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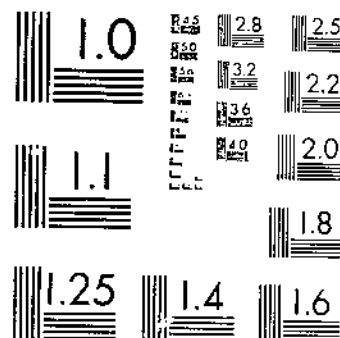
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LABORATORY AND FIELD TESTS OF CONCRETE EXPOSED TO THE ACTION OF SULPHATE  
MILLER, D. G. ; HANSON, P. W. 1 OF 1

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MICROCOPY RESOLUTION TEST CHART  
NATIONAL BUREAU OF STANDARDS-1963-A



MICROCOPY RESOLUTION TEST CHART  
NATIONAL BUREAU OF STANDARDS-1963-A



UNITED STATES DEPARTMENT OF AGRICULTURE  
WASHINGTON, D. C.

# LABORATORY AND FIELD TESTS OF CONCRETE EXPOSED TO THE ACTION OF SULPHATE WATERS<sup>1</sup>

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## INTRODUCTION

In the spring of 1919 the attention of the United States Department of Agriculture was called to a number of failures of concrete tile in the ground in southwestern Minnesota. At a conference between various interested organizations the Bureau of Public Roads<sup>2</sup> agreed to inves-

<sup>1</sup> A report of progress of experiments conducted under cooperative agreement between the Bureau of Agricultural Engineering of the U. S. Department of Agriculture, the University of Minnesota, and the Division of Drainage and Waters of the Department of Conservation, State of Minnesota. In the files of the university this report is Journal Series Paper No. 1106 of the Agricultural Experiment Station.

<sup>2</sup> Prior to the organization of the Bureau of Agricultural Engineering in 1931, the work of the U. S. Department of Agriculture in this investigation was done by the Division of Agricultural Engineering of the Bureau of Public Roads.

tigate the situation. A systematic field study was begun in the fall of 1919 and covered 23 counties in Minnesota and 4 counties in northern Iowa. Tile failures were located and samples of drain water and soil water analyzed. The study revealed a marked correlation between alkali<sup>3</sup> concentrations and failures of concrete tile. A laboratory was established at University Farm, St. Paul, Minn., in cooperation with the Agricultural Experiment Station and the Department of drainage and waters<sup>4</sup> of the State, to make an exhaustive investigation of the effect of alkali waters on concrete.

The act of the 1920 session of the Minnesota Legislature that provided funds for the draitile laboratory called for an "investigation of the causes of failure of agricultural draitile, the means of obviating such failures, and mapping of areas where extra precautions are necessary." In July, 1921, a bulletin (57)<sup>5</sup> based on many field examinations of draitile and on chemical analyses of 1,062 water samples and 150 soil samples, clearly showed the cause of failure to be the presence of alkali in the subsoil, in the form of the sulphates of magnesium ( $MgSO_4$ ) and sodium ( $Na_2SO_4$ ). In January, 1927, a bulletin (35) was published containing a map of Minnesota showing where extra precautions in using concrete tile had been found necessary.

This bulletin is intended to furnish to engineers, tile manufacturers, and tile users, additional information that will more nearly make it possible to completely obviate failures of agricultural draitile. At the same time the bulletin will make generally available the results of the work to date. It gives results of observations on the behavior of experimental specimens subjected to the action of artificial sulphate solutions in the laboratory, and the behavior of specimens installed under natural-field exposure conditions in Minnesota, North Dakota, and South Dakota. For this work more than fifty thousand 2 by 4 inch cement-concrete and cement-mortar cylinders, 1,000 cement-mortar briquets, 3,000 specially made concrete draitile, and numerous miscellaneous specimens, have been made. The experiments, while originally planned to aid in the general improvement of farm draitile, have a wide application to the use of concrete culverts, water and sewer pipe, irrigation structures, foundations, and all other types of concrete construction that, in service, must resist the action of soils or waters rich in sulphates.

#### EARLY STUDIES OF THE CONCRETE-ALKALI PROBLEM

Since the invention of Portland cement in 1824 many studies have been made and much research is still under way, looking toward solving the problem of deterioration of cement in contact with sulphate waters. It is believed that the following brief historical review will be helpful to workers in this field.

Among the many earlier European workers on this subject are Le Chatelier (30), in 1887, Michaelis (32) in 1891, and Feret (22), in 1890. Bied (10) in 1909 reported the results of 6 years laboratory tests of the action of sulphate solutions on mortars containing artificial pozzuolanas. Poulsen in 1923 issued a report (43) in which he

<sup>3</sup> The word "alkali" is used throughout this bulletin in the sense in which it is commonly used in arid and semiarid regions of the United States, and may mean any one or more, singly or in combination, of the sulphates, chlorides, and carbonates of sodium, magnesium, and calcium. In this sense sea water can be classed as "alkali" water.

<sup>4</sup> Now the Division of Drainage and Waters of the Department of Conservation.

<sup>5</sup> Italic numbers in parentheses refer to Literature Cited, p. 77.

describes a series of field experiments started on a comprehensive scale in 1896 by the Scandinavian Association of Manufacturers of Portland Cement, to determine the influence of various factors on the resistance of concrete exposed to sea water; Candlot (16) reviewed the results and conclusions by Vienne and other French engineers based largely on 40 years of investigations of experimental cubes installed between 1856 and 1875 in the harbor of La Rochelle. French engineers and chemists acknowledged the existence of the "alkali" problem three-quarters of a century ago. In fact, studies by both the French and English are older than Portland cement, for Vicat recognized the problem as far back as 1812 in connection with work on limes and natural cements reported upon in 1818 (50). Antedating the work of Vicat by more than a half century, Smeaton (44) experimented to secure a mortar that would best resist sea water, when he built the Eddystone Lighthouse in 1756-1759.

The concrete-alkali problem was not seriously considered in North America until nearly the beginning of the present century, after failures of important structures in widely separated parts of the country had occurred. Very evident deterioration of maritime structures along the North Atlantic seaboard, particularly in Boston Harbor, finally focused the attention of engineers and chemists of the eastern United States and resulted in an impressive series of field tests by the Aberthaw Construction Co. of Boston (2). This company, in 1909, made 24 concrete beams 16 feet long by 16 inches square and suspended them in Boston Harbor, in Charlestown Navy Yard, with their tops above high water and their bottoms below low water, so that they were subjected to the chemical action of sea water, to the mechanical effect of alternate wetting and drying, and to frost.

More or less contemporaneous with the Aberthaw tests, but entirely independent and possibly antedating them in some phases, were studies by the United States Bureau of Standards reported by Bates, Phillips, and Wig (8) in 1912. That paper was based on observations and chemical examinations of hollow test cylinders  $3\frac{1}{2}$  inches in outside diameter and 10 inches long through which were passed solutions of various kinds, and on compression tests of 8 by 16 inch cylinders installed in the open sea. These studies were fundamental in nature, and the behavior of mortar and concrete specimens of Portland, slag, iron-ore, and natural cements was observed.

About the time the Aberthaw tests in Boston Harbor were begun the interest of engineers and chemists throughout the western United States was aroused by disintegration of sections of the sewer system of Great Falls, Mont., as reported upon by Tannatt and Burke (47) in 1908. The first failure noted in this sewer system occurred in a 26 by 32 inch oval main shortly after its construction in 1890. This failure occurred about the time that extensive reclamation development began in the West. Troubles that developed with concrete structures built by the United States Reclamation Service in alkali soils and waters were reported by Jewett in 1908 (27). Also in 1908 was issued the pamphlet by Hadden (25) based chiefly on limited chemical work that followed observed failures of small drain tile in western Colorado. In 1910 a bulletin by Burke and Pinckney (14), containing chemical analyses and observed physical effects of storing briquettes in sulphate, carbonate, chloride, and other solutions, was

published. In 1915 the paper by Wig and Williams (54) based on results of the first year's tests of experimental tile installed in alkali soils in eight Western States was issued. Other reports on this work followed in 1917 (55), 1922 (58), and 1926 (59). Along the same general line as the paper by Burke and Pinckney in 1910, but broader, was the bulletin by Steik (46) published in 1917 and based on extended tests of briquettes in many solutions of several strengths for periods of as much as seven years.

A great many other papers reporting investigations and research on deterioration of concrete exposed to alkali have been printed since the inception of the Aberthaw tests and the report of the failure of the Great Falls sewer, coming from many sources in North America. Among them the following three are considered particularly noteworthy because of their historical and bibliographic matter: (1) A bibliography of the United States Department of Agriculture issued in 1925 (28) and supplemented in 1931<sup>\*</sup> is the most complete compilation of references on this subject. (2) Atwood and Johnson (6), trace the history of cement in sea water and analyze results obtained by various investigators. This paper cites 113 references. (3) A paper by Pagon (40), first published in 1915-16 is a comprehensive collection of the experiences and opinions of many workers, and has appended references to 145 printed articles.

#### ORGANIZATIONS MAKING CONCRETE-ALKALI INVESTIGATIONS

Although valuable reports on various phases of concrete-alkali investigations emanated from many sources between 1920 and 1930, there developed during this period an evident tendency in the United States and Canada to leave such work to organizations sufficiently interested properly to finance research that was broader and more fundamental in scope, for the most part, than much of the research previously undertaken. Some such organizations, now active, are:

The Bureau of Standards of the United States Department of Commerce makes investigations on the effect of alkali on Portland cement in cooperation with the Portland Cement Association, through a fellowship created in 1924 for fundamental studies of the constitution and hardening of Portland cement.

The research laboratory of the Portland Cement Association began in 1921 a comprehensive series of field and laboratory tests of Portland cement specimens exposed to alkali. In addition to doing the laboratory work, this organization has periodically examined some 2,000 concrete cylinders 10 inches in diameter and 24 inches long, after their exposure to sulphate soils and waters in Colorado, South Dakota, and western Canada.

The Engineering Institute of Canada in 1921 appointed a committee on deterioration of concrete in alkali soils, to continue the concrete-alkali investigations begun in 1918 by the Calgary branch of the institute and in 1919 by the University of Saskatchewan as field experiments carried on by exposing concrete blocks of known quality to sulphate waters. Between 1921 and 1928 the work was financed by the National Research Council of Canada, the Canadian Pacific Rail-

<sup>\*</sup> UNITED STATES DEPARTMENT OF AGRICULTURE, BUREAU OF AGRICULTURAL ENGINEERING. SUPPLEMENTARY BIBLIOGRAPHY RELATING TO THE DELETERIOUS ACTION OF SOIL ALKALIES AND OTHER CHEMICAL AGENTS ON CEMENT AND CONCRETE. 15 p. 1931. [Mimeographed.]

way, the Canada Cement Co., the city of Winnipeg, the three prairie Provinces, and the University of Saskatchewan, but in 1928 the National Research Council of Canada took this over and it is now carried on almost exclusively in the chemical laboratory of the University of Saskatchewan.

The Bureau of Public Roads has been conducting experiments with mortar and concrete cylinders surface treated or immersed in such preparations as water-gas tar, coal tar, and paraffin. In this work the physical effects of alkali action have been studied chemically and microscopically.

## GENERAL DESCRIPTION OF TESTS

### TEST SPECIMENS

Most of the test specimens were cylinders 2 inches in diameter by 4 inches long, used partly because the 2-inch diameter roughly approximates the wall thickness of many of the tile used in public drains in Minnesota and other States of the Middle West. A small number of standard briquets and commercial draitile were also tested.

The greater number of the test cylinders were made of concrete, not merely mortar, although only pebbles small enough to pass a  $\frac{3}{8}$ -inch square opening were used. The aggregate met all standard physical tests. It was separated into screen sizes and recombined, as shown in Table 1, to produce a fineness modulus of 4.67. The mineralogical composition of a sample of the combined materials is recorded in Table 2. Roughly, about 75 per cent of the aggregate may be classed as siliceous, 15 per cent argillaceous, and 10 per cent calcareous. The unit dry weight of the combined fine and coarse aggregate was 124 pounds per cubic foot. This was the highest unit weight of dry-rodded material that could be obtained by any combination of screen sizes, as was determined by repeated trials. The average weight of the concrete cylinders 24 hours after they were made, was about 505 grams and variations of individual cylinders from this average rarely exceeded 2 per cent.

TABLE 1.—Screen analysis of aggregate and quantity of each size used for 9-cylinder batch of 2 by 4 inch laboratory standard cylinders

Screen size	Passing screen	Retained on screen	Total coarser than screen	Required for batch
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Grams</i>
$\frac{3}{8}$ -inch.....	100.0	0	0	—
No. 4.....	50.3	43.7	43.7	1,543
No. 8.....	30.3	20.0	63.7	706
No. 16.....	24.4	11.0	75.6	420
No. 30.....	12.5	11.0	87.5	420
No. 50.....	3.1	9.4	95.9	332
No. 100.....	.4	2.7	99.5	110
Total.....			467.0	3,531
Fineness modulus.....			4.67	



TABLE 2.—*Petrological count of aggregate used in laboratory standard cylinders*

Component	Portion of entire sample	Portion of screen size			
		Passing 3/4-inch; retained on No. 4	Passing No. 4; retained on No. 10	Passing No. 10; retained on No. 20	Passing No. 20
	Per cent	Per cent	Per cent	Per cent	Per cent
Sandstone.....	27.5	33.0	30.0	22.0	10.0
Quartz.....	15.4	3.0	4.0	38.5	63.0
Slate.....	13.0	12.5	18.0	10.8	4.0
Granite.....	11.9	15.0	15.5	6.2	1.0
Rhyolite.....	10.8	12.5	13.0	7.0	5.0
Limestone.....	9.8	12.0	11.5	5.3	4.0
Chert.....	4.4	7.0	4.0	1.7	1.0
Feldspar.....	4.2	—	4.0	13.5	12.0
Gneiss.....	2.9	5.0	3.0	—	—
Total.....	100.0	100.0	100.0	100.0	100.0

E. C. E. Lord, Bureau of Public Roads, U. S. Department of Agriculture.

The cylinders with some exceptions were made in batches of nine. Each series consisted of five batches made on different days of the same week, the order of making being changed daily so that for the week it was the same for all series. Most of the cylinders were of 1:3 mix, with a relative consistency of 1.00 and a water-cement ratio of 0.62, which is 4.6 gallons of water per bag of cement. Each batch was mixed by hand at least 1½ minutes dry and 2 minutes after the water was added. After being mixed the materials were rodded in four layers in three 3-gang brass molds, each layer being tamped 20 times with a round-pointed steel rod ¾ by 15 inches in size. The cylinders were cured during the first 24 hours in a moist closet at room temperature. Following this routine, using local sand and gravel and storing the cylinders in distilled water until tested, produced concrete with high compressive strengths, generally in excess of 4,500 pounds at 28 days, and with an absorption of 6 per cent when tested in accordance with the American Society for Testing Materials standard specifications for draintile (5), which provide for oven drying at a temperature of not less than 230° F. followed by 5 hours in boiling water.

A limited number of mortar cylinders and standard briquets were made of standard Ottawa sand, and these fairly well represented very poorly graded aggregate characteristic of that used too frequently in small draintile. The compressive strength of the mortar cylinders that were mixed 1:3 ordinarily averaged between 2,500 and 3,000 pounds per square inch at 28 days, with an absorption of 10 per cent when the cylinders were tested like the others. Draintile of 5 and 6 inches diameter were made at three commercial tile plants.

#### EXPOSURE CONDITIONS

It was originally planned to base most of the conclusions on results obtained with cylinders stored in solutions in the laboratory. It became apparent, however, that this procedure would ignore factors encountered by concrete in service or would involve very great expense. The work was therefore broadened, and has included the following exposure conditions:

Cylinders and briquets were stored in the laboratory at ordinary room temperatures, in pure solutions of magnesium sulphate ( $\text{MgSO}_4$ ) and sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) ranging in strength from one-fourth of 1 per cent to 1.5 per cent. All solutions were held in covered 5-gallon earthenware jars, 10 liters (2.6 gallons) in each. The solutions were changed at intervals of one to four weeks. Never more than 20 cylinders were stored in any jar.

Cylinders and draitile were stored in the alkali water of Medicine Lake, 18 miles northwest of Watertown, S. Dak. This is a 300 or 400 acre body of clear water, some 30 to 40 feet deep, with stretches of gravel beach that afford excellent conditions for installing and examining specimens. As in many other lakes of the upper Mississippi River Basin, the water level of Medicine Lake has considerably receded during the last eight years. This increased the salt concentration from 2.34 per cent on April 29, 1924, to 7.42 per cent on October 21, 1931. Analyses of six water samples taken December 10, 1923; February 14 and April 29, 1924; and February 18, July 1 and October 21, 1931, are averaged in Table 3 and show a total salt content of 4.79 per cent, almost wholly magnesium and sodium sulphates. Medicine Lake freezes over, but all cylinders used in these experiments were installed at depths well below that to which the water froze.

TABLE 3.—Average of six analyses<sup>1</sup> of water from Medicine Lake, S. Dak., 1923 to 1931

Component	Quantity	Reacting value	Component	Quantity
Radicals:	Parts per million	Per cent	Anhydrous salts:	Parts per million
Na.....	4,313	11.50	$\text{NaNO}_3$ .....	Traces
Ca.....	1,030	3.48	$\text{NaCl}$ .....	1,053
Mg.....	5,323	35.02	$\text{Na}_2\text{SO}_4$ .....	12,041
$\text{NO}_3$ .....	Traces	.00	$\text{MgSO}_4$ .....	31,298
Cl.....	639	1.22	$\text{CaSO}_4$ .....	2,823
$\text{SO}_4$ .....	35,108	48.08	$\text{CaCO}_3$ .....	194
$\text{CO}_2$ .....	117	.27	$\text{Ca}(\text{HCO}_3)_2$ .....	492
$\text{HCO}_3$ .....	370	.43	Total.....	47,901
Total.....	47,901	100.00		

<sup>1</sup> Analyses by the water and beverage laboratory, Bureau of Chemistry and Soils, U. S. Department of Agriculture.

Draitile of 5 and 6 inches diameter were installed 6 to 7 feet deep as part of a tile system in alkali soil in Lyon County, southwestern Minnesota. About 50 feet of poorly made commercial 6-inch concrete tile had failed by disintegration at this location and were replaced in 1919 after but eight months' service. Soil conditions are represented by the water analysis first shown in Table 4. Draitile of 5 and 6 inches diameter were also buried 18 inches deep along the margin of an alkali slough in Cass County in southeastern North Dakota. The analysis of soil water taken from the trench in which these tile were installed is also shown in Table 4.

TABLE 4.—Analyses <sup>1</sup> of soil water samples taken at concrete drain tile installations

Component	Lyon County, Minn.		Cass County, N. Dak.		Component	Lyon County, Minn.		Cass County, N. Dak.	
	Quan- tity	React- ing value	Quan- tity	React- ing value		Quantity		Quantity	
Radicals:	<i>Parts per million</i>		<i>Parts per million</i>		Anhydrous salts:	<i>Parts per million</i>		<i>Parts per million</i>	
Na (calculat- ed).....	716	4.42	27,362	48.18	NaNO <sub>3</sub> .....	3		5,533	9
Ca.....	193	1.37	670	1.15	NaCl.....	2,206		77,770	
Mg.....	3,793	44.21	200	.87	Na <sub>2</sub> SO <sub>4</sub> .....	13,619		991	
NO <sub>3</sub> .....	0	.00	2	.00	MgSO <sub>4</sub> .....	78			
Cl.....	2	.01	3,360	3.84	MgCO <sub>3</sub> .....			1,168	
SO <sub>4</sub> .....	16,349	48.38	54,203	45.70	CaSO <sub>4</sub> .....	92			
CO <sub>3</sub> .....	111	.52			CaCO <sub>3</sub> .....	632		915	
HCO <sub>3</sub> .....	476	1.11	680	.46	Ca(HCO <sub>3</sub> ) <sub>2</sub> .....				
Total.....	21,630	100.00	86,336	100.00	Total.....	21,630		86,386	

<sup>1</sup> Analyses by the water and beverage laboratory, Bureau of Chemistry and Soils, U. S. Department of Agriculture.

## METHOD OF MEASURING ALKALI ACTION

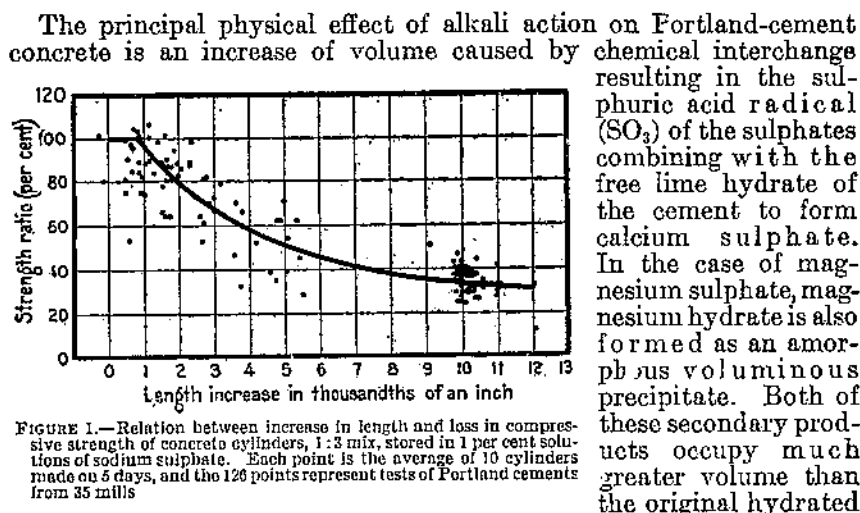
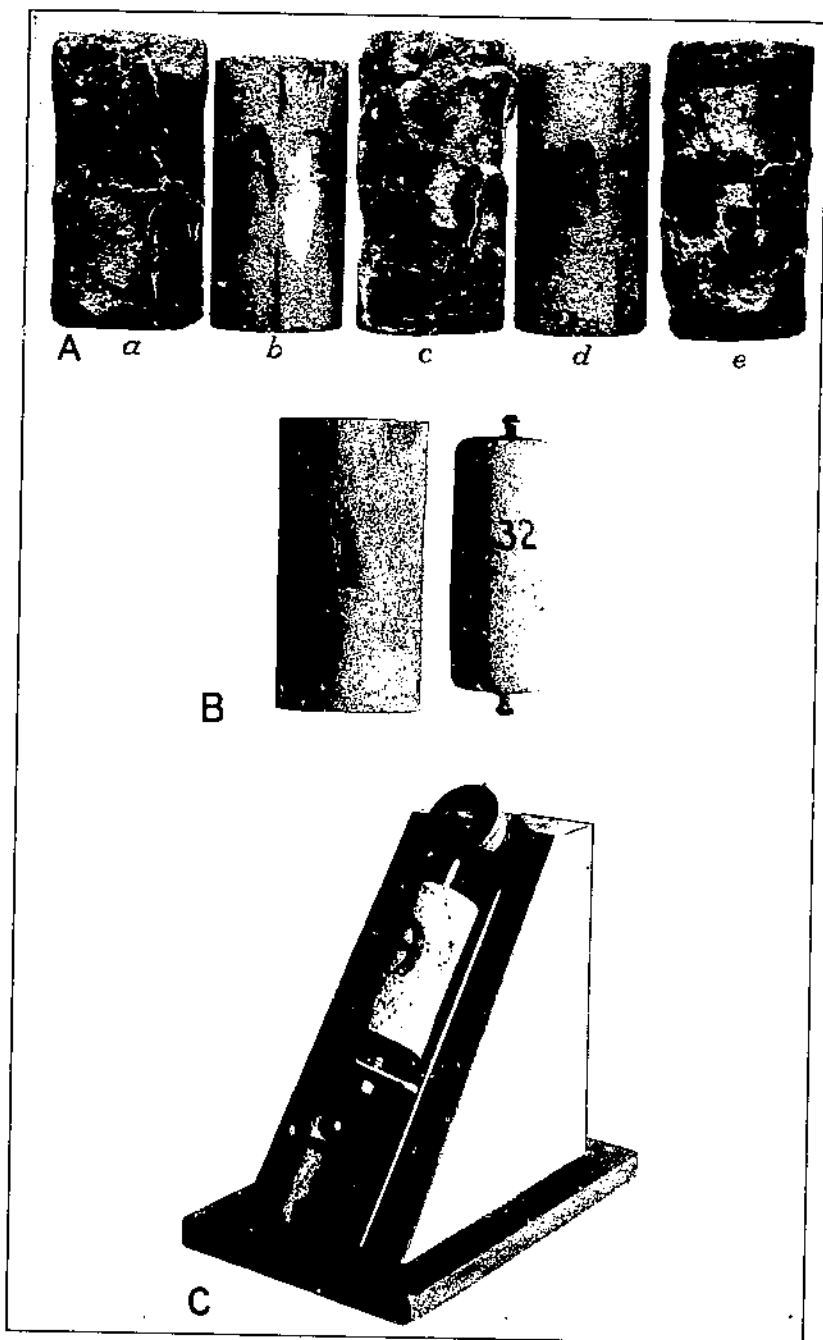


FIGURE 1.—Relation between increase in length and loss in compressive strength of concrete cylinders, 1:3 mix, stored in 1 per cent solutions of sodium sulphate. Each point is the average of 10 cylinders made on 5 days, and the 126 points represent tests of Portland cements from 35 mills

cement and free lime hydrate and are the chief causes for the physical rupture of the concrete illustrated in Plate 1, A. This characteristic was utilized in studying the progress of deterioration of test cylinders.

Round-head 1-inch brass screws were set in neat cement in the ends of one-third of the cylinders in the series exposed in the laboratory and of those in a few series exposed in Medicine Lake. Measurements between screw heads were taken in recording changes in the length of the cylinders. Both the amount and the rate of increase, as indicated by length changes, have been used in comparing behavior. Measurements were made with an Ames dial graduated to thousandths of an inch, and by interpolation, measurements were recorded to 0.0001 inch. The special mounting illustrated in Plate 1, C was devised to facilitate making the readings. Many readings made with this device by different observers indicate an accuracy of about 0.0002 inch.

The relation between increase in length and loss in compressive strength, based on tests of several hundred cylinders, is shown in



A. Three standard Ottawa sand cylinders (*a*, *c*, *e*) after storage in 1 per cent solutions of magnesium sulphate compared with two cylinders from the same lot (*b*, *d*) stored in distilled water, showing increase in volume due to sulphate action. B. Neat cement 2 by 1 inch cylinder after 10 years in 4 per cent solution of magnesium sulphate compared with cylinder stored in tap water. Cylinder from solution had but 38 per cent of its original volume. C. Special mounting for Ames dial used to measure length changes of 2 by 1 inch test cylinders.

Figure 1. The compression tests were made on blank-end cylinders from the same batches as the cylinders measured for length. The graph indicates that in these 2 by 4 inch cylinders of 1:3 mix and 0.62 water-cement ratio, an increase in length of 0.01 inch or 0.25 per cent predicated an average loss in strength of about 66 per cent. This relation was found to hold consistently with the different brands of Portland cement in mixes leaner than 1:2, and to vary only moderately with water-cement ratios between 0.44 and 0.73. Figure 2 shows the effect of the strength of sulphate solution upon the rate of increase in length of cylinders.

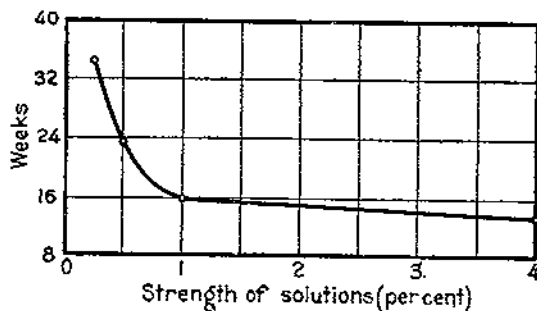


FIGURE 2.—Time required for mortar and concrete cylinders stored in magnesium sulphate solutions to increase in length 0.01 inch. Each point is the average for 25 cylinders in five series. (Cements A and B1 mixed in equal portions)

Because of the consistency of the results plotted in Figures 1 and 2, the practice of rating relative resistance of the test cylinders on the basis of time required to increase in length by 0.01 inch, and of considering the usefulness or "life" of all such cylinders to have ended when that increase in length had occurred, was adopted. For a mix of 1:1 and for neat cement, an increase of 0.01 inch in the cylinders was found to indicate a loss of strength considerably greater than the loss for the leaner mixes. The reason for this is discussed under "Quantity of cement in mix."

#### SULPHATE ACTION IN RELATION TO STRENGTH OF SOLUTION

The destructive action of solutions of magnesium sulphate and sodium sulphate increases with the strength of the solution but at a

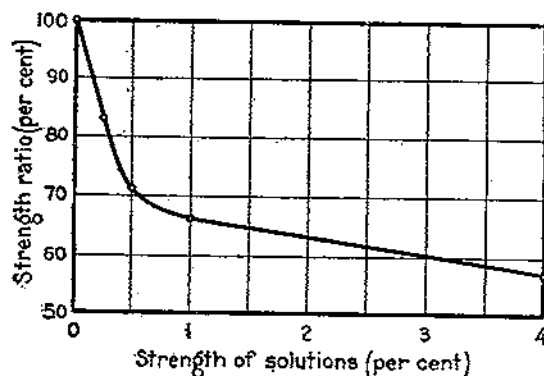


FIGURE 3.—Strength ratios of mortar and concrete cylinders after storage for 15 weeks in magnesium sulphate solutions of different strengths. Each point is the average for 25 cylinders in 5 series. (Cements A and B1 mixed in equal portions)

diminishing rate. Figures 2 and 3 show that with magnesium sulphate solutions between 0.25 and 0.5 per cent strength, the rate of action on the cylinders was somewhat proportional to the strength of the solution. With strengths greater than 1 per cent the rate of action increased less rapidly, and cylinders in 4 per cent solutions had compressive strengths averaging 86 per cent of those in 1 per cent

solutions. Table 5 and Figures 4 and 5 show similar results observed in briquets stored in 1, 5, and 15 per cent solutions of magnesium sulphate and of sodium sulphate, the destructive action of the 5 per cent solutions being in all cases much less than five times

that of the 1 per cent solutions and the action of the 15 per cent solutions not approaching three times that of the 5 per cent solutions.

TABLE 5.—Condition of standard Ottawa sand briquets stored for various periods in sodium sulphate and magnesium sulphate solutions of different strengths

[Conditions were rated visually, from 0 indicating complete disintegration to 10 indicating no apparent action of the solution upon the briquet]

Portland cement	Lots tested (number)	Condition of briquets immersed in solution for time stated											
		4 weeks						12 weeks					
		Na <sub>2</sub> SO <sub>4</sub>			MgSO <sub>4</sub>			Na <sub>2</sub> SO <sub>4</sub>			MgSO <sub>4</sub>		
		1 per cent	5 per cent	15 per cent	1 per cent	5 per cent	15 per cent	1 per cent	5 per cent	15 per cent	1 per cent	5 per cent	15 per cent
I.....	4	10	10	10	10	10	10	10	10	10	10	10	10
C.....	5	10	10	10	10	10	10	10	10	10	10	10	9
H.....	5	10	10	10	10	10	10	10	10	10	10	10	9
K <sub>2</sub> .....	4	10	10	10	10	10	10	10	10	10	10	10	9
J.....	1	10	10	10	10	10	10	10	10	10	10	10	10
G.....	5	10	10	10	10	10	10	10	10	10	10	10	9
E.....	2	10	10	10	10	10	10	10	9	8	10	9	7
F.....	1	10	10	10	10	10	10	10	10	9	10	10	9
D.....	6	10	10	9	10	10	9	9	7	6	10	9	3
M.....	2	10	10	9	10	10	9	9	6	3	10	9	2
B <sub>1</sub> .....	11	10	8	8	10	9	7	7	5	2	9	6	3
AA.....	2	10	10	10	10	10	10	10	8	7	10	9	8
P.....	1	10	10	10	10	10	9	10	9	2	10	9	5
A.....	7	9	5	5	10	9	3	3	2	1	8	4	2
½A and ½B <sub>1</sub> .....	5	10	9	8	10	10	9	8	6	4	9	7	5
Average of 61 lots.....		9.9	9.1	8.8	10.0	9.7	8.4	8.4	7.3	6.1	9.5	8.1	5.9

Portland cement	Lots tested (number)	Condition of briquets immersed in solution for time stated											
		20 weeks						26 weeks					
		Na <sub>2</sub> SO <sub>4</sub>			MgSO <sub>4</sub>			Na <sub>2</sub> SO <sub>4</sub>			MgSO <sub>4</sub>		
		1 per cent	5 per cent	15 per cent	1 per cent	5 per cent	15 per cent	1 per cent	5 per cent	15 per cent	1 per cent	5 per cent	15 per cent
I.....	4	10	10	8	10	9	8	10	10	8	10	8	7
C.....	5	10	10	9	10	9	6	10	10	8	10	8	6
H.....	5	10	8	8	10	9	6	10	7	6	10	8	4
K <sub>2</sub> .....	4	10	9	8	10	9	7	10	8	7	10	8	3
J.....	1	10	10	10	10	9	9	10	10	9	10	9	8
G.....	5	10	8	7	10	9	6	10	7	4	10	8	3
E.....	2	9	7	8	10	8	4	8	2	0	10	5	0
F.....	1	10	10	6	10	9	7	10	8	0	10	7	0
D.....	6	6	3	1	9	6	1	6	1	0	8	4	0
M.....	2	7	2	0	9	6	0	5	0	0	9	4	0
B <sub>1</sub> .....	11	5	2	0	8	4	1	4	0	0	7	3	0
AA.....	2	8	6	3	10	8	3	7	3	0	10	6	0
P.....	1	10	1	0	10	8	0	10	0	0	10	7	0
A.....	7	2	1	0	5	3	1	1	0	0	4	1	0
½A and ½B <sub>1</sub> .....	5	7	4	0	8	6	3	5	1	0	6	4	0
Average of 61 lots.....		7.3	5.3	3.7	8.8	6.7	3.6	6.8	3.8	2.6	8.2	5.3	1.9

The curves in Figures 2 and 3 are of the same general type as those in Figures 4 and 5. The actual difference in rate of action probably was due to difference in the type of the specimens, though it is possible that part of the difference was only apparent and was the result of using dissimilar methods of rating the cylinders and the briquets.

The general shape of the curves in Figures 2, 3, 4, and 5 is interpreted to mean that the rate of disintegration is dependent not only on the quantity of salt present but also on the quantity and availability within the specimen of those soluble constituents of the cement that react with the sulphates. This has great significance in the practical use of concrete which may be subjected to the action of sulphates, as it indicates that density of the concrete, to the greatest degree of impermeability obtainable, is a primary requisite of permanence. It suggests also that precautionary measures very nearly identical must be taken for all really serious sulphate-

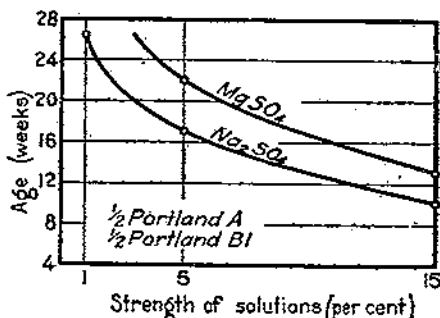


FIGURE 4.—Time required for standard Ottawa sand briquets stored in sulphate solutions to deteriorate 50 per cent. (Visual rating, see Table 5.) Each point is the average for five briquets

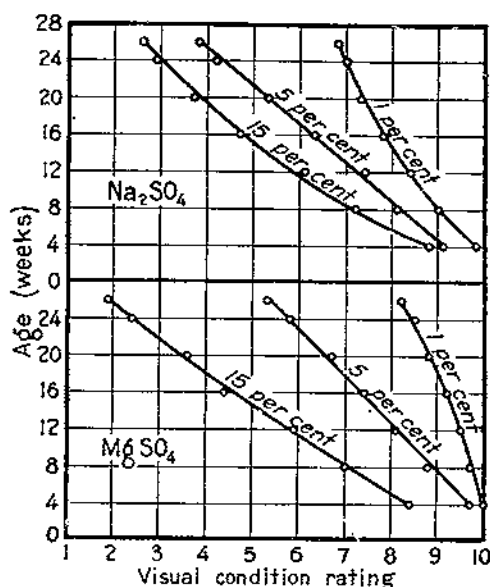


FIGURE 5.—Condition ratings based on appearance of standard Ottawa sand briquets stored in sulphate solutions for 26 weeks. Each point is the average for 61 briquets

exposure conditions, regardless of the actual quantities of sulphates present.

For consistent test results in any set of concrete-alkali laboratory experiments it is essential that the strengths of all solutions be maintained reasonably constant. Excessive variations of the time interval between changes may greatly influence the rate of action. Continually changing solutions would be ideal, but there are practical difficulties in such an arrangement. The effect of varying the time interval between renewals of 1 per cent solutions of sodium sulphate is shown in Figure 6. Two types of cylinders were used, one made of Minnesota sand and pebbles graded to produce a fineness modulus of 4.67, the other made of pit-run sand passing a No. 4 screen and having a fineness modulus of 2.83.

#### ACTIONS OF SODIUM SULPHATE AND OF MAGNESIUM SULPHATE COMPARED

The sodium and magnesium sulphates did not differ greatly in their effect upon the concrete, although, with most of the cements

used, the action of sodium sulphate was slightly more severe for solutions of the same strength. This is shown by Figures 4 and 5, which compare the effects of the two salts upon halves of identical briquets. The briquets used had been broken in the 7-day tensile tests, and the two halves of each briquet had been stored in different solutions of equal strength. In Figure 5 each curve was constructed by averaging

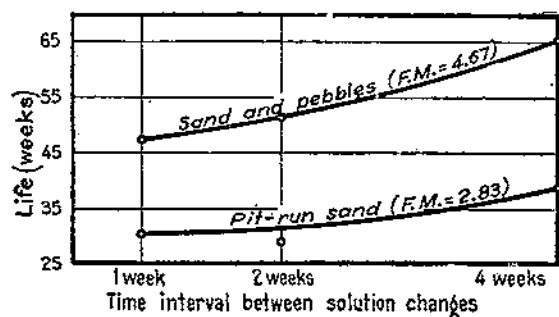


FIGURE 6.—Relation between the time elapsing between renewal of solutions and the life of two types of concrete cylinders stored in 1 per cent solutions of sodium sulphate. Each point is the average for 10 cylinders made on five days. (Cements A and B1 mixed in equal portions)

by sodium sulphate, while for other cements the reverse is true. The order of resistance to each salt is shown in Table 6. Plate 2 shows photographs of standard briquets made of 27 cements, after six months' exposure in 1 per cent solutions of the sulphates. All the data show clearly that a cement relatively high in resistance to one of the two salts is likewise high in resistance to the other, and that a cement low in resistance to one salt is low in resistance to the other.

TABLE 6.—Order of resistance to sulphate action of 14 standard Portland cements

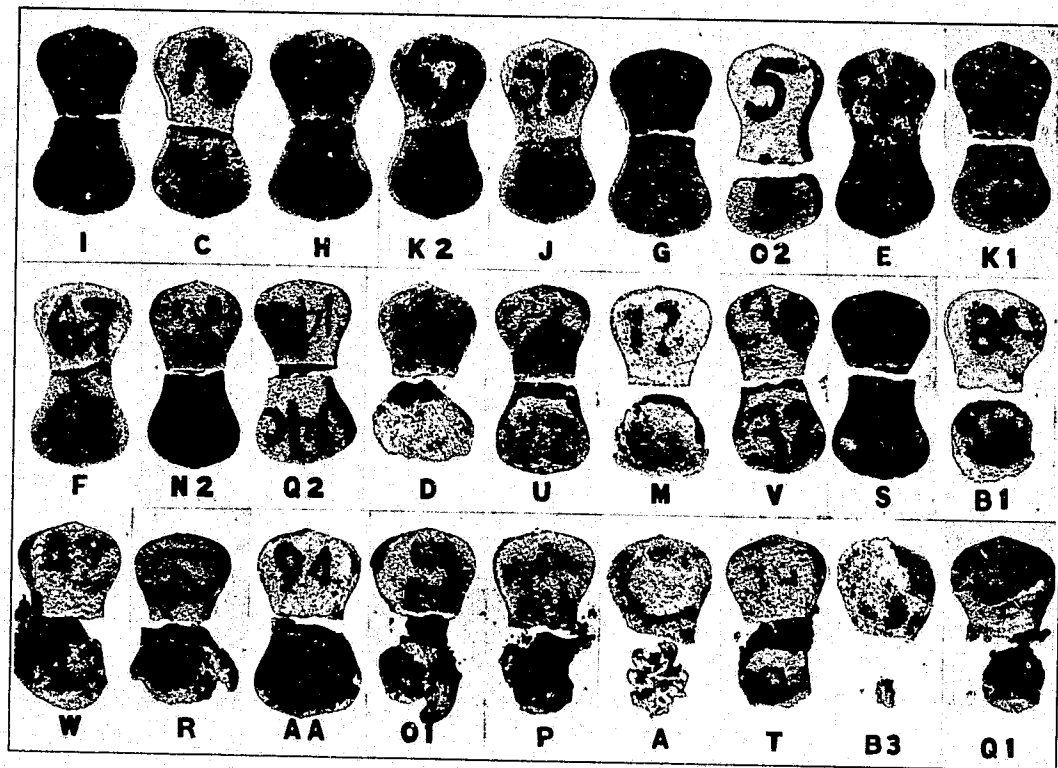
[For each sulphate this is the order of the sums of the condition ratings at 26 weeks for the 3 solution strengths, as shown in Table 5]

Portland cement	Lots tested (number)	Order of resistance to—		Portland cement	Lots tested (number)	Order of resistance to—	
		Sodium sulphate	Magnesium sulphate			Sodium sulphate	Magnesium sulphate
J.....	1	1	1	P.....	1	8	8
I.....	4	2	2	AA.....	2	9	9
O.....	5	3	3	B.....	2	10	10
K2.....	4	4	5	D.....	6	11	12
H.....	5	5	4	M.....	2	12	11
G.....	5	6	6	B1.....	11	13	13
F.....	1	7	7	A.....	7	14	14

#### EFFECT OF SHALE IN AGGREGATE

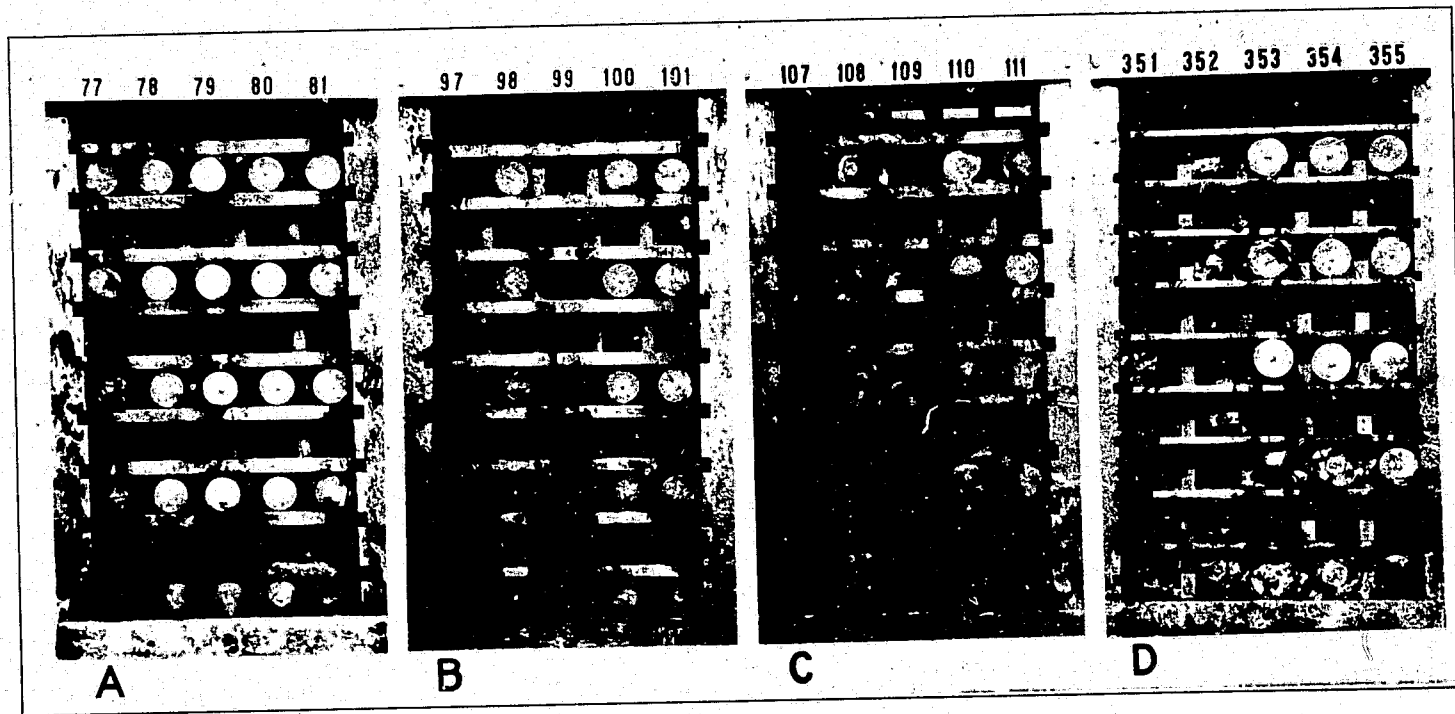
In some localities it is difficult to find deposits of sand and gravel entirely free of shale. The Minnesota Department of Highways permits 4 per cent of shale by volume in fine aggregate and 0.5 per cent in coarse aggregate for 1-course concrete. However, considerable drain tile in which the sand contained much more than 4 per





STANDARD BRIQUETS AFTER 6 MONTHS IN 1 PER CENT SULPHATE SOLUTIONS.

Upper halves were in magnesium sulphate and lower halves in sodium sulphate. A different standard Portland cement was used in each briquet



## CYLINDERS CURED IN STEAM OR WATER VAPOR AFTER STORAGE IN MEDICINE LAKE

A, Cured in steam at 212° F. after 7½ years in the lake. B, Cured in water vapor at 155° after 7½ years in the lake. Series 98, 100, and 101 were also cured in steam at 212°. C, Ottawa sand cylinders cured in water vapor at 155° after 7½ years in the lake. Series 108, 110, and 111 were also cured in steam at 212°. D, Cured in water vapor or steam for 12 hours at temperatures of 190°, 212°, 235°, 260° and 285°, respectively, after 6 years in lake.

cent shale has been marketed in Minnesota; one plant actually used sand containing 28 per cent shale and another plant 23 per cent. In experiments with the aggregates from these two plants it was found that removal of all the shale reduced the absorption of water by the concrete from 8.1 to 2.4 per cent in the one case and from 7.2 to 2.7 per cent in the other. It is evident that satisfactory drain tile can not be manufactured from such aggregates.

To indicate what influence shale may have on alkali resistance, cylinders made of a sand containing 9 per cent shale by volume were exposed, with cylinders of the same sand from which the shale had been removed by elutriation, to  $\frac{1}{4}$ ,  $\frac{1}{2}$ , 1, and 4 per cent solutions of magnesium sulphate. The relative resistance of two types of cylinders, with and without shale, is shown in Figure 7. It is clear that removing the 9 per cent of shale did not markedly alter the resistance of these specimens. However, removing 9 per cent of shale did reduce absorption by the aggregate from 3.1 to 1.6 per cent. This indicates the desirability of low shale content for any concrete subjected even to mild weathering agencies, irrespective of the influence of the shale on resistance to sulphates.

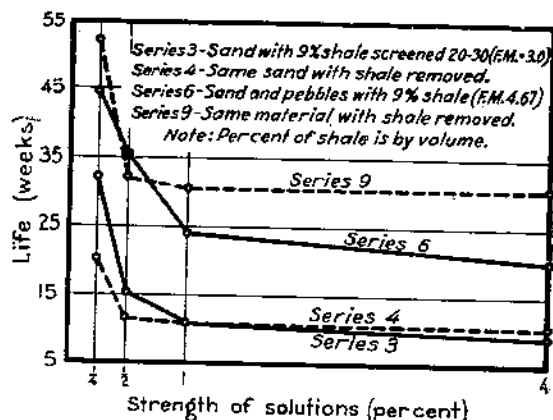


FIGURE 7.—Effect of shale in aggregate on the life of mortar and concrete cylinders stored in solutions of magnesium sulphate. Each point is the average for five cylinders made on different days. (Cements A and B1 mixed in equal portions)

#### QUANTITY OF WATER IN MIX

Because in the manufacture of small concrete drain tile it is common practice to remove the jackets immediately after tamping, an excess of mixing water is rarely used. On the contrary, there is a decided tendency to use too little water. At one Minnesota plant as little as 2.4 gallons of water per bag of cement was used regularly in making 6-inch tile. For this reason the cylinders in practically all the experiments herein reported have been made of concrete and mortar having low water-cement ratios.

To determine the influence of variations in the quantity of water on the resistance of dry-mixed concrete, the cylinders upon which Figure 8 is based were made and stored in 1 per cent solutions of magnesium sulphate. Cylinders in these five series had relative consistencies of 0.75, 0.90, 1.0, 1.10, and 1.25, and water-cement ratios of 0.44, 0.53, 0.59, 0.64, and 0.73, respectively. These water-cement ratios were equivalent to 3.3, 4.0, 4.4, 4.8, and 5.5 gallons of water per bag of cement.

Figure 8 shows that of this group of cylinders those with a relative consistency of 1.0, obtained with a water-cement ratio of 0.59, gave the best strength and absorption results and were most resistant to disintegration, although the cylinders with a relative consistency of

1.10, obtained with a water-cement ratio of 0.64, were only slightly less resistant. The driest-mixed cylinders gave the poorest strength and absorption results and had a life only 57 per cent of that of the

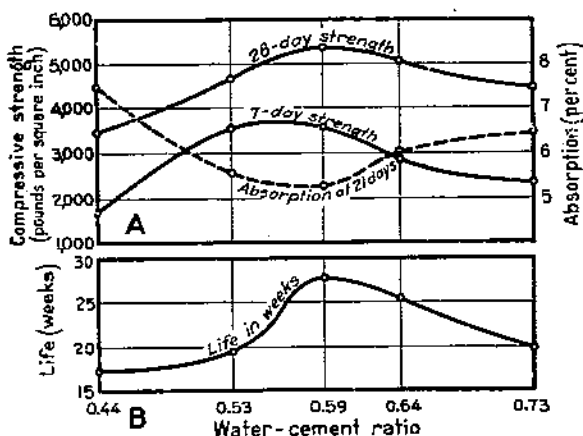


FIGURE 8.—Effect of water-cement ratio (A) on strength and absorption and (B) on life of concrete in 1 per cent solutions of magnesium sulphate. Each point is the average for five cylinders made on different days. (Cements A and B1 mixed in equal portions)

being the same. This is illustrated by the curves in Figure 9 based on tests of 1:5, 1:4, 1:3, 1:2, 1:1, and neat cement cylinders exposed to the action of 1 per cent solutions of magnesium sulphate. Enamered in the foregoing order, the relative resistances of the cylinders of this group were 1, 1.25, 3.3, 7, 36, and 66, the neat cement cylinders having a life 66 times that of the cylinders mixed 1:5. It is evident that only when the mix becomes as rich as 1:2 are outstanding results obtained, and the greatest value is not realized until it becomes as rich as 1:1.

Neat cement cylinders, after 10 years' exposure in 1 per cent magnesium sulphate solutions, showed an average decrease in length. Because of the density of neat cement the sulphate action was confined largely to the surfaces of the cylinders, and progressed continuously though slowly. This caused the cement to swell and fall off in very thin layers while, as indicated by length changes, the

best series. After these tests, it became the standard laboratory practice in mixing cylinders of this type to use a water-cement ratio of 0.59, which was later increased to 0.62 to improve workability and produce cylinders with smoother surfaces.

#### QUANTITY OF CEMENT IN MIX

The richer the mix the more resistant is concrete to sulphates, other factors

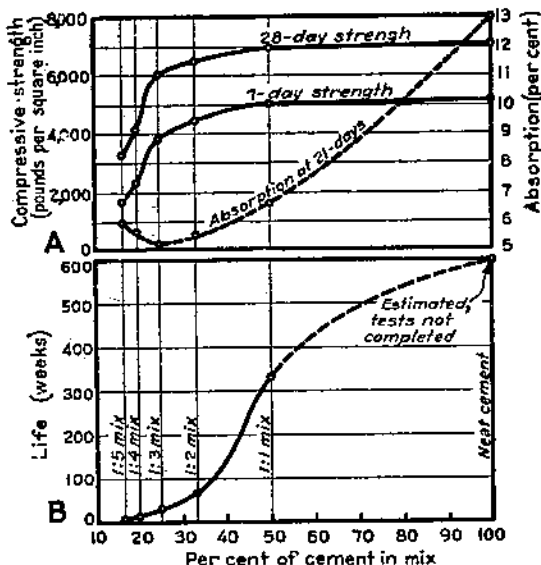


FIGURE 9.—Effect of quantity of cement in mix (A) on strength and absorption and (B) on life of concrete cylinders stored in 1 per cent solutions of magnesium sulphate. Each point is the average for five cylinders made on different days. (Cements A and B1 mixed in equal portions)

interiors of the cylinders were affected relatively little. An extreme example is illustrated in Plate 1, B by the cylinder stored 10 years in a 4 per cent solution. This cylinder had a calculated volume only 38 per cent of its original volume, yet the measured length between screw heads was only 0.0061 inch less than the original length.

Neat cement cylinders from the same lot as those shown in Plate 1, B, after storage for four years in a 1 per cent solution of magnesium sulphate had 70 per cent of normal strength—the average for cylinders stored the same length of time in tap water—whereas the strength of the 1:1 cylinders was but 50 per cent of normal strength. From these 4-year compression tests and from volume-change observations over a longer period, the life of the neat cement cylinders has been estimated at 600 weeks for comparison with the other cylinders of this group. (Fig. 9.) All the cylinders were stored at room temperatures and prevented from drying out, therefore surface crazing was avoided and such destructive action as took place was solely the result of chemical action by the magnesium sulphate.

### LENGTH OF TIME OF MIXING

It is generally agreed that after the ingredients for concrete have been combined intimately and uniformly, continuing the mixing has small value.

It is the practice in the laboratory to hand mix each batch of nine cylinders  $1\frac{1}{2}$  minutes dry and 2 minutes after adding the water. To determine the adequacy of such mixing, cylinders of three series were made and tested, with the results recorded in Table 7. The cylinders of these series were identical except as to the lengths of mixing time after adding the water, which were 1, 2, and 5 minutes. It is not evident that this difference in mixing affected strength and absorption more than slightly. However, the relative resistances of the three series as measured by length changes after storage in 1 per cent solutions of sodium sulphate were 1, 1.1, and 1.2 in favor of the 5-minute mix.

TABLE 7.—Effect of time of mixing on resistance of 2 by 4 inch concrete cylinders exposed in 1 per cent solutions of sodium sulphate ( $\text{Na}_2\text{SO}_4$ )

[Each test result is average for five cylinders made on different days]

Series	Mixing of materials	Fineness modulus	Water-cement ratio	Absorption at 21 days	Stored in tap water		Stored in $\text{Na}_2\text{SO}_4$			
					Age when broken	Strength in compression	Age when broken	Strength in compression	Strength ratio (1)	Time required to increase in length 0.010 inch
				Per cent	Weeks	Lbs. per sq. in.	Weeks	Lbs. per sq. in.	Per cent	Weeks
426	$1\frac{1}{2}$ minutes dry and 1 minute wet.....	4.67	0.62	6.5	1	2,910				
					4	4,230				
					52	5,120	62	2,560	51	
					74	5,170	74	1,700	26	79.9
427	$1\frac{1}{2}$ minutes dry and 2 minutes wet.....	4.67	.62	6.5	1	2,870				
					4	4,380				
					52	6,040	52	3,210	53	
					82	5,850	82	1,530	26	79.0
428	$1\frac{1}{2}$ minutes dry and 5 minutes wet.....	4.67	.62	6.4	1	3,100				
					4	4,700				
					52	5,740	62	3,760	66	
					90	5,380	90	2,030	39	87.1

<sup>1</sup> Ratio of the strength of cylinders stored in sodium sulphate to those of the same series stored in tap water.

## LONG-TIME WATER CURING

The effect of long-time water curing on the resistance of Portland-cement concrete and mortar cylinders to 1 per cent solutions of magnesium sulphate is shown in Table 8. The table compares cylinders from eight series cured 1 year in water and from 10 series cured 6 months in water with cylinders from the same series water cured 20 days. None of the cylinders had any hardening in air.

TABLE 8.—*Influence of long-time water curing on life of various types of Portland-cement mortar and concrete 2-by-4-inch cylinders stored in 1 per cent solutions of magnesium sulphate*

General description	Life in weeks of samples cured in distilled water for—		
	20 days	6 months	1 year
<b>Sand cylinders:</b>			
Ottawa standard sand	12.9		12.7
Minnesota sand, screened, 20-30	4.8		23.4
Minnesota sand, screened, 20-30	10.8		27.9
Minnesota sand, screened, 20-30, shale removed	10.8		23.1
Minnesota sand, pit run	15.6		28.3
Minnesota sand, special grading	23.8		40.0
Minnesota sand, screened, 20-30	8.5		20.7
Minnesota sand, special grading, shale removed	30.5		30.4
Average	14.7		26.6
<b>Laboratory standard cylinders:</b>			
Water-cement ratio, 0.44	17.3	29.3	
Water-cement ratio, 0.53	19.5	22.4	
Water-cement ratio, 0.59	27.7	28.6	
Water-cement ratio, 0.64	25.4	25.1	
Water-cement ratio, 0.73	19.8	20.0	
Mix 1:1	330.0	318.7	
Mix 1:2	68.3	60.0	
Mix 1:3	30.0	24.7	
Mix 1:4	11.4	13.1	
Mix 1:5	9.1	11.4	
Average	55.6	55.3	

The data in Table 8 show an average increase in the life of the mortar cylinders cured 1 year of 81 per cent over that of cylinders cured 20 days, but show slight change in the life of concrete cylinders cured 6 months. Just why cylinders cured 1 year in water should show greatly increased resistance while those similarly cured half as long should show no increased resistance is difficult to explain. However, the cylinders in the two groups were dissimilar in many respects. In general, those cylinders representative of the highest quality concrete seemed to have been benefited least by long-time water curing, whether the curing period was 1 year or 6 months, and differences in cylinders probably account for the apparent discrepancy in resistance after being water-cured 1 year and being water-cured 6 months. It is evident that resistance of low-quality concrete may be raised somewhat by long-time water-curing, but concrete of reasonably good quality will be improved very little by such treatment.

## LONG-TIME AIR HARDENING

Vicat (51) suggested as long ago as 1857 that the action of magnesium sulphate on cement mortar continues until all lime is acted upon except that which is combined with carbon dioxide. Feret (23),

Bates, Phillips, and Wig (8), Blount (12), and others have suggested carbonization of some of the free lime near the surface of concrete aged in air or water, as one cause for observed increased resistance to sulphate attack. Burke (15) in 1925 concluded, after an interesting series of experiments with carbon dioxide treated briquets, that "It seems logical to assume that if good concrete is allowed to set in the presence of  $\text{CO}_2$  or properly exposed thereafter, the surface would become very resistant to sulphate attack."

Air under ordinary conditions contains more or less carbon dioxide, and therefore it should follow that the resistance of concrete would be appreciably increased by hardening the concrete in air at ordinary temperatures, whether storage were under room-dry conditions or out of doors. It should also follow that greater resistance would result from storage in the air of an occupied heated room than from storage out of doors, because the  $\text{CO}_2$  content of the room air would generally be higher. Increased resistance of concrete usually does occur under these two conditions of air hardening, as is illustrated by the curves in Figure 10. Whether

this is due entirely to carbonation of some of the lime is not known. Figure 10 is based on the behavior in 1 per cent solutions of sodium sulphate, of three groups of cylinders for each of which the air-hardening periods, following 20 days in water, were 0, 2, 4, 8, and 49 weeks. One group was air-hardened out of doors, and two groups, one of which was of standard Ottawa sand, in the laboratory.

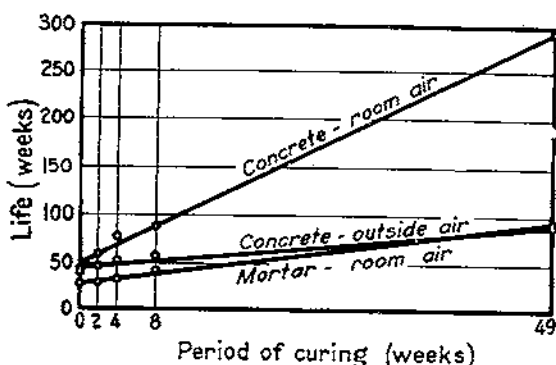


FIGURE 10.—Effect of air curing, after 20 days' storage in water, on life of concrete and Ottawa sand-mortar cylinders stored in 1 per cent solutions of sodium sulphate. Each point is the average for 10 cylinders made on five days. (Cements A and B1 mixed in equal portions)

The concrete cylinders exposed longest in air under room-dry conditions were most resistant of any of the cylinders, and their life was nearly seven times that of cylinders of the same group not air-hardened. Cylinders of the three groups were first exposed to air between March 7 and April 11, when temperatures were about average and humidities somewhat above average. The average relative resistances of cylinders from the three groups were 1, 1.2, 1.4, 1.7, and 4.3 for respective air-hardening periods of 0, 2, 4, 8, and 49 weeks.

Some of the curves in Figure 11 (series 23, 24, 25, 28, 29, 30, 33, 34, and 35) show the behavior of three groups of cylinders in each of which hardening in air for periods of 0, 14, and 18 days followed hardening in water for periods of 20, 6, and 2 days, respectively. One group was exposed to 1 per cent solutions of sodium sulphate and two groups, one of which was of standard Ottawa sand, to 1 per cent solutions of magnesium sulphate. The concrete cylinders stored in magnesium sulphate were exposed to air in the laboratory between July 10 and August 4, while the other cylinders were so exposed between March 5 and April 7. For all the cylinders in these three

groups, the relative average resistances of those air hardened for 0, 14, and 18 days respectively were 1, 2.6, and 4.0. These results are surprising in view of the fact that the cylinders in this group that were air-hardened for 18 days had an average relative resistance very

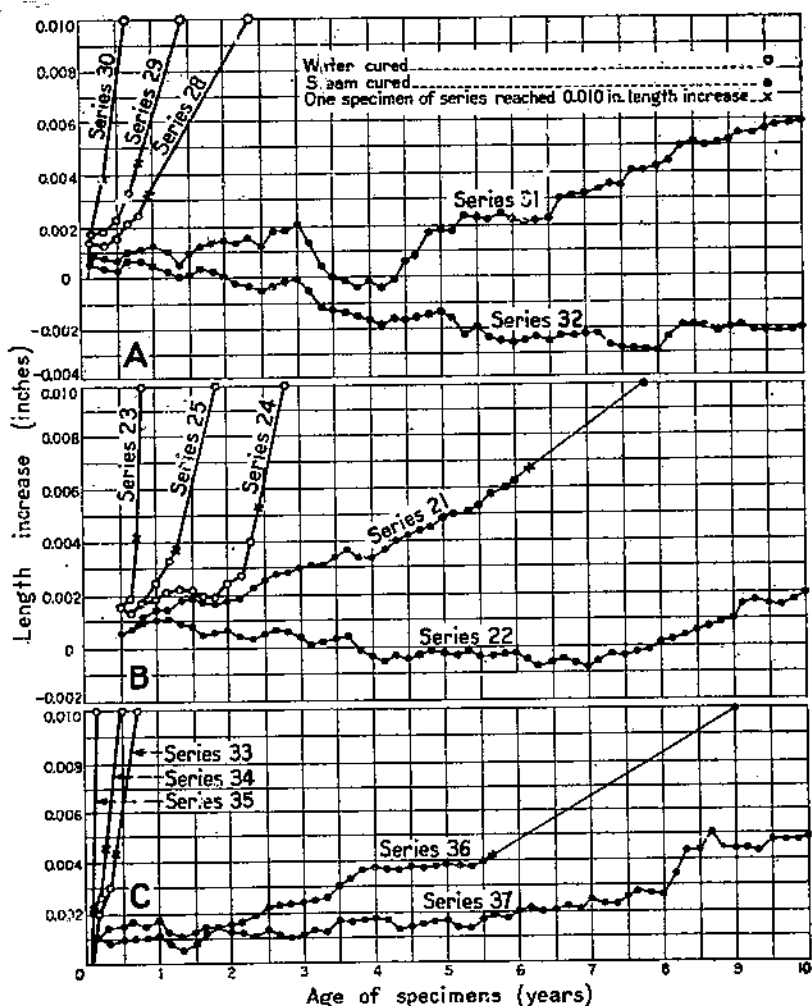


FIGURE 11.—Increase in length with age of 2-by-4-inch Portland-cement mortar and concrete cylinders, 1:3 mix, stored in sulphate solutions: A, Concrete cylinders in 1 per cent sodium sulphate; B, concrete cylinders in 1 per cent magnesium sulphate; C, Ottawa sand-mortar cylinders in 1 per cent magnesium sulphate. Curing conditions, following 1 day in moist closet, were: For series 24, 23, and 33, 2 days in distilled water and 18 days in air; for series 25, 20, and 34, 6 days in distilled water and 14 days in air; for series 23, 30, and 35, 20 days in distilled water; for series 21, 31, and 36, 2 days in water vapor at 212° F. and 18 days in air; for series 22, 32, and 37, 8 days in water vapor at 212° and 14 days in air. A length increase of 0.01 inch indicates 60 to 70 per cent loss in compressive strength. Each point is the average for 5 or 10 cylinders made on five days. (Cements A and B1 mixed in equal portions)

nearly as great as those shown in Figure 10 that were air-hardened 49 weeks.

Comparing the effects of air hardening as shown by Figures 10 and 11, it would appear that for the cements used in those experiments the most effective time for air hardening concrete that is to be subjected



to alkali action may be shortly after the final set of the cement and before, instead of after, curing in water. The reason for this is not apparent, but the seeming fact may explain why commercial concrete of very ordinary quality, to which little or no water was applied while it was curing, has frequently displayed a high degree of resistance to sulphate attack.

Different cements, however, may respond differently to the same variations in method of curing. Available data on this are limited to those obtained by comparative compression tests on cylinders exposed for one year to the alkali water of Medicine Lake, as recorded in Table 9. Eight different cements were included in those tests, and the comparison shown is that made between cylinders cured 20 days in water with no air hardening following, and cylinders cured 20 days in water and exposed for 35 days in air in the laboratory. Some of the cements were greatly benefited by air hardening, whereas others gave slightly contrary results. Cements A and B1 were those used for the tests presented in Figures 10 and 11, and Table 9 shows that cylinders of the same cements, air hardened 35 days, averaged 47 per cent stronger after one year in the lake than did similar cylinders not air hardened. This closely checks an average calculated value of 41 per cent for the cylinders in Figure 10, air hardened 28 days and stored in a 1 per cent solution of sodium sulphate.

TABLE 9.—Effect of air hardening on alkali resistance of concrete cylinders made of cements from different mills, as determined by compression tests after one year in Medicine Lake, S. Dak.

Portland cement		Results of compression tests				Increase in strength due to air hardening
		Without air hardening		Hardened in air 35 days		
Brand	Lots tested	Cylinders tested	Strength ratio	Cylinders tested	Strength ratio	
	Number	Number	Per cent	Number	Per cent	Per cent
A.....	6	60	24.0	60	35.7	48.8
B1.....	6	60	48.2	60	69.6	44.8
D.....	3	30	88.7	30	85.7	24.7
H.....	3	30	84.3	30	93.7	11.2
I.....	3	30	95.0	30	101.7	7.1
K2.....	3	30	92.7	30	93.7	1.1
C.....	3	50	92.3	30	87.3	-5.4
G.....	3	30	98.7	30	91.3	-7.5

The cements shown in Table 9 most benefited by air hardening were those having the least resistance to the action of alkali. (Pp. 38 to 44.) In fact, the order of recording the cements in Table 9, which is the order of increased resistance caused by air hardening, is very closely the order of least alkali resistance. The table shows that even after 35 days of air hardening those cements of lowest alkali resistance still made poor showings when compared with the more resistant cements. Whether earlier air hardening of the more resistant cements would show results more like those obtained with the cements represented by Figures 10 and 11 can not be stated definitely.

The full significance of these air-hardening experiments can not now be stated, but the following statement is conservative: For each increase of two weeks of air hardening, up to one year, the durability

of concrete made of low alkali-resisting cements may ordinarily be expected to increase by 10 or 20 per cent over that of concrete without air hardening, the degree of increase varying with different concretes and with temperature, humidity, and other conditions during storage. It has not been shown, however, that the life of concrete made of cements high in alkali resistance is greatly influenced by air hardening.

#### CURING IN WATER VAPOR

It was learned from experiments begun in 1922 that concrete cured in steam over water boiling at atmospheric pressure displayed remarkable resistance in the laboratory to solutions of magnesium and sodium sulphate (33, 37). Some of the cylinders of these earlier series are still under observation, and their condition as indicated by length changes is shown in Figure 11. For each of these groups, resistance to sulphate action was in the same order as duration of time in steam, whereas for the water-cured groups the resistance was in the order of time in air, as discussed in the preceding section.

Following the earlier laboratory work there were made for exposure in Medicine Lake a total of 6,525 cylinders in 145 series, of which 15 series were cured in water and air for comparison and 130 series were cured in water vapor at temperatures of 100°, 155°, 190°, 212°, 230°, 260°, 285°, 315°, and 350° F. for periods, at most temperatures, ranging from 45 minutes to 8 days. Five-year tests have been completed for 83 series, and 1 and 3 year tests for all series.

The data obtained (Table 10), although incomplete for many series cured at the higher temperatures, apparently justify the following conclusions regarding alkali resistance of concrete. These conclusions are also supported by the appearance of cylinders after exposure for periods up to seven and one-half years in Medicine Lake, as shown in Plate 3.

TABLE 10.—Tests of 2 by 4 inch concrete cylinders cured in water vapor at various temperatures and exposed to the action of sulphate water of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water

[Unless otherwise noted the fineness modulus of aggregate is 4.67 and the mix is 1:3. Each test result, with a few exceptions, is an average of 5 cylinders made on different days. Figures in parentheses, indicate per cent of normal strength based on parallel tests of cylinders from the same batches, stored in tap water]

## TEMPERATURE, 100° F.

Series	Cement laboratory No.	Portland cement	Water ratio	Curing method					Absorption at 21 days	Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in water vapor	Temperature of water vapor	Time in air		Tank specimens				Lake specimens		
										7 days	28 days	1 year	5 years	1 year	3 years	5 years
				Hours	Days	Hours	° F.	Days	Per cent							
87	11	½A and ½B1	0.50	3		69	100	25	6.6	3,730	4,120	5,110	5,470	1,430 (28)	0	0
88	11	do	.50	6		66	100	25	6.7	3,590	4,320	4,960	5,450	1,370 (28)	0	0
89	11	do	.50	12		60	100	25	6.7	4,050	4,680	5,270	5,200	1,290 (25)	0	0
90	11	do	.50	24		48	100	25	6.7	4,080	4,220	5,240	6,270	1,630 (31)	0	0
91	11	do	.50	48		24	100	25	6.8	3,510	4,060	5,010	4,970	1,920 (38)	0	0
92	11	do	.59	24	27			28	6.7	2,620	4,540	5,690	5,400	3,780 (66)	0	0
93	11	do	.59	72				25	7.0	3,380	3,560	5,120	5,140	3,150 (62)	0	0
94	11	do	.59	24		48	100	24	6.8	3,810	4,000	5,640	5,910	2,020 (36)	0	0
313	65	M	.60	24	20			35	5.9	3,150	3,950	6,050	5,680	2,250 (37)	0	0
315	65	M	.60	24		48	100	53	6.2	3,740	3,800	5,970	6,320	4,230 (71)	3,390	3,330 (53)
1112	17	½A and ½B1	.64	24	27			25	10.0	1,650	2,630	3,540	2,790	1,790 (51)	0	0
1113	17	do	.64	72				25	10.7	2,000	2,000	2,980	2,940	840 (28)	0	0
1114	17	do	.64	24		48	100	25	10.1	2,270	2,790	3,040	3,050	630 (21)	0	0

## TEMPERATURE, 155° F.

82	11	½A and ½B1	0.59	3	-----	69	155	25	6.9	3,300	4,210	5,380	5,550	1,500 (9)	0	0
83	11	do	.59	6	-----	66	155	25	6.9	3,840	4,720	5,700	5,620	1,490 (9)	0	0
84	11	do	.59	12	-----	60	155	25	6.7	4,440	4,060	5,250	5,920	1,030 (20)	0	0
85	11	do	.59	24	-----	48	155	25	6.6	4,350	4,550	6,400	5,880	1,220 (19)	0	0
86	11	do	.59	48	-----	24	155	25	6.7	3,540	4,210	6,250	6,320	1,660 (27)	0	0
92	11	do	.59	24	27	-----	-----	28	6.7	2,620	4,540	5,690	5,400	3,780 (66)	0	0
93	11	do	.59	72	-----	-----	-----	25	7.0	3,380	3,560	5,120	5,140	3,150 (62)	0	0
95	11	do	.59	24	-----	48	155	25	6.4	4,240	4,370	5,510	5,510	1,600 (30)	0	0
97	11, 8	do	.59	24	-----	48	155	25	6.3	3,900	4,770	5,690	5,970	1,880 (33)	0	0
99	11, 8	do	.59	24	-----	72	155	24	6.4	4,490	4,830	5,410	5,990	1,530 (28)	0	0

<sup>1</sup> Standard Ottawa sand cylinders.

TABLE 10.—Tests of 2 by 4 inch concrete cylinders cured in water vapor at various temperatures and exposed to the action of sulphate water of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water—Continued

TEMPERATURE, 155° F.—Continued

Series	Cement laboratory No.	Portland cement	Water ratio	Curing method					Absorption at 21 days	Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in water vapor	Temperature of water vapor	Time in air		Tank specimens				Lake specimens		
										7 days	28 days	1 year	5 years	1 year	3 years	5 years
				Hours	Days	Hours	° F.	Days	Per cent							
102	17	½A and ½B1	0.59	24		48	155	25	6.2	4,550	4,730	5,580	5,960	980 (18)	0	0
104	17	do.	.59	24		72	155	24	6.1	4,910	5,180	5,180	5,630	1,270 (25)	0	0
174	18	do.	.59	24		48	155	35	6.1	4,020	4,390	4,330	5,650	3,910 (90)		2,740 (48)
179	18	do.	.59	24		48	155	35	5.8	4,110	4,510	5,210	6,400	3,820 (73)		1,750 (27)
184	18	do.	.59	24		48	155	35	6.0	4,070	4,630	5,100	6,110	4,240 (83)		800 (13)
313	65	M	.60	24	20			35	5.9	3,150	3,950	6,050	5,680	2,250 (37)	0	0
314	65	M	.60	24		48	155	53	6.0	4,460	4,010	6,170	6,810	5,430 (88)	3,920	3,690 (54)
107	17	½A and ½B1	.64	24		48	155	25	9.8	2,660	3,080	3,580	3,580	770 (22)	0	0
109	17	do.	.64	24		72	155	24	9.6	2,670	3,220	3,490	3,580	930 (27)	0	0
112	17	do.	.64	24	27			25	10.0	1,650	2,630	3,540	2,790	1,790 (51)	0	0
113	17	do.	.64	72				25	10.7	2,000	2,000	2,980	2,940	840 (28)	0	0
115	17	do.	.64	24		48	155	25	10.1	2,730	2,900	3,400	3,020	890 (25)	0	0
127	17	do.	.64	24		48	155	53	9.1	2,530	2,710	3,460	3,290	2,280 (60)		0
129	17	do.	.64	24		72	155	52	9.0	2,700	3,120	3,320	3,230	1,070 (59)		0

TEMPERATURE, 100° F.

351	74	½ A and ½ B1	0.62	24	-----	12	190	54	6.6	2,650	2,940	5,320	6,870	1,730 (33)	0	0
391	74	do.	.62	24	20	-----	-----	35	6.3	3,130	4,690	6,060	6,430	2,480 (41)	0	0

TEMPERATURE, 212° F. (COMBINED IN SOME SERIES WITH 155° F.)

77	11	½A and ½B1	0.59	3	-----	69	212	25	7.9	1,110	1,250	1,250	2,810	1,210 (97)	1,340	2,630 (94)
78	11	do.	.59	6	-----	66	212	25	7.2	2,260	3,040	3,130	5,900	2,840 (91)	2,930	4,120 (70)
79	11	do.	0.59	12	-----	60	212	25	6.9	3,400	4,120	4,550	6,020	4,410 (97)	4,940	5,400 (90)
80	11	do.	.59	24	-----	48	212	25	6.9	3,000	4,020	4,600	5,790	4,330 (94)	4,860	5,150 (89)
81	11	do.	.59	48	-----	24	212	25	6.7	2,910	3,850	4,630	6,730	4,410 (95)	5,340	6,030 (89)
92	11	do.	.59	24	27	-----	-----	28	6.7	2,620	4,540	5,690	5,400	3,780 (66)	0	0

96	11	do.	.59	24	48	212	25	6.5	3,770	4,410	4,560	5,720	4,300 (94)	4,930 (86)
98	11.8	do.	.59	24	24	155	25	6.7	4,180	4,120	5,020	6,190	4,340 (87)	5,310 (86)
100	11.8	do.	.59	24	48	155	24	6.2	4,310	4,960	5,730	5,750	5,540 (97)	5,890 (102)
101	11.8	do.	.59	24	24	155	24	6.3	4,110	4,710	4,890	6,490	4,640 (95)	5,330 (82)
103	17	do.	.59	24	48	212	25	6.0	4,600	5,510	5,200	6,280	4,980 (94)	5,510 4,910 (78)
105	17	do.	.59	24	24	155	24	6.0	4,820	5,070	4,910	6,720	5,220 (106)	6,000 5,680 (85)
106	17	do.	.59	24	48	212	24	5.7	5,110	5,900	5,320	6,020	5,250 (99)	5,830 5,720 (95)
296	61	do.	.62	24	48	212	53	6.5	3,330	3,660	5,020	5,760	4,390 (88)	5,840 5,950 (103)
297	63	A	.60	24	48	212	53	6.4	3,650	3,060	4,680	6,020	4,600 (98)	4,960 5,480 (91)
298	62	B	.62	24	48	212	53	6.9	3,700	3,830	4,580	6,440	4,480 (98)	4,840 5,600 (87)
299	33	D	.60	24	48	212	53	6.4	3,690	3,770	4,620	6,330	4,270 (92)	4,910 5,900 (93)
300	34	C	.67	24	48	212	53	6.6	3,880	4,540	4,940	6,110	4,890 (99)	5,690 5,810 (95)
301	61	$\frac{1}{2}$ A and $\frac{1}{2}$ B1	.62	24	48	212	53	6.5	3,690	3,610	4,500	4,840	4,480 (100)	5,160 5,300 (110)
302	35	E	.60	24	48	212	53	6.2	3,540	3,530	4,550	5,040	4,450 (98)	4,660 4,640 (92)
303	37	F	.60	24	48	212	53	6.0	3,550	3,900	5,060	4,890	4,920 (97)	5,920 5,470 (112)
304	39	G	.60	24	48	212	53	6.2	3,740	3,920	4,990	5,870	4,680 (94)	5,060 5,060 (86)
305	41	H	.60	24	48	212	53	6.1	3,730	3,840	4,860	5,380	4,410 (91)	4,340 5,100 (95)
306	61	$\frac{1}{2}$ A and $\frac{1}{2}$ B1	.62	24	48	212	53	6.2	3,280	3,920	4,480	6,570	4,410 (98)	5,990 5,230 (80)
307	40	I	.60	24	48	212	53	5.9	3,420	4,150	5,090	5,850	4,190 (82)	4,990 4,530 (77)
308	42	J	.62	24	48	212	53	6.4	2,650	3,240	4,680	5,310	3,480 (74)	4,440 4,580 (86)
309	55	K1	.62	24	48	212	53	6.5	3,260	4,200	4,950	5,800	4,910 (99)	4,910 4,960 (86)
310	60	L	.60	24	48	212	53	6.2	3,260	3,870	5,060	6,380	4,460 (88)	4,720 5,770 (90)
311	65	M	.60	24	48	212	53	5.5	4,660	5,580	5,810	6,740	5,570 (96)	5,420 5,960 (88)
313	65	M	.60	24	20		35	5.9	3,150	3,950	6,050	5,680	2,250 (37)	0 0
352	74	$\frac{1}{2}$ A and $\frac{1}{2}$ B1	.62	24	12	212	54	6.9	2,450	2,910	5,230	6,070	4,240 (81)	3,850 0
391	74	do.	.62	24	20		35	6.3	3,130	4,890	6,060	6,430	2,480 (41)	0 0
584	139	do.	.62	24	20	1	35	6.3	3,740	5,150	5,790	6,270	3,350 (58)	0 0
585	139	do.	.62	24	20	1	55	6.4	2,770	2,860	5,430	6,610	5,370 (99)	0 0
586	139	do.	.62	24		1								
587	139	do.	.62	24	48	212	54	6.6	2,970	2,920	5,800	6,440	4,400 (76)	0 0
635	139	do.	.64	24			53	6.3	3,060	4,330	4,250	6,600	4,370 (103)	5,950 6,550 (96)
636	139	do.	.64	24	20		35	6.5	3,320	4,470	5,800	6,070	4,530 (78)	0 0
640	139	do.	.64	24		48	53	6.9	3,580	3,760	4,530	6,010	3,740 (83)	5,360 6,140 (102)
641	232	do.	.62	24	20		35	5.9	3,230	4,720	6,360		3,950 (62)	0 0
830	232	do.	.62	24		34	55	6.9	3,020	2,880	5,740		4,210 (73)	2,280
831	232	do.	.62	24		112	55	7.4	2,590	2,400	5,890		4,610 (78)	4,330
832	232	do.	.62	24		3	55	7.2	2,890	3,130	6,350		4,510 (71)	2,840
833	232	do.	.62	24		6	55	7.0	3,220	3,470	5,730		4,970 (87)	6,270
834	232	do.	.62	24		12	55	7.1	3,570	3,580	5,070		5,250 (104)	5,970
835	232	do.	.62	24		24	54	7.2	3,960	4,190	5,750		5,770 (100)	6,510
836	232	do.	.62	24		48	53	6.6	4,360	4,670	4,860		4,620 (95)	4,770
837	232	do.	.62	24		96	61	6.1	4,330	4,990	5,030		4,210 (84)	5,190
838	232	do.	.62	24		192	47	6.1	5,180	6,050	5,130		4,550 (89)	5,060

1 Standard Ottawa sand cylinder.

\* Minutes.

TABLE 10.—Tests of 2 by 4 inch concrete cylinders cured in water vapor at various temperatures and exposed to the action of sulphate water of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water—Continued

TEMPERATURE, 212° F. (COMBINED IN SOME SERIES WITH 155° F.)—Continued

TEMPERATURE, 212° F. (COMBINED)																
Series	Cement laboratory No.	Portland cement	Water ratio	Curing method					Absorption at 21 days	Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in water vapor	Temperature of water vapor	Time in air		Tank specimens				Lake specimens		
										7 days	28 days	1 year	5 years	1 year	3 years	5 years
				Hours	Days	Hours	° F.	Days	Per cent							
1108	17	½A and ½B1	0.64	24		24	155	25	9.8	2,590	2,910	2,930	2,880	2,540 (87)	3,390	2,580 (90)
1110	17	do	.64	24		24	155	24	9.5	2,600	3,160	2,940	2,590	2,720 (93)	3,160	2,360 (91)
1111	17	do	.64	24		24	155	24	9.7	2,510	3,130	2,630	3,020	2,450 (93)	2,820	2,320 (77)
1112	17	do	.64	24	27				10.0	1,650	2,630	3,540	2,700	1,790 (51)	0	0
1113	17	do	.64	72		24	212	25	10.7	2,000	2,000	2,980	2,940	840 (28)	0	0
1116	17	do	.64	24		48	212	25	10.3	2,220	2,470	2,500	3,250	2,430 (97)		2,460 (73)
1128	17	do	.64	24		24	155	53	9.1	2,350	2,480	2,830	3,340	3,180 (112)		2,040 (61)
1130	17	do	.64	24		24	155	52	9.1	2,750	2,720	2,830	3,130	3,080 (109)		1,740 (56)
1131	17	do	.64	24		24	155	52	9.2	2,630	2,470	2,600	2,940	2,560 (98)		1,730 (59)

TEMPERATURE, 230° F. GAGE PRESSURE, 6.1 POUNDS PER SQUARE INCH

353	74	½A and ½B1	0.62	24		12	235	54	7.3	1,900	2,750	4,780	5,410	3,800 (79)	3,970	2,480 (46)
391	74	do.	.62	24	20			35	6.3	3,130	4,890	6,060	6,430	2,480 (41)	0	0
811	219	do.	.62	24	20			35	5.5	3,620	4,930	6,350		0	0	0
812	219	do.	.62	24		34	230	55	7.0	2,060	2,170	5,530		5,160 (93)	5,120	
813	219	do.	.62	24		1½	230	55	6.9	1,830	2,190	5,620		4,530 (81)	5,220	
814	219	do.	.62	24		3	230	55	6.7	2,180	2,320	6,020		4,860 (81)	6,100	
815	219	do.	.62	24		6	230	55	6.6	2,390	2,990	5,520		5,320 (96)	6,070	
820	222	do.	.62	24		12	230	103	6.8	2,640	3,200	5,260		4,990 (95)	6,130	
821	222	do.	.62	24		24	230	102	6.6	3,560	3,250	4,740		4,520 (96)	6,080	
829	225	do.	.62	24		48	230	87	6.9	3,900	4,650	5,640		4,360 (94)	5,900	
855	232	do.	.62	24	20			35	5.7	3,840	5,310	5,540		4,070 (73)	0	0
869	232	do.	.62	24		96	230	46	6.1	4,480	4,600	4,480		4,600 (103)	5,330	
870	232	do.	.62	24		192	230	42	6.2	3,830	4,450	3,360		3,560 (106)	4,470	

TEMPERATURE, 260° F. GAGE PRESSURE, 20.7 POUNDS PER SQUARE INCH

354	74	1/2A and 1/2B1	0.62	24	12	260	54	7.1	2,270	2,400	4,880	5,570	3,700 (77)	4,160	3,320 (60)
391	74	do	.62	24	20		35	0.3	3,130	4,890	0,060	0,430	2,480 (41)	0	0
806	219	do	.62	24	20		35	5.8	3,450	4,020	0,110		1,620 (27)	0	0
807	219	do	.62	24		260	55	7.4	1,960	1,930	5,400		4,610 (85)	5,050	
808	219	do	.62	24	1 1/2		2,000	7.1	2,130	2,130	5,480		4,990 (91)	5,300	
809	219	do	.62	24	3	260	55	7.2	2,170	2,400	5,090		5,200 (102)	5,540	
810	219	do	.62	24	6	260	55	7.0	2,790	2,870	5,380		4,940 (92)	5,750	
818	222	do	.62	24	12	260	97	0.7	3,180	3,380	5,560		4,630 (83)	5,840	
819	222	do	.62	24	24	260	95	6.7	3,480	3,810	4,020		4,520 (98)	5,290	
828	225	do	.62	24	48	260	83	0.6	3,720	3,880	3,990		3,790 (95)	4,760	
855	232	do	.62	24	20		35	5.7	3,840	5,310	5,540		4,070 (73)	0	0
867	232	do	.62	24	96	260	48	6.4	3,820	3,810	3,460		3,550 (103)	3,830	
868	232	do	.62	24	192	260	44	6.6	3,260	3,690	3,420		2,940 (86)	3,400	

TEMPERATURE, 285° F. GAGE PRESSURE, 38.5 POUNDS PER SQUARE INCH

355	74	1/2A and 1/2B1	0.62	24	12	285	54	7.2	2,260	2,870	4,340	5,250	3,870 (89)	3,970	4,100 (80)
391	74	do	.62	24	20		35	6.3	3,130	4,890	0,060	0,430	2,480 (41)	0	0
430	97, 98	K1	.64	24	12	285	54	6.6	2,960	3,470	3,950	4,830	3,080 (93)	5,000	4,210 (87)
431	86, 99	L	.64	24	12	285	54	6.1	3,650	4,150	4,470	5,330	4,290 (96)	5,440	5,440 (102)
432	82, 95	O	.64	24	12	285	54	6.9	3,330	3,820	5,530	0,550	4,930 (89)	5,480	5,410 (83)
433	83	H	.64	24	12	285	54	6.5	3,460	3,950	5,110	0,520	4,840 (95)	5,510	5,980 (92)
801	219	1/2A and 1/2B1	.62	24	20		35	5.8	3,740	6,260	5,790		2,820 (49)	0	0
802	219	do	.62	24	34	285	55	7.3	1,780	1,740	5,240		5,570 (106)	5,980	
803	219	do	.62	24	1 1/2	285	55	7.2	2,010	2,220	6,560		5,560 (85)	5,980	
804	219	do	.62	24	3	285	55	7.3	2,420	2,590	5,560		5,650 (102)	5,700	
805	219	do	.62	24	6	285	55	6.8	3,080	3,370	5,860		5,600 (96)	6,720	
816	222	do	.62	24	12	285	87	6.5	3,320	3,420	4,940		4,510 (91)	5,350	
817	222	do	.62	24	24	285	85	6.3	3,740	4,160	4,540		4,750 (105)	4,790	
827	225	do	.62	24	48	285	64	6.7	3,300	3,600	3,200		3,170 (99)	4,060	
855	232	do	.62	24	20		35	5.7	3,840	5,310	5,540		4,070 (73)	0	0
865	232	do	.62	24	96	285	45	6.5	3,550	4,230	3,680		4,190 (118)	4,020	
866	232	do	.62	24	192	285	41	6.5	3,540	4,300	4,270		4,630 (100)	4,940	

TEMPERATURE, 315° F. GAGE PRESSURE, 68.7 POUNDS PER SQUARE INCH

840	232	1/2A and 1/2B1	0.62	24	20		35	5.8	3,810	5,050	6,010		3,060 (51)	0	0
841	232	do	.62	24	34	315	55	6.9	2,010	2,020	5,680		5,130 (90)	5,740	
842	232	do	.62	24	1 1/2	315	55	6.8	2,490	2,640	5,550		5,390 (97)	6,170	
843	232	do	.62	24	3	315	55	6.5	3,090	3,030	5,620		5,220 (93)	6,430	
844	232	do	.62	24	6	315	55	6.8	3,660	3,900	5,110		5,100 (100)	6,040	
845	232	do	.62	24	12	315	55	6.5	3,410	3,780	4,150		4,340 (105)	4,660	
846	232	do	.62	24	24	315	54	6.8	3,180	3,750	3,440		2,770 (81)	3,650	
847	232	do	.62	24	48	315	53	6.8	3,440	3,970	3,160		3,240 (103)	3,810	
848	232	do	.62	24	96	315	51	6.6	3,430	4,280	3,090		3,620 (117)	4,310	
849	232	do	.62	24	192	315	47	6.8	3,580	4,640	3,130		4,470 (143)	4,060	

<sup>1</sup> Standard Ottawa sand cylinders.

TABLE 10.—Tests of 2 by 4 inch concrete cylinders cured in water vapor at various temperatures and exposed to the action of sulphate water of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water—Continued

TEMPERATURE, 350° F. GAGE PRESSURE, 110.8 POUNDS PER SQUARE INCH

Series	Cement laboratory No.	Portland cement	Water ratio	Curing method					Absorption at 21 days	Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in water vapor	Temperature of water vapor	Time in air		Tank specimens				Lake specimens		
										7 days	28 days	1 year	5 years	1 year	3 years	5 years
				Hours	Days	Hours	° F.	Days	Per cent							
855	232	½A and ½B1	0.62	24	20			35	5.7	3,840	5,310	5,540		4,070 (73)	0	0
856	232	do.	.62	24		34	350	55	7.3	1,670	1,940	5,710		4,790 (84)	5,750	
857	232	do.	.62	24		1½	350	55	7.0	2,500	2,800	5,950		5,350 (90)	6,020	
858	232	do.	.62	24		3	350	55	6.7	3,210	3,310	5,010		5,170 (92)	6,160	
859	232	do.	.62	24		6	350	55	6.8	3,110	3,540	4,300		4,130 (96)	4,090	
860	232	do.	.62	24		12	350	55	6.7	3,280	3,470	3,880		3,760 (97)	4,400	
861	232	do.	.62	24		24	350	54	6.8	3,830	3,840	4,700		4,220 (90)	5,040	
862	232	do.	.62	24		48	350	53	6.5	3,530	4,710	4,580		4,800 (105)	5,910	
863	232	do.	.62	24		96	350	51	6.3	4,140	4,320	4,670		4,530 (97)	5,820	
864	232	do.	.62	24		192	350	47	6.2	3,770	4,260	5,000		4,310 (86)	5,520	



Curing in water vapor at temperatures ranging upward from 212° F. increases resistance to a remarkable degree. Full correlation of resistance, curing temperatures, and length of curing periods must await later results, but for tests between 212° and 285° F. resistance has been in the order of both temperature and length of curing period.

Curing in water vapor at temperatures between 100° and 190° F. does not increase resistance; on the contrary, in some cases decreased resistance follows. Exceptions to this occurred with cylinders containing certain admixtures as discussed on pages 59 to 74.

#### ABSORPTION AND STRENGTH

Those cylinders referred to in Table 10 that were cured continuously in water averaged lower in absorption than did the cylinders cured in water vapor. For identical curing periods, absorption was nearly constant, regardless of temperature, although there was a tendency for absorption to decrease as duration of the curing period increased.

The data in Tables 11, 12, and 13 do not indicate a general increase of strength of the fresh cements, with increase of temperatures above 100° F., with the single exception of those cylinders cured at 155°. On the other hand, those cylinders referred to in Table 11 that were made with the cements stored for one year, generally displayed a tendency to increase in strength as the curing temperature was increased. The 10 fresh cements used in these tests were purchased in the open market from warehouse stocks newly received, as indicated by invoices, while the stored cements were lots of the same brands that had been kept in their original bags in a winter-heated dry room and were not lumpy when tested.

TABLE 11.—*Compression strength of concrete cylinders cured in water vapor at temperatures between 100° and 350° F. for different periods*

[All cylinders were first cured 24 hours at room temperature in moist closet. Cylinders cured in water vapor were stored in air and tested dry in compression at seven days. Test results are, in all cases, the average for four or more cylinders. Italicized figures indicate retrogression in strength]

#### MADE WITH FRESH CEMENT

Series	Portland cement	Check cylinders cured in water		Compression tests at 7 days, pounds per square inch					
		Absorption (per cent) at 21 days	Compression tests, pounds per square inch		Cylinders cured in water vapor at 100° F.				
			7 days	28 days	1½ hours	6 hours	21 hours	96 hours	Average for 1½-96 hours
274	I	5.9	4,240	5,500	3,300	3,970	4,300	5,810	4,380
275	KI	5.8	4,010	6,340	3,550	3,770	4,730	4,540	4,150
276	H	0.1	3,680	5,210	3,110	3,450	3,030	4,610	3,780
277	C	0.2	3,640	5,620	2,610	3,190	3,800	4,170	3,440
278	E	5.9	3,560	5,700	2,900	2,900	3,070	4,110	3,400
279	F	5.5	5,110	6,700	3,780	4,090	5,370	5,040	4,560
280	G	0.2	4,240	5,270	2,880	3,800	4,260	4,280	3,830
281	M	5.8	4,040	5,270	3,310	3,810	4,280	4,680	4,020
282	P	5.5	4,420	6,580	3,610	3,830	4,730	4,370	4,140
284	D	5.5	4,470	0,050	3,670	4,150	4,990	6,090	4,730
Average		5.8	4,140	5,830	3,280	3,700	4,420	4,780	4,040

TABLE 11.—*Compression strength of concrete cylinders cured in water vapor at temperatures between 100° and 350° F. for different periods—Continued*

## MADE WITH CEMENT STORED FOR ONE YEAR

Series	Portland cement	Check cylinders cured in water		Compression tests at 7 days, pounds per square inch					
		Absorption (per cent) at 21 days	Compression tests, pounds per square inch		Cylinders cured in water vapor at 100° F.				
			7 days	28 days	1½ hours	6 hours	24 hours	96 hours	Average for 1½-96 hours
206	L	6.1	2,560	5,200	600	2,370	2,150	3,480	2,140
207	KI	6.5	2,560	4,510	960	1,870	2,260	2,820	1,980
208	H	6.5	2,910	4,620	1,940	2,010	3,150	5,000	2,520
209	C	6.8	2,140	4,520	830	1,310	2,280	2,140	1,640
210	E	6.3	3,430	4,890	1,970	2,060	3,420	3,900	2,070
211	F	6.2	3,390	4,900	2,450	2,480	3,640	3,890	3,150
212	O	8.3	2,930	4,740	1,870	2,260	3,250	3,560	2,730
213	M	6.6	2,760	4,510	2,120	2,080	3,210	3,430	2,710
215	P	6.1	3,860	4,670	2,880	2,990	4,160	5,730	3,440
217	D	6.3	3,360	5,220	1,690	2,290	3,180	3,880	2,760
Average		6.4	3,010	4,780	1,730	2,260	3,090	3,380	2,610

## MADE WITH FRESH CEMENT

Series	Portland cement	Compression tests at 7 days, pounds per square inch								
		Cylinders cured in water vapor at 155° F.					Cylinders cured in steam at 212° F.			
		1½ hours	6 hours	24 hours	96 hours	Average for 1½-96 hours	1½ hours	6 hours	24 hours	Average for 1½-96 hours
274	L	4,200	4,560	4,690	5,490	4,580	3,780	3,790	4,410	4,180
275	KI	3,920	3,700	4,610	4,460	4,170	3,310	3,410	4,440	3,820
276	H	3,710	4,490	4,770	5,690	4,670	2,620	3,080	3,160	3,260
277	C	3,020	3,710	4,490	5,350	4,140	2,580	3,050	3,940	3,690
278	E	3,240	3,930	4,790	5,550	4,380	2,740	2,680	4,230	3,560
279	F	4,330	5,370	5,200	5,850	5,190	3,790	4,050	4,620	4,260
280	G	3,550	4,160	5,130	5,740	4,650	2,840	3,510	3,950	3,670
281	M	3,460	4,610	4,410	5,600	4,540	3,170	3,000	4,430	3,820
282	P	3,670	4,820	4,620	5,650	4,640	3,770	3,860	4,180	4,360
284	D	3,800	4,960	6,070	6,420	5,330	3,800	4,000	4,610	4,490
Average		3,690	4,430	4,810	5,590	4,630	3,230	3,440	4,140	3,910

## MADE WITH CEMENT STORED FOR ONE YEAR

Series	Portland cement	Compression tests at 7 days, pounds per square inch								
		1½ hours	6 hours	24 hours	96 hours	Average for 1½-96 hours	1½ hours	6 hours	24 hours	Average for 1½-96 hours
206	L						1,530	1,470	2,290	2,850
207	KI	1,220	1,800	2,316	3,700	2,260	2,120	1,520	2,320	3,420
208	H	1,940	2,140	2,990	3,770	2,710	1,700	1,950	2,560	2,640
209	C						1,200	1,610	2,100	3,920
210	E	2,500	3,090	3,600	4,160	3,340	1,860	2,090	2,760	4,380
211	F	2,130	2,470	3,080	4,870	3,140	2,070	2,520	3,340	4,610
212	O						1,600	2,200	2,870	3,780
213	M	2,310	2,530	3,120	4,120	3,020	1,680	1,980	2,480	4,590
215	P						2,270	2,850	3,750	4,820
217	D	1,850	2,230	3,520	4,230	2,900	2,120	2,690	2,920	3,650
Average		1,960	2,380	3,100	4,140	2,900	1,800	2,090	2,740	4,030

1 Averages for 6 cements only.

TABLE 11.—*Compression strength of concrete cylinders cured in water vapor at temperatures between 100° and 350° F. for different periods—Continued*

## MADE WITH FRESH CEMENT

Series	Portland cement	Compression tests at 7 days, pounds per square inch							
		Cylinders cured in steam at 260° F.					Cylinders cured in steam at 315° F.		
		1½ hours	6 hours	24 hours	96 hours	Average for 1½-96 hours	1½ hours	6 hours	24 hours
274	I	3,100	3,630	4,010	3,630	3,610	2,970	4,070	3,810
275	K	2,710	3,040	4,550	3,830	3,530	2,860	3,840	3,300
276	H	2,450	2,820	3,440	3,780	3,120	2,290	3,430	4,270
277	C	2,250	2,680	4,240	4,760	3,490	2,220	3,590	3,960
278	E	2,300	2,970	4,090	4,440	3,470	2,100	4,030	4,950
279	F	3,470	4,120	5,310	4,330	4,310	3,320	4,230	4,100
280	G	2,760	2,920	4,260	2,650	3,000	2,710	4,100	2,820
281	M	2,340	2,840	3,770	5,240	3,550	2,050	3,700	4,470
282	P	3,040	3,040	5,330	4,750	4,270	3,050	4,170	4,540
284	D	3,180	4,120	4,890	3,600	3,960	3,250	4,190	3,010
Average		2,730	3,310	4,390	4,050	3,630	2,750	3,770	3,870

## MADE WITH CEMENT STORED FOR ONE YEAR

Series	Portland cement	1½ hours	6 hours	24 hours	96 hours	Average for 1½-96 hours	1½ hours	6 hours	24 hours
206	I	1,180	1,370	2,570	2,030	2,010	830	1,090	3,080
207	K	1,200	2,100	2,910	4,040	2,550	1,140	2,480	3,940
208	H	1,520	2,260	3,300	4,070	2,790	1,710	2,650	4,810
209	O	1,230	2,130	3,220	3,800	2,590	1,160	2,390	4,120
210	E	1,340	2,250	3,770	4,900	3,060	1,610	2,910	4,860
211	F	1,850	2,610	3,660	5,540	3,430	1,830	3,470	5,450
212	O	1,930	2,510	3,730	2,860	2,780	1,700	2,990	5,620
213	M	1,260	2,120	3,350	4,610	2,830	1,750	2,730	4,870
215	P	2,470	2,980	4,250	5,070	3,690	1,600	3,360	5,080
217	D	1,520	2,680	3,100	3,040	2,410	1,980	2,420	4,170
Average		1,570	2,240	3,300	4,090	2,820	1,530	2,740	4,100

## MADE WITH FRESH CEMENT

Series	Portland cement	Compression tests at 7 days, pounds per square inch							
		Cylinders cured in steam at 315° F.		Cylinders cured in steam at 350° F.					
		96 hours	Average for 1½-96 hours	1½ hours	6 hours	24 hours	96 hours	Average for 1½-96 hours	
274	I	3,460	3,440	3,150	3,520	4,610	4,610	3,970	
275	K	3,590	3,400	2,910	3,600	3,780	4,720	3,750	
276	H	3,710	3,430	2,060	4,320	4,570	4,180	3,780	
277	C	3,880	3,410	2,200	3,880	4,340	4,590	3,820	
278	E	4,910	4,010	2,100	4,460	5,330	4,100	4,080	
279	F	4,430	4,020	3,200	4,230	4,850	5,230	4,830	
280	O	3,750	2,920	2,660	2,970	4,110	4,550	3,550	
281	M	4,070	3,740	2,870	4,180	4,490	5,170	4,180	
282	P	4,900	4,170	3,020	4,040	4,930	5,630	4,410	
284	D	3,780	3,310	3,110	3,310	4,330	4,520	3,820	
Average		3,950	3,590	2,730	3,850	4,530	4,920	4,010	

## MADE WITH CEMENT STORED FOR ONE YEAR

Series	Portland cement	1½ hours	6 hours	24 hours	96 hours	Average for 1½-96 hours	1½ hours	6 hours	24 hours
206	I	2,920	2,210	1,250	2,540	3,020	4,200	2,750	
207	K	3,760	2,530	1,450	2,910	4,110	4,320	3,200	
208	H	4,550	3,430	1,720	2,850	4,790	5,940	3,330	
209	O	4,050	2,860	1,460	2,520	3,760	3,800	2,680	
210	E	4,980	3,400	1,440	2,960	4,880	4,090	3,320	
211	F	5,320	4,020	1,340	3,470	5,090	4,360	3,660	
212	O	4,310	3,150	2,070	2,820	4,050	5,340	3,570	
213	M	4,510	3,460	1,480	2,810	4,390	4,530	3,300	
215	P	4,990	3,260	2,680	3,340	4,710	4,710	3,800	
217	D	2,700	2,620	2,040	2,600	3,370	4,260	3,070	
Average		4,210	3,140	1,690	2,870	4,220	4,350	3,280	

TABLE 12.—*Compressive strength per square inch and absorption in 21 days of concrete cylinders cured in water vapor for periods of three-fourths of an hour to 8 days at various temperatures*

[All cylinders were first cured 24 hours in moist closet at room temperatures, and following curing in water vapor were stored in dry air until tested. Each test result is average for 4 or more cylinders. Cement used was equal parts brands A and B1]

Temperature of water vapor	Check cylinders cured in water			¾ hour			1½ hours	
	Absorption	Compression		Absorption	Compression		Compression	
		7 days	28 days		7 days	28 days	7 days	28 days
°F.	Per cent	Pounds	Pounds	Per cent	Pounds	Pounds	Pounds	Pounds
100.....		3,270			2,630		2,400	
155.....		3,550			2,940		3,240	
212.....	5.0	3,230	4,720	6.9	3,020	2,680	2,590	2,400
230.....	5.6	3,730	5,100	7.0	2,060	2,170	1,830	2,190
260.....	5.8	3,640	4,960	7.4	1,960	1,630	2,000	2,130
285.....	5.8	3,790	5,780	7.3	1,780	1,740	2,010	2,220
315.....	5.8	3,810	5,050	6.9	2,010	2,020	2,490	2,640
350.....	5.7	3,840	5,310	7.3	1,670	1,940	2,500	2,800
Average.....	5.8	3,670	5,150	7.1	2,080	2,110	2,240	2,400

Temperature of water vapor	1½ hours	3 hours		6 hours	
	Absorption	Absorption	Compression		Compression
			7 days	28 days	
°F.	Per cent	Per cent	Pounds	Pounds	Pounds
100.....			3,080		3,290
155.....			3,380		3,730
212.....	7.4	7.2	2,800	3,130	3,220
230.....	6.9	6.7	2,180	2,320	2,300
260.....	7.1	7.2	2,170	2,460	2,700
285.....	7.2	7.3	2,420	2,590	3,080
315.....	6.8	6.5	3,090	3,030	3,560
350.....	7.0	6.7	3,210	3,310	3,110
Average.....	7.1	6.9	2,660	2,510	3,030

Temperature of water vapor	12 hours			24 hours			2 days	
	Absorption	Compression		Absorption	Compression		Compression	
		7 days	28 days		7 days	28 days	7 days	28 days
°F.	Per cent	Pounds	Pounds	Per cent	Pounds	Pounds	Pounds	Pounds
100.....		3,470			4,260		4,220	
155.....		4,020			4,420		4,460	
212.....	7.1	3,570	3,580	7.2	3,960	4,190	4,360	4,670
230.....	6.8	2,040	3,200	6.6	3,560	3,250	3,000	4,050
260.....	6.7	3,190	3,380	6.7	3,480	3,810	3,720	3,880
285.....	6.5	3,320	3,420	6.3	3,740	4,100	3,300	3,600
315.....	6.5	3,410	3,780	6.8	3,180	3,750	3,440	3,970
350.....	6.7	3,280	3,470	6.8	3,830	3,840	3,630	4,710
Average.....	6.7	3,340	3,470	6.7	3,630	3,830	3,710	4,250

TABLE 12.—Compressive strength per square inch and absorption in 21 days of concrete cylinders cured in water vapor for periods of three-fourths of an hour to 8 days at various temperatures—Continued

Temperature of water vapor  °F.	2 days	4 days		8 days			
	Absorption	Absorption	Compression		Absorption	Compression	
			7 days	28 days		9 days	28 days
	Per cent	Per cent	Pounds	Pounds	Per cent	Pounds	Pounds
100.....			4,870			5,280	
155.....			4,720			4,820	
212.....	6.6	6.1	4,330	4,990	6.1	5,180	6,050
230.....	6.9	6.1	4,480	4,000	6.2	3,830	4,450
260.....	6.6	6.4	3,820	3,810	6.6	3,260	3,690
285.....	6.7	6.5	3,740	4,230	6.5	3,540	4,300
315.....	6.8	6.6	3,430	4,280	6.8	3,580	4,640
350.....	6.5	6.3	4,140	4,320	6.2	3,770	4,260
Average.....	6.7	6.3	3,990	4,370	6.4	3,860	4,570

TABLE 13.—Compressive strength in pounds per square inch of concrete cylinders cured for 12 to 32 days in water vapor at different temperatures

Temperature of water vapor	12 days	14 days	16 days	18 days	20 days	22 days	24 days	26 days	28 days	30 days	32 days
100° F.....	4,760	4,910	4,610	4,760	4,460	4,290	5,380	4,950	5,100	4,610	4,530
155° F.....	5,190	4,090	5,210	4,590	4,740	4,850	5,090	4,730	5,020	4,960	4,550
212° F.....	4,670	4,580	5,080	4,580	4,530	4,840	4,430	4,600	4,290	4,500	4,860
Average.....	4,870	4,720	4,970	4,640	4,580	4,660	4,940	4,760	4,800	4,680	4,650

From a comparison of Figures 12 and 13, it is evident that the strength tests were more uniformly consistent, although lower, for the stored cements than for the fresh cements, with the closest agreement in actual strengths for curing temperatures of 260°, 315°, and 350° F., for the 24 and 96 hour curing periods. While strengths of the steam-cured cylinders averaged lower than 28-day strengths of the water-cured check cylinders, the data in Tables 11 to 13 reveal that individually, in some one or more of the high temperatures, cylinders made of each of the cements either exceeded or approached the 28-day strengths of comparable water-cured ones. There was not, however, any tendency for the steam-cured cylinders to develop abnormally high compressive strengths.

These strength trends are somewhat contrary to those reported a number of years ago by Wig (53), whose report was based on tests made in 1907 and 1908 in the structural materials laboratory of the United States Geological Survey at St. Louis, Mo., in which it was found that:

A compressive strength considerably (in some cases over 100 per cent) in excess of that obtained normally after aging for six months may be obtained in two days by using steam under pressure for curing the mortar or concrete.

Woodworth (60) in 1930 and Pearson and Brickett (41) in 1932 published reports that substantiated these conclusions of Wig.

It is possible that the strength of steam-cured concrete is influenced by both the cement and the sand, since in the manufacture of sand-lime brick strengths upward of 8,500 pounds per square inch have

been obtained, by using steam pressures of 125 to 150 pounds per square inch (42). It is evident, therefore, that Portland-cement concrete cured at these high temperatures could reasonably be expected to be stronger than that normally cured, particularly at less

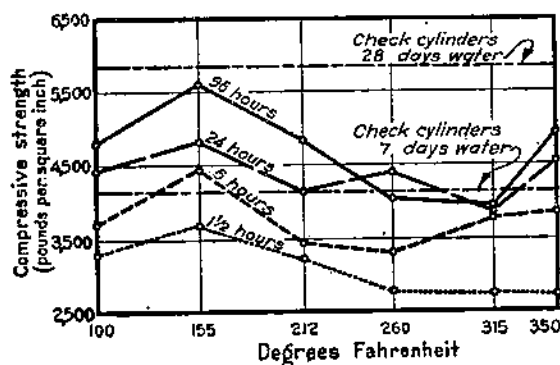


FIGURE 12.—Strength, at 7 days, of concrete cylinders made of fresh cements cured in water vapor at temperatures shown for 1½ to 96 hours. (Based on Table 11)

ages, if conditions were such that the free lime hydrate of the cement could combine readily with the silica of the sand which is active at these high temperatures. That some concretes cured at high temperatures do not develop strengths in excess of the strength of concrete cured normally may indicate that the active silica in the sand is not sufficient to bring this about.

Cylinders of all the cements, both fresh and stored, showed retrogression of strength for some time period at some of the temperatures, particularly for the 4-day (96 hours) period at temperatures of 260° and 315° F. Such retrogression was generally followed by full recovery of strength in tests at higher temperatures. Retrogression of the strength of cement steam cured under different conditions has been observed by a number of other workers. (9, 48, 56, 60.) The following explanation of this phenomenon has been suggested by Thorvaldson and Vigfusson (48) as a result of an interesting series of physical and chemical experiments at the University of Saskatchewan under the auspices of a research committee of the Engineering Institute of Canada.

It seems probable that the first action, causing a loss in tensile strength, is due to a change in the tricalcium aluminate of the cement and that this change is the primary cause of the increase in the sulphate resistance of the mortar. The second change, causing an increase in tensile strength, could then probably be due to hydration of the silicates, speeded up by the action of steam or possibly partly due to the formation of stable cementing substances from the aluminate.

Regardless of whatever physical or chemical changes in Portland cement follow curing in water vapor at temperatures of 212° F. and upward, hydration of the cement grains is very greatly accelerated. This is well shown in Plate 4 by the photomicrographs of thin sections

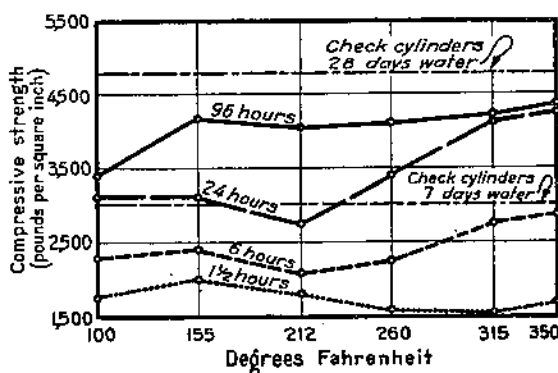
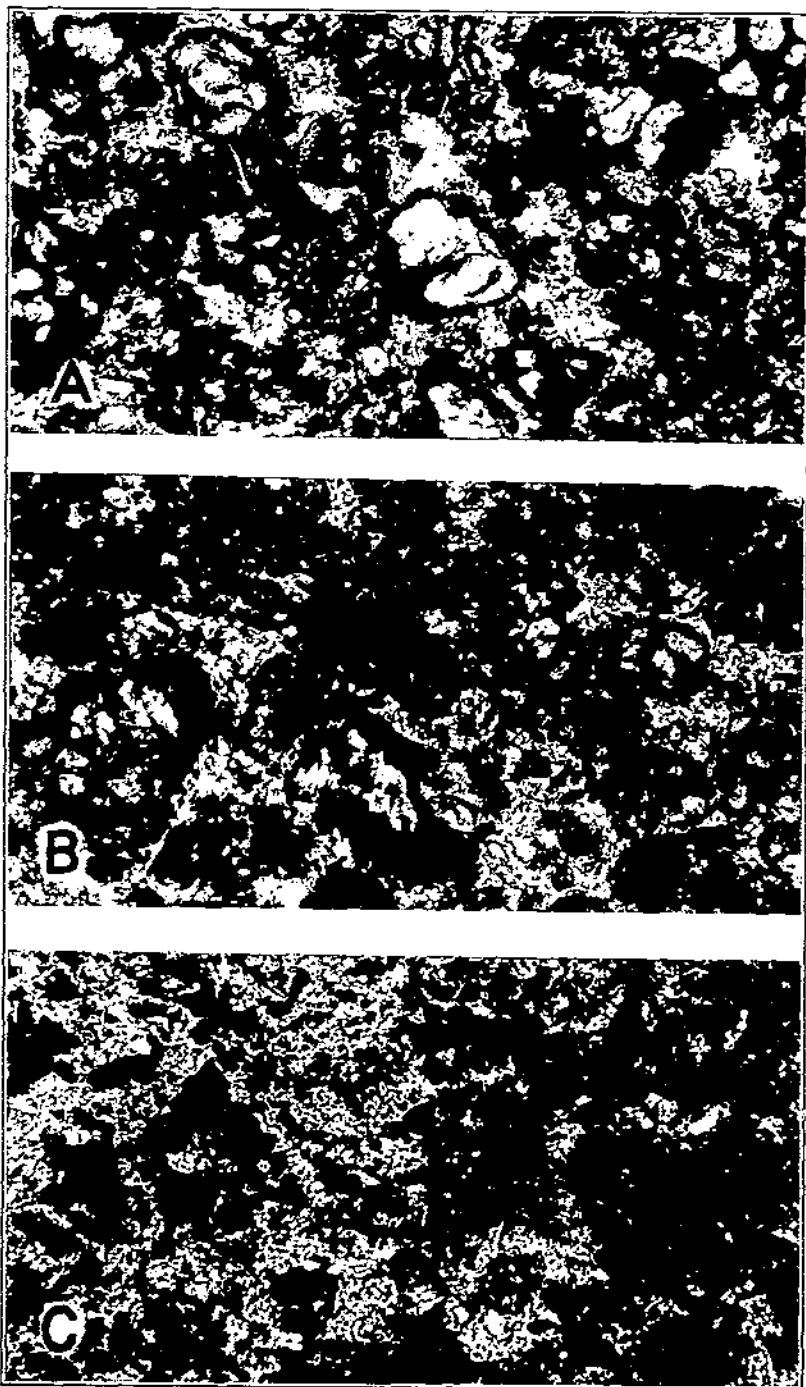
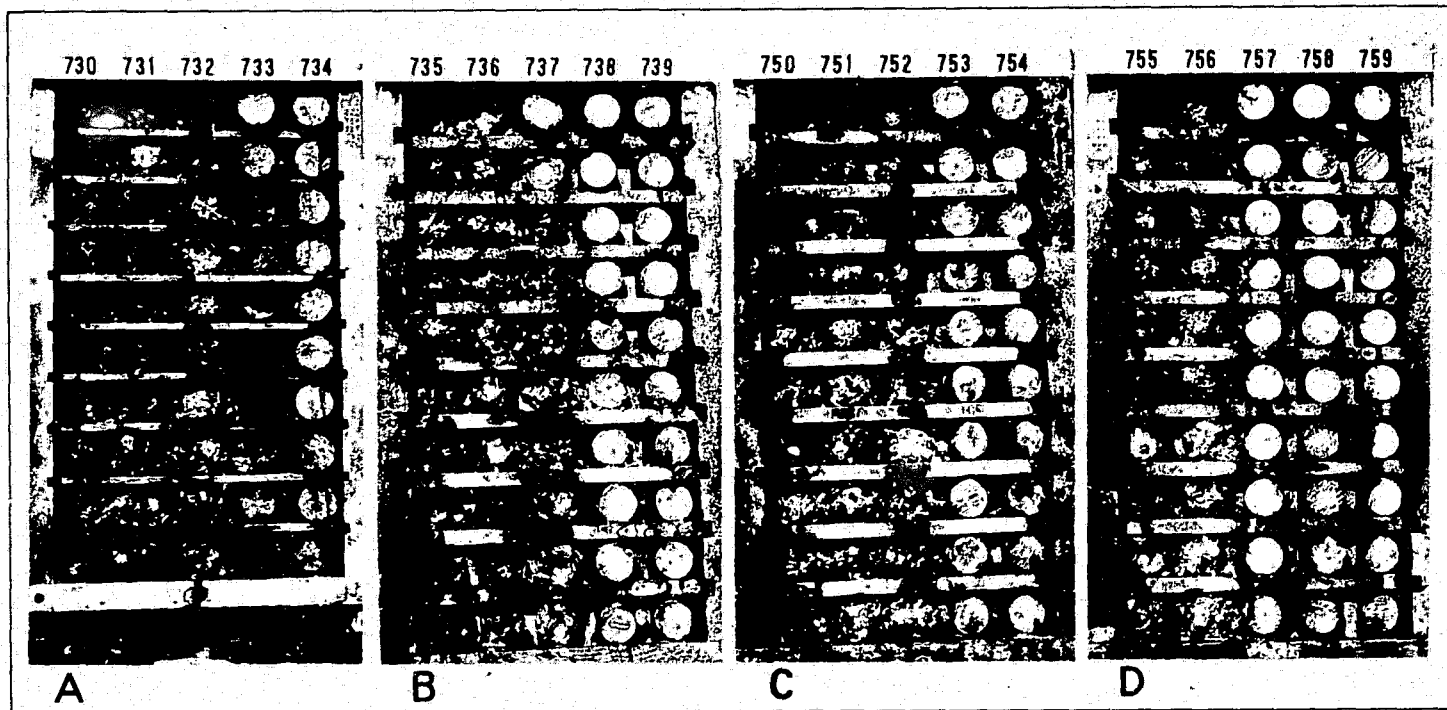


FIGURE 13.—Strength, at 7 days, of concrete cylinders, made of cements stored for 1 year, cured in water vapor at temperatures shown for 1½ to 96 hours. (Based on Table 11)



PHOTOMICROGRAPHS OF NEAT CEMENT BRIQUETS SHOWING PHYSICAL CHANGES RESULTING FROM DIFFERENT CONDITIONS OF CURING

A, Cured 12 days in water at room temperatures. B, Cured 6 hours in steam at 315° F. C, Cured 24 hours in steam at 315° F.  $\times 350$ .



CYLINDERS MADE OF TWO DIFFERENT LOTS OF STANDARD PORTLAND CEMENT AFTER STORAGE IN MEDICINE LAKE FOR THREE YEARS  
 From left to right the cements in each group are B1, A, D, G and L. A, Without air hardening. B, Air hardening five weeks. C, Without air hardening. D, Air hardening five weeks



of specimens. Within a few hours the steam-cured cement grains are greatly altered and reduced in size to a degree not approached in the water-cured specimens after 12 days.

Concrete cured in water vapor at all temperatures up to 350° F. and then stored in water, ordinarily gains strength much as it does when water-cured, although the rate of gain is somewhat retarded, the retardation depending on temperatures and duration of curing period. This statement is based on a study of the 7 and 28 day and 1 and 5 year test data in Table 10.

The effect on the 28-day strength of concrete, of applying water vapor at temperatures of 100°, 155°, and 212° F., at early ages, is illustrated in Figure 14. These graphs indicate that curing of concrete at temperatures above normal should preferably be commenced 12 to 24 hours after the concrete is made. Figure 14 is based on tests of cylinders made in 1923, entirely unrelated to those referred to in Figures 12 and 13, yet the cylinders cured at 155° were stronger in all cases than those cured at either 100° or 212°.

All temperatures in these steam-curing tests were those to which specimens were actually subjected during curing and, it will be noted, are considerably above those of 100° to 135° F. ordinarily used at commercial tile plants manufacturing "steam-cured" products. The maximum temperature of 350° is no greater, however, than has been used in the manufacture of sand-lime brick, and is therefore within the limit of practical application. A previous publication (87) gives more complete details about certain phases of concrete cured at high temperatures.

#### RESULTS WITH VAPOR-CURED CONCRETE

Concrete cured 12 to 24 hours in water vapor at temperatures between 100° and 350° F., then stored in dry air at room temperatures, had a compressive strength at seven days not greatly different from that of 7-day concrete water cured at room temperatures.

Curing prolonged beyond 48 hours had little effect on compressive strength of concrete cured in water vapor at temperatures between 190° and 350° F. Concrete cured 48 hours at these temperatures ordinarily attained a maximum and fairly constant strength equal to 80 to 90 per cent of that of 28-day concrete continuously water cured at room temperatures.

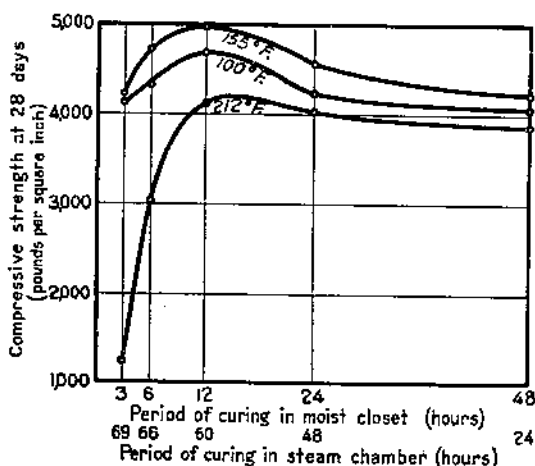


FIGURE 14.—Strength of concrete cylinders as affected by the time of their transfer from moist closet to steam chamber. The total curing period of 72 hours was followed by 25 days in air. Each point is the average for five cylinders made on different days and tested room-dry. (Cements A and B mixed in equal portions)

Variations in curing temperatures between the limits of 100° and 350° F. had no pronounced effect on strength, although specimens made with fresh cements were consistently somewhat stronger when cured at 155° than at the other temperatures. Specimens made of cements stored one year gave more uniform results but had lower strengths than those made of fresh cements. These statements are based on curing periods up to 8 days for all temperatures and up to 32 days for temperatures of 100°, 155°, and 212°. (Tables 12 and 13.)

With some exceptions, cylinders stored in tap water in the laboratory after being cured in high-temperature water vapor continued to increase in strength at a rate not essentially different from that of water-cured check cylinders.

To obtain the highest compressive strength of the concrete, the most favorable time for applying water vapor at temperatures of 100°, 155°, and 212° F. was 12 to 24 hours after making. Data for the other temperatures are not available.

The reactions of concrete made of Portland cements from different mills to curing under these special conditions has been essentially similar in both strength and resistance.

Concrete made of all the cements used in the strength tests showed retrogression of strength for some time period after being cured in water vapor at some temperature between 100° and 350° F. The retrogression was followed in most cases by full recovery of strength at higher temperatures or after longer curing periods. This phenomenon occurred most frequently and generally was of greatest magnitude in those groups of cylinders cured 96 hours at 260° and 315° F., with the result that tests were more uniform at 100°, 155°, 212°, and 350° than at either 260° or 315°.

With some exceptions the cements that lost most in strength during storage, as indicated by tests of cylinders following curing at all temperatures for the shorter time periods, were the cements that displayed greatest resistance to deterioration in Medicine Lake.

No gain in resistance to alkali followed when the temperature of the water vapor in which the concrete was cured was increased until 212° F. was reached. Between 212° and 285°, increased resistance followed increase of curing temperature. At a temperature of 212°, increased resistance followed lengthening of the curing period up to six days. Data for longer curing periods and for other temperatures are yet too incomplete to be conclusive although, up to 1932, specimens cured at the highest temperatures and for the longest periods have made the most favorable showings.

Absorption of concrete cured in water vapor at high temperatures is not a criterion of resistance to sulphate waters.

TABLE 14.—Tests of 2 by 4 inch concrete cylinders made from Portland cements from different mills after exposure for various periods in Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water

[Unless otherwise noted the fineness modulus of aggregate is 4.67 and the mix is 1:3. Each test result, with a few exceptions, is an average of 5 cylinders made on different days. Figures in parentheses, in compression test columns, indicate per cent of normal strength based on parallel tests of cylinders from the same batches, stored in tap water in the laboratory]

Series	Cement laboratory No.	Portland cement	Water ratio	Curing method			Absorption at 21 days	Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in air		Tank specimens				Lake specimens		
								7 days	28 days	1 year	5 years	1 year	3 years	5 years
172	18	½A and ½B1	0.59	Hours 24	Days 20	Days 35	Per cent 5.6	2,870	4,190	5,300	5,400	4,370 (82)		
177	18	do	.59	24	20	35	5.9	2,520	4,200	4,930	6,120	3,670 (74)		0
256	61	do	.59	24	20	35	5.8	2,780	3,950	5,200	6,520	1,480 (28)		0
391	74	do	.62	24	20	35	6.3	3,130	4,890	6,080	6,430	2,480 (41)	0	0
434	74	do	.66	24	20	35	6.4	2,690	4,030	5,050	5,830	4,870 (96)	0	0
554	129	do	.64	24	20	35	6.3	3,570	4,760	5,980	6,820	3,530 (59)	0	0
583	139	do	.62	24	20	35	6.3	3,810	4,950	6,420	7,820	3,650 (57)	0	0
640	139	do	.64	24	20	35	6.5	3,320	4,470	5,800	6,070	4,530 (78)	0	0
715	176	do	.62	24	20	35	5.6	3,830	5,100	6,420		4,550 (71)	0	0
771	204	do	.62	24	20	35	5.9	3,450	4,440	5,870		0	0	0
776	204	do	.62	24	20	35	6.2	3,400	4,420	6,480		0	0	0
801	219	do	.62	24	20	35	5.8	3,740	6,260	5,790		2,820 (49)	0	0
806	219	do	.62	24	20	35	5.8	3,450	4,620	6,110		1,620 (27)	0	0
811	219	do	.62	24	20	35	5.8	4,830	6,350			0	0	0
822	225	do	.62	24	20	35	5.5	3,620	4,830	6,350		0	0	0
830	232	do	.62	24	20	35	5.3	3,220	5,230	6,250		3,800 (61)	0	0
840	232	do	.62	24	20	35	5.9	3,230	4,720	6,360		3,950 (62)	0	0
855	232	do	.62	24	20	35	5.8	3,810	5,050	6,010		3,060 (51)	0	0
976	237	do	.62	24	20	35	5.7	3,840	5,310	5,540		4,070 (73)	0	0
258	62	do	.62	24	20	35	6.0	3,700	4,590	5,750		4,650 (81)	0	0
655	156	B1	.59	24	20	35	5.8	2,960	3,790	4,580	5,780	800 (17)	0	0
665	156	B1	.62	24	20	35	6.4	3,670	5,070	5,800		2,870 (50)	0	0
670	161	B1	.62	24	20	35	5.9	3,500	5,020	5,820		3,860 (66)	0	0
675	161	B1	.62	24	20	35	6.2	3,550	5,470	7,100		3,050 (43)	0	0
690	166	B1	.62	24	20	35	5.8	3,490	5,320	6,450		3,870 (60)	0	0
695	166	B1	.62	24	20	35	6.4	2,830	4,680	6,150		3,810 (62)	0	0
699	170	B1	.62	24	20	35	6.8	2,960	4,340	6,510		4,210 (65)	0	0
709	170	B1	.62	24	20	35	5.8	3,660	5,580	6,250		3,130 (50)	0	0
730	178	B1	.62	24	20	35	5.8	4,020	6,940	6,250		2,930 (42)	0	0
735	178	B1	.62	24	20	35	5.6	3,400	4,610	5,780		2,580 (45)	0	0
750	183	B1	.62	24	20	35	5.6	3,820	4,660	5,450		5,360 (98)	0	0
755	183	B1	.62	24	20	35	5.9	3,810	5,340	5,740		1,790 (31)	0	0
							5.7	3,500	5,190	5,650		5,430 (96)	0	0

TABLE 14.—Tests of 2 by 4 inch concrete cylinders made from Portland cements from different mills after exposure for various periods in Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water—Continued

Series	Cement laboratory No.	Portland cement	Water ratio	Curing method			Absorption at 21 days	Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in air		Tank specimens				Lake specimens		
								7 days	28 days	1 year	5 years	1 year	3 years	5 years
464	109	Portland B2	0.64	Hours 24	Days 20	Days 35	Per cent 5.8	3,740	5,010	7,070	5,940	5,950 (84)	920	0
465	110	do.	.64	24	20	35	5.5	3,660	4,920	6,910	6,070	4,850 (70)	0	0
466	111	Portland B3	.64	24	20	35	5.7	3,810	5,020	5,860	5,790	3,960 (68)	0	0
257	65	A	.59	24	20	35	5.3	2,720	4,290	5,490	6,440	3,190 (58)	0	0
657	158	A	.62	24	20	35	6.5	4,080	4,560	4,920		0	0	0
667	158	A	.62	24	20		6.0	3,450	4,670	0,280		790 (13)	0	0
672	163	A	.62	24	20		6.0	3,790	5,240	0,070		1,880 (31)	0	0
677	163	A	.62	24	20	35	5.9	3,670	4,460	0,070		1,630 (27)	0	0
692	168	A	.62	24	20		5.6	3,760	5,520	5,760		680 (12)	0	0
697	168	A	.62	24	20	35	5.9	3,970	5,440	5,820		1,670 (29)	0	0
700	171	A	.62	24	20	35	5.8	3,950	5,360	6,190		1,830 (30)	0	0
710	171	A	.62	24	20		5.6	4,090	4,700	5,260		1,940 (37)	0	0
731	179	A	.62	24	20		5.7	3,500	4,770	6,430		810 (13)	0	0
736	179	A	.62	24	20	35	5.8	3,680	5,030	5,080		2,620 (52)	0	0
751	184	A	.62	24	20		5.8	4,200	5,580	6,090		2,310 (38)	0	0
756	184	A	.62	24	20	34	5.7	3,830	5,140	5,850		4,420 (76)	0	0
369	86, 99	I	.64	24	20	35	6.1	3,590	4,370	6,110	6,650	4,470 (73)	2,500	0
703	174	I	.62	24	20	35	6.2	4,480	5,610	7,040		6,240 (89)	4,150	
713	174	I	.62	24	20		5.7	4,370	4,790	6,330		5,800 (92)	5,600	
734	182	I	.62	24	20		6.0	4,040	5,580	5,940		6,230 (105)	6,740	
739	182	I	.62	24	20	35	5.8	3,620	5,260	5,810		6,930 (119)	6,180	
754	187	I	.62	24	20		6.2	3,980	5,050	6,770		5,950 (88)	6,130	
759	187	I	.62	24	20	35	5.7	3,840	5,380	6,210		6,040 (97)	6,810	
761	199	I	.62	24	20	35	6.0	4,160	4,830	7,560		6,900 (91)	5,370	
372	97	K2	.64	24	20	35	6.4	3,120	4,440	5,720	7,350	4,600 (80)	2,260	1,450 (20)
658	159	K2	.62	24	20	35	6.5	3,990	5,170	6,290		5,490 (87)	1,740	
668	159	K2	.62	24	20		6.0	3,140	5,240	5,980		5,790 (97)	4,410	
673	164	K2	.62	24	20		6.0	4,260	5,340	6,800		5,830 (86)	2,690	
678	164	K2	.62	24	20	35	5.9	3,770	5,580	6,270		5,640 (90)	2,710	
693	169	K2	.62	24	20		6.2	3,280	5,370	5,740		5,460 (95)	5,060	
698	109	K2	.62	24	20	35	6.2	3,400	4,730	6,360		6,580 (104)	4,610	
373	98	K1	.64	24	20	35	6.1	4,220	4,970	7,050	6,520	3,400 (48)	0	0
364	81	E	.62	24	20	35	5.8	3,340	4,770	7,320	7,540	2,910 (40)	0	0
404	103	S	.62	24	20	35	6.0	3,010	4,380	5,470	6,310	3,160 (58)	1,230	0
413	105	Q2	.62	24	20	35	5.7	3,520	5,390	6,500	6,350	4,300 (66)	0	0
401	100	Q1	.62	24	20	35	6.4	2,900	5,310	5,110	5,960	0	0	0
370	87, 88	P	.64	24	20	35	6.1	3,730	5,040	6,540	6,700	810 (12)	0	0

TESTS OF CONCRETE EXPOSED TO SULPHATE WATERS 37

371	84	L1	.64	24	20	35	5.8	3,690	5,000	5,770	6,230	3,170 (55)	250	0
260	34	C	.66	24	20	35	0.1	3,340	4,540	5,980	6,850	5,090 (85)	4,390	4,290 (63)
367	82, 95	C	.67	24	20	35	0.8	3,220	4,320	5,120	7,700	4,790 (76)	2,280	0
664	155	C	.66	24	20	35	6.6	3,580	5,150	6,270		5,910 (95)	3,260	
664	155	C	.66	24	20	35	6.5	3,220	4,890	6,220		6,250 (95)	4,820	
669	160	C	.66	24	20	35	6.8	3,150	5,180	6,590		6,190 (105)	5,120	
674	160	C	.66	24	20	35	6.5	3,430	5,480	7,030		5,610 (80)	5,820	
689	165	C	.66	24	20	35	6.3	3,200	5,000	7,390		5,670 (77)	4,960	
694	165	C	.66	24	20	35	6.5	3,480	5,520	6,780		5,910 (87)	4,400	
385	83	H	.62	24	20	35	0.1	3,940	4,770	6,280	7,250	3,680 (69)	290	0
656	157	H	.62	24	20	35	6.5	3,230	5,090	5,670		4,510 (80)	0	0
666	157	H	.62	24	20	35	6.0	3,520	5,120	6,640		5,490 (83)	0	0
671	162	H	.62	24	20	35	6.7	3,380	4,680	6,260		5,550 (89)	4,140	
676	162	H	.62	24	20	35	6.5	3,330	4,460	5,830		5,870 (101)	3,900	
691	167	H	.62	24	20	35	0.9	2,360	3,760	5,040		4,150 (82)	610	0
696	167	H	.62	24	20	35	0.6	3,080	4,570	5,510		5,490 (100)	2,290	0
259	33	D	.59	24	20	35	5.4	3,340	4,780	5,900	6,230	1,590 (27)	0	0
308	85, 94	D	.64	24	20	35	6.4	3,390	4,130	5,730	6,770	2,986 (52)	0	0
701	172	D	.62	24	20	35	0.1	4,040	5,600	6,170		5,750 (93)	2,170	0
711	172	D	.62	24	20	35	5.8	3,950	5,380	7,050		4,980 (71)	2,070	0
732	180	D	.62	24	20	35	5.9	3,780	5,400	6,620		4,750 (72)	0	0
737	180	D	.62	24	20	35	6.2	3,960	5,130	5,580		4,780 (85)	1,100	0
752	185	D	.62	24	20	35	5.8	4,390	5,290	6,090		3,850 (63)	0	0
757	185	D	.62	24	20	35	5.7	4,080	5,530	6,160		4,860 (79)	3,490	0
763	196	BB	.62	24	20	35	5.9	4,030	5,060	6,360		2,830 (44)	0	0
361	80	F	.62	24	20	35	6.3	2,870	4,320	6,350	5,730	0	0	0
769	201	X	.62	24	20	35	6.3	3,050	4,960	6,440		4,440 (69)	0	0
576	133	AA	.62	24	20	35	6.0	3,460	4,820	5,850	6,520	3,440 (50)	0	0
765	197	AA	.60	24	20	35	5.8	3,750	5,430	6,820		0	0	0
402	101	R	.62	24	20	35	6.3	3,680	4,940	6,290	5,900	2,440 (39)	0	0
415	107	V	.62	24	20	35	6.5	2,830	4,540	5,920	5,550	2,940 (50)	0	0
555	128	Y	.64	24	20	35	6.0	3,780	4,670	5,480	6,610	3,510 (64)	0	0
557	128	Y	.73	24	20	35	6.4	2,760	4,250	4,690	5,550	1,800 (38)	0	0
313	65	M	.60	24	20	35	5.9	3,150	3,950	6,050	5,680	2,250 (37)	0	0
366	77, 96	G	.64	24	20	35	6.0	4,050	4,820	5,900	6,690	1,030 (33)	0	0
577	134	G	.62	24	20	35	6.3	3,910	5,610	6,730	7,320	6,140 (91)	3,340	1,300 (18)
702	173	G	.62	24	20	35	6.1	3,530	5,330	6,510		5,290 (81)	3,270	
712	173	G	.62	24	20	35	5.7	3,630	4,910	6,120		5,830 (96)	5,060	
733	181	G	.62	24	20	35	6.0	4,070	5,190	5,710		6,040 (100)	4,150	
738	181	G	.62	24	20	35	5.9	3,810	5,080	6,290		5,960 (95)	5,170	
753	186	G	.62	24	20	35	5.9	3,750	5,420	6,290		5,890 (94)	5,070	
758	186	G	.62	24	20	35	5.6	3,860	5,020	5,930		5,800 (98)	7,290	
363	89	O1	.62	24	20	35	5.9	4,010	5,220	6,730	6,430	670 (10)	0	0
468	113	O2	.64	24	20	35	6.3	2,140	3,940	6,000	5,260	5,590 (93)	5,580	3,550 (67)
767	198	CC	.62	24	20	35	6.2	3,440	5,540	6,650		5,330 (80)	2,380	0
405	104	T	.62	24	20	35	6.4	2,940	4,380	5,530	5,860	280 (5)	0	0
416	106	U	.62	24	20	35	5.8	3,670	5,010	6,510	5,240	1,490 (23)	0	0
414	106	U	.62	24	20	35	6.1	3,030	4,410	6,430	5,900	2,490 (39)	0	0
362	79	N1	.62	24	20	35	6.1	3,220	4,600	6,970	6,550	4,000 (67)	950	0
403	102	N2	.62	24	20	35	5.9	3,860	5,640	6,630	5,720	4,410 (67)	1,860	0
575	132	Z	.62	24	20	35	6.0	4,760	5,160	6,490	6,510	4,970 (77)	2,210	0

## PORTLAND CEMENTS FROM DIFFERENT MILLS

## RESISTANCE TO SULPHATE ACTION

Standard Portland cements from different manufacturing plants may differ greatly in sulphate resistance, as is well illustrated by the photographs of Plate 5 supported by the data given in Table 14. This fact was reported in 1926 (34) and subsequently (36, 38). The reason for this is not clear. Behavior in sulphate waters of concrete and mortars made of Portland cements from 35 mills has been observed. Each of these cements was tested under four distinct exposure conditions as follows: (1) Half of one of the standard 7-day tensile strength briquets was stored in the laboratory in a 1 per cent solution of sodium sulphate, and its condition at six months was rated according to its appearance; (2) the companion half of the briquet

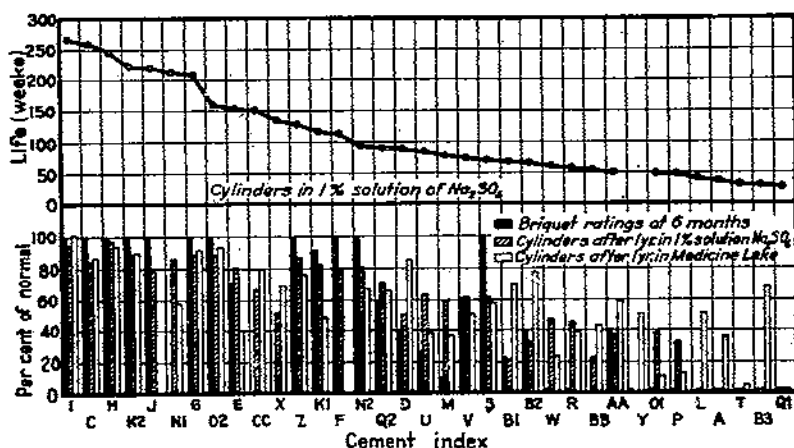


FIGURE 15.—Resistance to action of sulphate waters by cylinders and briquets made of standard Portland cements from 35 mills, as determined under different conditions of exposure (100 per cent normal represents: For cylinders, a strength ratio of 1.00; for briquets, a visual rating of 10). Note the general reliability of the briquet ratings by appearance after six months in 1 per cent solutions of  $\text{Na}_2\text{SO}_4$ , as compared with the change of volume and compression tests of the cylinders similarly exposed

exposed under the first condition was stored in the laboratory in a 1 per cent solution of magnesium sulphate and its condition at six months was rated according to its appearance; (3) cylinders that had not been air hardened were stored in the laboratory in 1 per cent solutions of sodium sulphate and their condition was rated by compression tests at one year and by length changes; (4) after five weeks of air hardening, cylinders were stored in Medicine Lake and their condition was rated by compression tests at one year.

The results of these tests are given in Table 15 and compared graphically in Figure 15. The values shown are average results where more than one lot of any cement was tested. Photographs of the briquets after six months in the sulphate solutions are reproduced in Plate 2. Study of Figure 15 shows that those cements that best resisted sulphate action under one condition of exposure ordinarily were resistant to action under the three other conditions.

Based on numerous repeat tests, the assumption that resistance of a cement is a characteristic fully as constant as any other property, seems justified.

## PHYSICAL AND CHEMICAL TESTS

Chemical analyses of one lot of each of 35 different brands of cement used in these experiments are given in Table 16. The average results of standard tests of these cements for time of setting, tensile strength, and fineness are shown graphically in Figure 16. Special determinations for fineness of 7 of the cements differing widely in resistance are recorded in Table 17.<sup>6</sup>

TABLE 15.—Sulphate resistance of 35 standard Portland cements under different exposure conditions listed in order of resistance as indicated by life of cylinders

[Excepting only E, F, M, and L, the same lots of cements were used for all tests]

Portland cement	Cylinders stored in 1 per cent Na <sub>2</sub> SO <sub>4</sub>		Medicine Lake, S. Dak., cylinders strength ratios at 1 year	Briquets rated visually at 6 months		Portland cement	Cylinders stored in 1 per cent Na <sub>2</sub> SO <sub>4</sub>		Medicine Lake, S. Dak., cylinders strength ratios at 1 year	Briquets rated visually at 6 months	
	Life <sup>1</sup>	Strength ratios at 1 year		Na <sub>2</sub> SO <sub>4</sub> 1 per cent	MgSO <sub>4</sub> 1 per cent		Life <sup>1</sup>	Strength ratios at 1 year		Na <sub>2</sub> SO <sub>4</sub> 1 per cent	MgSO <sub>4</sub> 1 per cent
	Weeks		Per cent				Weeks		Per cent		
L	266.0	94.7	101.6	10.0	10.0	M	77.6	60.0	37.0	1.0	8.0
C	257.0	85.0	87.0	10.0	10.0	V	75.0	61.0	50.0	6.1	7.0
H	246.0	97.9	94.0	10.0	10.0	S	72.3	62.0	58.0	10.0	10.0
K2	222.5	90.0	90.0	10.0	10.0	B1	70.0	21.3	69.8	2.2	4.0
J	220.0	79.0		10.0	10.0	B2	65.9	32.5	77.0	4.0	7.0
N1	211.8	86.0	57.0			W	62.0	47.0	23.0	0	7.0
G	209.0	88.0	91.0	10.0	10.0	R	58.6	45.0	39.0	0	7.0
O2	199.7	87.0	93.0	10.0	10.0	BB	55.7	21.0	44.0		
E	152.6	81.0	40.0	7.0	10.0	AA	51.3	37.0	59.0	4.0	10.0
CC	150.9	66.0	80.0			Y			51.0	0	4.0
X	137.1	32.0	69.0			O1	49.6	33.0	10.0	0	3.0
Z	127.9	87.0	77.0	10.0	10.0	P	48.7	33.0	12.0	0	3.0
K1	118.1	63.0	48.0	9.0	10.0	L	41.6	0	51.0		
F	115.1	60.0		10.0	10.0	A	36.9	0	35.7	0	3.5
N2	95.3	81.0	67.0	10.0	10.0	T	30.0	0	5.0	0	5.0
Q2	91.6	70.0	68.0	6.0	10.0	B3	29.2	0	68.0	0	4.0
D	91.6	50.0	86.0	4.0	8.7	Q1	27.0	0	0	0	3.0
U	86.1	64.0	39.0	2.6	6.0						

<sup>1</sup> Time required to increase 0.01 inch in length.

TABLE 16.—Chemical analyses of Portland cements used

[Chemical analyses by the Division of Tests, Bureau of Public Roads, U. S. Department of Agriculture, except for cements CC, X, and BB, which were analyzed by Bureau of Standards, U. S. Department of Commerce. Only one lot of each cement analyzed]

Portland cement	Silica (SiO <sub>2</sub> )	Iron (Fe <sub>2</sub> O <sub>3</sub> )	Alumina (Al <sub>2</sub> O <sub>3</sub> )	Lime (CaO)	Magnesia (MgO)	Sulphuric anhydride (SO <sub>3</sub> )	Loss on ignition	Total
	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
L	21.71	3.39	6.53	62.50	1.59	1.77	1.84	99.33
C	22.43	3.20	5.03	62.54	3.75	1.91	1.20	100.06
H	21.13	2.73	8.89	62.40	.40	1.73	1.29	98.71
K2	21.45	2.65	6.45	62.59	2.67	1.82	1.95	99.79
J	20.47	3.58	6.24	61.82	3.94	1.85	1.95	99.85
N1	22.85	4.27	4.93	61.70	1.92	1.25	3.10	100.02
G	20.82	3.10	7.52	62.75	1.12	1.64	2.10	99.05
O2	24.87	2.39	4.67	61.30	1.26	1.58	3.60	99.57
E	21.93	3.42	6.31	62.29	3.44	1.33	1.22	99.94
CC	22.30	2.90	6.10	61.70	.70	1.70	1.10	99.50
X	22.00	2.70	5.50	61.90	4.60	1.80	1.00	99.50
Z	21.05	2.98	5.42	62.55	4.20	1.51	2.35	100.06
Average	21.92	3.11	6.12	62.41	2.50	1.66	1.89	99.62

<sup>6</sup> Throughout this discussion of cements from different mills, the arrangement of the material in all tables and figures is that of sulphate resistance as determined by the time required for 2 by 4 inch cylinders to increase in length 0.01 inch, recorded as the "life" in the second column of Table 15.

TABLE 16.—Chemical analyses of Portland cements used—Continued

Portland cement	Silica (SiO <sub>2</sub> )	Iron (Fe <sub>2</sub> O <sub>3</sub> )	Alumi- na (Al <sub>2</sub> O <sub>3</sub> )	Lime (CaO)	Magne- sia (MgO)	Sulphu- ric an- hydride (SO <sub>3</sub> )	Loss on igni- tion	Total
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
K1	22.90	2.82	5.88	63.70	0.94	1.72	1.85	99.91
F	21.55	2.89	8.51	60.64	1.85	1.94	2.09	99.47
N2	21.74	3.10	6.38	63.06	1.99	1.76	1.47	99.50
Q2	22.45	2.33	5.92	62.70	1.79	1.87	2.70	99.76
D	21.12	2.48	6.82	61.24	5.24	2.01	1.90	100.31
U	22.40	3.22	6.83	62.65	1.34	1.22	2.15	99.81
M	21.80	2.57	7.69	62.05	2.46	2.16	1.18	99.91
V	21.50	2.80	6.30	62.08	1.21	1.80	3.70	99.39
S	21.52	3.30	5.63	61.40	5.00	1.34	1.53	99.72
B1	20.53	2.14	9.61	63.06	1.56	1.72	1.26	99.88
B2	22.40	2.42	6.48	63.05	1.76	1.49	2.20	99.80
W	21.15	2.70	7.40	63.35	1.77	1.37	2.25	99.99
Average.....	21.76	2.73	6.95	62.42	2.24	1.70	2.03	99.83
R	21.70	2.70	6.59	64.19	1.27	1.66	1.55	99.66
BB	21.30	2.70	7.20	64.80	.90	1.80	1.30	100.10
AA	20.65	3.61	8.86	62.51	1.75	1.49	1.04	99.94
Y	21.85	3.10	6.45	62.65	1.64	1.48	2.65	99.82
O1	21.60	3.46	5.04	62.75	2.68	1.68	2.75	99.96
P	22.50	3.06	6.39	62.95	1.30	1.56	2.25	100.01
L	22.22	3.20	8.06	62.30	.59	1.47	1.46	99.60
A	22.66	2.51	8.12	61.87	1.05	1.72	1.60	99.53
T	20.73	2.85	6.23	61.16	4.95	1.61	2.06	99.59
B3	20.97	4.19	6.71	63.43	1.45	1.65	1.54	99.94
Q1	21.28	3.14	7.72	61.33	2.65	1.53	1.90	99.55
Average.....	21.59	3.14	7.03	62.75	1.84	1.60	1.83	99.79
General average.....	21.76	2.99	6.69	62.52	2.20	1.66	1.92	99.74

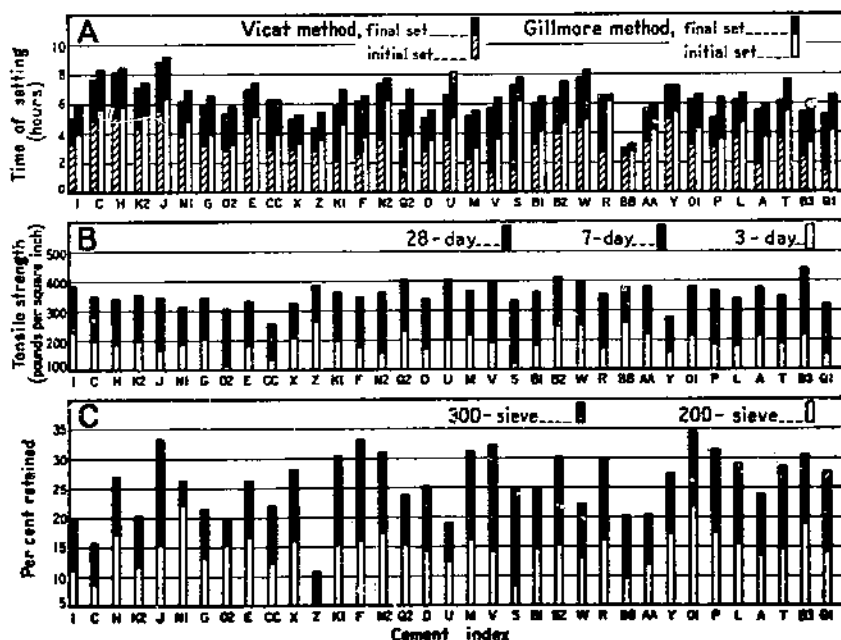


FIGURE 16.—Results of standard tests of 35 cements: A, Time of set; B, tensile strength; C, fineness. The order of the cements, from the left, is that of the life of cylinders in 1 per cent solutions of sodium sulphate. (Table 15)



Examination of the data fails to disclose any trends that appear sufficiently significant to account for differences in resistance displayed by the cements. Perhaps the nearest approach to a trend is indicated by the chemical analyses in the quantities of alumina and iron considered together. Long ago Le Chatelier (30) advanced the theory that cements low in alumina and high in iron resist sea water best. Analyses in Table 16 support this conclusion when considered generally, but show many exceptions for individual cements.

#### COMPOUNDS IN THE CEMENTS

The actual composition of hydrated Portland cement is a subject outside the scope of this bulletin. It was deemed worth while, though, to calculate the compounds recorded in Table 18, after an adaptation by Bogue (13) of a method suggested by Colony (1), in order to bring out possible trends. The results are disappointing in that the table shows no decided trends, although wide differences in resistance are displayed by individual cements. Contrariwise, Thorvaldson (49), following a carefully conducted series of tests in which he used a few commercial cements and a number of specially prepared laboratory cements made of pure raw materials, stated among other conclusions that—

The higher the lime content of the cement (i.e. the higher the percentage of tricalcium silicate), the aluminum remaining the same, the lower is the resistance to the action of the sulphates.

This did not consistently hold for the 35 commercial cements of Table 18.

TABLE 17.—Fineness of seven Portland cements differing widely in resistance to sulphate waters

[Analyses other than No. 200 sieve were made by Bureau of Standards, U. S. Department of Commerce]

Portland cement	Life in 1 per cent Na <sub>2</sub> SO <sub>4</sub>	Retained by No. 200 sieve	Retained by No. 325 sieve	Greater than 60 microns	Greater than 40 microns	Greater than 20 microns	Greater than 10 microns
	Weeks	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
I.....	266	15.3	25.3				83.5
C.....	257	9.1	17.2				81.8
H.....	246	15.9	25.4	33.8	47.9	66.0	84.1
K1.....	118	18.2	24.7	34.7	48.1	65.7	83.0
D.....	92	17.9	28.1				84.1
B1.....	70	17.0	17.4	32.3	46.9	63.4	80.8
A.....	37	18.2	29.7				83.8

#### RAW MATERIALS

Whether or not the degree of resistance of a commercial standard Portland cement is influenced by the constitution of the raw materials is difficult to determine, because many factors are involved in manufacturing the cements. However, there does appear to be a tendency for cements from adjacent plants, or from plants known to use similar raw materials, to behave alike in concrete exposed to sulphate action.

TABLE 13.—*Calculated compounds (in per cent) of the Portland cements for which chemical analyses are shown in Table 16*

[For bases of these calculations refer to Paper No. 21 of the Portland Cement Association fellowship at the U. S. Bureau of Standards, Department of Commerce]

Group and Portland cement	Ignition loss	Free MgO	Free CaO	CaSO <sub>4</sub>	4CaOAl <sub>2</sub> O <sub>3</sub> Fe <sub>2</sub> O <sub>3</sub>	3CaOAl <sub>2</sub> O <sub>3</sub>	3CaOSiO <sub>2</sub>	2CaOSiO <sub>2</sub>
Group 1:								
I.....	1.84	1.59	0.35	3.0	10.3	11.5	33.3	37.4
C.....	1.20	3.75	.14	3.2	9.7	7.9	39.6	34.5
H.....	1.29	.40	.35	3.2	8.3	18.9	22.5	43.6
K2.....	1.95	2.97	.46	3.1	8.1	12.6	37.1	33.6
J.....	1.95	3.94	(?)	3.1	10.9	10.4	43.6	25.8
N1.....	3.10	1.92	(?)	2.1	13.0	5.6	35.2	38.0
G.....	2.10	1.12	.92	2.8	9.4	14.7	33.7	34.4
O.....	3.60	1.26	.52	2.7	7.3	8.1	19.5	56.7
E.....	1.22	3.44	(?)	2.3	10.4	10.9	35.6	36.0
CC.....	1.10	.70	(?)	2.9	8.8	11.2	43.8	31.0
X.....	1.00	4.60	(?)	3.1	8.2	10.0	38.7	34.0
Z.....	2.35	4.20	(?)	2.6	9.1	9.3	49.5	23.6
Average.....	1.69	2.50	.46	2.8	9.5	10.2	36.0	35.7
Group 2:								
K1.....	1.95	.94	(?)	2.9	8.6	11.8	38.6	38.1
F.....	2.09	1.85	.64	3.3	8.8	17.6	13.4	51.7
N2.....	1.47	1.99	.29	3.0	9.4	11.7	37.9	33.9
Q2.....	2.70	1.79	1.12	3.2	7.1	11.7	31.4	40.1
D.....	1.90	5.24	.88	3.4	7.5	13.9	39.0	38.0
U.....	2.15	1.34	(?)	2.1	9.8	12.6	30.6	41.2
M.....	1.18	2.46	Trace.	3.7	7.8	16.0	25.2	43.6
V.....	3.70	1.21	(?)	3.1	8.5	11.9	37.8	33.2
S.....	1.53	5.00	(?)	2.3	10.0	9.3	39.8	21.9
B1.....	1.26	1.58	.38	2.9	6.3	22.0	26.6	38.9
B2.....	2.20	1.76	(?)	2.5	7.4	13.1	35.0	37.9
W.....	2.25	1.77	(?)	2.3	8.2	15.0	39.5	30.9
Average.....	2.03	2.24	.55	2.9	8.3	13.9	32.0	37.5
Group 3:								
R.....	1.55	1.27	(?)	2.6	8.2	12.9	43.3	29.6
BB.....	1.30	.90	(?)	3.1	8.2	14.5	44.4	27.7
AA.....	1.04	1.75	(?)	2.5	11.1	17.3	28.4	37.8
Y.....	2.65	1.64	(?)	2.5	9.4	11.8	36.7	35.0
O1.....	2.75	2.68	.52	2.9	10.5	7.5	45.4	27.8
P.....	2.25	1.30	(?)	2.6	9.3	11.7	33.4	39.4
L.....	1.45	.59	(?)	2.5	9.7	15.9	22.9	46.5
A.....	1.60	1.05	.52	2.9	7.0	17.2	14.4	54.2
T.....	2.90	4.95	1.60	2.7	8.7	11.7	34.2	33.7
B3.....	1.54	1.45	(?)	2.8	12.7	10.7	42.9	27.8
Q1.....	1.90	2.65	(?)	2.6	9.5	15.1	27.1	40.6
Average.....	1.83	1.84	.88	2.7	9.5	13.3	33.9	36.4
General average.....	1.92	2.20	.58	2.8	9.1	12.7	34.0	36.5

1 No data.

The 35 cements of Table 15 came from plants located in 15 States of the United States, mostly in the upper Mississippi River basin and the far West, and from two Canadian Provinces. Nearly all the highly resistant cements came from the relatively small area in Illinois, Iowa, Kansas, and Missouri indicated by cross-hatching in Figure 17, whereas none of the low-resistance cements came from this area. Of the 10 cements tested from plants within this area, 6 were among the 7 most resistant of the 35, and all 10 were among the 14 most resistant. It may be significant or merely coincidental that 9 of these 10 cements came from plants using raw materials from the Carboniferous geological system. It has not been possible to obtain complete information on the geological origin of all raw materials used at each plant, but apparently limestone from the Carboniferous system was used at not more than 13 of the 35 plants and shale from the same system at possibly 10 plants.

With so high a proportion of the resistant cements coming from plants using raw materials of the same geological age taken from a restricted area, and considering the other data, it seems logical to conclude that the raw materials used in the manufacture of a standard Portland cement are a factor in its resistance to sulphate action.

#### SUGGESTED TEST FOR SULPHATE RESISTANCE

To eliminate cements very low in resistance from consideration for concrete that is to be exposed to the action of sodium sulphate or magnesium sulphate, the following test routine is suggested:

One-half of each of the three briquets used in the standard 7-day tensile test should be stored in a 5 per cent solution of sodium sulphate and the companion half in a 5 per cent solution of magnesium sulphate. To make these 5 per cent solutions, on the basis of anhydrous salts, requires 3 ounces of room-dry salt per gallon of water. Not more than 15 briquet halves should be stored in each gallon of solution, which should be renewed completely every 4 weeks. It is desirable that the temperature of the solutions be maintained as near 70° F. as practicable. Earthenware jars, covered to reduce evaporation, are satisfactory and convenient containers.

Briquets made of highly resistant cements and stored under the conditions prescribed will show little or no visible action in either solution in less than 16 weeks excepting, perhaps, a slight rounding of the edges. Briquets made of cements very low in resistance, when subjected to this test, will have almost completely disintegrated in 16 weeks. The value of the test as outlined will be greatly increased if briquets made of cements from several mills are included in order to give a basis for directly comparing behavior. If this is done, the failure of any cement falling well below average will be more convincing.

The feasibility of speeding up this 16-week test by increasing the strength of the solution, by keeping the solution at higher temperatures, by using leaner mixes, and in numerous other ways, has been tried without satisfactorily consistent results. A more accelerated test of equal reliability is greatly to be desired, but can not yet be offered.

#### SUMMARY OF RESULTS WITH DIFFERENT PORTLAND CEMENTS

Standard Portland cements from different mills may differ greatly in resistance to the action of sulphate waters. Under identical exposure conditions, concrete and mortar made of resistant cements may

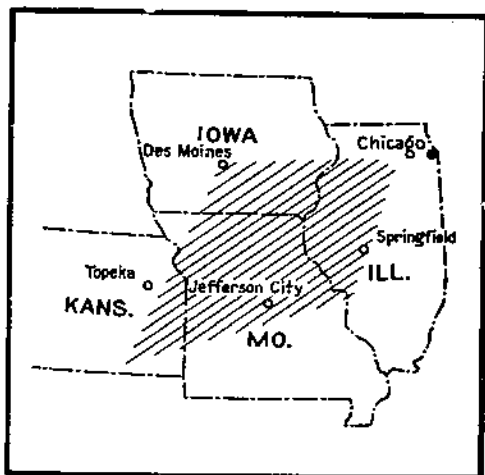


FIGURE 17.—Portland cements from 10 mills in the cross-hatched area were among the 14 most resistant to sulphate attack of 35 cements used in the tests

last 10 times as long as that made of cements of low resistance. The reason for this is not known.

Results of standard physical tests give no indication of the resistance of a cement to sulphates, nor do ordinary chemical analyses. Differences in the raw materials associated with the geological formations from which they come may be factors.

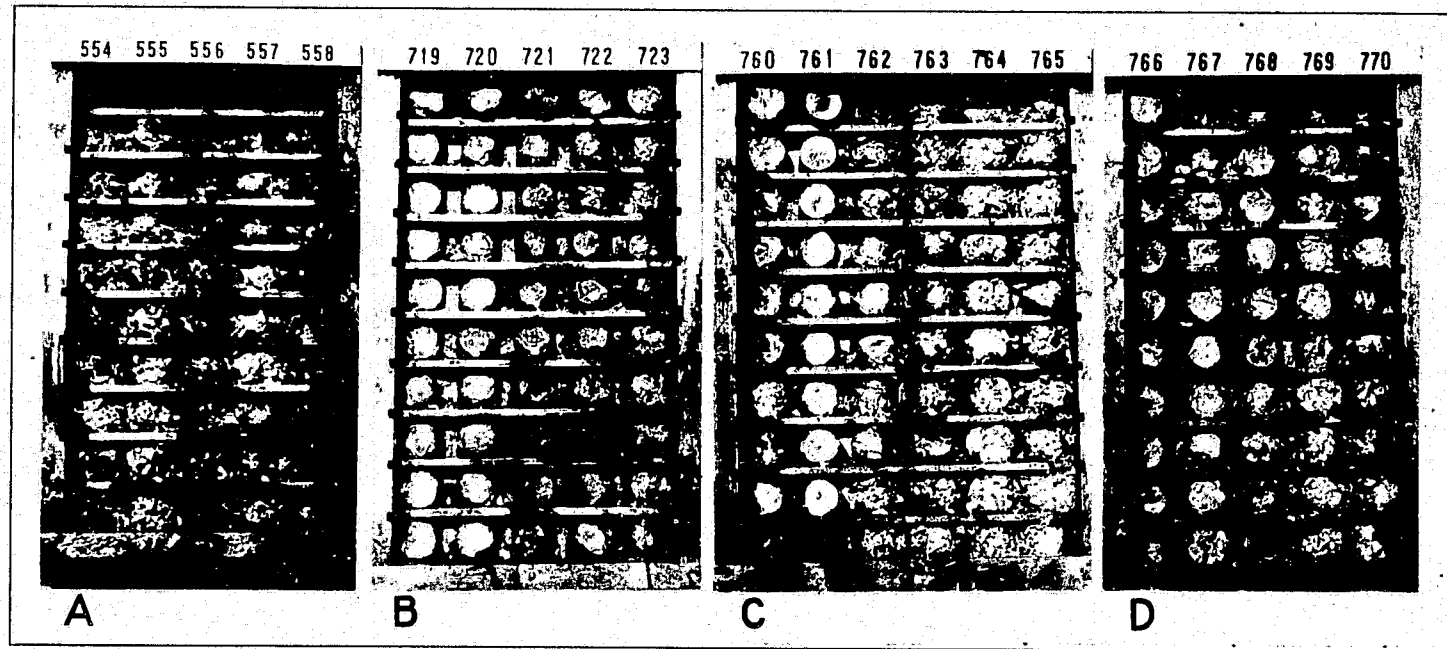
With Portland cements differing so greatly in resistance to sulphate action, certainly the first consideration for all concrete that is to be so subjected should be the cement itself, and, regardless of all other precautions, the use of any cement of low resistance should be avoided. Until a more accelerated test of equal reliability is developed, the 16-week test outlined on page 43 herein is recommended.

### SPECIAL CEMENTS OTHER THAN ALUMINA CEMENTS

The alkali resistance of 14 special cements was investigated. Cylinders of 10 of these cements were stored both in laboratory solutions and in Medicine Lake, while cylinders of the other 4 were tested under either one or the other of these conditions. For comparison, cylinders made of 7 Portland cements were tested with this group. Standard physical tests of the special cements are recorded in Table 19, and chemical analyses of seven of them in Table 20. Table 21 shows the resistance of these special cements and of some companion Portland cements under three exposure conditions. There follows a general description of essential characteristics of these special cements together with a summarized account of behavior, based on the data in Table 21 and in Table 22. Plate 6 shows photographs of several of the Medicine Lake series.

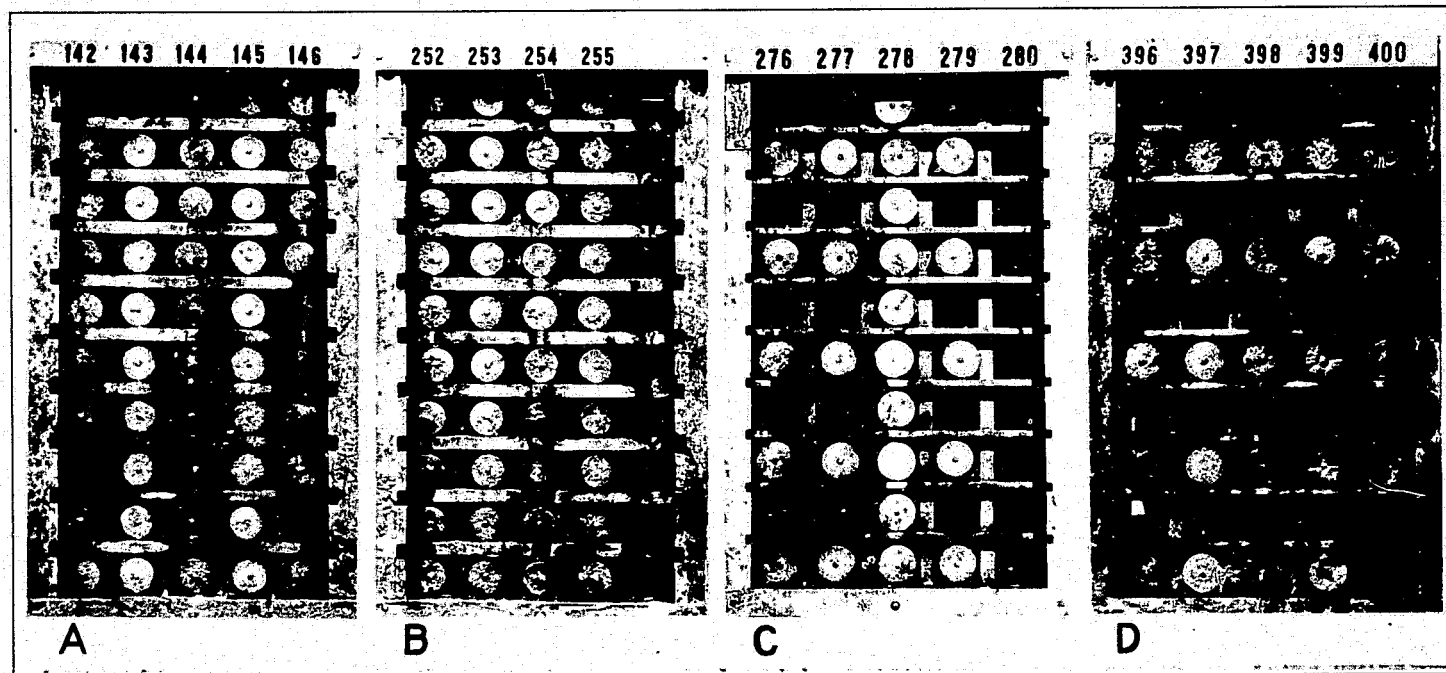
TABLE 19.—Standard physical tests of 14 special cements

Special cements	Time of set								Sound-ness	Fineness		Nor-mal con-sist-ency	Spe-cific grav-ity	Tensile strength of briquets		
	Vicat				Gillmore					Ret-ained by No. 200 sieve	Ret-ained by No. 300 sieve			3 days	7 days	28 days
	Initial		Final		Initial		Final									
	H.	M.	H.	M.	H.	M.	H.	M.		Per cent	Per cent			Lbs. per sq. in.	Lbs. per sq. in.	Lbs. per sq. in.
X	4	0	9	45	5	45	10	30	O. K.	4.0	19.5	38.75	2.81	290	384	577
B	3	0	5	50	3	40	6	15	do.	5.5	14.5	24.75	3.13	225	270	381
H	3	20	4	40	3	10	4	45	do.	3.2	37.4	28.00	-----	281	423	466
I	1	40	3	40	2	35	3	50	do.	.4	1.1	33.00	-----	325	362	371
A2	4	40	18	30	5	30	-----	-----	do.	7.7	29.1	24.25	3.14	122	184	324
A3	5	20	8	35	5	35	9	5	do.	8.0	15.5	23.25	3.18	127	208	330
A1	6	10	6	40	5	50	7	5	do.	12.0	17.9	24.25	3.12	145	206	319
C	3	15	5	45	3	55	6	10	do.	10.2	19.3	24.50	3.16	210	276	388
G	3	40	6	15	4	45	6	30	do.	6.4	14.5	33.00	-----	167	213	305
D	1	0	3	5	1	40	3	35	do.	1.9	28.1	28.25	-----	372	430	465
E	3	10	4	40	3	0	4	30	do.	6.4	51.1	26.00	-----	293	325	366
F	1	25	3	30	2	35	5	10	do.	4.5	39.3	28.75	-----	365	403	446
L	9	-----	17	-----	20	-----	36	-----	do.	11.4	20.2	33.50	-----	99	109	136
K	4	5	7	45	4	15	7	5	do.	7.4	18.8	27.00	3.13	201	263	-----



CYLINDERS MADE OF SPECIAL CEMENTS OTHER THAN HIGH ALUMINA, AFTER STORAGE IN MEDICINE LAKE

A, After five years. Series 554—one-half Portland A, one-half Portland II; Series 555 and 557—Portland V; series 556 and 558—special A2. B, After four years special cement X. Series 719, mix 1: 0.94; series 720 and 721, mix 1: 1.88; series 722, mix 1: 2.82; series 723 mix 1: 4.70. C, After two and one-half years. Series 761, 763 and 765, standard Portland cements from different mills; series 760, 762 and 764 special cements from the same mills, respectively. D, After two and one-half years. Series 767 and 769, standard Portland cements from different mills; series 766 and 768, special cements from the same mills, respectively.



## CYLINDERS MADE OF HIGH ALUMINA CEMENTS AFTER STORAGE IN MEDICINE LAKE

- A, Ottawa sand mortar cylinders made of alumina cement Ac1 after seven years in the lake. Series 142 to 146, respectively, stored in distilled water 27 days, in moist air 72 hours, in water vapor at 100° F. for 48 hours, in water vapor at 155° F. for 48 hours, and in steam at 212° for 48 hours. B, Made of alumina cement Ac2 after six and a half years in the lake. C, Ottawa sand mortar cylinders made of alumina cement Ac3 after six years in the lake. Mixes for series 276 to 280, respectively, were 1:2, 1:3, 1:3, 1:4, and 1:5. D, Made of alumina cement Ac3 after six years in the lake. Water ratios for series 396 to 400, respectively, were 0.44, 0.53, 0.59, 0.73, and 0.88.

TABLE 20.—Chemical analyses <sup>1</sup> of 7 special cements

Special cement	Silica (SiO <sub>2</sub> )	Iron (Fe <sub>2</sub> O <sub>3</sub> )	Alumina (Al <sub>2</sub> O <sub>3</sub> )	Lime (CaO)	Magne- sia (MgO)	Sulphur- ic anhy- dride (SO <sub>3</sub> )	Loss on ignition	Total
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
X.....	35.85	3.60	6.14	48.60	1.01	1.53	3.20	99.99
B.....	21.40	3.93	5.32	52.29	1.86	1.98	2.68	99.86
A2.....	22.37	3.43	5.95	52.51	2.57	1.73	1.36	99.92
C.....	21.16	3.26	11.01	58.46	1.77	1.87	2.47	99.99
D.....	20.50	2.54	5.86	64.35	1.01	2.37	3.40	100.03
L.....	21.60	3.95	11.45	51.20	.53	4.12	6.85	99.70
K.....	21.70	.61	7.01	66.00	0.67	1.46	2.14	99.59

<sup>1</sup> Analyses by the Division of Tests, Bureau of Public Roads, U. S. Department of Agriculture.

TABLE 21.—Resistance to the action of sulphate waters of 14 special cements under different conditions of exposure and of some companion Portland cements

Cement	Stored in 1 per cent solution of Na <sub>2</sub> SO <sub>4</sub>		Strength ratio at 1 year in Medicine Lake	Stored in tap water	
	Life	Strength ratio at 1 year		Age	Length increase
	<i>Weeks</i> ( <i>°</i> )	<i>Per cent</i>	<i>Per cent</i>	<i>Weeks</i>	<i>Inch</i>
Special X.....	.....	106	100	220	0.0016
Special B.....	235.0	95	105	236	.0015
Portland F <sup>1</sup> .....	115.1	80	.....	115	.0007
Special H.....	208.0	84	88	208	.0011
Portland CC.....	150.9	66	80	151	.0006
Special I.....	198.0	99	83	198	.0029
Portland I.....	( <i>°</i> )	93	91	.....	.....
Special A2.....	.....	.....	90	.....	.....
Special A3.....	.....	81	87	198	— .0007
Special A1.....	197.5	.....	75	.....	.....
Portland Y.....	.....	.....	51	.....	.....
Special C.....	163.5	85	93	164	.0021
Portland F <sup>1</sup> .....	115.1	80	.....	115	.0007
Special G.....	156.0	66	74	156	.0012
Portland X.....	137.1	52	69	137	.0010
Special D.....	125.9	97	90	126	.0006
Special E.....	121.0	72	60	122	.0021
Portland AA.....	33.4	0	9	33	.0003
Special F.....	110.3	* 60	58	110	.0006
Portland BB.....	55.7	21	44	56	— .0003
1/2 special L, 1/4 Portland A, and 1/4 Portland B1.....	80.3	53	.....	80	— .0026
Special K.....	25.0	0	.....	.....	.....

<sup>1</sup> Life not yet determined. Strength ratio at 5 years, 94 per cent.<sup>2</sup> Cylinders with Portland cement F were made 25 months before the cylinders with special cements B and C, and direct comparisons of resistance are therefore not satisfactory.<sup>3</sup> Not yet determined.

TABLE 22.—Tests of 2 by 4 inch cylinders made of special cements other than high alumina and exposed to the action of sulphate water in Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water

[Unless otherwise noted the fineness modulus of aggregate is 4.67 and the mix is 1:3. Each test result, with a few exceptions, is an average of 5 cylinders made on different days. Figures in parentheses indicate per cent of normal strength based on parallel tests of cylinders from the same batches, stored in tap water]

Series	Cement laboratory No.	Cement	Water ratio	Curing method				Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in air	Absorption at 24 days	Tank specimens				Lake specimens		
								7 days	28 days	1 year	5 years	1 year	3 years	5 years
				Hrs.	Days	Days	Per cent							
573	130	Special B	0.62	24	20	35	5.3	4,140	4,800	5,610	8,270	5,880 (105)	3,710	610 (7)
714	175	Special D	.64	24	20	35	5.0	5,080	6,040	6,250	—	5,620 (90)	0	0
574	131	Special C	.62	24	20	35	5.0	3,490	4,580	6,100	7,220	5,690 (93)	3,360	1,130 (16)
764	191	Special E	.62	24	20	35	3.8	4,480	5,290	5,990	—	3,570 (60)	0	0
785	197	Portland A.A.	.60	24	20	35	5.8	750	5,430	4,820	—	0	0	0
762	200	Special F	.62	24	20	35	5.0	5,390	6,610	5,690	—	3,830 (58)	0	0
763	190	Portland B.B.	.62	24	20	35	5.9	4,030	5,670	6,360	—	2,830 (44)	0	0
768	193	Special G	.62	24	20	35	5.9	2,910	4,410	5,930	—	4,410 (74)	810	0
769	201	Portland X	.62	24	20	35	6.3	8,060	9,900	6,440	—	4,440 (69)	0	0
467 <sup>1</sup>	112	Special X	.64	24	20	35	—	2,830	4,390	4,830	4,550	4,830 (100)	3,570	2,640 (61)
719	177	do.	.44	24	20	35	—	2,830	4,390	4,830	—	4,940 (87)	1,520	0
720-721	177	do.	.34	24	20	35	8.0	3,000	4,000	5,100	—	4,340 (85)	6,730	0
722	177	do.	.61	24	20	35	6.6	2,690	4,020	4,220	—	3,710 (83)	5,500	0
723	177	do.	.90	24	20	35	8.2	860	1,530	2,230	—	1,700 (76)	210	0
724	177	do.	.42	24	20	35	9.5	4,280	6,900	6,680	—	0,070 (91)	6,220	0
725-726	177	do.	.51	24	20	35	9.4	2,460	4,080	2,400	—	3,660 (93)	3,760	0
727	177	do.	.70	24	20	35	13.3	1,200	2,370	3,050	—	2,150 (70)	1,400	0
728	177	do.	1.08	24	20	35	15.8	320	790	990	—	0	0	0
706	162	Special H	.62	24	20	35	6.0	6,360	6,480	7,570	—	6,520 (86)	5,160	0
787	108	Portland G.C.	.62	24	20	35	6.2	3,440	5,540	6,450	—	5,330 (80)	2,390	0
790	190	Special I	.64	24	20	35	4.7	4,920	5,570	5,780	—	4,810 (83)	2,590	0
761	199	Portland J	.62	24	20	35	6.4	1,600	4,830	7,500	—	6,930 (91)	5,370	0
243-247	28	Special A1	.62	24	20	35	6.2	2,910	3,770	5,370	5,960	4,030 (75)	0	0
248-250	10	Special A3	.63	24	20	35	9.5	1,430	2,730	3,760	3,620	3,280 (87)	800	0
556	127	Special A2	.64	24	20	35	6.0	3,200	4,720	5,050	7,080	5,330 (90)	0	0
555	128	Portland A2	.64	24	20	35	6.0	3,780	4,670	5,480	6,010	3,510 (64)	0	0
558	127	Special A2	.73	24	20	35	6.6	2,280	4,480	6,200	6,070	3,680 (58)	0	0
557	128	Portland A2	.73	24	20	35	6.4	2,760	4,250	4,690	5,550	1,800 (38)	0	0
561	127	Special A2	.62	24	7 20	35	7.0	3,260	4,720	5,860	5,770	6,090 (104)	4,120	0
560	128	Portland A2	.62	24	7 20	35	8.2	3,550	5,160	6,120	6,140	5,270 (86)	4,010	0
563	127	Special A2	.71	24	7 20	35	9.1	2,200	4,520	4,960	6,100	3,960 (80)	1,140	0
562	128	Portland A2	.71	24	7 20	35	9.2	2,720	4,290	5,450	5,810	3,920 (72)	900	0

<sup>1</sup> Mix 1:1.88.<sup>2</sup> Mix 1:0.94.<sup>3</sup> Mix 1:2.82.<sup>4</sup> Mix 1:4.70.<sup>5</sup> Standard Ottawa sand cylinders.<sup>6</sup> Mix 1:2.25 (fineness modulus=3.10).<sup>7</sup> Time in damp sand.

Special cement X is an imported mason's cement containing about 33½ per cent diatomaceous silica mixed with the cement clinker before grinding. Tests with this cement are of unusual interest both because of the siliceous nature of the admixture and because of the method of adding. Therefore the cement was differently proportioned in a number of mixes. The results of the tests are interpreted to indicate that, weight for weight, resistance of this special cement is about equal to that of the more resistant Portland cements although, since one-third of the material added is without cementaceous properties by itself, the concrete under all conditions is somewhat lower in unit strength. No check tests of this cement without the admixture are available and therefore conclusions on the exact effect of the admixture on resistance are impossible. However considered, special cement X made an excellent showing in 1 per cent solutions of sodium sulphate and did reasonably well in Medicine Lake.



Special cement B is reground Portland cement F, and special cement C is the same as B with a carborundum preparation added during regrinding. No test exactly parallel to that made on special cement B was made on Portland cement F, and therefore the specific effect of regrinding is not known. The resistance of special cement B was somewhat greater than that of special cement C, although neither displayed greater resistance than that of the more resistant Portlands.

The five special cements, D, E, F, H, and I have been developed in recent years to meet demands for high-early-strength concrete. The last four of these have been tested parallel with companion Portland cements AA, BB, CC, and I, respectively. No companion Portland cement was available for testing with special cement D, but the resistance displayed by this cement has been, at best, no greater than that of an average Portland cement. As evidenced by the tests, special cements E, F, and H did not display outstanding resistance, although they were somewhat more resistant than their companion Portland cements. Special cement I was slightly less resistant than its highly resistant companion Portland cement I.

Special cements A1, A2, and A3 are standard Portland cements, from different mills, to which tannic acid treated with gypsum was added during grinding. Portland cement Y is the same cement as special cement A2 without the gypsum. Results of tests at one year are slightly more favorable for the treated cement, but after three years no difference between the treated and untreated cement was apparent.

Special cement G is a soap-treated water-repellent product, otherwise the same as Portland cement X with which parallel tests were made. The treated cement displayed resistance differing very little from that of the untreated.

Special cement L is a natural cement somewhat higher in alumina and  $\text{SO}_3$  and lower in lime than normal Portland cement. Its use with 50 per cent Portland cement is recommended. Used in this manner, it produced a concrete of slightly greater resistance than that of concrete made with only Portland cement from the same lot.

Special cement K is a white cement very low in iron and high in magnesia, with other constituents about the same as those of normal Portland cement. This product made a very poor showing in alkali resistance.

With the possible exception of cement X, none of the special cements tested showed increased resistance to sulphate action in a degree warranting preference over the more resistant of the Portland cements.

#### ALUMINA CEMENTS

Alumina cements, according to Bied (11) were discovered by him in 1908 "in seeking a binder which would not be attacked either by sea water or by sulphated waters". In the United States, Spackman (46) in 1910 reported upon the aluminates, their properties and possibilities in cement manufacture, basing his report on experiments begun in 1902 and resulting, in November, 1908, in the making of 1,000 pounds of calcium aluminate which was used experimentally for many purposes for which Portland cement is used (17). Therefore there is some question as to who should have the credit for discovering alumina cements, although there is no question that they

were first manufactured and utilized commercially by the French. Because they possessed the property of early hardening, although setting no more quickly than standard Portland cement, the French Army used alumina cements in foundations for gun platforms and in other emergency construction during the World War. Thereafter the use of these cements extended rapidly to many other types of engineering structures.

Manufacture of alumina cement was begun in the United States early in 1924, and in June of the same year cylinders were made in the draitile laboratory at University Farm. These cylinders were exposed when 8 weeks old to the sulphate water of Medicine Lake, in which cylinders of one of the French alumina cements were already exposed.

#### CHEMICAL COMPOSITION

The raw materials of which alumina cements are made are bauxite and limestone, or bauxite and lime, and these cements are essentially calcium aluminates very low in silica (7, 20, 45), whereas standard Portland cements are essentially calcium silicates low in alumina. The chemical composition of the three alumina cements used in these experiments is shown in Table 23, in which is also shown the average of the chemical analyses of the 35 standard Portland cements of Table 16. It will be noted in Table 23 that the oxides of calcium, silicon, aluminum, and iron average approximately 63, 22, 7, and 3 per cent in the Portland cements as compared with 38, 8, 42, and 11 per cent in the high-alumina cements. As evidenced by these analyses, the three alumina cements are strikingly similar in composition and have consistently behaved similarly in the various tests to which subjected.

TABLE 23.—Chemical analyses of 3 brands of alumina cements compared with average for 35 Portland cements

Radical	Portland cements (average)	Alumina cements			
		Ac1	Ac2	Ac3	Average
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
Silica (SiO <sub>2</sub> ).....	21.76	9.42	8.01	4.71	7.68
Iron (Fe <sub>2</sub> O <sub>3</sub> ).....	2.99	14.02	8.44	10.86	11.14
Alumina (Al <sub>2</sub> O <sub>3</sub> ).....	6.69	40.60	41.59	42.72	41.64
Lime (CaO).....	62.52	35.23	40.92	39.06	38.40
Magnesia (MgO).....	2.20	.50	.35	.66	.50
Sulphuric anhydride (SO <sub>3</sub> ).....	1.66	Trace.		.35	.12
Loss on ignition.....	1.92	.15	.08	1.84	.69
Total.....	99.74	99.92	100.29	100.30	100.17

#### EXPOSURE CONDITIONS FOR ALUMINA CEMENTS

Each of the three alumina cements tested was used in cylinders exposed in Medicine Lake, while the behavior of two of them was also observed in cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate. Supplementing the experiments with cylinders, draitile of alumina cement Ac3 were manufactured at two commercial tile plants. Some of the tile were placed in Medicine Lake and others were buried in alkali soils in southwestern Minnesota and southeastern North Dakota. In addition to check cylinders stored in tap water in the laboratory according to the regular practice check cylinders

of the two alumina cements Ac1 and Ac3 were buried below frost in two soils free of acids and alkalis, as were the check daintile of Ac3 cement. Curing temperatures to which the alumina specimens were subjected were normal except that cylinders of the cement Ac1 were cured also in water vapor at 100°, 155°, and 212° F.

#### UNUSUAL PROPERTIES REVEALED BY LONG-TIME TESTS

It became evident during the work with alumina cements, that these cements had other properties besides early hardening that were

not common to standard Portland cements. For instance, check cylinders stored in tap water frequently expanded nearly as much as cylinders in sulphate solutions in the same room, thereby largely nullifying the value of such tests as an index of sulphate action on alumina cements. In Figures 18 and 19 the length increases of alumina concrete and mortar cylinders stored in tap water are shown in comparison with those of standard Portland cement cylinders stored in the same tank. The compression tests of these alumina-cement cylinders, shown in the same figures, revealed that all these alumina cements lost strength with age as they increased in volume but, contrary to what would be expected, while alumina cement Ac3 increased most in volume it showed the least loss of strength.

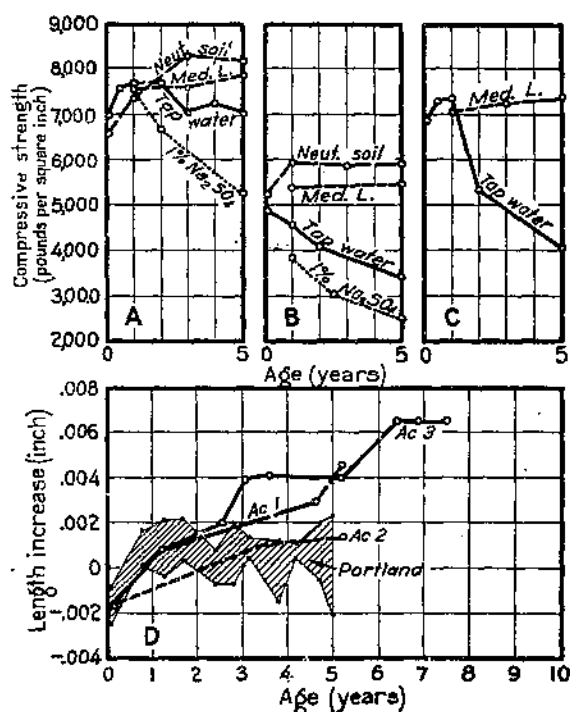


FIGURE 18.—Strength and length changes of standard laboratory cylinders made of alumina cements: A, Cylinders of alumina cement Ac3 stored in neutral soil, in Medicine Lake, in tap water, and in sodium sulphate solution; B, cylinders of alumina cement Ac1 stored in neutral soil, in Medicine Lake, in tap water, and in sodium sulphate solution; C, cylinders of alumina cement Ac2 stored in Medicine Lake and in tap water; D, length changes in cylinders of alumina cements stored in tap water compared with range of length changes of Portland cement cylinders made in 98 series from 35 brands. Each point for the alumina cements is the average for 5 to 35 cylinders.

Other graphs of Figures 18 and 19 show compression tests of cylinders made with two of the alumina cements buried below frost in two soils with neutral reactions for periods up to five years. None of these cylinders lost strength with age; there was instead a very definite tendency toward increase in strength. Difference in behavior of the laboratory cylinders stored in tap water and of the cylinders buried in damp soils out-of-doors could be attributed either to dissimilar tem-

peratures or to the only obvious alternative, some deleterious effects produced by water curing but not by moist soil. The latter hypothesis, however, is scarcely tenable because, as shown in Figures 18 and 19, the alumina-cement cylinders in the highly mineralized but relatively cool water of Medicine Lake invariably had 5-year strengths exceeding those of the cylinders in the comparatively warm laboratory solutions in which the salt content was much lower.

Cylinders of alumina cement Ac1 were subjected to air hardening in the laboratory for periods of 0, 2, 4, 8, and 49 weeks without appreciably different effects on compressive strength at any period up to five

years, as shown in Figure 20. The effect of air hardening on volume change was likewise inappreciable, except for the 49-week period which markedly reduced expansion.

With very evidently negative reactions to mildly high room temperatures for three alumina cements, it is interesting to note the effect produced by curing cylinders of cement Ac1 in water vapor at temperatures of 100°, 155°, and 212° F., as shown by Figure 20. These data reveal that in the cylinders stored in tap water the greatest increases of volume followed curing at 100° and very slight to negative increases followed curing at 155° and 212°. The cylinders cured at 155° behaved essentially the

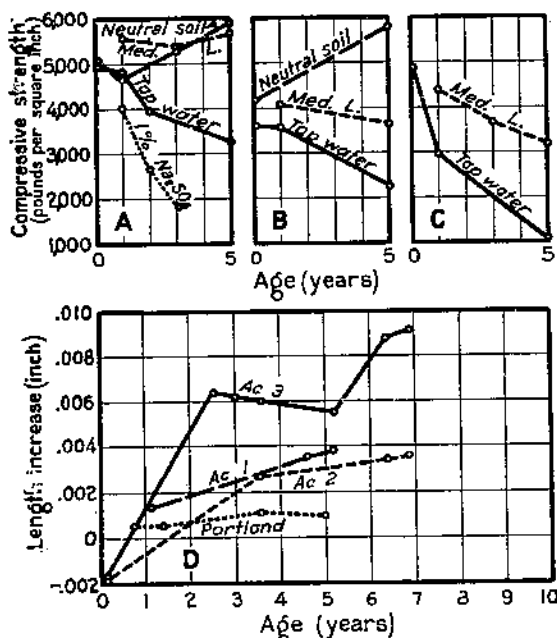


FIGURE 19.—Strength and length changes of mortar cylinders made of alumina cements and standard Ottawa sand: A, Cylinders of alumina cement Ac3 stored in neutral soil, in Medicine Lake, in tap water, and in sodium sulphate solution; B, cylinders of alumina cement Ac1 stored in neutral soil, in Medicine Lake, and in tap water; C, cylinders of alumina cement Ac2 stored in Medicine Lake and in tap water; D, cylinders of alumina cements compared with those of a brand of standard Portland cement. Each point is the average for 5 to 10 cylinders made on 5 different days

same as those of many Portland cements cured at ordinary room temperatures. High-temperature curing reduced compressive strengths at all periods tested up to 5 years, excepting only 100° curing at 1 year and less, although the cylinders cured at 155° and 212° most nearly maintained their 28-day strengths for 5 years.

With cylinders of the alumina cement Ac3, varying the water-cement ratio and the quantity of cement in the mix gave entirely consistent results as illustrated in Figure 21.

#### SULPHATE RESISTANCE OF ALUMINA CEMENTS

Under the conditions imposed by the field tests, the degree of resistance displayed by the three alumina cements approached the ideal, whereas the results obtained in the laboratory were considerably less satisfactory. Conclusions as summarized are:

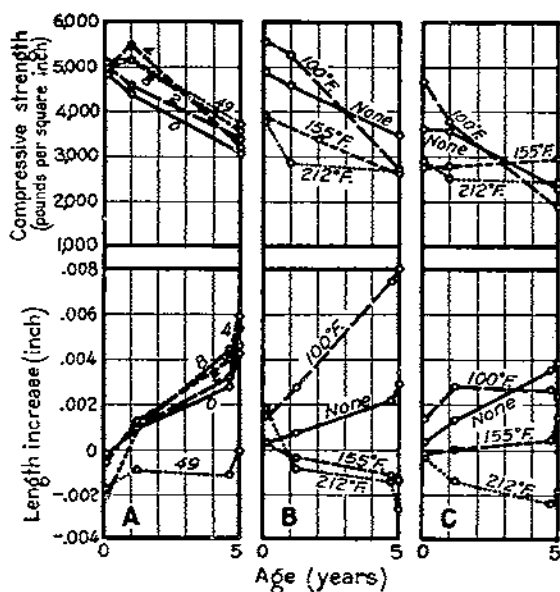


FIGURE 20.—Compressive strengths and length changes of cylinders of alumina cement Ac1 stored in tap water, as influenced by varying the curing conditions: A, Concrete cylinders hardened in air for the number of weeks indicated, after 20 days curing in water; B, concrete cylinders cured 48 hours in water vapor at temperatures indicated; C, mortar cylinders cured 48 hours in water vapor at temperatures indicated. Each point is the average for five cylinders made on different days

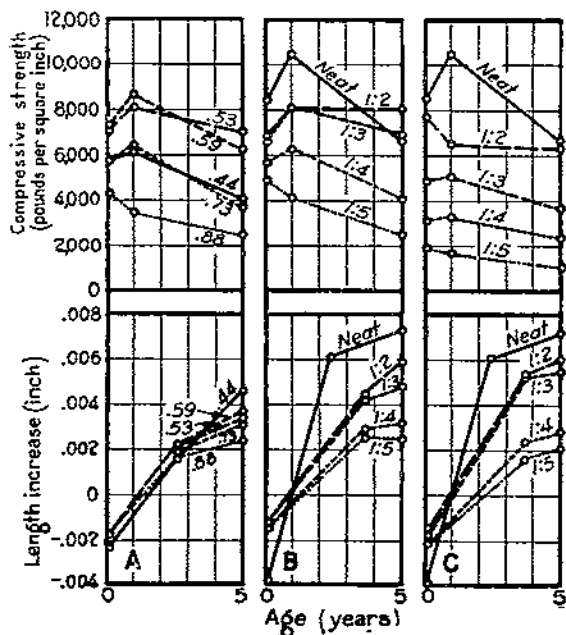


FIGURE 21.—Compressive strengths and length changes of cylinders of alumina cement Ac3 stored in tap water: A, Concrete cylinders with water-cement ratios as shown; B, neat cement cylinders and concrete cylinders of mixes as shown; C, neat cement cylinders and mortar cylinders of mixes as shown. Each point is the average for five cylinders made on different days

Medicine Lake water was resisted remarkably well by the three alumina cements. This is clearly shown by the compression tests up to five years and by appearances up to seven and one-half years. (Table 24 and pl. 7.) Resistance was so pronounced that for 34 of the 53 series of alumina cement cylinders crushing strengths were higher after 5 years in the lake than they were at 1 year, while the loss of strength was less than 9 per cent in 10 of the remaining 19 series. Of the other 9, the cylinders of series 142, 147, and 156, without air hardening previous to exposure, showed strength losses of 10, 15, and 12 per cent, respectively, during the 4 years; those of 1:5 concrete of series 291 had lost 22 per cent; and those of series 254-255, of standard Ottawa sand in 1:3 mortar, had lost 27 per cent of their strength. Cylinders of series 280 and 292, of standard Ottawa sand in 1:5 mix, with high water-cement ratios, failed the first year. The wet-mixed 1:3 concrete cylinders of series 400 were weaker at 3 and 5 years than at 1 year and were showing evidence of considerable deterioration, but tested only 16 per cent weaker at 5 years than at 1 year.

TABLE 24.—Tests of 2 by 4 inch cylinders made of high alumina cements cured in water and water vapor and exposed to the action of sulphate waters of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water

Unless otherwise noted the fineness modulus of aggregate is 4.67 and the mix is 1:3. Each test result, with a few exceptions, is an average of 5 cylinders made on different days. Figures in parentheses, in compression-test columns, indicate per cent of normal strength based on parallel tests of cylinders from the same batches, stored in tap water in the laboratory]

Series	Cement Laboratory No.	Cement	Water ratio	Curing method					Ab-sorp-tion at 21 days	Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in water vapor	Tem-perature of water vapor	Time in air		Tank specimens				Lake specimens		
										7 days	28 days	1 year	5 years	1 year	3 years	5 years
137	14	Alumina Acl.....	0.04	Hours 24	Days	Hours 48	°F. 155	Days 25	Percent 6.2	3,250	3,550	2,830	2,850	3,400 (120)	4,510	4,230 (151)
138	14	do.....	.04	24	{	24	155	25	6.2	3,150	3,670	2,860	2,510	3,320 (116)		4,960 (198)
139	14	do.....	.04	24		24	212	25	6.1	3,160	3,840	2,640	2,400	3,790 (144)		4,560 (185)
140	14	do.....	.04	24		48	155	24	6.1	3,190	3,890	2,730	2,570	3,790 (139)		5,040 (196)
141	14	do.....	.04	24	{	24	155	24	6.1	3,360	3,800	3,020	2,700	3,670 (121)		4,920 (182)
147	14	do.....	.02	24		27			6.3	4,560	4,890	4,560	3,460	5,220 (114)	1 6,410	4,460 (129)
148	14	do.....	.02	72				25	7.0	5,020	4,810	5,310	3,710	5,960 (112)	1 6,300	6,230 (168)
149	14	do.....	.02	24		48	100	25	6.5	5,370	5,510	5,240	2,660	5,450 (104)	1 5,300	5,330 (201)
150	14	do.....	.02	24		48	155	25	6.2	3,360	3,870		2,640	3,400	1 4,060	3,690 (140)
151	14	do.....	.02	24		48	212	25	6.1	3,300	3,750	2,840	2,660	3,650 (129)	1 4,110	4,090 (154)
152	14	do.....	.02	24	20			344	6.3	4,570	5,040	5,100	3,780	5,040 (99)		6,240 (165)
153	14	do.....	.02	24	20			50	6.5	4,830	4,910	5,120	3,690	6,280 (123)		6,720 (182)
154	14	do.....	.02	24	20			28	6.5	4,760	4,850	5,450	3,380	5,620 (103)		6,460 (191)
155	14	do.....	.02	24	20			14	6.5	4,250	4,960	4,570	3,590	5,770 (126)		6,420 (179)
156	14	do.....	.02	24	20				6.3	4,710	4,860	4,350	3,160	4,960 (113)		4,390 (130)
132	14	do.....	.05	24		48	155	25	8.1	2,250	2,910	2,760	2,830	2,790 (101)	1 2,930	2,940 (104)
133	14	do.....	.05	24	{	24	155	25	8.0	2,280	2,680	2,080	2,690	2,900 (139)	1 3,140	2,840 (106)
134	14	do.....	.05	24		24	212	25	8.0	2,470	2,850	2,440	2,640	2,840 (116)	1 3,120	3,090 (117)
135	14	do.....	.05	24		48	155	24	7.8	2,620	2,960	2,650	2,910	2,590 (98)	1 2,930	3,340 (115)
136	14	do.....	.05	24	{	24	155	24	7.8	2,550	2,930	2,630	2,650	2,580 (98)	1 3,170	3,030 (114)
142	14	do.....	.03	24		27			8.7	2,910	3,600	3,590	2,240	4,060 (113)		3,640 (162)
143	14	do.....	.03	72				25	9.3	4,220	4,430	4,220	2,160	4,310 (102)		5,880 (272)
144	14	do.....	.03	24		48	100	25	9.1	4,320	4,040	3,750	1,880	4,490 (120)		4,120 (219)

1 2-year tests.

TABLE 24.—Tests of 2 by 4 inch cylinders made of high alumina cements cured in water and water vapor and exposed to the action of sulphate waters of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water—Continued

Series	Cement laboratory No.	Cement	Water ratio	Curing method					Ab-sorption at 21 days	Average compression tests (pounds per square inch)						
				Time in moist closet	Time in water	Time in water vapor	Tem-perature of water vapor	Time in air		Tank specimens				Lake specimens		
										7 days	28 days	1 year	5 years	1 year	3 years	5 years
				Hours	Days	Hours	°F.	Days	Percent							
145	14	Alumina Ac1.....	0.63	24	-----	48	155	25	8.1	2,200	2,700	2,780	2,030	2,620 (94)	-----	3,700 (128)
146	14	do.....	.63	24	-----	48	212	25	7.8	2,500	2,870	2,500	2,400	3,010 (120)	-----	3,960 (165)
208-212	25	Alumina Ac3.....	.51	24	20	-----	-----	35	4.9	6,700	6,780	6,200	7,770	7,420 (118)	7,240	8,050 (115)
276	27	do.....	.44	24	20	-----	-----	35	6.3	6,210	7,080	6,490	6,390	8,000 (123)	7,370	7,970 (125)
277-278	27	do.....	.50	24	20	-----	-----	35	8.3	5,050	4,830	5,110	3,640	5,560 (100)	5,350	5,690 (150)
279	27	do.....	.73	24	20	-----	-----	35	10.2	3,380	3,090	3,250	2,380	3,730 (115)	3,350	3,830 (101)
280	27	do.....	.90	24	20	-----	-----	35	11.8	2,160	1,020	1,080	1,130	700 (42)	0	0
286	27	do.....	.44	24	20	-----	-----	35	5.1	7,490	6,820	8,080	8,100	8,400 (105)	8,080	8,210 (101)
287-288	27	do.....	.53	24	20	-----	-----	35	4.9	7,080	6,620	8,160	6,880	8,210 (101)	6,710	7,830 (114)
289	27	do.....	.67	24	20	-----	-----	35	5.3	6,560	5,080	6,290	4,000	6,100 (98)	5,400	6,310 (154)
290	27	do.....	.81	24	20	-----	-----	35	5.8	4,640	4,840	4,150	2,520	5,080 (122)	4,080	4,770 (189)
396	70, 71	do.....	.44	24	20	-----	-----	35	5.6	5,300	5,760	6,110	4,040	5,780 (95)	6,040	5,920 (147)
397	70, 71	do.....	.53	24	20	-----	-----	35	4.9	6,660	7,120	8,100	7,040	7,680 (94)	8,000	7,710 (110)
398	70, 71	do.....	.59	24	20	-----	-----	35	5.3	6,890	7,210	8,030	6,280	7,390 (86)	8,300	6,780 (108)
399	70, 71	do.....	.73	24	20	-----	-----	35	6.3	5,950	5,710	6,440	3,700	5,400 (84)	6,690	5,790 (150)
400	70, 71	do.....	.88	24	20	-----	-----	35	7.6	5,140	4,230	3,480	2,550	3,790 (109)	2,950	3,200 (125)
429	70, 71	do.....	.35	24	20	-----	-----	35	9.7	6,400	8,420	10,440	6,620	6,330 (89)	10,160	8,510 (120)
435	70, 71, 74	Alumina Ac3, 5 per cent.....	.60	24	20	-----	-----	35	6.3	2,630	3,880	5,490	5,970	4,100 (75)	0	0
		Portland A, 47.5 per cent.....														
		Portland B1, 47.5 per cent.....														
436	70, 71, 74	Alumina Ac3, 10 per cent.....	.60	24	20	-----	-----	35	6.2	2,260	3,760	4,820	5,420	2,720 (50)	0	0
		Portland A, 45 per cent.....														
		Portland B1, 45 per cent.....														
437	70, 71, 74	Alumina Ac3, 20 per cent.....	.60	24	20	-----	-----	35	7.0	1,360	2,830	3,640	3,000	3,320 (91)	0	0
		Portland A, 40 per cent.....														
		Portland B1, 40 per cent.....														
438	70, 71	Alumina Ac3.....	.60	24	20	-----	-----	35	5.8	7,080	7,700	7,820	6,440	7,220 (92)	8,130	7,810 (121)
252-253	58	Alumina Ac2.....	.53	24	20	-----	-----	35	5.4	7,730	7,230	7,480	6,030	7,010 (94)	7,230	7,340 (182)
291	58	do.....	.70	24	20	-----	-----	35	6.5	4,360	4,830	2,910	2,190	4,370 (150)	3,330	3,410 (150)
254-255	58	do.....	.60	24	20	-----	-----	35	9.3	5,010	4,850	2,920	2,020	4,340 (149)	3,690	3,170 (157)
292	58	do.....	.90	24	20	-----	-----	35	12.3	2,130	1,520	1,130	1,230	920 (81)	0	0

1 Standard Ottawa sand cylinders.

2 Mix 1:2.

4 Mix 1:4.

5 Mix 1:5.



Solutions of 1 per cent sodium sulphate, in which cylinders of Ac1 and Ac3 cements were stored in the laboratory, afforded a test of alumina cement much more severe than did Medicine Lake water which contained several times as much sulphate. (Table 3.) It becomes evident, however, by comparing the strength curves of cylinders stored in solutions of sodium sulphate with the curves of those in tap water (figs. 18 and 19), that only about one-half the loss in strength of the cylinders in the solutions was directly attributable to sulphate action, the remaining loss being due to temperature conditions of the laboratory as discussed on page 50. It appears, therefore, that alumina-cement cylinders normally weaken in tap water in the laboratory and that this weakening is considerably accelerated when sulphate action is introduced.

Drain tile of 5 and 6 inch diameters made of Ac3 cement at two commercial plants were tested for resistance under field-exposure conditions in comparison with Portland-cement tile that were essentially similar except that they were mixed 1:3 whereas the alumina-cement tile were mixed 1:4. General conditions of exposure and strength tests of the tile up to five years are recorded in Table 25 and test trends are shown in Figure 22. Both tabulated data and graphs clearly indicate a much higher degree of resistance for the tile of alumina cement than for those of Portland cement, fully supporting the results obtained in field tests of cylinders.

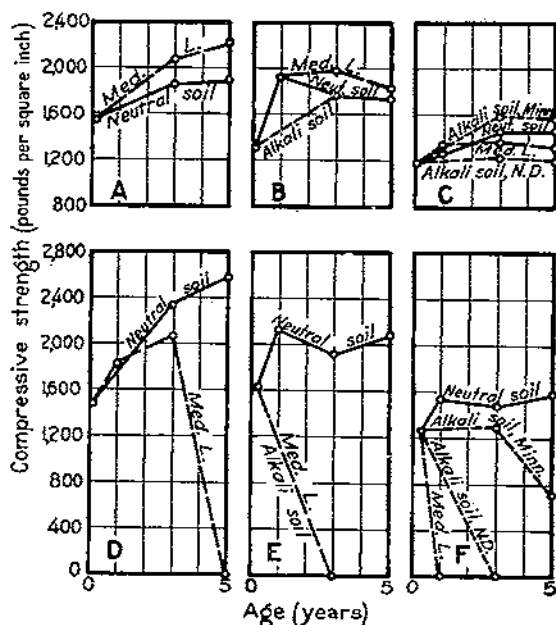


FIGURE 22.—Breaking tests up to five years of 5 and 6 inch concrete drain tile made of alumina and Portland cements, exposed in Medicine Lake, S. Dak., in a neutral soil, and in alkali soils: A and B, Tile of alumina cement Ac3 and Wisconsin aggregate; C, tile of alumina cement Ac3 and Minnesota aggregate; D and E, Portland cement M and Wisconsin aggregate; F, Portland cement B and Minnesota aggregate. Each point is the average for 5 to 10 tile.

TABLE 25.—Tests up to 5 years of drain tile made of alumina and Portland cements and subjected to various conditions of exposure

(The neutral Minnesota and Wisconsin soils were very low in soluble salts, containing, respectively, but 0.04 and 0.05 per cent)

## WISCONSIN AGGREGATE

Location and exposure conditions				Mix	Tile tested	Strength and absorption tests							
Installation	Salt content		1 to 4 months			1 year		3 years		5 years			
	Lake and soil waters	Reacting values SO <sub>4</sub>	Strength			Absorption	Strength	Absorption	Strength	Absorption	Strength	Absorption	
	Parts per million	Per cent		Number	Pounds per lin. ft.	Per cent	Pounds per lin. ft.	Per cent	Pounds per lin. ft.	Per cent	Pounds per lin. ft.	Per cent	
Alumina cement Ac3: <sup>1</sup>			1:4	8	1,540	9.3							
Stock pile (2 months).....			1:4	5	1,560	8.5							
Stock pile (3 months).....			1:4	5					1,850	7.9	1,890	8.3	
Neutral soil, Wisconsin.....			1:4	5					2,080	7.0	2,220	7.0	
Medicine Lake, S. Dak.....	47,901	48.08	1:4	5									
Portland cement M: <sup>2</sup>			1:3	8	1,210	0.2							
Stock pile (1 month).....			1:3	5	1,480	0.1							
Stock pile (2 months).....			1:3	5					2,350	7.4	2,570	7.7	
Neutral soil, Wisconsin.....			1:3	5			1,830	7.4	2,070	6.8	2,270	7.0	
Medicine Lake, S. Dak.....	47,901	48.08	1:3	5									
Alumina cement Ac3: <sup>3</sup>			1:4	10	1,310	0.6							
Stock pile (1 month).....			1:4	5			1,020	9.1	1,780	9.1	1,740	9.0	
Neutral soil, Minnesota.....			1:4	5					1,760	8.4	1,750	8.1	
Alkali soil, North Dakota.....	86,386	45.70	1:4	5			1,030	8.6	1,980	7.5	1,830	8.0	
Medicine Lake, S. Dak.....	47,901	48.08	1:4	5									
Portland cement M: <sup>4</sup>			1:3	10	1,620	0.4							
Stock pile (3 months).....			1:3	5			2,120	0.4	1,920	7.9	2,080	8.1	
Neutral soil, Minnesota.....			1:3	5					0		0		
Alkali soil, North Dakota.....	86,386	45.70	1:3	5			1,130	9.5	0		0		
Medicine Lake, S. Dak.....	47,901	48.08	1:3	5									

## MINNESOTA AGGREGATE

Alumina cement Ac3: 1			1:4	10	1,180	10.4						
Stock pile (1 month).....			1:4	5			1,270	9.4	1,460	9.1	1,470	9.1
Neutral soil, Minnesota.....			1:4	5					1,580	9.0	1,570	9.6
Alkali soil, Minnesota.....	21,630	48.36	1:4	5					1,230	8.2	1,200	8.8
Alkali soil, North Dakota.....	86,386	45.70	1:4	5			1,350	9.1	1,360	8.4	1,320	9.2
Medicine Lake, S. Dak.....	47,901	48.08	1:4	5								

## Portland cement B1: \*

Stock pile (4 months).....			1:3	10	1, 260	10.1							
Neutral soil, Minnesota.....			1:3	5									
Alkali soil, Minnesota.....	21, 630	48.36	1:3	5			1, 540	9.0	1, 480	8.6	1, 550	8.9	
Alkali soil, North Dakota.....	86, 380	45.70	1:3	5					1, 290	8.0	710	9.5	
Medicine Lake, S. Dak.....	47, 901	48.08	1:3	5			0		0		0		

- 1 Tile (6-inch) cured 24 hours moist room, 72 hours water vapor at 120° F., 8 weeks air.  
 2 Tile (5-inch) cured 24 hours moist room, 48 hours water vapor at 120° F., 4 weeks air.  
 3 Tile (5-inch) cured 24 hours moist room, sprinkled 24 hours, 3 weeks air.  
 4 Tile (5-inch) cured 24 hours moist room, 48 hours water vapor at 120° F., 4 weeks air.  
 5 Tile (6-inch) cured 24 hours moist room, sprinkled 2 weeks, 8 weeks air.  
 6 Tile (5-inch) sprinkled 24 hours in moist room, 2 weeks air.

## MIXTURES OF ALUMINA AND PORTLAND CEMENTS

Alumina and Portland cements were mixed in various proportions to indicate the possibility of developing combinations relatively high in resistance and lower in cost than alumina cement. Results of different phases of this work are recorded in Tables 24, 26, 27, and 14. The data for series 434 in Table 14 and series 435 to 438 in Table 24 give little encouragement to this endeavor. All the cylinders containing 5, 10, and 20 per cent alumina cement and, respectively, 95, 90, and 80 per cent Portland cement, had lower strength ratios after one year in Medicine Lake than did those in which only Portland or alumina cement was used. At three years all cylinders in these series except those of 100 per cent alumina cement had failed completely.

TABLE 26.—*Effect of combining alumina cement Ac3 and Portland cement on compressive strength of concrete cylinders, normally cured and stored in tap water*

[Each test is the average of 4 or 5 cylinders made on 2 and 5 days]

Series No.	Cement		Water ratio	Absorption at 21 days	Average of compression tests					
	Alumina	Portland			1 day	3 days	7 days	28 days	1 year	5 years
	Per cent	Per cent		Per cent	Lbs. per sq. in.	Lbs. per sq. in.	Lbs. per sq. in.	Lbs. per sq. in.	Lbs. per sq. in.	Lbs. per sq. in.
381	0	100	0.84		770	1,780	2,540	3,710	5,400	
382	5	95	.64		640	1,570	2,360	3,640	4,720	
383	10	90	.64		420	1,460	2,270	3,610	5,540	
384	15	85	.65		390	520	1,530	3,100	5,600	
385	20	80	.66		590	650	1,220	2,780	4,070	
386	25	75	.65		440	440	620	1,240	1,750	
387	35	65	.81		1,370	1,150	1,400	2,090	2,350	
388	50	50	.75		2,300	1,790	1,410	1,760	2,720	
389	75	25	.60		4,510	4,870	4,120	3,680	3,970	
390	100	0	.59		6,380	6,420	7,150	7,190	7,620	
434	0	100	.66	6.4			2,590	4,080	5,050	5,830
435	5	95	.66	6.3			2,630	3,880	5,490	5,970
436	10	90	.66	6.2			2,260	3,700	4,820	5,420
437	20	80	.66	7.0			1,360	2,830	3,640	3,060
438	100	0	.66	5.8			7,080	7,790	7,820	6,440

TABLE 27.—*Effect of length changes of combining alumina cement Ac3 and Portland cement in concrete cylinders normally cured and stored in tap water*

[Each test is the average for 5 cylinders made on 5 days]

Series	Cement		Length increase at age <sup>1</sup>			
	Portland	Alumina	6 weeks	8 weeks	120 weeks	200 weeks
	Per cent	Per cent	Inch	Inch	Inch	Inch
434	100	0	-0.0017	-0.0020	-0.0007	+0.0008
435	95	5	-.0018	-.0021	-.0003	+.0008
436	90	10	-.0020	-.0023	-.0003	-.0002
437	80	20	-.0026	-.0029	-.0006	+.0002
438	0	100	-.0016	-.0019	+.0018	+.0028

<sup>1</sup> Initial readings made at end of three weeks.

## CONCLUSION REGARDING ALUMINA-CEMENT CONCRETE AND MORTAR

Under conditions of these tests, each of three alumina cements resisted sulphate action to a high degree but was unstable when used in concrete and mortar stored for long periods in tap water at room temperatures.

## ADMIXTURES

Mixing special ingredients with Portland-cement concrete to increase resistance to sulphate attack, although long advocated and practiced, has been from the first a matter of extended controversy. That early opinions regarding admixtures were divergent is not difficult to comprehend, for there is a great range in the resistance of Portland cements from different mills as well as decided variations in the resistance of concrete due solely to variations of curing conditions, as has been discussed. Therefore, these factors had to be carefully weighed in comparing the behavior of concretes with and without admixtures, otherwise the results might be so contradictory as to be valueless or even misleading. Random comparisons based on examinations of field structures are likely to be deceiving, since exposure conditions are not identical for any two structures and may vary greatly even for different parts of the same structure. With the hope of somewhat reducing the doubts about the effect on sulphate resistance of some of the more common types of admixtures, the series of experiments here reported upon were outlined, although it was manifestly impracticable to test more than a very few of the large number of products that have been suggested.

In these experiments with admixtures 26 products were used, including 9 siliceous materials, natural and artificial; 2 high-iron products; 3 miscellaneous chemical compounds and mixtures; and 7 water-repellents, including 3 soap preparations, an organic oil, a mineral oil, kerosene, and water-gas tar. The essential chemical composition of each admixture is recorded in Table 28, and the results of fineness tests of the siliceous materials are given in Table 29. There follows a brief description of the noteworthy characteristics of the materials of each group and a summary of the observed effects on compressive strength and particularly on alkali resistance of concrete. (Tables 30 and 31, and pls. 8 and 9.) Numerous workers have reported on these and similar products used in concrete for other specific purposes (3, 4, 26, 29, 52).

TABLE 28.—*Essential chemical composition of admixtures*

(Analyses by the Division of Tests, Bureau of Public Roads, U. S. Department of Agriculture, except as noted. All values are given in per cent)

Admixture	Silica (SiO <sub>2</sub> )	Alumina (Al <sub>2</sub> O <sub>3</sub> ) and iron (Fe <sub>2</sub> O <sub>3</sub> )	Metallic iron	Lime (CaO)	Magnesia (MgO)	Carbon (C)	Carbon dioxide (CO <sub>2</sub> )	Sulphur (S)	Sulphuric anhydride (SO <sub>3</sub> )	Sodium oxide (Na <sub>2</sub> O)	Calcium chloride (CaCl <sub>2</sub> )	Barium chloride (BaCl <sub>2</sub> )	Soaps	Loss on ignition
<b>Siliceous materials:</b>														
Barnsdall admix.	96.80	1.70												1.50
Blast-furnace slag	33.37	16.50		45.50	2.54			1.80						.27
Celite	86.40	6.90												6.70
Colloxy	65.70	10.00		2.20	1.30									9.66
Fuel ash	44.50	34.00		2.80	1.14	12.70		.18	0.08					4.55
Haydite	64.35	31.70												
Omicron <sup>1</sup>	64.25	18.30												13.30
Trass	55.60	30.10		1.65	.90			8.00						
Volcanic ash	72.45	13.55		.70	(?)					6.02				6.10
<b>High-iron products:</b>														
Ironite <sup>2</sup>	5.25		83.33											
Metallicron <sup>3</sup>	10.00		90.00											
<b>Miscellaneous chemical compounds and mixtures:</b>														
Barium chloride (certified product) <sup>4</sup>												99.5		
Cal	1.58	.72		46.92	1.21				(?)		21.2			27.44
Calcium chloride (certified products) <sup>4</sup>											99.6			
Caseln <sup>5</sup>														
Earthcrete <sup>6</sup>		.75		(?)	(?)			8.55	3.45					
Hydrated lime (high CaO) <sup>7</sup>														
Hydrated lime (high MgO) <sup>7</sup>	1.49	.80		44.27	28.20		23.95		1.59					.57
Sulphur (certified product) <sup>8</sup>								100.00						
<b>Water repellents:</b>														
Alkagel <sup>9</sup>														
Medusa waterproofing <sup>11</sup>													23.0	
Truscon waterproofing paste <sup>12</sup>													21.5	
Linseed oil <sup>9</sup>														
Automobile oil <sup>9</sup>														
Kerosene <sup>9</sup>														
Water-gas tar <sup>13</sup>														

<sup>1</sup> Also small amounts of lime, magnesia, and alkali salts.<sup>2</sup> Trace.<sup>3</sup> Iron and iron oxide equivalent to 83.33 per cent metallic iron; manganese and manganese oxide equal to 0.56 per cent metallic manganese.<sup>4</sup> About 90 per cent metallic iron; balance siliceous material resembling clay.<sup>5</sup> Analysis by manufacturer.<sup>6</sup> None.<sup>7</sup> Calcium caseinate (milk product).<sup>8</sup> Unaccounted balance consisted of a mixture of sodium chloride and potassium nitrate.<sup>9</sup> Not analyzed.<sup>10</sup> Lost 80.87 per cent on drying; residue consisted of copper and iron soaps with paraffin; ammonia present.<sup>11</sup> Soap about 23 per cent; balance was partly hydrated and carbonated lime with about 6 per cent magnesia.<sup>12</sup> Water about 78 per cent; considerable free ammonia.<sup>13</sup> Thin liquid with 70.09 per cent soft-pitch residue at 300° C.

TABLE 29.—Fineness tests of the nine siliceous admixtures for which chemical analyses are shown in Table 28

[Analyses other than those of the 200 and 300 sieves were made by the Bureau of Standards, U. S. Department of Commerce.]<sup>1</sup>

Admixture	Retained by No. 200 sieve	Retained by No. 300 sieve	Greater than 60 microns	Greater than 40 microns	Greater than 20 microns	Greater than 10 microns
	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
Barnsdall admix.	0.2	0.5		(?)	38.2 (21)	86.0
Blast furnace slag	12.7	19.4	32.8	58.0	76.9	84.0
Celite	1.9	2.9				
Colloxy	10.5	18.1	26.8 (59)	40.6 (41)	71.7 (21)	83.8 (10)
Fuel ash	3.4	5.4	(?)	14.1	30.2	55.2
Haydite	3.1	6.0	11.8	27.5 (38)	52.3 (21)	83.0
Omicron	2.8	4.4		(?)	33.5 (22)	85.4
Trass	28.3	32.8	33.0	45.0 (40)	69.7	79.2
Volcanic ash	0.4	11.3				

<sup>1</sup> Note by the Bureau of Standards: Fractionations were made in an air elutriator calibrated for Portland cement which has a density of about 3.1. As the density of the admixtures differs from this value the limiting sizes of each fraction will also differ from the nominal sizes listed above, which are for Portland cement. Microscopic measurements of the limiting sizes of separation were made in some cases. The values obtained have been placed in parentheses after the per cent residue in the table. These values are averages of measurements on about 40 particles. No measurements were made on the samples of blast furnace slag and powdered fuel ash because the particles were very irregular in size and shape. When the residue was less than 10 per cent the results obtained were not reliable because the material remaining in the bulb was not sufficient for proper agitation.

<sup>2</sup> Less than 10 per cent.

TABLE 30.—Effect of admixtures on resistance of 2 by 4 inch concrete cylinders exposed in the laboratory to the action of solutions of sodium sulphate ( $\text{Na}_2\text{SO}_4$ )

[Cements A and B1 mixed in equal portions. Each test result is the average for 5 or more cylinders made on 5 days]

Series	General description	Fineness moduli	Water-cement ratios	Absorption at 24 days	Stored in tap water		Stored in 1 per cent solutions of $\text{Na}_2\text{SO}_4$			
					Age tested	Breaking strengths	Age tested	Breaking strengths	Per cent of normal strength	Time required to increase in length 0.010 inch
				Per cent	Weeks	Lbs. per sq. in.	Weeks	Lbs. per sq. in.	Per cent	Weeks
51	{ Check cylinders without admixture.	4.67	0.59	5.1	1	3,200	46	1,800	34	43.4
					4	4,760				
					46	5,400 (100)				
52	{ 1½ per cent Alkagal.	4.67	.60	5.7	1	2,800	34	1,600	28	31.0
					4	4,150				
					34	5,660 (100)				
53	{ 3 per cent Alkagal.	4.67	.63	6.0	1	3,300	28	1,610	35	25.0
					4	4,070				
					28	4,660 (100)				
61	{ Check cylinders without admixture.	4.67	.59	5.5	1	3,430	38	1,970	32	25.4
					4	4,290				
					38	6,130 (100)				
62	{ 5 per cent blast-furnace slag.	4.67	.60	5.4	1	3,370	43	1,900	30	40.0
					4	4,920				
					43	6,440 (100)				
63	{ 10 per cent blast-furnace slag.	4.67	.62	5.6	1	3,110	59	1,410	23	55.7
					4	4,710				
					50	6,200 (100)				
64	{ 20 per cent blast-furnace slag.	4.67	.65	5.8	1	3,460	70	1,720	26	67.1
					4	4,800				
					70	6,650 (100)				
65	{ 40 per cent blast-furnace slag.	4.67	.71	5.5	1	3,230	49	3,890	54	178.6
					4	4,340				
					49	7,170 (100)	182	2,230	30	
					182	7,350 (100)				
46	{ Check cylinders without admixture.	4.67	.59	5.6	1	3,000	48	1,570	27	44.7
					4	4,620				
					48	5,880 (100)				

TABLE 30.—*Effect of admixtures on resistance of 2 by 4 inch concrete cylinders exposed in the laboratory to the action of solutions of sodium sulphate ( $\text{Na}_2\text{SO}_4$ )—Con.*

[Cements A and B mixed in equal portions. Each test result is the average for 5 or more cylinders made on 5 days]

Series	General description	Fineness moduli	Water-cement ratios	Absorption at 21 days	Stored in tap water		Stored in 1 per cent solutions of $\text{Na}_2\text{SO}_4$			
					Age tested	Breaking strengths	Age tested	Breaking strengths	Per cent-age of normal strength	Time required to increase in length 0.010 inch
				Per cent	Weeks	Lbs. per sq. in.	Weeks	Lbs. per sq. in.	Per cent	Weeks
50	4 per cent cal.	4.67	0.50	6.2	1	2,850				
					4	4,200	62	1,510	26	59.0
					62	5,840 (100)				
46	Check cylinders without admixture.	4.67	.50	5.6	1	3,090				
					4	4,620	48	1,570	27	44.7
					48	5,880 (100)				
49	4 per cent calcium chloride.	4.67	.61	7.0	1	2,720				
					4	3,970	60	2,480	52	66.0
					60	4,790 (100)				
56	Check cylinders without admixture.	4.67	.50	5.8	1	3,370				
					4	4,590	40	1,630	25	37.1
					40	6,030 (100)				
57	5 per cent hydrated lime, high calcium.	4.67	.61	6.0	1	3,520				
					4	4,510	33	1,760	31	30.3
					33	5,680 (100)				
58	10 per cent hydrated lime, high calcium.	4.67	.63	6.7	1	2,860				
					4	4,440	29	1,540	27	26.3
					29	5,640 (100)				
59	5 per cent hydrated lime, high magnesium.	4.67	.61	5.9	1	3,170				
					4	4,620	34	1,770	27	30.9
					34	6,490 (100)				
60	10 per cent hydrated lime, high magnesium.	4.67	.63	6.3	1	3,650				
					4	4,980	32	1,670	33	29.3
					32	5,000 (100)				
46	Check cylinders without admixture.	4.67	.59	5.6	1	3,000				
					4	4,620	48	1,570	27	44.7
					48	5,880 (100)				
47	5 per cent ironite.	4.67	.50	5.8	1	3,040				
					4	4,910	54	1,560	27	30.7
					54	5,850 (100)				
48	20 per cent ironite.	4.67	.61	6.2	1	3,030				
					4	4,130	61	1,590	30	68.0
					61	5,380 (100)				
41	Check cylinders without admixture.	4.67	.50	5.9	1	2,690				
					4	4,540	44	1,410	24	40.6
					44	5,840 (100)				
42	2½ per cent volcanic ash.	4.67	.61	6.4	1	2,370				
					4	4,420	32	1,440	29	26.6
					32	4,990 (100)				
43	5 per cent volcanic ash.	4.67	.63	6.2	1	2,440				
					4	4,020	33	1,730	32	30.0
					33	5,480 (100)				
44	10 per cent volcanic ash.	4.67	.65	6.3	1	2,650				
					4	3,630	49	1,700	32	45.6
					49	5,240 (100)				
45	20 per cent volcanic ash.	4.67	.74	7.0	1	1,780				
					4	3,290	52	1,560	34	267.2
					52	5,050 (100)				
51	Check cylinders without admixtures.	4.67	.59	5.1	1	3,200				
					4	4,760	46	1,560	34	43.4
					46	5,490 (100)				
64	5 per cent water-gas tar.	4.67	.55	4.7	1	2,420				
					4	3,980	31	1,590	35	28.1
					31	4,520 (100)				
55	20 per cent water-gas tar.	4.67	.51	5.7	1	1,140				
					4	2,140	60	1,140	60	65.8
					60	2,270 (100)				

1 Estimated.



TABLE 31.—Tests of 2 by 4 inch concrete cylinders containing various admixtures, cured by different processes and exposed to action of sulphate waters of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water

[Unless otherwise noted the fineness modulus of aggregate is 4.67 and the mix is 1:3. Each test result, with a few exceptions is an average of five cylinders made on different days. Figures in parenthesis, in compression test columns, indicate per cent of normal strength based on parallel tests of cylinders from the same batches, stored in tap water in the laboratory]

Series	Cement laboratory No.	Portland cement	Water ratio	Admixture		Curing method					Absorption at 21 days	Average compression tests (pounds per square inch)						
				Amount	Ingredient	Time in moist closet	Time in water	Time in water vapor	Temperature of water vapor °F.	Time in air		Tank specimens				Lake specimens		
												7 days	28 days	1 year	5 years	1 year	3 years	5 years
				Per cent		Hours	Days	Hours		Days	Per cent							
182	18	½A and ½B1	0.50		None	24	20			35	6.0	2,800	4,010	5,780	5,790	4,450 (77)	12,100	0
183	18	do	.03	3	Alkagol A	24	20			35	5.3	2,260	3,560	4,570	4,070	3,520 (77)	12,140	0
184	18	do	.59		None	24		48	155	35	6.0	4,070	4,580	5,100	6,110	4,240 (83)	14,700	0
185	18	do	.63	3	Alkagol A	24		48	155	35	5.0	3,280	3,940	4,570	5,050	3,300 (72)	13,900	800 (13)
186	18	do	.63	3	do	24		48	100	35	5.4	3,120	3,730	3,820	4,420	3,550 (93)	13,060	1,090 (22)
554	129	do	.64		None	24	20			35	6.3	3,570	4,760	5,980	6,820	3,530 (59)	0	0
581	129	do	.62	6	Barium chloride	24	20			35	6.5	3,530	4,570	5,870	6,220	3,730 (64)	0	0
582	129	do	.62	12	do	24	20			35	6.7	3,240	4,460	5,100	6,050	4,420 (87)	2,800	0
822	225	do	.62		None	24	20			35	5.3	3,220	5,230	6,250		3,800 (61)	0	0
823	225	do	.62	3.75	Barnsdall admix	24	20			35	5.2	3,440	4,660	7,090		3,650 (43)	0	0
824	225	do	.64	7.5	do	24	20			35	5.5	2,970	5,070	6,480		3,000 (46)	0	0
825	225	do	.72	15	do	24	20			35	6.0	3,060	4,490	5,350		2,130 (40)	0	0
826	225	do	.79	30	do	24	20			35	6.8	2,310	3,790	5,410		1,450 (27)	0	0
981	263	do	.64		None	24	27			28	5.9		5,440	7,100		5,030 (71)		
982	263	do	.67	4	Barnsdall admix	24	27			28	6.1		5,030	6,610		4,590 (66)		
983	263	do	.69	6	do	24	27			28	6.0		5,140	6,050		4,060 (67)		
984	263	do	.71	8	do	24	27			28	6.1		5,560	5,950		3,800 (64)		
985	274	I	.64		None	24	27			28	5.9		5,330	7,100		7,170 (100)		
986	274	do	.67	4	Barnsdall admix	24	27			28	6.0		5,370	7,890		6,170 (78)		
987	274	do	.69	6	do	24	27			28	6.1		5,480	7,680		5,460 (71)		
988	274	do	.71	8	do	24	27			28	6.3		5,310	7,080		6,010 (93)		
989	263	½A and ½B1	.80		None	24	27			28	6.2		4,850	5,540		1,570 (23)		
990	263	do	.82	4	Barnsdall admix	24	27			28	6.6		5,010	5,770		630 (11)		
991	263	do	.83	6	do	24	27			28	6.4		4,770	5,340		980 (18)		
992	263	do	.84	8	do	24	27			28	6.2		4,660	6,050		570 (9)		
993	274	I	.80		None	24	27			28	6.5		3,980	6,610		3,970 (60)		
994	274	do	.82	4	Barnsdall admix	24	27			28	6.4		4,740	6,540		4,650 (71)		
995	274	do	.83	6	do	24	27			28	6.8		4,440	5,990		4,850 (81)		
996	274	do	.84	8	do	24	27			28	6.6		4,500	6,010		4,260 (71)		
997	263	½A and ½B1	.94		None	24	27			28	6.7		3,040	4,370		2,840 (65)		

<sup>1</sup> 2-year tests.

<sup>2</sup> Special high-silica aggregate, 1:3 mix; 2 by 4 inch cylinders cured in damp sand.

<sup>3</sup> Special high-silica aggregate, 1:2:3 mix; 2 by 4 inch cylinders cured in damp sand.

<sup>4</sup> Special high-silica aggregate, 1:2:4 mix; 4 by 8 inch cylinders cured in damp sand.

TABLE 31.—Tests of 2 by 4 inch concrete cylinders containing various admixtures, cured by different processes and exposed to action of sulphate waters of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water—Continued

Series	Cement laboratory No.	Portland cement	Water ratio	Admixture		Curing method					Absorption at 21 days	Average compression tests (pounds per square inch)						
				Amount	Ingredient	Time in moist closet	Time in water	Time in water vapor	Temperature of water vapor	Time in air		Tank specimens				Lake specimens		
												7 days	28 days	1 year	5 years	1 year	3 years	5 years
				Per cent		Hours	Days	Hours	°F.	Days	Per cent							
* 998	263	½A and ½B1.	0.95	4	Barnsdall admix.	24	27			28	0.8		3,570	4,040		2,700 (58)		
* 999	263	do.	.96	6	do.	24	27			28	0.9		4,020	4,510		1,510 (33)		
* 1,000	263	do.	.97	8	do.	24	27			28	0.9		3,610	4,050		830 (18)		
* 1,001	274	I.	.94		None	24	27			28	0.5		3,370	4,880		4,360 (89)		
* 1,002	274	do.	.95	4	Barnsdall admix.	24	27			28	0.6		3,480	4,730		4,080 (86)		
* 1,003	274	do.	.96	6	do.	24	27			28	0.6		3,560	4,300		3,950 (91)		
* 1,004	274	do.	.97	8	do.	24	27			28	0.7		3,570	4,780		5,020 (105)		
157	18	½A and ½B1.	.74	40	Blast furnace slag.	24	20			35	6.5	2,420	3,060	6,030	6,200	4,230 (70)	12,340	0
158	18	do.	.03	10	do.	24	20			35	6.0	2,880	4,180	5,440	6,260	5,170 (95)	13,930	0
159	18	do.	.74	40	do.	24	20			35	6.7	2,410	4,190	5,670	5,600	5,560 (93)	5,220	690 (12)
160	18	do.	.03	10	do.	24		48	155	35	6.4	3,850	4,010	5,300	4,060	4,800 (91)	3,540	0
161	18	do.	.74	40	do.	24		48	155	35	6.9	3,800	4,020	5,450	5,710	4,830 (89)	5,640	2,760 (48)
172	18	do.	.59		None	24	20			35	5.6	2,870	4,190	5,300	5,400	4,370 (82)	12,050	0
167	18	do.	.59	4	Cal.	24	20			35	5.7	3,200	4,150	4,860	5,600	3,310 (80)	11,380	0
168	18	do.	.90	8	do.	24	20			35	5.8	3,180	4,290	5,670	6,370	4,020 (83)	13,090	1,570 (25)
169	18	do.	.59	4	do.	24	20			35	5.8	3,250	4,280	4,550	5,580	4,800 (105)	13,620	1,090 (20)
170	18	do.	.90	8	do.	24		48	155	35	6.0	4,090	4,670	5,030	5,960	5,010 (100)	10,200	5,540 (93)
171	18	do.	.59	4	do.	24		48	155	35	6.0	4,550	4,690	5,140	5,730	4,980 (97)	15,560	4,090 (82)
172	18	do.	.59		None	24	20			35	5.6	2,870	4,190	5,300	5,400	4,370 (82)	12,050	0
162	18	do.	.03	4	Calcium chloride.	24	20			35	5.7	2,870	4,330	5,630	5,130	3,910 (71)	11,710	0
163	18	do.	.79	8	do.	24	20			35	7.1	2,200	3,010	4,280	4,220	3,550 (83)	13,130	1,040 (25)
164	18	do.	.03	4	do.	24	20			35	5.9	2,940	4,000	5,450	5,790	5,050 (93)	15,240	2,200 (38)
165	18	do.	.79	8	do.	24		48	155	35	7.1	3,800	3,640	4,000	4,620	3,300 (72)	13,860	3,770 (82)
166	18	do.	.63	4	do.	24		48	155	35	5.8	4,840	4,920	4,900	5,970	4,460 (80)	15,840	4,090 (84)
167	18	do.	.59		None	24	20			35	5.6	2,870	4,190	5,300	5,400	4,370 (82)	12,050	0
172	18	do.	.59		do.	24	20			35	5.8	4,930	6,580	6,420		3,540 (55)		
1181	294	do.	.62	¼	Casein	24	20			35	0.3	4,300	5,030	6,020		3,170 (48)		
1182	294	do.	.62	¼	do.	24	20			35	7.0	4,020	5,050	5,720		3,250 (57)		
1183	294	do.	.62	¼	do.	24	20			35	0.7	3,870	4,760	5,480		3,440 (93)		
1184	294	do.	.62	¼	do.	24	20			35	7.9	2,490	3,680	4,290		2,600 (52)		
1185	294	do.	.62	1	do.	24	20			35	0.4	3,530	5,050	6,070	6,280	3,760 (56)	0	0
412	105	Q2.	.66	2½	Celite	24	20			35	5.7	3,520	5,390	6,500	6,350	4,300 (66)	0	0
413	105	do.	.62		None	24	20			35	0.3	3,810	4,950	6,420	7,820	3,650 (57)	0	0
583	139	½A and ½B1.	.62		do.	24	20			35	0.1	3,260	5,340	6,180	6,390	5,290 (86)	0	0
634	139	do.	.64	2	Celite	24	20			35	0.1	3,260	5,340	6,180	6,390	5,290 (86)	0	0
635-636	139	do.	.64		None	24				35	0.3	3,960	4,330	4,250	6,600	4,370 (103)	5,950	6,530 (99)

637-638	139	do	.64	2	Celite	24	48	212	53	6.2	4,160	4,730	5,300	6,010	4,410 (83)	5,020	6,250 (90)
639	139	do	.64	2	Colloy	24	20		35	6.5	3,630	4,610	5,250	5,800	4,630 (88)	0	0
640	139	do	.64		None	24	20		35	6.5	3,320	4,470	5,800	6,070	4,530 (78)	0	0
641	139	do	.64		do	24	48	212	53	6.9	3,690	3,760	4,630	6,010	3,740 (83)	5,369	6,140 (102)
642-643	139	do	.64	2	Colloy	24	48	212	53	6.8	3,870	4,100	4,420	6,270	3,800 (86)	5,510	5,840 (93)
776	204	do	.62		None	24	20		35	6.2	3,400	4,420	6,480		0	0	
777	204	do	.64	2	Colloy	24	20		35	6.4	3,160	4,040	5,270		0	0	
778	204	do	.65	4	do	24	20		35	6.4	3,290	4,540	0,110		0	0	
715	176	do	.62		None	24	20		35	5.6	3,830	5,100	6,420	4,550 (71)	0	0	
716	176	do	.62	.27	Earthcrete	24	20		35	5.6	4,460	5,160	5,780	4,630 (80)	0	0	
717	176	do	.62	1.06	do	24	20		35	5.8	3,530	5,120	0,950	4,860 (70)	0	0	
718	176	do	.62	.27	do	24		48	155	5.6	5,210	5,550	5,370	5,150 (96)	0	0	
554	129	do	.64		None	24	20		35	6.3	3,570	4,760	5,980	6,820	3,530 (59)	0	0
504-505	129	do	.62	2½	Fuel ash	24	20		35	6.3	3,580	5,100	6,710	7,410	2,840 (42)	0	0
506	129	do	.64	5	do	24	20		35	6.4	3,300	4,760	5,850	7,260	2,670 (46)	0	0
507-508	129	do	.67	10	do	24	20		35	6.3	3,300	5,080	6,570	6,370	3,660 (56)	0	0
1180	205	A	.77		None	24	27			8.2	2,410	3,790	4,290		1,870 (44)		
1187	205	do	1.13	43	Haydite	24	27			0.0	1,680	3,070	5,330		3,370 (63)		
1188	205	do	2.05	150	do	24	27			10.2	690	1,700	4,460		3,170 (72)		
1189	206	Special F	.77		None	24	27			8.3	4,750	5,540	6,040		4,930 (82)		
1190	206	do	1.13	43	Haydite	24	27			8.2	3,340	4,140	5,980		4,920 (82)		
1191	206	do	2.05	150	do	24	27			11.2	1,260	2,600	4,660		2,960 (66)		
1192	204	½A and ½B1	.62		None	24	27			5.7	4,880	6,060	0,500		4,760 (73)		
1193	204	do	.90	43	Haydite	24	27			6.1	3,100	4,740	6,370		5,470 (86)		
1194	204	do	1.02	150	do	24	27			6.5	1,630	2,620	5,260		3,920 (75)		
1195	204	do	.71	30	do	24	27			5.8	4,390	5,840	7,450		6,750 (91)		
172	18	do	.59		None	24	20		35	5.6	2,870	4,190	5,300	5,400	4,370 (82)		0
173	18	do	.61	20	Ironite	24	20		35	5.9	2,950	3,980	4,890	5,800	5,020 (102)		1,590 (27)
174	18	do	.59		None	24		48	155	6.1	4,020	4,390	4,330	5,050	3,910 (90)		2,740 (48)
175	18	do	.61	20	Ironite	24		48	155	6.1	3,880	4,440	5,510	5,630	4,050 (84)		5,280 (94)
176	18	do	.61	20	do	24		48	100	6.1	3,080	4,220	5,250	5,770	5,190 (99)		5,020 (87)
1141	287	do	.62	1	None	24	20		35	5.6	4,560	5,400	7,160		4,440 (92)		
1142	287	do	.62	2	Kerosene	24	20		35	5.3	3,930	5,790	0,160		3,230 (52)		
1143	287	do	.62	4	do	24	20		35	5.1	3,790	5,740	5,940		3,850 (95)		
1144	287	do	.62	8	do	24	20		35	4.4	3,800	5,530	5,930		3,350 (50)		
1145	287	do	.62		do	24	20		35	4.8	3,740	4,870	5,520		2,070 (48)		
1156	204	do	.62		None	24	20		35	5.9	4,670	5,400	5,900		2,250 (38)		
1157	204	do	.62	½	Linseed oil	24	20		35	4.5	3,910	5,700	0,100		3,330 (55)		
1158	204	do	.62	1	do	24	20		35	3.6	4,240	5,470	5,350		4,190 (78)		
1159	204	do	.62	2	do	24	20		35	3.5	3,120	4,550	5,600		5,050 (89)		
1160	204	do	.62	4	do	24	20		35	3.1	1,820	3,970	5,100		4,960 (97)		
709	201	X	.62		None	24	20		35	6.3	3,050	4,960	6,440		4,440 (60)	0	0
770	201	X	.62	2	Medusa waterproofing	24	20		35	5.0	3,220	4,750	5,930		3,020 (51)	0	0
779	201	X	.62	1	do	24	20		35	5.2	3,030	4,690	5,480		25,70 (50)	0	0

\* Special high-silica aggregate, 1 : 2 : 3 mix; 2 by 4 inch cylinders cured in damp sand.

\* Special high-silica aggregate, 1 : 2 : 4 mix; 4 by 8 inch cylinders cured in damp sand.

\* Mix 1 : 2½; special aggregate—fineness modulus 2.95.

\* Mix 1 : 3½; special aggregate—fineness modulus 2.95.

\* Mix 1 : 6; special aggregate—fineness modulus 2.95.

\* Mix 1 : 4½.

\* Mix 1 : 7½.

TABLE 31.—Tests of 2 by 4 inch concrete cylinders containing various admixtures, cured by different processes and exposed to action of sulphate waters of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water—Continued

Series	Cement laboratory No.	Portland cement	Water ratio	Admixture		Curing method					Absorption at 21 days	Average compression tests (pounds per square inch)						
				Amount	Ingredient	Time in moist closet	Time in water	Time in water vapor	Temperature of water vapor	Time in air		Tank specimens				Lake specimens		
						Hours	Days	Hours	°F.	Days		7 days	23 days	1 year	5 years	1 year	3 years	5 years
780	201	X	0.65	Per cent 4	Medusa waterproofing.	24	20			35	4.0	2,780	4,700	5,340		2,180 (41)	0	0
1106	204	½A and ½B1.	.62		None	24	20			35	5.4	4,760	6,380	6,740		4,460 (60)		
1107	204	do.	.64	20	Metallicron	24	20			35	5.3	5,000	6,480	6,730		5,200 (77)		
1108	204	do.	.07	40	do	24	20			35	5.4	4,050	6,800	6,880		4,870 (71)		
1199	204	do.	.62		None	24		48	165	53	5.5	5,100	5,000	6,120		5,580 (91)		
1200	204	do.	.64	20	Metallicron	24		48	165	53	5.6	5,520	4,970	6,030		5,300 (80)		
1146	287	do.	.62		None	24	20			35	5.3	4,280	5,960	7,200		3,370 (47)		
1147	287	do.	.62	1	Oil	24	20			35	2.8	4,580	5,200	7,290		3,070 (41)		
1148	287	do.	.62	2	do	24	20			35	2.4	4,460	5,900	6,520		4,450 (68)		
1149	287	do.	.62	4	do	24	20			35	2.0	4,180	5,530	5,860		3,940 (67)		
1150	287	do.	.62	8	do	24	20			35	1.8	3,390	4,720	6,010		2,090 (35)		
771	204	do.	.62		None	24	20			35	5.0	3,450	4,440	5,870		0	0	0
772	204	do.	.65	3.75	Omicron	24	20			35	6.1	3,510	4,480	6,010		0	0	0
773	204	do.	.69	7.5	do	24	20			35	6.5	3,430	4,710	5,370		0	0	0
774	204	do.	.74	15.0	do	24	20			35	7.6	3,430	4,340	5,510		3,440 (62)	0	0
775	204	do.	.80	30.0	do	24	20			35	9.9	3,050	4,220	4,980		4,430 (80)	3,870	0
10 203	19	do.	.64		None	24	20			35	9.0	1,420	2,240	3,850	3,740	2,140 (56)	0	0
10 294	19	do.	.07	10	Sulphur	24	20			35	9.7	1,150	1,250	1,870	2,610	0	0	0
391	74	do.	.62		None	24	20			35	6.3	3,130	4,580	6,060	6,430	2,480 (41)	0	0
392	74	do.	.76	33	Truss	24	20			35	7.3	2,710	3,830	5,760	6,620	5,040 (87)	2,810	1,750 (31)
392a	74	do.	.85	33	do	24	20			35	7.1	2,430	3,450	5,260	5,540	4,170 (79)	2,060	1,300 (23)
393	74	do.	.93	60	do	24	20			35	9.1	1,030	3,350	4,006	5,106	4,400 (96)	3,510	2,140 (42)
393a	74	do.	1.09	60	do	24	20			35	8.4	1,440	2,970	4,270	4,740	3,660 (86)	2,650	1,830 (30)
394	86	I	.78	33	do	24	20			35	6.8	3,330	4,600	6,620	7,150	5,300 (80)	2,640	1,200 (18)
394a	86	I	.87	33	do	24	20			35	6.7	3,000	3,850	6,170	5,410	4,140 (67)	1,080	0
395	86	I	.95	60	do	24	20			35	7.6	2,710	3,770	6,060	6,970	4,500 (71)	2,570	2,110 (42)
395a	86	I	1.09	60	do	24	20			35	7.3	2,280	3,380	4,090	4,900	3,510 (93)	1,620 (32)	0
554	129	½A and ½B1.	.64		None	24	20			35	6.3	3,570	4,760	5,980	6,820	3,530 (59)	0	0
578	129	do.	.62	1	Truscon	24	20			35	4.7	3,370	4,700	5,320	6,460	3,750 (70)	0	0
579	129	do.	.62	2	do	24	20			35	4.1	3,370	4,580	4,980	5,360	3,280 (66)	0	0
580	129	do.	.62	4	do	24	20			35	3.7	3,020	3,980	4,630	5,230	2,420 (52)	0	0
177	18	do.	.69		None	24	20			35	6.0	2,520	4,200	4,930	6,120	3,670 (74)	1,010	0
178	18	do.	.74	20	Volcanic ash	24	20			35	6.8	1,900	3,210	5,450	6,670	2,060 (54)	1,070	0
179	18	do.	.69		None	24		48	165	35	5.8	4,110	4,510	5,210	6,460	3,820 (73)	2,380	1,750 (27)
180	18	do.	.74	20	Volcanic ash	24		48	165	35	7.6	3,240	3,340	4,440	5,500	3,600 (81)	1,410	1,760 (32)
181	18	do.	.74	20	do	24		48	100	35	7.3	2,710	2,820	5,130	5,670	4,290 (84)	1,560	2,430 (43)

10 Standard Ottawa sand cylinders.

11 Mix 1 : 4.

12 Mix 1 : 5.

In preparing the mix for the test specimens, all admixtures were added to the batch except as otherwise noted, the proportions of the admixtures being calculated on a basis of their weight added to that of the cement in the mix.

#### SILICEOUS MATERIALS

Finely divided silicas of volcanic origin, diatomaceous earths, ground burned clays, ground blast-furnace slags, and similar substances, because of their pozzuolanic properties, have been used and recommended by engineers of continental Europe, for concrete exposed to sea water, and in recent years have been favorably considered by a number of English cement chemists and engineers (12, 19). Engineers of the United States and Canada, however, have never generally accepted as a proven fact the practical value of siliceous admixtures. As is well known, the use of a pozzuolana is based on the theory that active silica of the admixture will slowly combine with free lime of the set cement to form relatively insoluble compounds of calcium silicate.

The chemical composition of the siliceous admixtures tested differs greatly in silica ( $\text{SiO}_2$ ) content, as shown in Table 28, although blast-furnace slag with 33.37 per cent and fuel ash with 44.5 per cent were the only ones containing less than 50 per cent of silica. In the other materials the range was from 55.6 per cent for trass to 96.8 per cent for Barnsdall admix. These chemical analyses mean little when considered alone, if the theory underlying the use of siliceous admixtures is sound, since only the silica active at ordinary temperatures is of value, and that must be finely divided to combine readily with the free lime of the cement.

A completely satisfactory method for determining active silica has not been agreed upon. The method used in these experiments was that suggested by Cowper (18, p. 46-49), in which 0.6 gram of the siliceous material was combined with 0.4 gram of hydrated lime, placed in a test tube, and shaken at intervals of 12 hours. The pozzuolanic activity was judged by inspecting the tubes at different times after shaking and noting the increase in volume of the solid matter. The results are shown in Table 32. Another method has been suggested by Blount (12) who stated that normally the active silica is regarded as that which can be extracted from the material by a weak alkali such as 1 per cent caustic soda solution, but there is no fixed method. A third method (24) is based on the electric resistance, measured to 0.1 ohm, of a lime water solution of known strength to which is added the siliceous material and the pozzuolanic activity calculated on a basis of decreased conductivity due to removal of some of the lime as a result of chemically combining with active silica.

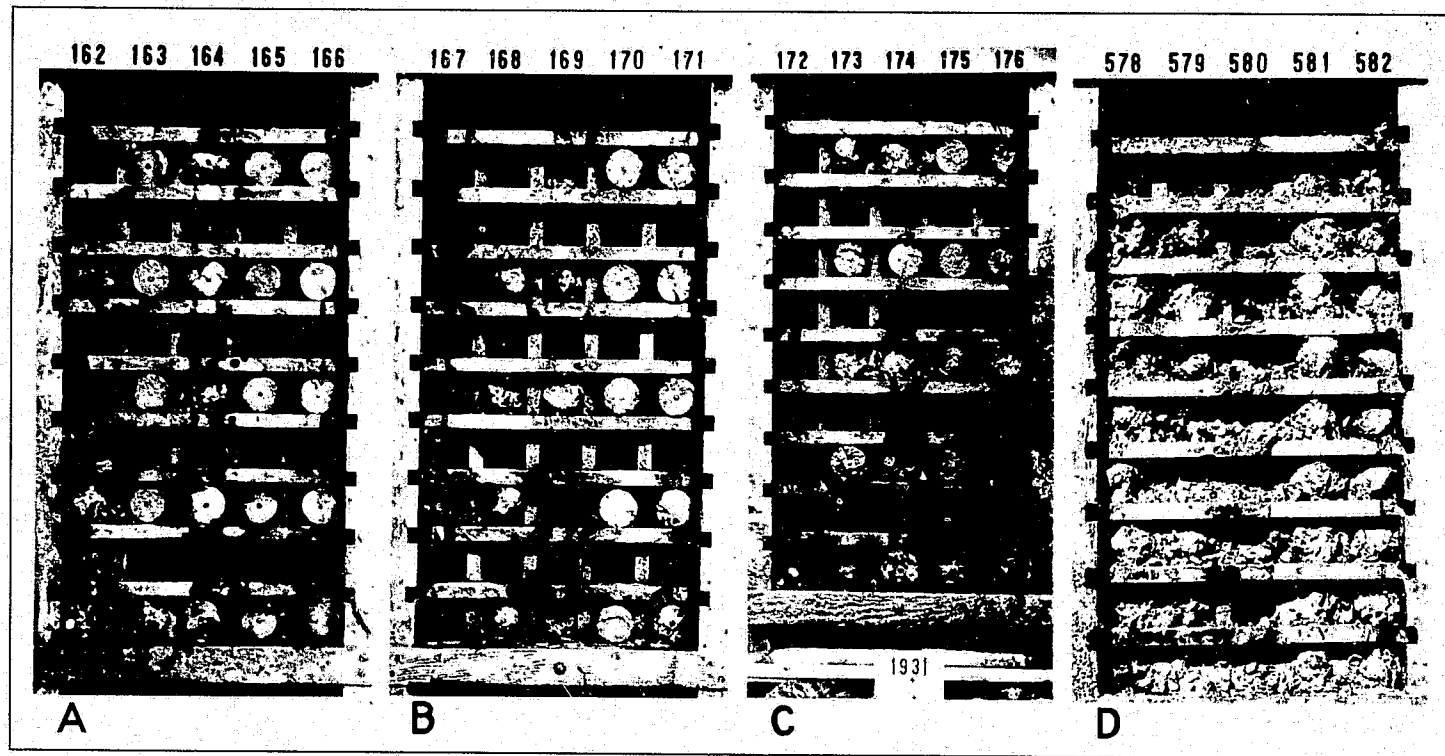
TABLE 32.—*Pozzuolanic activity of the nine siliceous admixtures, compared by volume increase*

Days	Barnsdall admix	Blast-furnace slag	Cellite	Colloy	Fuel ash	Haydite	Omitron	Trass	Volcanic ash
	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
1	31	103	192	244	83	22	300	31	18
2	40	232	223	322	117	53	684	80	30
3	40	267	338	377	133	88	940	107	60
4	42	285	346	414	150	105	932	133	85
5	50	322	369	432	147	119	924	143	110
6	60	340	338	469	150	133	876	170	127
7	60	304	365	524	158	122	924	170	118
8	50	322	323	450	167	105	908	179	118
9	60	336	392	460	171	135	872	188	135
10	60	340	377	469	184	152	874	188	135
11	60	327	368	469	179	152	876	206	145
12	60	324	350	450	201	140	829	198	135
13	60	324	342	456	217	156	812	197	150
14	60	322	346	450	217	156	748	206	148

Of the nine siliceous admixtures tested, blast-furnace slag, trass, and volcanic ash are the only ones that definitely increased resistance. The effect of Haydite is still undetermined. It is not seen how the pozzuolanic activity recorded in Table 32 or the fineness tests of Table 29 could have been used to predict the relative effect of the siliceous materials of this group on resistance of concrete to sulphate action. Therefore, it appears that, regardless of the methods used to determine the active properties of a pozzuolana, justification for its use to increase the resistance of a Portland-cement concrete must depend upon careful experimentation.

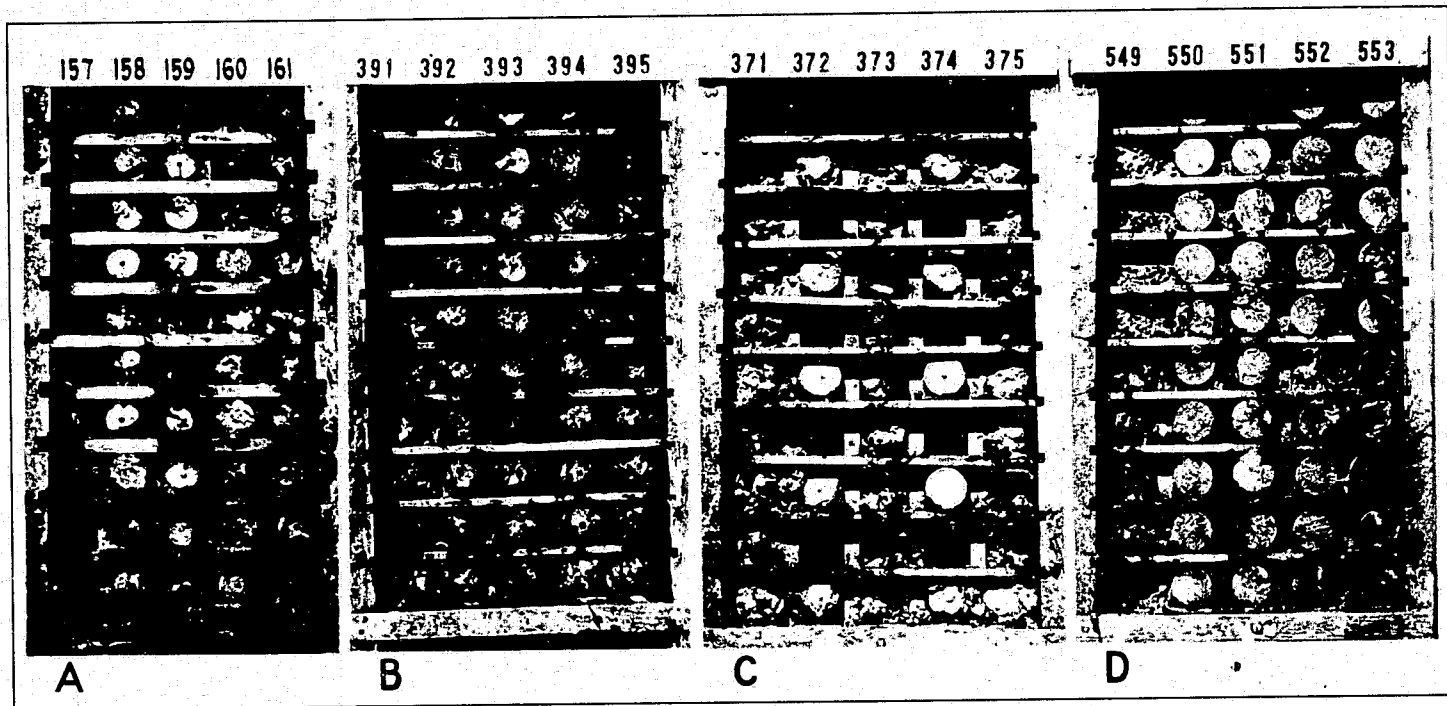
Barnsdall admix has the appearance of pulverized chert and is described by the producers as a "pure, finely ground, meta-colloidal Tripoli silica." This product was used in quantities varying from 3.75 to 30 per cent and in 1:3, 1:2:3, and 1:2:4 concretes. Two aggregates, a combination of two cements of low resistance, and a resistant cement were used in the tests. It is not apparent from tests up to three years (Table 31) that this material increased resistance; some of the data seemed to indicate decreased resistance. The effect of 8 per cent and less of the admixture on the 28-day strength of concrete was small, but was slightly advantageous with the leaner mixes. The effect on the strength at one year was inappreciable. Fifteen and thirty per cent of the admixture decreased compressive strengths at 28 days and at 1 year.

Blast-furnace slag, finely ground, was used in proportions of 5, 10, 20, and 40 per cent in cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate and in proportions of 10 and 40 per cent in cylinders exposed in Medicine Lake. Some of these cylinders were cured in water at room temperatures and others in water vapor at 155° F. This slag did not appreciably affect the 28-day strength of normally cured concrete. The data definitely indicate that the strength increased at one and five years for cylinders containing 10 and 40 per cent, the only ones of which long-time tests were made. Resistance to the laboratory solutions increased with quantity of slag used, the cylinders containing 40 per cent of slag having a life 400 per cent longer than the check group. Stored in Medicine Lake the normally cured cylinders containing 40 per cent slag definitely increased in resistance and the cylinders containing 10 per cent slag increased slightly. Cylinders with 40 per cent slag but with no air hardening showed little or no increased resistance, nor did cylinders containing 10 per cent slag cured at 155° F. Cyl-



CYLINDERS CONTAINING VARIOUS ADMIXTURES AFTER STORAGE FOR SEVEN YEARS IN MEDICINE LAKE

A, Calcium chloride admixed as follows: Series 162, 164, and 166—4 per cent; series 163 and 165—8 per cent. Series 165 and 166 cured 48 hours in water vapor at 155° F. B, Cal, same treatment as in A. C, Series 172—no admixture; 173—20 per cent ironite; 174—no admixture but cured 48 hours in water vapor at 155° F.; 175—20 per cent ironite, cured 48 hours in water vapor at 155° F.; 176—20 per cent ironite, cured 48 hours in water vapor at 100° F. D, Series 578, 579, and 580 contained 1, 2, and 4 per cent, respectively, of Truscon waterproofing paste. Series 581 and 582 contained 6 and 12 per cent, respectively, of baryum chloride.



CYLINDERS CONTAINING SILICEOUS ADMIXTURES AND CYLINDERS' SURFACE TREATED, AFTER STORAGE IN MEDICINE LAKE

- A. Blast-furnace slag admixed in the following percentages, after five years in the lake. Series 157, 159, and 161 40 per cent; series 158 and 160 10 per cent. Series 160 and 161 were cured 48 hours in water vapor at 155° F. B. Series 391 contained no admixture. Series 392 and 394 contained 33 per cent trass and series 393 and 395 contained 66 per cent trass. After four and one-half years in the lake. C. Series 371 to 373 standard Portland cement series 374 and 375 concrete and mortar, respectively, surface treated with Inertol. After five and one-half years in the lake. D. Series 549 to 553, respectively, surface-treated by dipping as follows, after four and one-half years in the lake; boiling water one-half minute; one coat linseed oil 70° F. one-half minute; two coats linseed oil 70°, one-half minute; one coat linseed oil 225° one-half minute; two coats linseed oil 225° one-half minute.



inders containing 40 per cent slag and cured at 155° tested 48 per cent of normal at five years, this series making considerably the best showing in Medicine Lake of any of the slag group. (Pl. 9.)

Celite, a diatomaceous earth, used in the proportions of 2 and 2½ per cent, did not appreciably affect either strength or resistance of normally cured cylinders.

Colloy, probably benonite clay, used in proportions of 2 and 4 per cent, resulted in concrete weaker at one year than check cylinders with no admixture. The effect of either quantity on strength and resistance was negligible.

Fuel ash, used in proportions of 2½, 5, and 10 per cent, apparently had no pronounced effect on the compressive strength or resistance of cylinders up to five years.

Haydite, a burned clay ground until 94 per cent passed a No. 300 sieve, was used both as a substitute for some of the cement and as an addition to the batch. This product was substituted for 30 and 60 per cent of the cement in the cylinders of most series, but 30 per cent was added to the cement for one series. As used, this material increased absorption and considerably reduced the 7 and 28 day strengths, but between 7 and 28 days there was a pick-up, so that cylinders in which 30 per cent of the cement was substituted averaged slightly stronger than the respective check cylinders and in those having 60 per cent substitution the strength recoveries were pronounced. The strongest and most resistant 1-year cylinders were those with the 30 per cent addition. (Table 31.) Tests with Haydite are being continued in a number of new series not here reported.

Omicron is the trade name for a finely divided product which was added in quantities up to 30 per cent. The effect on strength at one year was inappreciable for 3.75 per cent, but for 7.5, 15, and 30 per cent was a slight reduction. The cylinders in which 15 and 30 per cent was used showed increased resistance at one year, but no effect was indicated for 3.75 and 7.5 per cent. The only cylinders exposed in Medicine Lake that were made with this material and showed any compressive strength at three years were those containing 30 per cent admixture.

Trass, long used in hydraulic limes and cements, was imported from Germany. Trass is described by Eckel (21) as an ancient volcanic mud composed of earthy or compact pumiceous dust with fragments of pumice, trachyte, carbonized wood, etc. This material, finely powdered, was used both as an addition to the batch and as a substitute for some of the cement, with surprisingly similar effects on the compressive strength of the concrete for all periods up to five years. The quantities added were equivalent to 33 and 66 per cent of the weight of the cement, which by volume were almost exactly 50 and 100 per cent the volume of the cement; the quantities substituted replaced 25 and 40 per cent of the cement by weight. In both cases and at all ages, 66 per cent of trass reduced the strength of the concrete by about 25 per cent and 33 per cent reduced the strength by about half as much. The different additions increased resistance of the concrete about equally, with the result that cylinders containing trass averaged about 34 per cent of normal strength at five years, as against 0 per cent at three years, as compared with the check group without trass. While results were far from ideal, it was evident that trass did definitely increase resistance of concrete under extremely severe conditions of exposure. (Pl. 9.)

Volcanic ash from northwestern Nebraska was added to the mix in quantities of 2.5, 5, 10, and 20 per cent for cylinders stored in 1 per cent solutions of sodium sulphate in the laboratory and 20 per cent for cylinders stored in Medicine Lake. Twenty per cent ash increased the resistance of the laboratory cylinders more than 500 per cent, but lesser quantities had very little effect. Twenty per cent ash did not, however, increase the resistance of the cylinders exposed in Medicine Lake; on the contrary, it slightly reduced their resistance. In all cases, this product reduced the compressive strength of concrete at 7 and 28 days, loss of strength being in the same proportion as quantity of admixture. The only long-time strength data are 1 and 5 year tests of cylinders with 20 per cent ash, which averaged 11 per cent stronger at one year and 7 per cent weaker at five years than did normal check cylinders. This might be interpreted in a number of ways, but has been assumed to indicate that 20 per cent of this volcanic ash had slight effect on strength beyond one year. It is evident that more tests of this product might profitably be made, as regards both its effect on resistance and its effect on long-time strength.

#### HIGH-IRON PRODUCTS

Ironite is the trade name for finely ground iron and iron oxide, equivalent to 83.33 per cent metallic iron, mixed with ammonium chloride to hasten oxidation when used as a brush coat. Twenty per cent of this material was added to the batch for cylinders tested in Medicine Lake and 20 and 5 per cent to cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate. Special water-vapor curing at temperatures of 100° and 155° F. was used for some of the cylinders exposed in Medicine Lake. The effect of Ironite on compressive strength of normally cured cylinders was inappreciable at all ages up to 5 years. The effect on resistance was beneficial, particularly for the cylinders cured at 100° and 155° which had respective strength ratios of 87 and 94 per cent after five years in Medicine Lake. (Pl. 8.)

Metallicron is a trade product consisting of about 90 per cent finely divided metallic iron and the balance a siliceous material resembling clay. Cylinders with 20 per cent admixture were slightly stronger at 7 and 28 days than the check cylinders, and those with 40 per cent were slightly weaker, although at one year the strengths of all cylinders were essentially similar. At one year the effect of Metallicron on resistance, while positive, was not pronounced.

#### MISCELLANEOUS CHEMICAL COMPOUNDS AND MIXTURES

Barium chloride, among other things, was recommended by Michaelis (32) as long ago as 1891 for increasing resistance of concrete in sea water. Six and 12 per cent of barium chloride dissolved in the mixing water reduced the strength of concrete at all ages up to five years, but somewhat increased the resistance to sulphate. Cylinders containing 12 per cent tested 87 per cent of normal strength after one year in Medicine Lake, and not far from 50 per cent after three years. All of the barium chloride cylinders tested in Medicine Lake, however, failed before five years.

Cal is a material obtained by pulverizing the dried or undried product resulting from a mixture of either quick lime or hydrated

lime, calcium chloride, and water (61). Additions of 4 per cent of this material were used in cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate, and 4 and 8 per cent additions were used both in cylinders cured normally and in cylinders cured in water vapor at 155° F. for Medicine Lake exposure tests. The effect of 4 and 8 per cent of Cal on compressive strength was not very pronounced, although cylinders with the admixture generally tested slightly stronger than those of plain concrete. The cylinders containing 4 per cent Cal tested 14 per cent weaker than the check groups at one year, but stronger at five years. Additions of Cal increased resistance, under the conditions of the tests, except in cylinders without air hardening of one Medicine Lake series containing 4 per cent. Cylinders containing 4 and 8 per cent Cal when cured at 155° F. had the high values of 82 and 93 per cent, respectively, of normal strength at five years. (Pl. 8.)

Calcium chloride equivalent in weight to 4 and 8 per cent of the cement was dissolved in the mixing water. Four per cent was added for cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate, while 4 and 8 per cent were used in cylinders exposed in Medicine Lake, some cured normally and some cured in water vapor at 155° F. Four per cent calcium chloride had slight effect on compressive strength at any age between 7 days and 5 years, but 8 per cent reduced the strength at all ages, the average reduction being in excess of 20 per cent. The sulphate resistance of cylinders was increased by the calcium chloride in all cases except one Medicine Lake series with 4 per cent and without air hardening. Cylinders containing 4 and 8 per cent calcium chloride when cured at 155° gave the high strength ratios of 84 and 82 per cent, respectively, at five years. (Pl. 8.)

Casein, or calcium caseinate, is a finely powdered milk derivative. It was used as a concrete admixture in small quantities of 0.125, 0.25, 0.5, and 1 per cent of the weight of the cement. All quantities decidedly reduced the 7 and 28 day strengths, with 1 per cent causing reductions of more than 40 per cent. At 1 year, the effect on strength was less pronounced than at 7 and 28 days, and a very slight tendency toward increasing the resistance to sulphates was evident.

Earthcrete is the trade name of a powdered mixture consisting essentially of sodium chloride and potassium nitrate. Added in proportions of 0.27 and 1.06 per cent of the cement, it did not appreciably increase the resistance of cylinders exposed in Medicine Lake. As used, the effect on compressive strength was not pronounced at 7 days, 28 days, or 1 year.

Hydrated limes, both high-calcium and high-magnesium, behaved so similarly that in this discussion they need not be considered separately. Five and ten per cent of these limes had slight effect on compressive strengths at 7 and 28 days. The only long-time compression tests made were at ages between 32 and 43 weeks, when all cylinders containing hydrated lime showed loss of strength except those with 5 per cent high-magnesium lime which tested about normal. The effect on resistance of cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate was negative in all cases.

Sulphur used in the proportion of 10 per cent decreased compressive strength at all ages from 7 days to 5 years, and very definitely decreased the resistance of cylinders stored in Medicine Lake.

## WATER REPELLENTS

The group of water repellents includes three patented soap preparations, boiled linseed oil, automobile oil of medium viscosity, kerosene, and water-gas tar. They very generally decreased absorption of water by the concrete. Four of them, in some one or more of the quantities used, reduced the absorption by more than one-third. This effect of oils has long been known (39). However, it is not evident from the results obtained with these water-repellents and also with some of the other materials, that the absorption of concrete is by itself of any value whatever as an index of sulphate resistance.

Alkagel A is the trade name of a colloidal paste of copper and iron soaps together with paraffin, which smelled strongly of ammonia and lost 81 per cent of its weight on drying. Alkagel A was added to the mixing water in proportions of 1.5 and 3 per cent of the weight of the cement in cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate, while for cylinders exposed in Medicine Lake 3 per cent was used in normally cured cylinders and in cylinders cured in water vapor at 100° and 155° F. This product caused decreases in compressive strength at all ages from 7 days to 5 years except with one series which had normal strength at 7 days. The effect on sulphate resistance ranged from an inappreciable increase to a decided decrease. Curing cylinders containing this paste at 100° and 155° F. gave no positive results.

Medusa waterproofing is a powder consisting of 23 per cent soap mixed with hydrated and carbonated lime and about 6 per cent magnesia. Used in proportions of 1, 2, and 4 per cent, it caused no increase in sulphate resistance of cylinders stored in Medicine Lake, though absorption by those cylinders decreased with increase in quantity of admixture. The compressive strength was affected inappreciably at seven days and was considerably reduced at one year.

Truscon waterproofing paste, concentrated, added to the mixing water at rates of 1, 2, and 4 per cent of the weight of the cement, did not appreciably increase the resistance of any of the cylinders in which it was used, although absorption by the cylinders decreased as the quantity of admixture was increased. The effect on compressive strength ranged from inappreciable and adverse at 7 and 28 days to adverse at 1 and 5 years. (Pl. 8.)

Linseed oil, boiled, added in proportions of 0.5, 1, 2, and 4 per cent of the weight of the cement, retarded hardening, as evidenced by the 7-day tests. The cylinders with 2 and 4 per cent then tested only 67 and 39 per cent, respectively, of normal. At 28 days and 1 year, the strengths of the linseed-oil cylinders more nearly approached normal, and at 1 year the cylinders with 4 per cent oil were less than 14 per cent weaker than the check cylinders. Absorption definitely decreased as the quantity of linseed oil was increased, and the 1-year tests of Medicine Lake cylinders indicated that resistance increased with quantity of linseed oil used.

Automobile oil, with Society of Automotive Engineers viscosity classification of 30, when used in quantities of 1, 2, 4, and 8 per cent very markedly reduced absorption. However, the effect of this oil on compressive strengths was not pronounced, except at 7 and 28 days for cylinders containing 8 per cent and 1 year for cylinders containing 4 and 8 per cent, which all averaged 19 per cent lower

in strength than the check cylinders without oil. The effect on resistance to sulphates was hardly appreciable at one year, except on the cylinders with 8 per cent oil which made the poorest showing.

Kerosene used in quantities of 1, 2, 4, and 8 per cent appreciably reduced absorption by the cylinders and somewhat reduced 7-day strengths. In proportions under 8 per cent, kerosene had slight effect on 28-day strengths but apparently reduced the strength at one year. The effect of kerosene on resistance to sulphates was inappreciable at one year.

Water-gas tar, a thin liquid with 70 per cent soft pitch residue at 300° C., when added with the mixing water in proportions equivalent to 5 and 20 per cent of the weight of the cement, greatly reduced compressive strengths at 7 and 28 days. The resistance of cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate was below normal for those with 5 per cent of water gas tar, but very definitely above normal for those with 20 per cent.

#### RECAPITULATION OF RESULTS WITH ADMIXTURES

The 26 admixtures used did not, as a whole, increase the resistance of concrete to sulphate attack enough to justify their use for this purpose. A few, however, seemed to show definite possibilities, particularly in conjunction with the relatively high curing temperatures of 100° and 155° F., which are comparable to those used at many plants making draitile and other concrete products. Tests of cylinders from a number of the admixture series have not been made for exposure periods beyond one year, but the results of the 1-year tests do not indicate that any of those admixtures will show definitely beneficial results at five years. The admixtures which the 5-year tests indicate as holding most promise are the following:

Ironite, 20 per cent, used in cylinders cured at 155° and 100° F., which tested, respectively, 94 and 87 per cent of normal strength after five years in Medicine Lake.

Cal, 4 and 8 per cent, used in cylinders cured at 155° F., which tested, respectively, 82 and 93 per cent of normal strength after five years in Medicine Lake.

Calcium chloride, 4 and 8 per cent, used in cylinders cured at 155° F., which tested, respectively, 84 and 82 per cent of normal strength after five years in Medicine Lake.

Blast-furnace slag, 40 per cent, used in cylinders cured at 155° F., which made an excellent showing in 1 per cent solutions of sodium sulphate in the laboratory and after five years in Medicine Lake showed 48 per cent of normal strength.

Trass additions of 33 and 66 per cent, which definitely increased the resistance of cylinders stored in Medicine Lake, although no series averaged more than 42 per cent of normal strength. Substituted for 25 and 40 per cent of the cement, trass gave about the same results as the somewhat larger quantities added to the batch.

Volcanic ash, 20 per cent, which gave excellent results in water-cured cylinders stored in the laboratory in 1 per cent solutions of sodium sulphate, but failed to develop increased resistance under the more severe exposure conditions of Medicine Lake except for the cylinders vapor-cured at 100° F., which tested 43 per cent of normal strength at five years.

Moler, a diatomaceous silica used to replace 33½ per cent of the cement by mixing with the cement clinker before grinding, apparently caused an increase in resistance although, under the conditions tested, definite conclusions are not possible. (See special cement X, Table 22.)

#### SURFACE TREATMENTS

Treating the surface of concrete to protect it against sulphate action must be complete to be long effective. Even very slight disintegration destroys the bond between concrete and coating, and the action progresses at an increasing rate as the area of loosened coating becomes larger. Coatings that are more or less water-tight therefore rarely do more than somewhat retard early action. Comparatively few tests of this type are here reported, as experiments with only four products were conducted. The results of these tests are given in Table 33. The appearance of cylinders treated with two of these substances, after some years in Medicine Lake, is shown in Plate 9. The report by Lord (31) on experiments with surface-treated concrete cylinders in Medicine Lake is of interest in this connection.

TABLE 33.—Tests of 2 by 4 inch concrete cylinders given various surface treatments, cured in water and air and exposed to the action of sulphate water of Medicine Lake, S. Dak., as compared with similar cylinders stored in tap water

[Unless otherwise noted the fineness modulus of aggregate is 4.67 and the mix is 1 : 3. Each test result, with a few exceptions is an average of five cylinders made on different days. Figures in parentheses, in compression test columns, indicates per cent of normal strength based on parallel tests of cylinders from the same batches, stored in tap water in the laboratory]

Series	Cement laboratory No.	Cement	Water ratio	Surface treatment or impregnation	Curing method			Absorption at 21 days	Average compression tests (pounds per square inch)						
					Time in moist closet	Time in water	Time in air		Tank specimens				Lake specimens		
					Hours	Days	Days	Per cent	7 days	28 days	1 year	5 years	1 year	3 years	5 years
374	74	½A and ½B1	0.02	Inertol, first coat at 22 days, second at 26 days.	24	20	35	6.7	2,930	4,870	5,570	6,200	5,390 (97)	3,310	5,320 (86)
375	74	do.	.04	do.	24	20	35	10.5	1,430	2,790	3,040	3,740	2,970 (82)	0	0
391	74	do.	.02	None.	24	20	35	6.3	3,130	4,890	6,060	6,430	2,480 (41)	0	0
549	129	do.	.02	None. Dipped in boiling water ½ minute at 28 days and ½ minute at 31 days.	24	20	35	5.8	3,790	4,770	6,550	7,750	1,620 (25)	0	0
550	129	do.	.02	Boiled linseed oil at 70° F., dipped ½ minute at 28 days.	24	20	35	5.8	3,540	4,960	6,040	6,520	5,520 (91)	5,220	4,210 (65)
551	129	do.	.02	Boiled linseed oil at 70° F., dipped ½ minute at 28 days and ½ minute at 31 days.	24	20	35	5.8	3,730	5,200	5,520	6,970	5,730 (104)	5,560	2,930 (42)
552	129	do.	.02	Boiled linseed oil at 225° F., dipped ½ minute at 28 days.	24	20	35	5.8	3,430	4,080	5,970	6,820	5,330 (89)	5,260	2,410 (35)
553	129	do.	.02	Boiled linseed oil at 225° F., dipped ½ minute at 28 days and ½ minute at 31 days.	24	20	35	5.8	3,750	4,960	5,510	6,830	5,650 (103)	5,050	1,940 (28)
554	129	do.	.02	None.	24	20	35	6.3	3,570	4,700	5,980	6,820	3,530 (59)	0	0
934	245	do.	.02	McEverlast special paving coating, 1 brush coat at 24 hours.	24	20	55	4.0	4,370	5,270	0,030	—	5,010 (93)	3,020	—
935	245	do.	.02	McEverlast special paving coating, 1 brush coat at 21 days.	24	20	35	5.8	4,340	5,040	5,440	—	5,710 (105)	0	0
976	237	do.	.02	None.	24	20	35	6.0	3,700	4,590	5,750	—	4,650 (81)	0	0
977	237	do.	.02	McEverlast penetration, 1 brush coat at 24 hours followed by 1 brush coat concrete cover coat at 48 hours.	24	20	55	2.7	2,890	4,180	5,660	—	4,710 (83)	4,780	—
978	237	do.	.02	McEverlast penetration, 1 brush coat at 21 days followed by 1 brush coat concrete cover coat at 22 days.	24	20	35	2.1	3,670	4,050	5,740	—	5,550 (97)	3,120	—
979	237	do.	.02	McEverlast paving special, 1 brush coat at 24 hours followed by 1 brush coat concrete cover coat at 48 hours.	24	20	55	4.5	3,200	4,410	5,020	—	4,920 (88)	3,770	—
980	237	do.	.02	McEverlast paving special, 1 brush coat at 21 days followed by 1 brush coat concrete cover coat at 22 days.	24	20	35	2.0	3,410	4,450	5,780	—	5,250 (91)	3,450	—
1293	19	do.	.04	None.	24	20	35	9.9	1,420	2,240	3,850	3,740	2,140 (56)	0	0
1294	19	do.	.04	Sulphur impregnated.	24	20	35	9.9	1,350	2,210	3,130	2,610	1,500 (48)	0	0

1 Standard Ottawa sand cylinders.

The results of the study of surface treatments are very generally summarized as follows:

Inertol appreciably retarded sulphate action on concrete cylinders, but at the end of five years this coating afforded only slight protection.

Boiled linseed oil, both one coat and two coats, whether applied at room temperatures or heated to 225° F., appreciably retarded action on concrete cylinders up to four years and apparently afforded a slight measure of protection beyond about five years.

McEverlast afforded some protection up to two years and indicated a slight protection beyond about three years.

Sulphur impregnation afforded concrete no protection against disintegration in Medicine Lake.

### CONCLUSIONS

Detailed conclusions on the effect of many factors on resistance of concrete to sulphate action are incorporated in various sections of this bulletin dealing with those factors.

The severity of action on concrete of pure solutions of either magnesium or sodium sulphate increases with the strength of solution, but at a diminishing rate for strengths greater than 1 per cent.

The destructive action of magnesium sulphate does not differ greatly from that of sodium sulphate in solutions of equal strength, although the latter averaged slightly more severe with most of the 35 Portland cements used in these tests.

The 28-day strength is a fair index of resistance for concrete of any given cement and given curing conditions, but may have no significance for comparing concretes made of cements from different mills or when the concretes are cured under widely different conditions.

Under identical exposure conditions, concrete made of a highly resistant Portland cement may last 10 times as long as that made of a cement of low resistance. Neither standard physical tests nor ordinary chemical analyses give any indication of the resistance of a cement to sulphate action. Qualities of the raw material associated with the geological formations from which it comes may be factors in the resistance of a cement.

Resistance of concrete is markedly increased by curing in water vapor at temperatures of 212° to 350° F., almost to the point of immunity to action for the most favorable temperatures and curing periods. Resistance is not increased, however, by raising the curing temperatures until 212° is reached, except in connection with the use of certain admixtures.

The admixtures Ironite, Cal, calcium chloride, blast-furnace slag, trass, moler, and possibly volcanic ash have appreciably retarded sulphate action on concrete cured at room temperatures. Results were outstanding, however, only as the relatively high curing temperatures of 100° and 155° F. were used in conjunction with Ironite, and 155° with Cal and calcium chloride. Under these conditions, cylinders had the highly satisfactory values of 82 to 94 per cent of normal strengths after five years in Medicine Lake, S. Dak.

Special cements other than alumina cements have not shown a degree of resistance that would justify preference over the more resistant of the Portland cements, except possibly an imported mason's cement containing 33½ per cent diatomaceous silica (moler) mixed with the cement clinker before grinding.

Each of the three alumina cements tested resisted sulphate action to a degree that approached the ideal, but displayed definite indica-



tions of instability when used in concretes and mortars stored for long periods in tap water at room temperatures.

### RECOMMENDATIONS AND SUGGESTIONS

Some of the following recommendations apply to all types of concrete to be exposed to sulphate attack, and some are applicable only to concrete pipe and other products similarly manufactured.

The resistance of a cement which is to be exposed to sulphate action should be tested in accordance with the routine suggested on page 43. Cements very low in resistance may then be rejected. Only cements that are above the average in resistance should be considered for use where sulphates are known to be present.

With any given cement and any predetermined conditions of curing, care should be observed in all particulars to obtain the highest practicable 28-day strength. That strength, although fallible for comparing different concretes, has much value as an index of the permeability and sulphate resistance of the products of the same cement and method of manufacture, particularly with rich mixes.

Concrete should be kept from intimate contact with sulphates until it has had opportunity to dry and harden in air for the longest time practicable. Depending on the particular cement used, air hardening may greatly increase resistance and, as a precautionary measure, should be continued for 30 days if possible, and 90 days or longer is desirable.

To develop the highest resistance in drain tile, sewer pipe, and other products of concrete, they should be steam cured when 12 to 24 hours old at temperatures of 212° F. or higher for 48 hours or longer.

Alumina cement may be used advantageously for concrete structures subject to extremely severe conditions of sulphate exposure if the concrete will be continuously moist at temperatures generally below 60° F. and rarely exceeding 70° F. These moisture and temperature conditions are about the average for drain tile after installation.

The following results of the experiments suggest methods of increasing the resistance of concrete to sulphate attack, but check tests are too limited to justify basing definite recommendations on them.

Very resistant concrete has been made by using curing temperatures of 100° and 155° F. in conjunction with additions of the commercial high-iron product Ironite, and equally good results have followed the use of curing temperatures of 155° in conjunction with additions of calcium chloride and of the calcium chloride product Cal. These results appear to hold some promise, as it is now common practice at many tile plants to use curing temperatures between 100° and 155°.

Concrete containing certain quantities of one of the admixtures Ironite, Cal, calcium chloride, trass, blast-furnace slag, and moler, after curing in water vapor at room temperatures, when exposed in Medicine Lake displayed resistance sufficiently increased to make the use of those materials seem justifiable where conditions of sulphate exposure are only moderately severe. These admixtures might have some merit in sea-water construction.

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