THESIS

A COMPARITIVE ANALYSIS OF MICHIGAN CEMENTS

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Cement - Testing

A COMPARITIVE ANALYSIS

OF

MICHIGAN CREEKS

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The Authors.

HISTORY.

A curious, great bridge which has come down from the days of the ancients stands in the south of France, not far from the old city of Nimes. It is called the Pont du Gard and it is still referred to by the natives of the region as one of the seven wonders of thw world. It spans the deep valley of the Gard and it is builded in three tiers of stone arches. There are six arches of the lowest tier, ranging from sixty to seventy-five feet in width, eleven seventy-five foot arches in the second tier, and thirty-five smaller ones in the third, carrying the so-called "specus", or water trough, through which the Romans twenty centuries ago were wont to supply the fountains of their beloved Nemaunus, about a dozen miles distant. It is this trough that the Emperor Augustus once lined with cement.

Today, after twenty centuries, that lining remains one of the hardest substances in the world. The many years that it actually carried water only added to the encasing. The accretions of lime from the water itself gradually narrowed the specus until today, many centuries after the Pont du Gard was abandoned as an aqueduct, one finds that in places it is hard to walk through the narrowed space.

In the latter days of the Empire, the Romans grew extremely expert in the use of a form of concrete. They awoke gradually to the tenacious powers of lime rock, and before the great fabric of their high civilization crumbled, they were working quite extensively in a fabric that would outlive many civilizations-in a plastic stone that would not and could not crumble. The traces of their work in natural cement are still existent everywhere within the lands that they once

occupied. Their day was the early dawn of the Concrete Age.

For nineteen centuries the Concrete Age, although born, lingered in a dreary immaturity. For it was not until about one hundred years ago that an Englishman - Joseph Aspdin of Leeds - invented Portland Cement, so naming it from a widely used building stone from the Isle of Portland, to which his invention, once thoroughly set, bore a close resemblance in color.

Aspdin's method was through application to the cement raw materials of an intense heat. It had been of course, the traditional custom to burn the lime rock, to complete through fire the transformance of its carbonate of lime to an oxide of lime. But the kilns were crude and the process simple. Moreover, no matter how carefully one ground the rock before pouring it into the kilns, there was bound to result great quantities of hard slag, or "clinker", as it became known to the lime burners. This was sifted out from the finished product and thrown away. But its very presence was a constant annoyance.

Into Aspdin's mind there flashed the thought one day, a century ago, that perhaps the despised clinker might possibly contain certain cement qualities of real value. He began experimenting with it, pulverizing it into the finest sort of powder, and mixing it with water and a variety of aggregates of sand and stone. It responded to the tests. It made an artificial rock, of an appearance and a quality far superior from that obtained through the natural cement, with the clinker carefully eliminated.

Aspdin went further. He experimented with heats much in excess of those previously in use in lime-kiln practice upon

lime rock, until the product of the kiln was entirely clinker, which was just the thing he really desired.

Aspdin's chief contribution was not in the advanced use which he made of the clinker, but rather that he was the first one to realize the importance of the proper mixture of the materials --he made a science of the entire business.

But even with his work done, portland cement did not come into its own quickly. For while it was used in many parts of England and France as long ago as the 'fifties', it was not until 1870 that long experiments were made to produce portland cement in the United States.

Yet, once invented, portland cement moved but slowly toward the fulness of its possibilities. The process for its production was so intricate, the mechanisms for carrying out the process so much more complicated than those required for the production of natural cement or for slacking lime that men passed it by. Then, about a quarter of a century ago, some things began to happen. First and foremost important of all, prices of material things in the United States began to go up, as well as labor. This was especially true in the case of building materials, all save cement, in which there was a decided lowering of price, due in a large measure to the introduction of the rotary kiln. To an appreciable extent the Concrete Age has come into its own through dire labor necessities. The fact that in this day and age we of the United States are long in electricians and garage helpers and very short in good masons - either brick or stone - has been a powerful factor in its recent great growth.

In 1896 the annual production of portland cement in this

country first reached the then tremendous figure of a million barrels. Yet this was but a beginning. Four years later - at the beginning of the present century - it had come to 8,482,000 barrels in a single twelvemonth. These were still more beginnings. In 1920 more than 100,000,000 barrels of portland cement were manufactured in the United States. In 1922 the production was nearly 115,000,000 barrels. The Concrete Age has truly arrived.

The above is an extract from "Observations on an Outstanding American Industry" by Edward Hungerford.

The History of the Cement Industry in Michigan.

The first attempt to manufacture portland cement in the United States was made in Michigan in 1872, when an experimental vertical kiln plant was constructed in Kalamazoo, using marl and clay in the process. The venture was a failure and exercised little or no influence in the New York and Pennsylvania developments beginning in 1875.

After the failure of the Kalamazoo venture, no second attempt was made to re-establish the industry in Michigan until 1896 in which year the Peerless Portland Cement Co. erected a vertical kiln plant at Union City, Branch county, and began the successful manufacture of portland cement from marl and shale.

In 1897 the Bronson Portland Cement Co. erected a plant aat Bronson, Branch county, and in 1898 the Coldwater Cement Company, now the Wolverine Portland Cement Co., built plants at Coldwater and Quincy.

In 1900 Michigan with six plants attained sixth third place

in the production of cement in the United States.

Since 1896, thirty-six different cement plants have been projected or built in Michigan and eleven are in operation today. Of the eleven plants, five are reported to be using marl and clay, five using lime stone and clay or shale, and one using clay and the waste from the plant of the Michigan Alkali Co. All plants use coal as fuel, nine manufacture cement by the "wet process" and two by the dry process.

The Petoskey plant which was erected on the limestone deposits on Little Traverse Bay is pronounced "the best and most efficient wet process plant".-Geological Report for Michigan prior to 1921.

The boom years of the cement industry in Michigan were between 1899 and 1901, the production growing from less than 350,000 barrels in 1899 to more than 1,000,000 barrels in 1901. By 1920 the production had increased to more than 4,000,000 barrels per year.

In 1920 Michigan stood seventh in the production and shipment of cement.

OBJECT.

This thesis was taken up by the authors with two objects in view:

First, to determine if all the brands of Portland Cement manufactured in Michigan are made to conform to the standard specifications of the American Society for Testing Materials.

Second, to determine if possible from results obtained if there is any marked preference between the different brands of cement as to their quality and fitness, and if so in our opinion, which brand this is.

In order to insure that a fair sample of each brand would be secured, three sacks of each were obtained.

Atlas and Universal brands of cement were used as a controls because of their long standing as being among the best portland cements manufactured in this country.

DEFINITION.

Portland cement is the product obtained by finely pulverizing clinker produced by calcining to incipient fusion an intimate and properly proportioned mixture of argillaceous and calcareous materials, with no additions subsequent to calcination excepting water and calcined or uncalcined gypsum.

SPECIFICATIONS.

The specifications used are those of the American Society for Testing Meterials and are of two distinct parts, Chemical and Physical.

Chemical Properties.

Loss on ignition, per cent	4.00
Insoluble residue, per cent	0.85
Sulphuric anhydride (SO_3) per cent	2.00
Magnesia (MgO), per cent	5.00

These limits are not to be exceeded.

Physical Properties.

The specific gravity of cement shall be not less than 3.10 (3.07 for white Portland cement). Should the test of cement as received fall below this requirement a second test may be made upon an ignited sample. The specific gravity test will not be made unless specifically ordered.

The residue on a standard No. 200 sieve shall not exceed 22 per cent by weight.

A pat of neat cement shall remain firm and hard, and show no signs of distortion, cracking, checking, or disintegration in the steam test for soundness.

The cement shall not develop initial set in less than 45 minutes when the Vicat needle is used or 60 minutes when the Gillmore needle is used. Final set shall be attained within 10 hours.

The average tensile strength in pounds per square inch of not less than three standard mortar briquettes composed of one part cement and three parts standard sand, by weight, shall be equal to or higher than the following:

Age at Test Days	Storage of Briquettes	Tensile Strength lb. per sq. in.
7	1 day in moist air, 6 days in water	200
28	1 day in moist air, 27 days in water	300

The average tensile strength of standard mortar at 28 days shall be higher than the strength at 7 days.

SAMPLING.

Tests may be made on individual or composite samples as may be ordered. Each test sample should weigh at least 8 pounds.

The samples for tests made in this thesis were made by taking a scoop full from each sack and thoroughly mixing. The sample used in the test was then taken from this mixture. The average weight of cement taken from each sack was about 5 pounds making the total sample about 15 pounds.

CHEMICAL ANALYSIS.

Loss on Ignition.

Method.

One gram of cement shall be heated in a weighed covered platinum crucible, of 20 to 25 cc. capacity, as follows:

The crucible shall be placed in a hole in an asbestos board, clamped horizontally so that about three-fifths of the crucible projects below, and blasted at a full red heat for 15 minutes with an inclined flame; the loss in weight shall be checked by a second blasting for 5 minutes. Care shall be taken to wipe off particles of asbestos that may adhere to the crucible when withdrawn from the hole in the board. Greater

neatness and shortening of the time of heating are secured by making a hole to fit the crucible in a circular disk of sheet platinum and placing the disk over a somewhat larger hole in an asbestos board.

A permissable variation of 0.25 will be allowed, and all results in excess of the specified limit but within this permissable variation shall be reported as 4 per cent.

Remarks: In making this test in this thesis we were unable to get the platinum crucibles and fused silica crucibles of the specified size were used in their place. In heating the cement we made one blast of one-half hour rather than the two heats of 15 minutes and 5 minutes as specified. From our conversations with Chief Chemists at some of the plants in Michigan we found that this is the method that is used altogether in commercial testing.

Insoluble Residue.

Method.

To a 1 gram sample of cement shall be added 10 cc. of water and 5 cc. of concentrated hydrochloric acid; the liquid shall be warmed until effervescence ceases. The solution shall be diluted to 50 cc. and digested on a steam bath or hot plate until it is evident that decomposition of the cement is complete. The residue shall be filtered, washed with cold water, and the iilter paper and contents digested in about 30 cc. of a 5 percant solution of sodium carbonate, the liquid being held at a temperature just short of boiling for 15 minutes. The remaining residue shall be filtered,

washed with cold water, then with a few drops of hot hydrochloric acid, 1:9, and finally with hot water, and then ignited at a red heat and weighed as the insoluble residue.

A permissable variation of 0.15 will be allowed, and all results in excess of the specified limit but within this permissable variation shall be reported as 0.85 per cent.

Remarks: This test is very seldom made except on the re-check of cement which has been rejected. Statement made by Chief Chemist of a large cement company which has plants in several states, to the authors of this thesis.

Sulphuric Anhydride.

Method.

One gram of the cement shall be dissolved in 5 cc. of concentrated hydrochloric acid diluted with 5 cc. of water, with gentle warming; when solution is complete 40 cc. of water shall be added, the solution filtered, and the residue washed thoroughly with water. The solution shall be diluted to 250 cc., heated to boiling and 10 cc. of a hot 10 per cent colution of barium chloride shall be added slowly, drop by drop, from a pipette and the boiling continued until the precipitate is well formed. The solution shall be digested on the steam bath until the precipitate has settled. The precipitate shall be filtered, washed, and the paper and contents placed in a weighed platinum crucible and the paper slowly charred and consumed without flaming. The barium sulphate shall then be ignited and weighed. The weight obtained multiplied by 34.3 gives the percentage of sulphuric anhydride.

The acid filtrate obtained in the determination of the insoluble residue may be used for the estimation of sulfuric anhydride instead of using a separate sample.

A permissable variation of 0.10 will be allowed, and all results in excess of the specified limit but within this permissable variation shall be reported as 2.00 per cent.

Remarks: In making this test the authors used 10 cc. of concentrated hydrochloric acid and 10 cc. of water to dissolve the cement in instead of 5 cc. of each as specified. The authors spent one day in working and studying methods used in chemical analysis in a cement plant and we found that they used the amounts stated. The extra amounts used would of course have no effect on the amounts of constituents found, being used only to dissolve the cement.

Magnesia.

Method:

To 0.5 gram of the cement in an evaporating dish shall be added 10 cc. of water tp prevent lumping and then 10 cc. of concentrated hydrochloric acid. The liquid shall then then be gently heated and agitated until attack is complete. The solution shall be evaporated to complete dryness on a steam or water bath. To hasten dehydration the residue may be heated to 105 or even 200° C. for one half to one hour. The residue shall be treated with 10cc. of concentrated hydrochloric acid diluted with an equal amount of water. The dish shall be covered and the

solution digested for ten minutes on a steam bath or water bath. The diluted solution shall be filtered and the separated silica washed thoroughly with water. Five cubic centimeters of concentrated hydrochloric acid and sufficient bromine water to precipitate any manganese which may be present, shall be added to the filtrate (about 250cc). This shall be made alkaline with ammonium hydroxide, boiled until there is but a faint odor of ammonia, and the precipitated iron and aluminum hydroxides, after settling, shall be washed with hot water, once by decantation and slightly on the filter. Setting aside the filtrate, the precipitate shall be transferred by a jet of hot water to the precipitating vessel and dissolved in 10 cc. of hot hydrochloric acid. The paper shall be extracted with acid, the solution and washings being added to the main solution. The aluminum and iron shall then be reprecipitated at boiling heat by ammonium hydroxide and bromine water in a volume of about 100 cc., and the second precipitate shall be collected and washed on the filter used in the first instance if this is still intact. To the combined filtrates from the hydroxides of iron and aluminum, reduced in volume if need be, 1 cc. of ammonium hydroxide shall be added, the solution brought to boiling, 25 cc. of a saturated solution of boiling ammonium oxalate added, and the boiling continued until the precipitated calcium oxalate has assumed a well defined granular form. The precipitate after one hour shall be filtered and washed, then with the filter shall be

placed wet in a platinum crucible, and the paper burned off over a small flame of a Bunsen burner; after ignition it shall be redissolved in hydrochloric acid and the solution diluted to 100 cc. Ammonia shall be added in slight excess, and the liquid boiled. The lime shall then be repricipitated by ammonium oxalate, allowed to stand until settled, filtered and washed. The combined filtrates from the calcium precipitates shall be acidified with hydrochloric acid, concentrated on the steam bath to about 150 cc., and made slightly alkaline with ammonium hydroxide, boiled and filtered (to remove a little aluminum and iron and perhaps calcium). When cool, 10 cc. of saturated solution of sodium-ammonium-hydrogen phosphate shall be added with constant stirring. When the crystallin ammonium-magnesium orthophosphate has formed, ammonia shall be added in moderate excess. The solution shall be set aside for several hours in a cool plate, filtered and washed with water containing 2.5 per cent of NH_3. The precipitate shall be dissolved in a small quantity of hot hydrochloric acid, the solution diluted to about 100 cc., 1 cc. of a saturated solution of sodium-ammonium- hydrogen phosphate added, and ammonia drop by drop, with constant stirring, until the precipitate is again formed as described and the ammonia is in moderate excess. The precipitate shall then be allowed to stand about two hours, filtered and washed as before. The paper and contents shall be placed in a weighed platinum crucible, the paper slowly charred, and

the resulting carbon carefully burned off. The precipitate shall then be ignited to constant weight over a Mekerr burner, or a blast not strong enough to soften or melt the pyrophosphate. The weight of magnesium pyrophosphate oabtined multiplied by 72.5 gives the percentage of magnesia. The precipitate so obtained always contains some calcium and usually small quantities of iron, aluminum, and manganese as phosphates.

A permissable variation of 0.4 will be allowed, and all results in excess of the specified limit but within this permissable variation shall be reported as 5.00 per cent.

Remarks: In our tests for magnesia we did not perform the second precipitation and filtration as is called for in some places in this test. We followed more closely the method used in commercial practice which is the same in all ways except the second precipitation. In "Portland Cement" by Mead, in chapter on "Tests" the method is also given which does not call for the second precipitation but which is the same in all other respects. The time required for a magnesia test previous to the cooling of the solution is somewhat over four hours, therefore we believe our course in omitting the parts mentioned is justified., inasmuch as we only had four hours in a continuous period in which to perform this test.

TABLE OF RESULTS OF CHEMICAL TESTS.

Brand	Loss on Ignition	Insoluble Residue	Sulphuric Anhydride	Magnesia (MgO)
Atlas	2.41	.65	1.72	3.67
Alpha	.90	.70	1.18	1.66
Huron	1.41	.84	1.85	3.28
New Aetna	1.00	.85	2.00	2.34
Newaygo	.85	.64	1.80	3.84
New Egyptian	1.40	.85	1.08	3.26
Peerless	.73	.72	1.71	3.44
Petoskey	1.48	.76	1.92	3.91
Peninsular	.86	.85	1.74	3.93
Michigan	2.10	.76	1.95	3.75
Wyandotte	1.25	.85	1.86	2.72
Wolverine	1.16	.74	1.36	3.97
Universal	1.71	.73	1.40	4.02

As the above table shows, all of the Michigan cements come well under the specifications required by the American Society for Testing Materials, as to chemical analysis.

The results given should not be considered as being absolute, but they may be considered as being comparitive as all tests were run under the same conditions and with the same degree of accurateness throughout the entire tests. In the tests all computations were carried to the fourth place except in final result which was reduced to two decimal places as shown.

PHYSICAL ANALYSIS

Determination of Specific Gravity.

Apparatus: The determination of specific gravity shall be made with a standardized Le Chatelier apparatus. This apparatus is standardized by the United States Bureau of Standards. Kerosene free from water, or benzine not lighter then 62° Baume', shall be used in making this determination.

Method: The flask shall be filled with either of these liquids to a point on the stem between zero and one cubic centimeter, and 64 g. of cement, of the same temperature as the liquid, shall be slowly introduced, taking care that the cement does not adhere to the inside of the flask above the liquid and to free the cement from air by rolling the flask in an inclined position. After all the cement is introduced, the level of the liquid will rise to some division of the graduated neck; the difference between readings is the volume displaced by 64 g. of the cement.

The specific gravity shall then be obtained from the formula

$$\text{Specific gravity} = \frac{\text{Weight of cement (g.)}}{\text{Displaced volume (cc.)}}$$

The flask, during the operation, shall be kept immersed in water, in order to avoid variations in the temperature of the liquid in the flask, which shall not exceed 0.5° C. The result of repeated tests should agree within 0.01

The determination of specific gravity shall be made on the cement as received; if it falls below 3.10, a second

determination shall be made after igniting the sample as in the test for Loss on Ignition.

Remarks: In making this test the authors used kerosene free from water for the liquid. The kerosene was placed in the flasks and allowed to stand in water for one day before it was used.

The few samples of cement which did not at first pass this test went well under the test after the sample had been ignited.

Determination of Fineness.

Wire cloth for standard sieves for cement shall be woven (not twilled) from brass, bronze, or other suitable wire, and mounted without distortion on frames not less than 1½ in. below the top of the frame. The sieve frames shall be circular, approximately 8 in. in diameter, and may be provided with a pan and cover.

A standard No. 200 sieve is one having nominally an 0.0029 in. opening and 200 wires per inch standardized by the U. S. Bureau of Standards, and conforming to the following requirements:

The No. 200 sieve should have 200 wires per inch, and the number of wires in any whole inch shall not be outside the limits of 192 to 208. No opening between adjacent parallel wires shall be more than 0.0050 in. in width. The diameter of the wire should be 0.0021 in. and the average diameter shall not be outside the limits 0.0019 to 0.0023 in. The value of the sieve as determined by seiving tests made in conformity with the standard specifications for these

tests on a standardized cement which gives a residue of 25 to 20 per cent on the No. 200 seive, or on other similarly graded material, shall not show a variation of more than 1.5 per cent above or below the standards maintained at the Bureau of Standards.

Method: The test shall be made with 50 g. of cement. The seive shall be thoroughly clean and dry. The cement shall be placed on the No. 200 seive, with pan and cover attached, if desired, and shall be held in a slightly inclined position so that the sample will be well distributed over the seive, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the up stroke. The seive shall be turned every 25 strokes about one-sixth of a revolution in the same direction. The operation shall continue until not more than 0.05 g. passes thru in one minute of continuous seiving. The fineness shall be determined from the weight of the residue on the seive expressed as a percentage of the weight of the original sample.

Mechanical seiving devices may be used, but the cement shall not be rejected if it meets the fineness requirements when tested by the hand method just described.

Remarks: In making this test the authors used the hand method described in all cases.

Mixing Cement Pastes and Mortars.

The quantity of dry material to be mixed at one time shall not exceed 1000 g. nor be less than 500 g. The

proportions of cement or cement and sand shall be stated by weight in grams of the dry materials; the quantity of water shall be expressed in cubic centimeters. The dry materials shall be weighed, placed upon a non-absorbent surface, thoroughly mixed dry if sand is used, and a crater formed in the center, into which the proper percentage of clean water shall be poured; the material on the outer edge shall be turned into the crater by the aid of a trowel. After an interval of ½ minute for the absorption of the water the operation shall be completed by continuous, vigorous mixing, squeezing and kneading with the hands for at least one minute. During the operation of mixing, the hands should be covered by rubber gloves.

The temperature of the room and the mixing water shall be maintained as nearly as practicable at 21° C (70° F).
Remarks: In mixing the mortar used in these tests the temperature of the water was kept as nearly as possible at 21° C. but the temperature of the room could not be controlled and there was much variation.

Normal Consistency.

Apparatus: The Vicat apparatus consists of a frame bearing a movable rod, weighing 300 g., one end being 1 cm. in diameter for a distance of 6 cm., the other having a removable needle, 1 mm. in diameter, 6 cm. long. The rod is reversible, and can be held in any desired position by a screw, and has midway between the ends a mark which moves under a scale attached to the frame. The paste is held in

a conical, hard-rubber ring, 7 cm. in diameter at the base, 4 cm. high, resting on a glass plate about 10 cm. square.

In making the determination, 500 g. of cement, with a measured quantity of water, shall be kneaded into a paste, as described previous to this, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained about 6 in. apart; the ball resting in the palm of one hand shall be pressed into the larger end of the rubber ring held in the other hand, completely filling the ring with paste; the excess at the larger end shall then be removed by a single movement of the palm of the hand; the ring shall then be placed on its larger end on a glass plate and the excess paste at the smaller end sliced off at the top of the ring by a single oblique stroke of a trowel held at a slight angle with the top of the ring. During these operations care shall be taken not to compress the paste. The paste confined in the ring, resting on the plate, shall be placed under the larger end of the rod, the end of the rod brought in contact with the paste; the scale shall then be read and the rod quickly released. The paste shall be of normal consistency when the rod settles to a point 10 mm. below the original surface in ½ minute after being released. The apparatus shall be free from vibrations during the test. Trial pastes shall be made with varying percentages of water until the normal consistency is obtained. The amount of water required shall be expressed in percentage by weight of the dry cement.

The consistency of standard mortar shall depend on

the amount of water required to produce a paste of normal consistency from the same sample of cement. Having determined the normal consistency of the sample, the consistency of standard mortar made from the same sample shall be as indicated in the following table, the values being in percentage of the combined dry weights of the cement and standard sand.

PERCENTAGE OF WATER FOR STANDARD MORTARS.

Percentage of Water for Neat Cement Paste of Normal Consistency	Percentage of Water for One Cement, Three Standard Ottawa Sand.		
L%			
15	9.0	23	10.3
16	9.2	24	10.5
17	9.3	25	10.7
18	9.5	26	10.8
19	9.7	27	11.0
20	9.8	28	11.2
21	10.0	29	11.3
22	10.2	30	11.5

The Vicat apparatus seems to be used very little in cement plant labratories. The men who do the testing every day prefer the ball method for testing for normal consistency . It is claimed by them that their results are more accurate than with the Vicat apparatus. It is interesting to note that our results for normal consistency were higher in nearly every case than the normal consistency used for testing at the plants with which we came in contact.

Determination of Soundness.

A steam apparatus, which can be maintained at a temperature between 98 and 100° C. is recommended. The capacity of this apparatus may be increased by using a rack for holding the pats in a vertical or inclined position.

A pat of cement paste of normal consistency about 3 in. in diameter, ½ in. thick at the center, and tapering to a thin edge, shall be made on clean glass plates about 4 in. square, and stored in moist air for 24 hours. In molding the pat, the cement paste shall first be flattened on the glass and the pat then formed by drawing the trowel from the outer edge toward the center.

The pat shall then be placed in an atmosphere of steam at a temperature between 98 and 100° C. upon a suitable support 1 in. above boiling water for 5 hours.

Should the pat leave the plate, distortion may be detected best with a straight edge applied to the surface which was in contact with the plate.

Determination of Time of Setting.

The Gillmore needles were used in making this test. Method: The time of setting shall be determined as follows: A pat of neat cement paste about 3 in. in diameter and ½ in. in thickness with a flat top, mixed to a normal consistency, shall be kept in moist air at a temperature maintained as nearly as practicable at 21° C. The cement shall be considered to have acquired its initial set when the pat will bear, without appreciable indentation, the Gillmore needle

1/12 in. in diameter, loaded to weigh ¼ pound. The final set has been acquired when the pat will bear without appreciable indentation, the Gillmore needle 1/24 in. in diameter, loaded to weigh 1 pound. In making the test, the needles shall be held in a vertical position and applied lightly to the surface of the pat.

The initial set should not be acquired in less than 60 minutes with the Gillmore needle, and the final set should be acquired within 10 hours.

Tension Tests.

In making briquettes for tension tests the gang molds were used. Molds were cleaned before use and oiled enough to prevent cement from sticking to the sides.

The molds shall be made of non-corroding metal and have sufficient material in the sides to prevent spreading during molding.

The sand to be used shall be natural sand from Ottawa Ill,, screened to pass a No. 20 sieve and retained on a No. 30 sieve.

This sand, having passed the No. 20 sieve, shall be considered standard when not more than 5 g. pass the No. 30 sieve after one minute of continuous sieving of a 500 g. sample.

The sieves shall conform to the following specifications: The No. 20 sieve shall have between 19.5 and 20.5 wires per whole inch of the warp wires and between 19 and 21

wires per whole inch of the shoot wires. The diameter of the wire should be 0.0165 in. and the average diameter shall not be outside the limits of 0.0160 and 0.0170 in.

The No. 30 sieve shall have between 29.5 and 30.5 wires per whole inch of the warp wires and between 28.5 and 31.5 wires per whole inch of the shoot wires. The diameter of the wire should be 0.0110 in. and the average diameter shall not be outside the limits 0.0105 to 0.0115 in.

Immediately after mixing, the standard mortar shall be placed in the molds, pressed in firmly with the thumbs and smoothed off with a trowel without ramming. Additional mortar shall be heaped above the mold and smoothed off with a trowel; the trowel shall be drawn over the mold in such a manner as to exert a moderate pressure on the material. The mold shall then be turned over and the operation of heaping, thumbing and smoothing off repeated.

Tests shall be made with any standard machine. The briquettes shall be tested as soon as they are removed from the water. The bearing surfaces of the clips and briquettes shall be free from grains of sand or dirt. The briquettes shall be carefully centered and the load applied continuously at the rate of 600 lb. per minute.

Testing machines should be frequently calibrated in order to determine their accuracy.

Briquettes that are manifestly faulty, or which give strengths differing more than 15 per cent from the average value of all test pieces made from the same sample and broken at the same period, shall not be considered in determining the tensile strength.

Remarks: As the tensile strength is watched very closely by persons buying cement and as this is one of the most important tests we were very careful to see that all clips and briquettes were perfectly clean and that rolls were oiled. In no cases did the briquettes break in such a way as to appear due to dirt or grit on the machine or briquette.

Storage of Test Pieces.

The moist closet may consist of a soapstone, slate or concrete box, or a wooden box lined with metal. If a wooden box is used, the interior should be covered with felt or broad wicking kept wet. The bottom of the moist closet should be provided with non-absorbent shelves on which to place the test pieces, the shelves being so arranged that they may be withdrawn readily.

Unless otherwise specified all test pieces, immediately after molding, shall be placed in the moist closet, for from 20 to 24 hours.

The briquettes shall be kept in molds on glass plates in the moist closet for at least 20 hours. After 24 hours in moist air the briquettes shall be immersed in clean water in storage tanks of non-corroding material.

The air and water shall be maintained as nearly as practicable at a temperature of 21° C. (70°F).

Remarks: All test pieces were stored as specified except that we could not keep the air and water at the desired temperature. This may have had some effect on the tensile strength, but all briquettes were in the same storage tank so that the results are strictly comparative.

RESULTS OF PHYSICAL TESTS.

Brand	Specific Gravity	Fineness %	Soundness	Normal Consist.
Alpha	3.14	8518	OK	26.75
Atlas	3.15	80.7	OK	23.0
Huron	3.09 *3.23	81.6	OK	24
Michigan	3.06 *3.20	80.6	OK	27
New Aetna	3.08 *3.16	81.4	OK	27.25
Newaygo	3.10	80.4	OK	23
New Egyptian	3.10	85.7	OK	27
Peerless	3.11	78.04	OK	24.5
Peninsular	3.07 *3.18	83.6	OK	28.5
Petoskey	3.08 *3.21	89	OK	27
Universal	3.06 *3.17	84	OK	26
Wolverine	3.16	82.4	OK	23.5
Wyandotte	3.13	87.4	OK	24.5

* Refers to the test made for specific gravity after the sample had been ignited for 45 minutes.

RESULTS

TIME OF SET

Brand	Initial Set	Final Set
Alpha	4 hrs. 39 min.	9 hrs. 24 min.
Atlas	2 hrs. 16 min.	5 hr. 52 min.
Huron	4 hrs. 15 min.	8 hrs. 6 min.
Michigan	4 hrs. 20 min.	7 hrs. 50 min.
New Aetna	5 hrs. 9 min.	8 hrs. 8 min.
Newaygo	3 hrs. 15 min.	8 hrs. 15 min.
New Egyptian	4 hrs. 19 min.	9 hrs. 8 min.
Peerless	3 hrs. 35 min.	7 hrs. 40 min.
Peninsular	4 hrs. 56 min.	9 hrs. 32 min.
Petoskey	4 hrs. 25 min.	7 hrs. 23 min.
Universal	5 hrs. 28 min.	8 hrs. 43 min.
Wolverine	3 hrs. 54 min.	8 hrs. 00 min.
Wyandotte	3 hrs. 5 min.	7 hrs. 23 min.

From the above results it is seen that all brands come well within the limits of 60 minutes to 10 hrs.

TENSILE STRENGTH TESTS.

Brand	Period 7 day	28 day	3 mo.	6 mo.	9 mo.	1 yr.
Alpha	350	300				
	310	300				
	320	315				
Av.						
Av.	326	325				
Atlas	250	335				
	210	275				
	225	310				
	215	305				
Av.	222	306				
Huron	210	305				
	230	335				
	250	325				
	190	300				
Av.	220	320				
Michigan	255	320				
	220	275				
	185	315				
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Av.	220	305				
New Aetna	300	345				
	250	310				
	240	375				
	275	300				
Av.	266	320				
Newaygo	255	350				
	210	440				
	207	380				
	260	410				
Av.	235	395				
New Egyptian	245	350				
	200	345				
	240	340				
	210	340				
Av.	224	344				
Peerless	240	305				
	230	305				
	185	340				
		325				
Av.	218	326				

TENSILE STRENGTHS.
(Continued)

Brand	Period					
	7 day	28 day	3 mo.	6 mo.	9 mo.	1 yr.
Peninsular	265	365				
	230	360				
	235	320				
	295	360				
Av.	256	351				
Petoskey	250	320				
	295	340				
	290	305				
	310	355				
Av.	286	330				
Universal	260	280				
	210	315				
	180	315				
	--	350				
Av.	216	315				
Wolverine	180	280				
	200	270				
	190	280				
	190	260				
Av.	192	272				
Wyandotte	190	275				
	180	275				
	210	260				
	230	285				
Av.	202	274				

GENERAL REMARKS.

It was the original intention of the authors to make a complete analysis of the chemical properties of the cements which would include determination of silica, iron and aluminum, calcium, and magnesia. This would have made a more complete comparison possible. Due to lack of time we were forced to abandon this idea after a few had been completly analysed.

Cements high in aluminum reach their full strength in less time than low aluminum cements. With a complete analysis we might have been able to predict with some degree of accuracy how the curve of tensile strengths would appear for a period of one year. Without this analysis not much can be said with only the 7 and 28 day tests completed.

Cements high in aluminum also set quicker than low aluminum cements.

Cements which are high in silica are slow setting but show a good tensile strength, also a progressive gain in tensile strength. It was our intention, with a complete chemical analysis, to check the percentage of silica against the time of setting.

CONCLUSION.

In making our conclusions, since all brands passed the specifications required in all tests except tensile strength, and since this test together with soundness is watched most closely by purchasers of cement, we are using these tests as a basis for comparison. As all the cements passed the soundness test in a satisfactory manner, this makes the comparison rest on the tensile strength.

The Newaygo brand of cement shows a high tensile strength for the seven and 28 day tests, in which the 28-day test shows a considerable increase over the 7-day test. The result of the time of setting shows very good for working conditions.

The Alpha brand of cement shows a very high 7-day strength but there is no gain in the 28-day strength.

The Wolverine brand of cement is the only one which failed in tensile strength. This brand failed in both the 7-day and 28-day tests. Seven of the eight briwuettes failed to come up to the required strength.

All of the Michigan brands of cement, with the one exception, appear to stand up very favorable in comparison with the two eastern brands used in these tests, and passed all the specifications required by the American Societh for Testing Materials.