THE EFFECT OF pH ON THE WORKABILITY

OF CONCRETE

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ABSTRACT

It was believed that the pH of concrete would affect the workability. The effect of pH on the workability was determined experimentally by adding chemical admixtures to a cement mortar mix, measuring the pH and workability.

pH was measured by a Beckman Glass Electrode pH Meter equipped with a standard Calomel electrode and a special type "42" glass electrode. The use of the type "42" glass electrode made it possible to place the electrodes in wet cement mortar without damage.

The workability was determined by measuring the penetration of a three inch diameter cylinder with a hemispherical tip. This ball penetration measure of workability has a linear relationship to that measured by the slump test.

From these tests it was found that the pH has no effect on the workability of cement mortar. The admixtures changed both the pH and the workability, however, the variations in pH and workability were independent of each other. It is believed that the major factor which affects the workability of concrete is the dispersing effect of the admixture on the cement.

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INTRODUCTION

The workability of concrete, if a properly designed mix is used, can affect the cost of a concrete structure more than any other one item. Workability is defined as the ease with which given proportions of rock, sand, cement, and water can be mixed into concrete and subsequently handled, transported, and placed with a minimum loss of homogeneity. (1)

Assuming the proper proportion of fine and coarse aggregate has been selected and the proper water-cement ratio for the desired strength of the concrete has been chosen, then a variation in cost is affected by the following: First, the amount of cement paste used for a given amount of aggregate; and second, the cost of placing the concrete, which depends on the ease of placing or simply the workability. As greater amounts of cement paste are used, the concrete cost rises; but with this larger amount of cement paste, the mix becomes more workable and therefore the cost of placing the concrete will be reduced. The quantity of cement that is required for strength is usually smaller than that needed for good workability. If for a mix, an inexpensive admixture can be used to increase the workability without decreasing the strength or give any other undesirable effects, the concrete can be placed at a lower cost than before the admixture was added. The question still remains unsolved as to the reason why certain admixtures cause workability to increase. It was believed that the resulting pH of the concrete might be a criterion of their effectiveness.

pH is defined as the negative logarithm of the concentration of the hydrogen ion in gram atoms per liter. For a neutral solution the hydrogen ion concentration is equal to 10^{-7} , therefore, the pH value is 7. Acid solutions have a pH below 7 while basic solutions have a pH above 7.

The idea that the pH of concrete may affect its workability arose when the composition of a commercial chemical admixture was obtained and a chemist stated the admixture would only change the pH of the concrete. It is, therefore, the purpose of this thesis to determine if the pH of concrete has any effect on its workability.

PORTLAND CEMENT

Composition and Constitution of Portland Cement

The chemical analysis of portland cement reveals three fundamental constituents; lime, silica, and alumina. In addition, there are generally small proportions of iron oxide, magnesia, sulphur trioxide, alkalies, and carbon dioxide. The usual limits in oxide composition for normal cements produced in the United States, from published analyses of a large number of cements, are given in the following table: (2)

Oxide	Usual Percentage Limits Normal Portland Cement
Lime (CaO)	62-65
Silica (SiO ₂)	19-22
Alumina (Al ₂ O ₃)	4-7
Iron Oxide (Fe ₂ O ₃)	2-4
Magnesia (MgO)	1-4
Sulphur Trioxide (SO ₃)	1.5-2
Alkalies (K ₂ O - Na ₂ O)	0.3-1
Water (H_2O) and Carbon Dioxide (C	O ₂) 1-3

In well-burned portland cement, it has been shown that there are four principal mineral compounds, which are: Tricalcium silicate $3CaO \cdot SiO_2$ symbolized by C_3S ; Dicalcium silicate $2CaO \cdot SiO_2$ symbolized by C_2S ; Tricalcium aluminate $3CaO \cdot Al_2O_3$ symbolized by C_3A ; Tetracalcium alumino- $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ symbolized by C_4AF .

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Under various circumstances the alumina may be partially combined as $5CaO \cdot 3Al_2O_3$, symbolized by C_5A_3 , and some uncombined or "free" lime may be present.

Setting and Hardening of Portland Cement

The setting and hardening of portland cement is a very intricate process and many of the details are still uncertain. However, there seems to be good evidence from the study of Bogue and Lerch (3) in 1933, that the following principles govern the set of cements.

Initial set is a formation of either tricalcium aluminate hydrate or tricalcium silicate hydrate. The time required for the initial set being governed by the time necessary for either one or the other hydrate to appear. In high tricalcium aluminate cements with no admixtures to retard the set, the tricalcium aluminate goes into solution very rapidly and the tricalcium aluminate hydrate is formed rapidly. This formation may be of sufficient magnitude to produce rigidity of the cement paste and it is said to have a "flash set". Rapid hydration of tetracalcium alumino-ferrite may also cause a flash set. Two to three percent of gypsum reacts with the hydrated aluminates to form fine crystalline needles of calcium sulphoaluminate, which action retards crystallization of the tricalcium aluminate and thus delays the set. With the calcium aluminate hydration retarded, the more slowly reactive tricalcium silicate will be given time to go into solution and precipitate as the calcium silicate hydrates. In this case, the latter hydrate will establish the set and the structure of the cement paste. The hydrolysis of the tricalcium silicate grains is well under way in 24 hours and has a marked advance in 7 days.

Dicalcium silicate is hydrolyzed into a gel at a very low rate, its influence on strength and hardness is small at ages less than a month but at one year it contributes proportionately as much strength as does the tricalcium silicate.

A decrease in the OH ion concentration, lower pH, caused by the presence of soluble calcium salts will accelerate the solution of the calcium silicates. Also, they increase the Ca ion concentration causing a more rapid precipitation of dicalcium silicate hydrate. (4)

Dissolved aluminates placed in a cement paste, however, retard the formation of the dicalcium silicate hydrate by precipitating a film of insoluble material around the grains of the silicates, (4), thus acting as a retarder for the dicalcium silicate hydrate.

The chemical action of admixtures on cement paste are very complicated and it is almost impossible to predict their

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effect on the workability of concrete. A method to determine the effect of pH on the workability of concrete will be to change the pH by various chemical admixtures and measure the corresponding change in workability.

TEST MIX

A cement and sand mortar mix of 1 to 3 parts by weight was used for all tests, each batch containing 9 pounds of cement and 27 pounds of sand. The amount of water used was slightly different for some batches. This was done to keep the initial workability approximately the same when there was a variance in the cement used, also when a commercial admixture was used which increased the workability considerably. See Tables I to V for data on amount of water used for various chemicals mixed.

Portland Cement manufactured by the California Portland Cement Company of Colton, California, was used. Cement used in the study was purchased at two different times, therefore, making the variance mentioned above. Tests 1 to 17 were run from the first purchase and tests 18 to 21 from the second purchase.

Two separate supplies of San Gabriel river sand were used, tests 1 to 17 from the first supply and 18 to 21 from the second. The sand was surface dried before splitting and remained so until used. It was split by standard methods until each part weighed 27 pounds, it was then sacked and stored until used.

The fineness modulus of the sand for tests 1 to 17 was 3.52 and for tests 18 to 21 was 3.54, see Figure 1, Page 8, for the fineness gradation of the sand.

METHODS OF MEASURING pH AND WORKABILITY

pН

A Beckman Glass Electrode pH Meter, Model M, was used to measure the pH of the mortar as it was mixed and tested. A special Beckman type "42" glass electrode was used with a standard Calomel electrode which allowed the electrodes to be placed in the wet mortar without damage. The type "42" glass electrode has a small 5 mm. pH-sensitive glass hemisphere which gives the electrode extraordinary strength. This electrode can be used for measurements in the range 1 to 14 pH, correction for sodium-ion is not usually required at room temperatures.

Workability

Workability, defined as ease with which concrete can be handled, transported, and placed with a minimum loss of



Figure 1 Gradation of the sound used

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homogeneity, is intangible and relative and can not be measured as pure workability as it depends on a number of properties which cannot be satisfactorily measured; there is, in fact, no general agreement as to what all these properties are. However, there have been several methods used to approximate a measurement of workability. Some of these methods for approximating the measurement of workability are:

- 1. Slump test (5)
- 2. Flow test (5)
- 3. Powers remolding test (6)
- 4. Compacting factor test (7)
- 5. Wigmore consistometer test (8)
- 6. Ball penetration test (9)

These tests all have advantages and disadvantages when used to approximate a value for the workability of concrete. The slump and flow tests have been standardized by The American Society of Testing Materials in specifications D 138-32T and C 124-36T respectively. The slump test has been used in the field to a greater extent than the flow test inasmuch as it is easier to use and less equipment is required. It is, therefore, generally accepted for its true representation of an approximation of the workability of concrete. The Powers remolding test and the compacting factor test have not been standardized as have the slump and flow tests. Also, they are not used in the field because they require equipment too elaborate for fast and easy measurements of workability. Values of workability measured by the slump test are equally as usable as those obtained from the Powers remolding and compacting factor tests.

The Wigmore consistometer test was introduced in England and little information is obtainable concerning its test results. However, in the test referred to in reference (8) no correlation exists between the slump test and the Wigmore consistometer test. For different harshness of mix with the same slump, different consistency factors were measured. Also, preliminary stiffening of the concrete occurred within 3 to 4 minutes after discharge from the mixer and caused the consistency factor to increase while the slump remained constant.

The ball penetration test measures the same properties of the concrete as does the slump test. Apparatus used by Kelly and Haavik (9) gave a linear relationship between the slump and penetration, one inch of penetration corresponding to two inches of slump. The greatest advantage of this test over the slump test is the ease of measuring the workability of con crete.

Since the ball penetration test is a simpler method to use to measure the workability of concrete than any of the other methods explained above; and since this test has very good correlation with the slump test, the ball penetration test was used. However, in these tests the size of the penetrometer was changed from that used by Kelly and Haavik (9) to enable small batches of mortar to be used.

The penetrometer, which was used, can be seen in Figure 2, Page 12. The plunger has a 3 inch hemispherical tip and weighs 10 pounds including the handle. Measurements were taken with the concrete in a 10 inch diameter cylinder 8 inches high.

Measuring the workability consisted of observing the penetration of the plunger into the level surface of cement mortar. The sliding stirrup resting on the edge of the container gives a reference line enabling the penetration to be read off the graduated handle.

TEST PROCEDURE

The mortar was thoroughly mixed by hand with a trowel before being placed in the testing container. After the mortar was placed in the container, it was jarred five times by means of a drop table which had drops of 3/8 inch. The excess mortar was struck off with a straight edge, and again jarred by two 3/8 inch drops. This procedure was followed to insure uniform



Figure 2 Ball Penetrometer

placement of the mortar in the testing container. The workability was measured by use of the penetrometer, after which the pH was measured. The mortar was then remixed, placed in the testing container and another set of measurements taken. For each amount of admixture placed in the mortar, three sets of measurements were taken and the average value of these measurements was used.

A linear relationship existed between the slump and the penetration with mortar placed in the testing container and measured as stated above. This can be seen in Figure 3, Page 14. Workability from this point on will mean a measurement of the properties of mortar determined by the penetration test.

Various chemicals were added to the concrete as admixtures to bring about a change in the pH and workability. Other effects which the chemical admixtures have on the mortar were not considered as they are not within the scope of this thesis.

Chemicals were added as follows: A batch of mortar was mixed and the initial workability and pH were measured three times, each at intervals of approximately two minutes. Then a small amount of chemical was added, the mortar was thoroughly

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mixed and the workability and pH measurements were again taken three times, then more of the same chemical was added and the workability and pH measurements were taken again. This process was repeated several times. The amount of the chemical added as an admixture was proportional to the chemical's molecular weight, usually 0.2 of the molecular weight in grams being added each time. This procedure was followed for each admixture.

TEST RESULTS

The values of the pH and workability varied in the different batches of mortar just after mixing and before an admixture was added even though care was taken to obtain a similar mix at all times. Causes for the variations are not known and have not been determined as they were not considered to be of sufficient importance. The pH and workability of a batch of plain mortar, with no admixture, did not vary with time. This is seen in test 14, where the mortar was mixed and the pH and workability were measured over a period of time. Before each measurement the mortar was mixed for one minute. The pH and workability were measured only once for each time after the mixing. Therefore, there will be a larger spread in the data than if average values were used.

Time after mixing (Minutes)	рН	Workability (Inches of penetration)
0	12.35	1 3/16
11	12.45	1 1/4
26	12.35	1 5/8
37	12.35	1 1/4
47	12.35	1 5/16
56	12.35	1 1/4

Test results are shown in the following table:

With the addition of an admixture, the pH and workability changed slightly with time, however, only around an average value. Test 15 was run similar to test 14, however, with 85 grams of sodium nitrate used as an admixture. The test results are as follows:

Time after mixing (Minutes)	рH	Workability (Inches of penetration)
No NaNO ₃ added	12.2	1 1/4
85 Gr. NaNO ₃ added		
13	12.05	17/8
27	12.00	2 1/4
37	12.10	2 3/16
48	12.00	2
58	12.15	2 1/16
Average value with a	dmixture	
	12.06	2 1/16

This test shows that with sodium nitrate used as an admixture, the pH and workability varied slightly with time

around an average value. However, since only one set of readings was taken at a time, it is justifiable to assume that the differences were errors in making the measurements. It will, therefore, be assumed that when an average value is used the pH and workability will not vary with time. Also, it will be assumed that neither the pH nor the workability will change with time when other admixtures are used. This is partially verified when the three separate sets of readings are taken and these readings vary around an average value instead of continually increasing or decreasing.

With neither the pH nor the workability depending on the time after which an admixture is added, it can be seen that the method of adding a small portion of admixture at intervals will not change the test results as only the effect pH has on the workability of mortar is of interest in this study.

Potassium nitrate, potassium hydroxide and potassium carbonate, used as admixtures, increased the pH of the mortar. Potassium nitrate and potassium hydroxide increased the workability while potassium carbonate decreased the workability. When compared with potassium hydroxide, potassium nitrate increased the workability to a greater extent but had a smaller effect on the pH. See Figure 4, Page 24, for the test results.

It should be remembered that the amount of admixture added is increased with time. Each point along the curve, in the direction of the arrow, represents an addition of admixture from that used at the previous point. For the amounts of admixture used at the various points, see Tables I to V. This procedure was used for the graphs on Figures 4, 5, 6, 7, and 8.

Sodium nitrate, sodium hydroxide and sodium carbonate used as admixtures, decreased the pH of the mortar. The workability was increased by both sodium nitrate and sodium hydroxide while it was decreased by sodium carbonate. Sodium hydroxide increased the workability to a greater degree than sodium nitrate. This is just the reverse as compared with the use of potassium salts. Sodium nitrate decreased the pH more than sodium hydroxide. See Figure 5, Page 25, for the test results.

Calcium salts used as an admixture did not react in the same manner as either the potassium or the sodium salts. Calcium nitrate, calcium hydroxide and calcium chloride increased the workability of the mortar but calcium carbonate decreased the workability. The pH was decreased by calcium nitrate, calcium chloride and calcium carbonate while calcium hydroxide increased the pH. See Figure 6, Page 26, for test results. Aluminum and magnesium hydroxide had very little effect on either the pH or the workability. Magnesium carbonate did not change the pH appreciably but did decrease the workability. Magnesium chloride decreased the pH and increased the workability. See Figure 7, Page 27, for test results.

From the test results of the different chemicals it will be noted that the pH alone did not affect the workability of the mortar, nor was it the positive or negative ion of the chemical which changed the pH and workability. The workability was apparently affected by the total chemical effect which the admixture had on the mortar. The only visible chemical action of the various admixtures on the mortar was with the addition of calcium nitrate, when ammonia gas was given off. It was later found the calcium nitrate was impure and the ammonia came from the impurity.

Two commercial admixtures, Pozzolith and Tricosal, were used. These admixtures were used as recommended by the manufacturer and had little effect on the pH. However, both changed the workability a large amount. See Figure 8, Page 28. Pozzolith, however, had more effect on the pH than did the Tricosal. The Pozzolith decreased the pH linearly while the Tricosal tended to increase the pH slightly and then remain constant. With three times the recommended amount, the pH decreased. This is shown in the following table:

Percent admixture added by weight	p H
Pozzolith - Test 18	
0 1.2	12.35
Tricosal - Test 19	
0 0.06 to 0.38 1.1	12.3 12.35 12.3

Amount recommended by manufacturer: Pozzolith 1 percent by weight Tricosal 0.18 percent by weight

Tricosal is a calcium salt of Protalbinic and Lysalbinic acids which results from the hydrolysis of albumin. (10) Because it is believed the presence of proteins will tend to pull the pH toward neutral position when either in alkalin or acid solution, and since Tricosal is a protein product, admixtures of two raw proteins were used. Dried rabbit serum obtained from rabbits blood and Bovine \mathcal{K} globulin obtained from steer's blood were the protein admixtures used. Results of these can be seen in Figure 8, Page 28. It will be noted that in both cases the workability was decreased. The pH was decreased with the addition of rabbit serum and increased with Bovine \mathcal{K} globulin. It should be noted that with both tests, all the raw protein did not go into solution, as small globules of protein were observed in the dried mortar. As a final test, the commercial detergent, Tide, was used as an admixture. Tide is a dispersing agent. The test results from this can be seen in Figure 8, Page 28. It is interesting to note that the curve of workability versus pH was very similar to, and was between those for the commercial admixtures of Pozzolith and Tricosal. With more than 0.25 percent by weight of Tide used as an admixture, the set was retarded slightly.

PRELIMINARY INVESTIGATION OF COMPRESSION STRENGTH OF MORTAR WITH THE ADDITION OF ADMIXTURES

Compression tests were made on mortar using Pozzolith, Tricosal, and Tide as admixtures. All mixes for the compression test had the same water-cement ratio. The compression tests were made on standard 3 inch diameter cylinders which had been cured for a period of seven days. The percent of admixture added, the pH, and the reduction in strength caused by the admixture can be seen in the following table. After adding the admixture, the mortar was too workable for the ball penetration test to be used to measure the workability of the mortar.

Admixture	% by Weight	pH	Reduction in Strength
No admixture	0	12.4	0
Pozzolith	1	12.1	1 %
Tricosal	0.18	12.35	11%
Tide	- 1	12.1	17%
Tide	0.5	12.1	25%

From these preliminary investigations it can be seen that some admixtures, even though they cause a good workable mix, cannot be used inasmuch as the strength of the mortar is greatly decreased.

Tide, which is a good dispersing agent, seems to have a secondary effect which decreased the strength of the mortar. Two tests were made and the comparison of them shows that with an increased amount of admixture, the strength of the mortar also increased.

When the cylinders with Tide used as an admixture were removed, a thin layer of cement adhered to the forms, leaving the outside of the test specimens with the appearance of sandstone. This is believed to be caused by the reaction of Tide with the oil used on the cylinder forms.

CONCLUSION

From these test data, it is concluded that pH has no effect on the workability.

Various chemicals used as admixtures to change the pH of the cement mortar also changed the workability; however, the pH and workability changed independently of each other. The change in pH and workability could not be traced to any single chemical component, but was the result of the chemical action the admixture had on the mortar. Commercial admixtures had a greater effect on the workability than the simple chemical admixtures, however, the pH was changed less. Before the test, it was believed that Tricosal, being manufactured from proteins, would change the pH considerably, but it remained almost constant. Various raw proteins had different effects on the pH; rabbit serum decreased the pH but Bovine f globulin increased the pH while the workability was decreased by both.

Manufacturers of the commercial admixture used in these tests, state that their admixtures act as dispersing agents. Tide, which is also a dispersing agent, changes the workability to a similar degree in comparison to the commercial admixture, however, the strength was decreased considerably. It is, therefore, believed the major factor which affects the workability of concrete is the dispersion of the cement particles and not the pH of the concrete.

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Figure 6 Workability vs. pH. with calcium salt admixtures used

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	Total Admixture Added		
Point No.	Proportion of	Weight	
Shown on graph	Molecular Weight	(grams)	
Potassium Carbo	nate Molecular weight 138.21 - Water added: 2700 ml.	Test No. 2	
1	0	0	
2	0.2	27.6	
3	0.4	55.2	
Potassium Nitrat	e Molecular weight 101.11 - T	est No. 3	
	water added: 2700 ml.		
1	0	0	
2	0.2	20.2	
3	0.4	40.4	
4	0.6	60.6	
5	0.8	80.8	
6	1.0	101.0	
7	1.4	141.4	
Potassium Hydro	xide Molecular weight 56.11 - Water added: 2700 ml.	Test No. 4	
1	0	0	
2	0.2	11.2	
3	0.4	22.4	
4	0.6	33.6	
5	0.8	44.8	
6	1.0	56.1	

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Tante	11

	Total Admixture Added		
Point No.	Proportion of	Weight	
Shown on graph	Molecular Weight	(grams)	
Sodium Nitrate Molecu	lar weight 85.01 - Te	st No. 1	
W	ater added: 2650 ml.		
1	0	0	
2	0.2	17	
3	0.4	34	
4	0.6	51	
5	0.8	68	
6	1.0	85	
Sodium Hydroxide Mole	ecular weight 40.01 -	Test No. 5	
V	later added: 2700 ml.		
1	0	0	
2	0.2	8	
3	0.4	16	
4	0.6	24	
5	0.8	32	
6	1.0	40	
Caliner Combonata Mal	aular maight 104 01	Test No. 6	
Sodium Carbonate Moto	Water added: 2700 ml	· Test No. 0	
V	valer auleu. 2100 ml.		
1	0	0	
2	0.2	21	

	-	-	-	
11.4	a h	10	- N	11
1 (2 L	1C	-	77

	Total Admixture Added	
Point No.	Proportion of	Weight
Shown on graph	Molecular weight	(grams)
Λ.		
Calcium Carbonate	Molecular weight 100.09 ·	- Test No. 7
W	ater added: 2700 ml.	
1	0	0
2	0.2	20
3	0.4	40
4	0.6 60	
5	0.7	70
Calcium Nitrate Mo	olecular weight 164, 10 - T	est No. 12
Valeranii Milliade Mil	Vater added: 2700 ml.	
1	0	0
2	0.2	32.8
3	0.4	65.6
4	0.6	98.4
Calcium Hydroxide	Molecular weight 74.10 -	Test No. 13
, v	Vater added: 2700 ml.	
1	0	0
2	0.2	14.8
3	0.4	29.6
4	0.6	44.4
5	0.8	59.2
6	1.0	74.0
Calcium Chloride M	íolecular weight 111.00 - 1	Test No. 21
T	Water added: 2500 ml.	
1	0	0
2	0.2	22.2
3	0.4	44.4
4	0.8 66.6	
5	1.0 111.0	

2	Total Admix	ture Added			
Point No	Proportion of	Weight			
Shown on graph	Molecular Weight	(grame)			
JIIOWII OII graph	Moleculai Weight	(grams)			
Magnesium Carbona	ate Molecular weight 84.33	3 - Test No. 8			
Water added: 2700 ml.					
1	0	0			
2	0.2 16.9				
3	0.4	33.8			
4	0.6	50.7			
Magnesium Hydroxi	de Molecular weight 58.64	4 - Test No. 9			
W	ater added: 2700 ml.	*			
1	0	0			
2	0.2	11.7			
3	0.4	23.4			
4	0.6	35.1			
5	0.8	46.8			
6	1.0	58.3			
7	1.4	81.7			
Magnesium Chloride W	e Molecular weight 95.24 ater added: 2700 ml.	- Test No. 11			
1	0	0			
2	0.2	19.1			
3	0.4	38.2			
4	0.6	47.2			
5	0.8	76.3			
6	1.0	95.3			
7	1.2	104.4			
Aluminum Hydroxid	e Molecular weight 78.00	- Test No. 10			
W	ater added: 2700 ml.	1000100010			
	<u>^</u>	0			
1	0	0			
2	0.2	15.6			
3	0.4	31.2			
4	0.6	46.8			
5	0.8	62.4			
6	1.0	78.0			
7	1.4	109.2			

Table V

Point N	10.	% Admixture	Total Admixture Used	
Shown on	graph	Used	Weight (grams)	
Pozzolith	Recommen	nded use - 1% b	y weight - Test 16 ml.	
1		0	0	
2		0.33	12.2	
Tricosal Recommended use - 0.18% by weight - Test 17 Water added: 2600 ml.				
1		0	0	
2		0.07	2.7	
3		0.13	5.3	
4		0.23	9.5	
Rabbiț S er	um – Te	st 18		
	Wat	er added: 2500 r	ml.	
1			0	
2			5	
3			10	
4			19.6	
Bovine & Globulin - Test 19				
	Wat	er added: 2500 r	nl.	
1			0	
2			5	
3			10	
Tide -	Test 20		· ·	
	Wat	er added: 2500 p	ml.	
1			0	
2			5	
3			10	
4			20	

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