A study of the effect of quinhydrone on the time of set of silicic acid gells and of the effect of silicic acid gel on the determination of P_h by the quinhydrone electrode.

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Introduction-

The studies discussed in this report are supplementary to the work being done at Union College on silicic acid gels. In as much as much data has been taken in this work using P_h as determined by the quinhydrone electrode, it was desired that a study be made of the effect of quinhydrone on the gel and of gel on the quinhydrone determinations. The P_h of gels during their setting has been worked on and the first part of the experiment deals with the effect of quinhydrone on the time of set of a silicic acid gel. Then it was desired to know wether a gel had any effect on the determination of the P_h by means of the quinhydrone set up. It had been thought that there was no effect in either case but there was no experimental evidence to show proof.

Apparatus-

The quinhydrone electrode which is used in measuring the P_h of a solution consists of a calomel half cell and a platinum electrode both dipping in the solution, saturated with quinhydrone, whose P_h is to be determined. A potentiometer is used to measure the E. M. F. of the cell. This potential is related to the P_h in the following relationship-

$$E = -\frac{RT}{NF}P_h + C$$

This is a linear relation and so the P_h may be easily read off from the graph when the value of the E. M. F. has been found. It is found that it takes several minutes for the solution to become saturated with the solid quinhydrone. This may be easily remedied by using a solution of quinhydrone in acetone. Much of the work in other experiments has been done using the solid substance so this method was adhered to in the present case.

Effect of quinhydrone on the time of set of silicic acid gel-

To study the effect of quinhydrone on the time of set of a gel a number of gels of the same composition were mixed at the same time and allowed to set in a water bath. Some of the gels contained an excess of quinhydrone and the others contained none. The solution of water glass used was made up from the Philadelphia Quartz Co. "E" brand, diluted with distilled water to 1.06 specific gravity. The first two runs

were made using 0.75 normal acetic acid and the rest with 2 normal acetic acid. The solutions were brought to a temperature of 25 degrees before mixing and the gels were kept in the water bath at 25 degrees while setting. Runs were made covering both acid and basic gels.

Effect of washed gel on P_h determinations-

The idea followed in this part of the work was to add a washed gel to a buffer solution of known P_h and note whether the gel affected the determination of the P_h with the quinhydrone electrode.

The first problem was to obtain buffer solutions to cover a P_h range of 4 th 8. The first set tried was McIlvaines Standard solutions which consisted of varios mixtures of 0.1 molar citric acid and 0.2 molar disodium phosphate. This covered the desired range but it was found that a mold formed in the citric acid after a few days destroying its usefullness. The only way of preventing the mold formation was by sterilization which was impractical. The buffers finally used were composed of mixtures of 0.2 acid potassium phthalate and 0.2 potassium hydroxide which covered a P_h range of 4 to 6.2 and mixtures of 0.2 molar sodium dihydrogen phosphate and 0.2 molar sodium hydroxide which covered the

range from 5.8 to 8. These solutions were found to be entirely satisfactory.

The producing of a gel which had been washed enough to take out the excess acid or base and their salts was found to be quite difficult. The gel had to be made either acid or basic in order that it would be possible to tell when the gel had been washed till it was neutral and all the salts had been removed. The gel was set and then broken up into small pieces to facilitate washing. It was then thoroughly mixed with distilled water. This water was renewed frequently but it took about two weeks to obtain the neutral gel. The criterion of neutrality of the gel was taken as that point when the wash water gave a P_h of 7. This was further complicated by the fact that the distilled water used rarely gave a neutral reading.

When a neutral gel was obtained, it was freed of most of the wash water and broken into small pieces. The gell was then added in lots of 5c.c. to a buffer solution of known P_h and the P_h redetermined with the quinhydrone set up. The amount of buffer used in each case was 50c.c. and the gell was well stirred and allowed to settle before each determination. A control run was made for each test run. This consisted of adding distilled water in 5c.c. lots to a buffer solution

of the same P_h as that of the solution to which gel was added. For some time the runs were giving results which were not expected. The addition of the gel caused a considerable rise in the P_h, the increase seeming to be proportional to the amount of gel added. Uppn investigation however it was discovered that the gel had not been washed long enough and was still basic. When a neutral gel was finally obtained several runs were made with the small amount of gel that remained and the results found to be satisfactory. Work is being continued and more complete data will be appended later.

Table #1

Run No.	C.C.Acid	C.C.Waterglass	Qh.	Order Set
1	20	30	0	1
	"	"	x	2
	"	"	x	3
2	20	30	0	3
	"	"	x	1
	"	"	0	4
	"	"	x	2
3	10	30	x	-
	"	"	x	-
	"	"	x	-
	"	"	0	-
	"	"	0	-
4	8	30	x	-
	"	"	x	-
	"	"	x	-
	"	"	0	-
	"	"	0	-
	"	"	0	-
5	9	30	x	5
	"	"	x	3
	"	"	x	1
	"	"	0	2
	"	"	0	5
	"	"	0	4

x denotes the presence of quinhydrone in the gel.

O denotes the absence of quinhydrone in the gel.

All gels set in a thermostat at 25 degrees centigrade.

Runs 3 and 4 each showed no difference in the time of set.

Table #2

Run No.	C.C.Acid	C.C.Waterglass	Qh.	Time of set
1	9	30	0	16 min.
	"	"	0	17 "
	"	"	x	19 "
	"	"	x	22 "
	"	"	0	23 "
	"	"	x	23 "
2	8	30	x	5 min.
	"	"	x	" "
	"	"	x	" "
	"	"	0	" "
	"	"	0	" "
	"	"	0	" "

These gels were set under the same conditions as those in Table #1.

In all gels in each table there was 30c.c. of distilled water in the mixture.

Table #3

Run No.	C.C. Gel	Millivolts	Ph
1	0	-194	4.37
	5	-194	4.37
	10	-194	4.37
	15	-194	4.37
2	0	-150	5.21
	5	-150	5.21
	10	-150	5.21
	15	-150	5.21
3	0	-150.5	5.21
	5	-150.5	5.21
	10	-150.5	5.21
	15	-150.5	5.21

Table #3 cont'd

Run No.	C.C. Gel	Millivolts	Ph
4	0	-154	5.12
	5	-154	5.12
	10	-154	5.12
	15	-154	5.12
5	0	-99	6.1
	5	-99	6.1
	10	-99	6.1
	15	-99	6.1
6	0	-55	6.75
	5	-55	6.75
	10	-55	6.75
	15	-55	6.75

The control runs made with distilled water instead of gel gave constant readings.

Measurements were made at 20 degrees centigrade.

50c.c. of buffer solution were used in each run, saturated with solid quinhydrone.

Discussion of results-

The results of the work on the time of set of gels indicate that the quinhydrone has no apparent effect on the time of set of a silicic acid gel. In several runs the gels set all at one time and in the other cases the indications were that the quinhydrone had no effect on the time but that the discrepancy was due to experimental error. The runs covered both acid and basic gels and the number of gels set in each run was as large as convenient so as to get an average result and minimize the error from outside sources.

The work on the P_h of buffer solutions containing varying amounts of gel is also conclusive. The voltmeter readings give a more sensitive indication than interpolated P_h readings and these show no change at all when a quantity of washed gel is added to the buffer solution. From the data recorded we may conclude that a silicic acid gel does not affect the determination of P_h as measured by the quinhydrone electrode.