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# The effect of electrolytes on the viscosity and time of set of silicic acid gels

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THE EFFECT OF ELECTROLYTES  
ON THE VISCOSITY AND TIME OF SET OF  
SILICIC ACID GELS

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THE EFFECT OF ELECTROLYTES  
ON THE VISCOSITY AND TIME OF SET OF  
SILICIC ACID GELS

A thesis presented to the Department  
of Chemistry at Union College in partial  
fulfillment of the requirements for the  
degree of Bachelor of Science in Chemistry,  
by

John W. Rhoades

Approved by Charles B. Hurd.

April, 1942

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## INTRODUCTION

The phenomenon of gelation of silicic acid gels has been found to be influenced by seven variables.

The factors determining the time of set have been summarized by Flemming (1), Hurd and Carver (2), and Hallstrom (3). These factors are:

1. The silica concentration
2. The acid concentration
3. pH
4. Temperature
5. Agitation
6. Added soluble material
7. Type of silicate

Of the above factors all were held constant with the exception of (6), which was being studied. The time of set was determined by the tilted rod method, but before the gel set the time of flow was observed at frequent intervals, using an Ostwald viscometer. The Ostwald viscometer has been used with success by Prasad, Mehta, and Desai (5), Santora (4), and Gormley (6). By using this viscometer a study of the complete period of gelation can be made from the time of mixing until the gel becomes too viscous to flow through the capillary.

This thesis contains the results of experiments on the effect of electrolytes on the viscosity and time of set of silicic acid gels.

## HISTORICAL

Prakash and Dahr (7) have shown that viscosity measurements of solution of sodium silicate and an acid are an indication of what they believe happens as a gel sets. As mentioned by Santora, Dahr and Chakravarty (8) have noted the effects of salts on the viscosity of silicic acid solutions, and found that with increasing quantities of electrolytes the viscosity first falls and then rises. Dahr came to the conclusion that the rise in viscosity was a measure of the degree of hydration of a particle. The viscosity curves in this thesis are similar to those obtained by Santora and Prasad, Mehta, and Desai.

Fraser (9) conducted viscosity experiments with both the Ostwald and the Stormer type viscometer and found that although the Stormer type had great possibilities, the Ostwald viscometer was better to use because of its simplicity. A. Einstein and E. Hatschek, as mentioned by Gormley, devised an equation to calculate the viscosity of a dispersed system, provided that: (1) the particles are spherical and not deformable; (2) the dimensions of the particles are large in comparison to the molecular dimensions of the dispersion medium; (3) the particles must be small in comparison to the measuring instrument; and (4) the movement of the dispersion medium in the

direct vicinity of the particles may not be influenced by the presence of other particles. The hydration has been calculated, although these conditions are not fulfilled in the case of silica gels.

## PROCEDURE

### Preparation of Solutions

A quantity of E brand sodium silicate, a product of the Philadelphia Quartz Company, was diluted with recently boiled distilled water. The resulting solution was titrated with standard sulfuric acid and its equivalent as NaOH determined as 1.295 N, using methyl orange as an indicator.

The acetic acid was prepared by diluting 99.5% glacial acetic acid with recently boiled distilled water. The normality of the acetic acid as determined by the standard sodium hydroxide, and using phenolphthalein as an indicator, was 1.908 N.

The electrolytic solutions were prepared by dissolving pre-determined amounts of the salts in distilled water and mixing the gel solution with the proper amount of salt solution to give a final solution of always 155 c.c. and .3 M with respect to the salt.

### Temperature Control

The temperature was controlled by a thyritron circuit and a mercury-alcohol thermo-regulator. As all

experiments were performed at 25° C., once the temperature was set the thermo-regulator needed no more adjustment. The temperature of the bath was only slightly above room temperature and the thermo-regulator worked very well, not varying more than  $\pm 1.1^{\circ}$  C.

### Preparation of Gels

In order that the viscosity measurements are to have any meaning, it is necessary that the gel-forming solutions are accurately controlled and recorded. In the procedure, the silicate solution was measured into a dry, clean 250 c.c. beaker with a pipette. The acid solution and the third constituent were measured into another 250 c.c. beaker with pipette. The two solutions were placed in the water and allowed to remain there until they reached the temperature of the water bath. The silicate solution was then poured into the acid solution and the two were well mixed by rapidly pouring back and forth. The viscosity measurements were then ready to be made.

### Time Measurements

The time after mixing was measured by an ordinary wrist watch with a sweep second hand, and was measured from the time the silicate solution was first poured into the acid solution. The time of flow in the viscometer

was measured by means of a stopwatch.

### pH Measurements

It has been shown by Hurd and Griffeth (10) that the quinhydrone potentiometer gives reliable results in an acid gel mixture. It was used in this case as all measurements were in the acid region. A calomel cell with saturated KCl solution and a platinum wire electrode were prepared and used in the normal way. The pH measurements were taken in beakers and, since the quinhydrone was added, this solution was not used in the viscosity measurements.

### The Ostwald Viscometer

Since only relative instead of absolute viscosities were required, the Ostwald Viscometer was used. This type has previously been used by many workers with success.

The Ostwald Viscometer is essentially a U tube with a capillary in one arm. Near the top of the capillary there is a small bulb of about 2 or 3 c.c. Above and below the bulb are marks on the capillary tube to serve as reference marks. A definite volume of gel solution is pipetted into the large arm and sucked up a short way above the top reference mark. As the liquid goes back down, the time from which the meniscus passes the top reference mark until it passes the lower reference mark



is measured by the stopwatch. The instrument is calibrated by determining the time of flow of a liquid whose viscosity is known at that temperature at which the run is made. Distilled water and propyl alcohol were used in this case. The viscometer used on these experiments had a large capillary and for that reason the time of flow was short. If a small capillary is used in the case of sillicic acid gel, the results are not very satisfactory, as the size of the capillary is too near the size of the coagulated particles.

#### Determination of Time of Set by Tilted Rod

The time of set was determined by the tilted rod method, as described by Hurd and Letteron (11). The glass rod should be 3 m.m. in diameter and 10 cm. long, one end of which is drawn to a point. The glass rod is placed in the gel at an angle of about  $20^\circ$  to the vertical and the gel is said to be set when it is stiff enough to hold the rod in the tilted position. The time of set as determined by this method is longer than the time of set determined by the viscometer. That is, when the gel has ceased to flow in the viscometer, it is not yet stiff enough to hold the rod in the tilted position. In the following experiments, the time of set was determined by the tilted rod method.

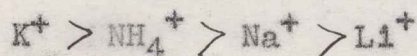
## EXPERIMENTAL

The process of gel-forming was studied by the indirect method of noting the effects of adding electrolytes in addition to the  $\text{NaC}_2\text{H}_3\text{O}_2$  formed naturally from the combination of sodium silicate and acetic acid. By adding predetermined amounts of salt solution, the final concentrations were made .3 molar with respect to the salts, with the exception of  $\text{K}_2\text{SO}_4$  and  $\text{Na}_2\text{SO}_4$ , which were not soluble enough. The solutions were prepared as mentioned before and 10 c.c. pipetted into the thermostated viscometer. The time of flow and the time after mixing was noted as well as the pH and the time of set as determined by the tilted rod method. The data obtained is recorded at the end of the paper in Tables I through XXIII. Viscosity runs were made, using as the third constituent:  $\text{H}_2\text{O}$ ,  $\text{NaCl}$ ,  $\text{KCl}$ ,  $\text{LiCl}$ ,  $\text{NH}_4\text{Cl}$ ,  $\text{NaBr}$ ,  $\text{KBr}$ ,  $\text{LiBr}$ ,  $\text{NH}_4\text{Br}$ ,  $\text{NaI}$ ,  $\text{KI}$ , and  $\text{NH}_4\text{I}$ . All the viscosity curves (Plates I-IV) have the same general shape and therefore the viscosity runs were discontinued and merely the time of set noted on the rest of the salts. The viscosity curves came out as one would expect. That is, the viscosity increases slowly at first but, as time goes on, it increases rapidly. The salts were so chosen that the effect of both the anion and cation could be observed and studied. In addition to the before-mentioned

salts, the time of set was measured when the following salts were used:  $\text{NaNO}_3$ ,  $\text{NH}_4\text{NO}_3$ ,  $\text{KNO}_3$ ,  $\text{NaCNS}$ ,  $\text{KCNS}$ ,  $\text{NH}_4\text{CNS}$ ,  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{K}_2\text{SO}_4$ , and  $\text{Na}_2\text{SO}_4$ .

### RESULTS AND DISCUSSION

When the salt solutions were used in place of pure water, the time of set was very markedly decreased and the viscosity curves sweep up much sooner, as is shown by Plates I-IV. The order of the coagulating power of the cations as determined by these experiments is:



For the anions, the order is:



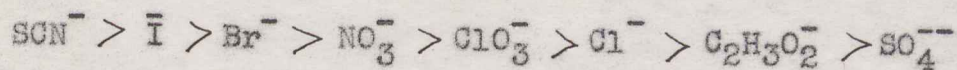
These results compare very well with the work done by Santora and others on silicic acid gels and also with the work done by Klobusitzky (12) on the speed of blood coagulation.

The process of gel formation is not fully understood and, for that reason, it is difficult to say how the salts affect the gel. One theory is that a lattice is set up and traps the water molecules so that the solution solidifies. Another is that the particles coagulate and grow larger and larger until the sol becomes immobile. The decrease in the time of set when a salt is present is said to be due to either the fact that the salts act as a catalyst or that the salts neutralize the charged particles so

that they do not repel each other and therefore coagulate more rapidly and become hydrated. As the particles become more and more hydrated they become larger and, also, the dispersion liquid decreases so that both facts tend to make the solution become more solid. It seems reasonable that viscosity curves have their shape because of the increasing size of the particles. When the particles are small (as when the gel is first mixed), a comparatively large increase in size would not affect the viscosity greatly unless the particle size was approaching the size of the capillary tube in which case a small increase in size would greatly increase the time of flow. This would account for the sudden up-sweep in the viscosity curves and also for the fact that the viscosity depends on the size of the capillary used.

Although the formation of the gel may be due to the lattice structure or the coagulation and hydration, it is very likely that it is a combination of the two effects (perhaps others) which actually cause the gel to solidify.

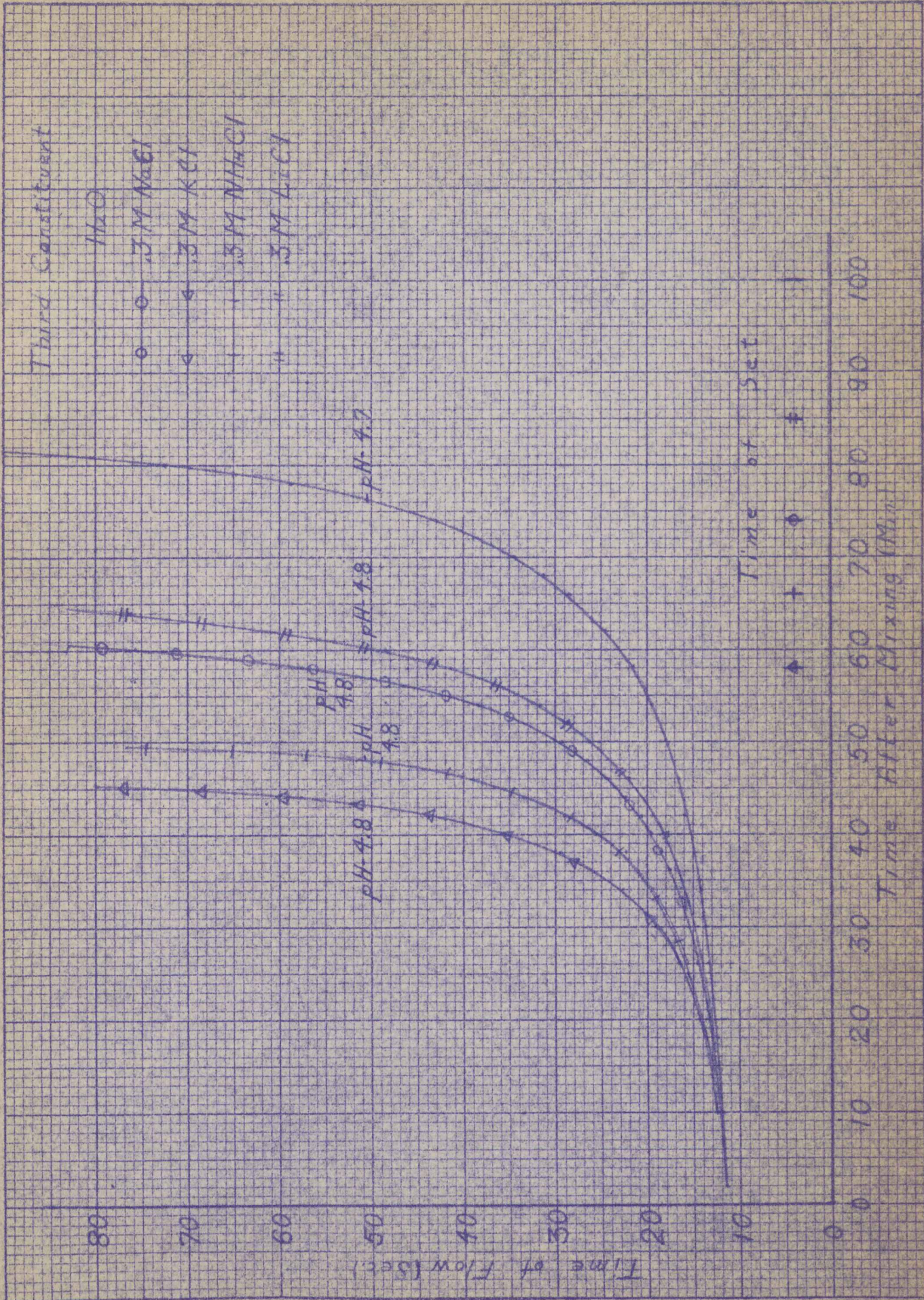
Hofmeister experimented on the lowering of the temperature of gelation and arrived at the following order:

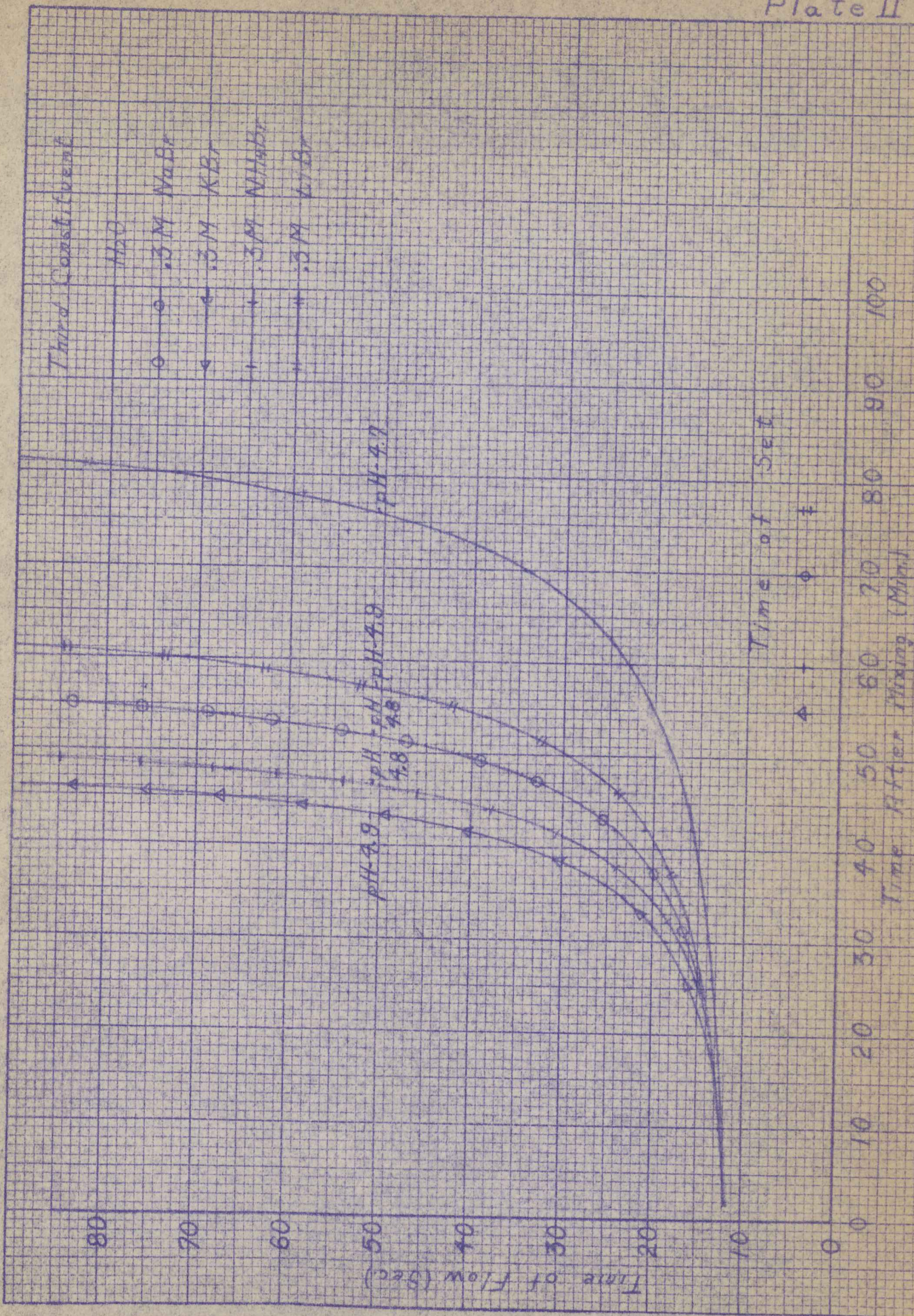


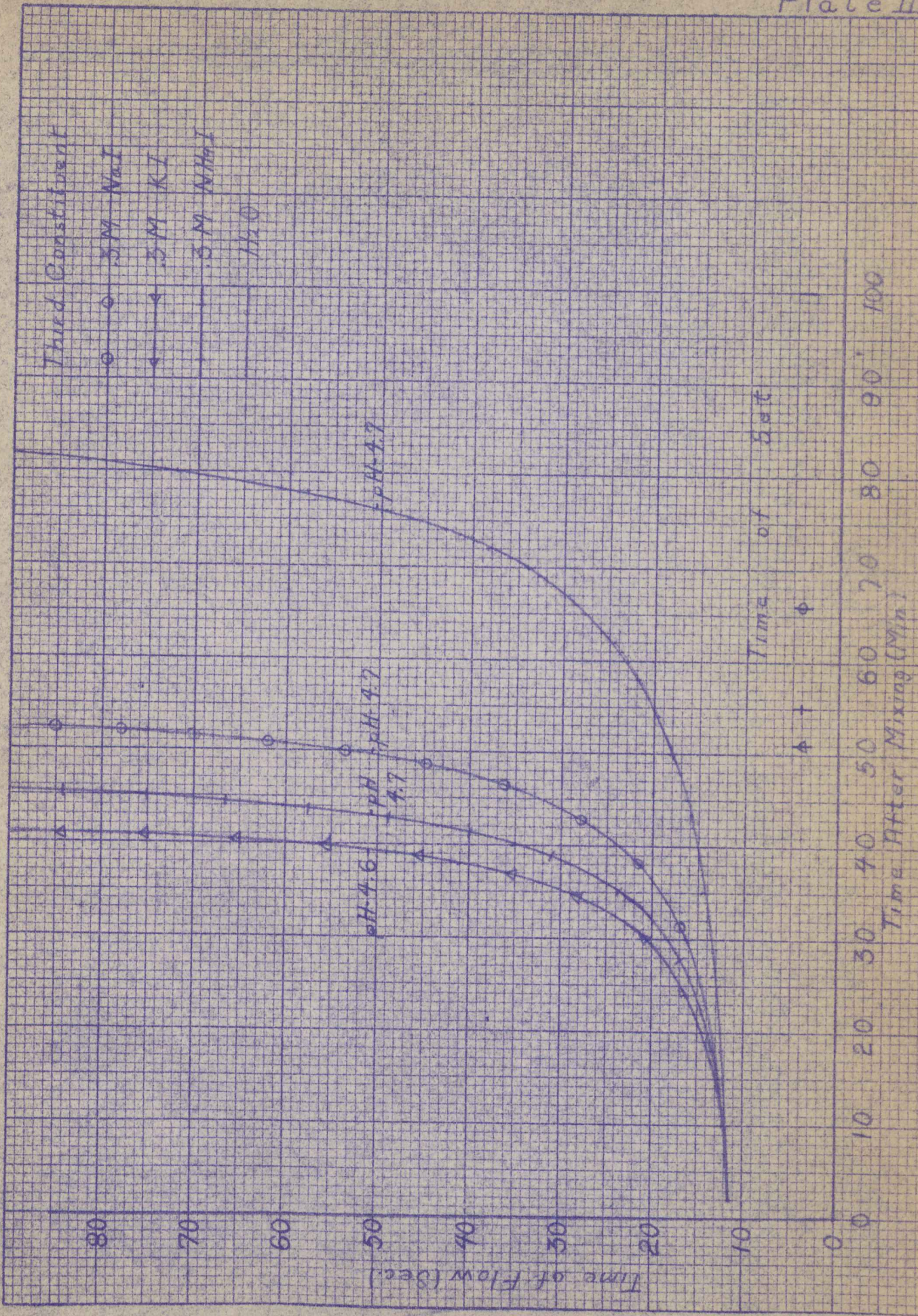
with the first having the greatest influence and the last the least influence (13). This series is known as a Hofmeister or Lyotropic series and agrees well with the work reported in this thesis.

## SUMMARY

1. The Ostwald viscometer has been used to study the viscosity during the process of gelation.
2. The time of set of the gels has been shown to decrease in a typical lyotropic series when salts are added.
3. Some ideas of how a gel sets and how the salts affect the time of set have been stated.
4. The order of the salts has been shown to agree with the work of others.









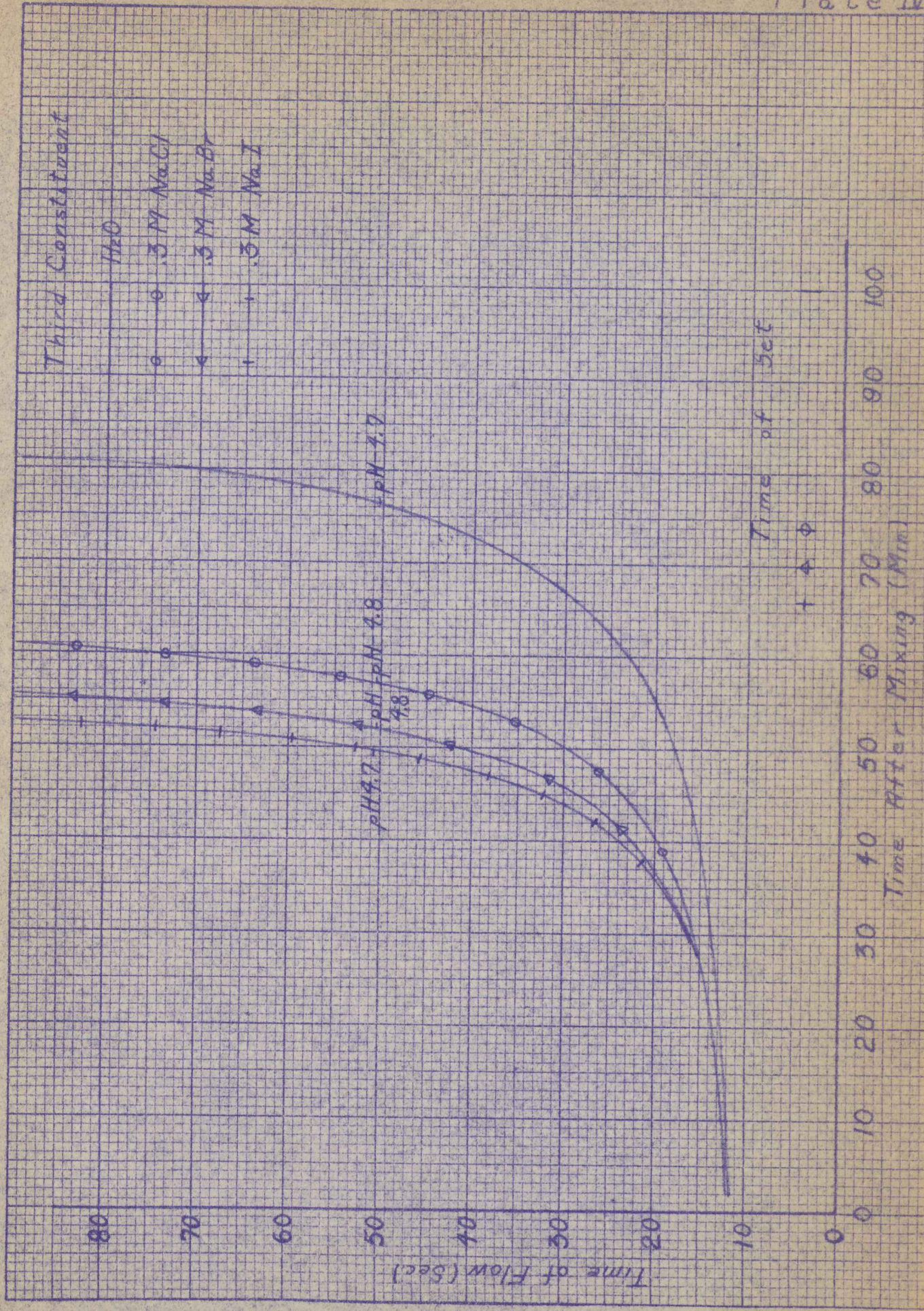


TABLE I

| Calibration of Viscometer                | Time of Flow (Sec.) |
|--|---------------------|
| Pure distilled H <sub>2</sub> O - 25° C. | 9.0                 |
|  | 9.0                 |
|  | 9.0                 |
|  | 9.0                 |
|  | Ave. <u>9.0</u>     |
| Propyl Alcohol - 25° C.                  | 22.0                |
|  | 22.0                |
|  | 22.0                |
|  | 21.9                |
|  | Ave. <u>22.0</u>    |

In the following tables, the total volume is always 155 c.c. In each case 10 c.c. was pipetted into the viscometer and the temperature held constant at 25° C.

TABLE II

Mixture as follows:

55 c.c. HAc  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
50 c.c. H<sub>2</sub>O

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> |
|------------------------------|----------------------------|
| 2                            | 11.6                       |
| 10                           | 12.0                       |
| 19                           | 12.5                       |
| 26                           | 13.2                       |
| 34                           | 14.4                       |
| 42                           | 15.8                       |
| 46                           | 16.8                       |
| 50                           | 18.3                       |
| 54                           | 19.8                       |
| 58                           | 21.8                       |
| 62                           | 25.0                       |
| 66                           | 28.6                       |
| 68                           | 31.4                       |
| 70                           | 34.6                       |
| 72                           | 38.6                       |
| 74                           | 43.6                       |
| 76                           | 50.0                       |
| 78                           | 59.1                       |
| 80                           | 72.7                       |
| 82                           | 96.4                       |
| 100                          | Set                        |

TABLE III

Mixture as follows:

55 c.c. HAc  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
50 c.c. H<sub>2</sub>O

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> |
|------------------------------|----------------------------|
| 2                            | 11.6                       |
| 10                           | 12.0                       |
| 18                           | 12.5                       |
| 26                           | 13.4                       |
| 34                           | 14.1                       |
| 42                           | 16.0                       |
| 46                           | 16.6                       |
| 50                           | 18.0                       |
| 54                           | 19.7                       |
| 58                           | 21.7                       |
| 62                           | 24.8                       |
| 66                           | 28.7                       |
| 68                           | 31.2                       |
| 70                           | 34.6                       |
| 72                           | 38.4                       |
| 74                           | 43.0                       |
| 76                           | 50.4                       |
| 78                           | 58.9                       |
| 80                           | 72.6                       |
| 82                           | 96.5                       |
| 100                          | Set                        |

TABLE IV

Mixture as follows:

55 c.c. HAc            pH - 4.7  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
50 c.c. H<sub>2</sub>O

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.8                       |                      |
| 10                           | 12.2                       | -168                 |
| 18                           | 12.6                       |                      |
| 26                           | 13.4                       |                      |
| 35                           | 14.7                       | -174                 |
| 42                           | 15.9                       |                      |
| 50                           | 18.2                       |                      |
| 54                           | 19.7                       |                      |
| 58                           | 21.9                       |                      |
| 62                           | 24.6                       |                      |
| 66                           | 28.6                       | -174                 |
| 70                           | 34.0                       |                      |
| 72                           | 38.3                       |                      |
| 74                           | 43.0                       |                      |
| 76                           | 49.5                       |                      |
| 78                           | 58.0                       |                      |
| 80                           | 72.2                       |                      |
| 82                           | 91.2                       |                      |
| 100                          | Set                        | -174                 |

TABLE V

Mixture as follows:

55 c.c. HAc                      pH - 4.8  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
40 c.c. H<sub>2</sub>O  
10 c.c. 4.65 M NaCl

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.8                       |                      |
| 10                           | 12.4                       |                      |
| 18                           | 13.4                       | -168                 |
| 26                           | 14.8                       |                      |
| 32                           | 16.0                       |                      |
| 36                           | 17.6                       |                      |
| 40                           | 19.6                       |                      |
| 42                           | 20.8                       | -170                 |
| 44                           | 22.4                       |                      |
| 46                           | 24.4                       |                      |
| 48                           | 26.4                       |                      |
| 50                           | 29.4                       |                      |
| 52                           | 33.7                       |                      |
| 54                           | 38.4                       |                      |
| 56                           | 46.4                       | -170                 |
| 58                           | 57.6                       |                      |
| 60                           | 75.7                       |                      |
| 74                           | Set                        | -170                 |

TABLE VI

Mixture as follows:

55 c.c. HAc                      pH - 4.8  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M KCl

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.6                       |                      |
| 10                           | 12.4                       | -170                 |
| 18                           | 13.8                       |                      |
| 26                           | 16.6                       |                      |
| 30                           | 19.1                       |                      |
| 34                           | 22.8                       | -184                 |
| 38                           | 29.6                       |                      |
| 40                           | 35.1                       |                      |
| 42                           | 44.0                       |                      |
| 44                           | 57.7                       |                      |
| 46                           | 94.6                       | -177                 |
| 58                           | Set                        | -172                 |

TABLE VII

Mixture as follows:

55 c.c. HAc                      pH - 4.8  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M LiCl

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 12.0                       |                      |
| 10                           | 12.4                       | -170                 |
| 18                           | 13.4                       |                      |
| 26                           | 14.6                       |                      |
| 30                           | 15.5                       |                      |
| 34                           | 16.6                       |                      |
| 38                           | 18.0                       | -170                 |
| 42                           | 19.6                       |                      |
| 46                           | 22.0                       |                      |
| 50                           | 26.1                       |                      |
| 52                           | 28.5                       |                      |
| 54                           | 31.8                       |                      |
| 56                           | 36.0                       |                      |
| 58                           | 41.3                       | -172                 |
| 60                           | 48.4                       |                      |
| 62                           | 59.4                       |                      |
| 64                           | 76.2                       |                      |
| 66                           | 128.4                      |                      |
| 85                           | Set                        | -172                 |

TABLE VIII

Mixture as follows:

55 c.c. HAc                      pH - 4.8  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M NH<sub>4</sub>Cl

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.8                       |                      |
| 10                           | 12.4                       | -166                 |
| 18                           | 13.8                       |                      |
| 26                           | 15.8                       |                      |
| 34                           | 19.6                       |                      |
| 38                           | 23.2                       |                      |
| 40                           | 25.6                       | -169                 |
| 42                           | 29.1                       |                      |
| 44                           | 33.4                       |                      |
| 46                           | 39.0                       |                      |
| 48                           | 51.7                       |                      |
| 50                           | 61.5                       |                      |
| 52                           | 104.0                      |                      |
| 66                           | Set                        | -170                 |



TABLE IX

Mixture as follows:

55 c.c. HAc                      pH - 4.8  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M NaBr

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.8                       |                      |
| 10                           | 12.4                       |                      |
| 18                           | 13.2                       | -148                 |
| 26                           | 15.0                       |                      |
| 30                           | 16.0                       |                      |
| 34                           | 18.0                       | -158                 |
| 38                           | 20.5                       |                      |
| 42                           | 24.2                       |                      |
| 44                           | 26.6                       |                      |
| 46                           | 30.2                       |                      |
| 48                           | 34.8                       |                      |
| 50                           | 41.4                       |                      |
| 52                           | 51.8                       |                      |
| 54                           | 66.2                       | -169                 |
| 56                           | 106.0                      |                      |
| 70                           | Set                        | -168                 |

TABLE X

Mixture as follows:

55 c.c. HAc                      pH - 4.9  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M KBr

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.6                       |                      |
| 10                           | 12.4                       |                      |
| 18                           | 13.5                       | -158                 |
| 26                           | 16.2                       |                      |
| 30                           | 18.6                       |                      |
| 32                           | 20.2                       |                      |
| 34                           | 22.2                       |                      |
| 36                           | 25.2                       | -164                 |
| 38                           | 28.8                       |                      |
| 40                           | 34.1                       |                      |
| 42                           | 42.8                       |                      |
| 44                           | 55.8                       |                      |
| 46                           | 89.2                       |                      |
| 55                           | Set                        | -163                 |

TABLE XI

Mixture as follows:

55 c.c. HAc                      pH - 4.9  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M LiBr

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.8                       |                      |
| 10                           | 12.6                       | -164                 |
| 18                           | 13.4                       |                      |
| 26                           | 14.9                       |                      |
| 34                           | 16.8                       |                      |
| 38                           | 18.6                       | -166                 |
| 42                           | 21.0                       |                      |
| 46                           | 24.2                       |                      |
| 48                           | 26.4                       |                      |
| 50                           | 29.7                       |                      |
| 52                           | 33.4                       |                      |
| 54                           | 38.4                       |                      |
| 56                           | 45.6                       |                      |
| 58                           | 55.7                       |                      |
| 60                           | 71.2                       |                      |
| 62                           | 117.6                      |                      |
| 77                           | Set                        | -166                 |

TABLE XII

Mixture as follows:

55 c.c. HAc                      pH - 4.8  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M NH<sub>4</sub>Br

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.6                       |                      |
| 10                           | 12.4                       |                      |
| 18                           | 14.5                       | -168                 |
| 26                           | 15.2                       |                      |
| 34                           | 19.8                       |                      |
| 38                           | 24.3                       | -170                 |
| 40                           | 27.4                       |                      |
| 42                           | 31.6                       |                      |
| 44                           | 38.5                       |                      |
| 46                           | 48.5                       |                      |
| 48                           | 66.2                       |                      |
| 60                           | Set                        | -170                 |

TABLE XIII

Mixture as follows:

55 c.c. HAc                      pH - 4.7  
 50 c.c.  $\text{Na}_2\text{SiO}_3$   
 30 c.c.  $\text{H}_2\text{O}$   
 20 c.c. 2.325 M NaI

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.6                       |                      |
| 10                           | 12.2                       |                      |
| 18                           | 13.2                       | -178                 |
| 26                           | 14.8                       |                      |
| 30                           | 16.3                       |                      |
| 34                           | 18.5                       | -176                 |
| 38                           | 21.3                       |                      |
| 42                           | 26.4                       |                      |
| 44                           | 30.0                       |                      |
| 46                           | 34.9                       |                      |
| 48                           | 42.4                       |                      |
| 50                           | 53.6                       |                      |
| 52                           | 74.8                       |                      |
| 66                           | Set                        | -174                 |

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TABLE XIV

Mixture as follows:

55 c.c. HAc                      pH - 4.6  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M KI

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.4                       |                      |
| 10                           | 12.2                       | -174                 |
| 18                           | 13.8                       |                      |
| 22                           | 15.2                       |                      |
| 26                           | 17.3                       | -174                 |
| 30                           | 20.4                       |                      |
| 32                           | 23.1                       |                      |
| 34                           | 27.0                       |                      |
| 36                           | 32.0                       |                      |
| 38                           | 41.1                       |                      |
| 40                           | 56.1                       | -173                 |
| 51                           | Set                        | -173                 |

TABLE XV

Mixture as follows:

55 c.c. HAc                      pH - 4.6  
50 c.c. Na<sub>2</sub>SiO<sub>3</sub>  
30 c.c. H<sub>2</sub>O  
20 c.c. 2.325 M NH<sub>4</sub>I

| <u>Time after Mix (min.)</u> | <u>Time of Flow (sec.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------------|----------------------|
| 2                            | 11.4                       |                      |
| 10                           | 12.0                       | -192                 |
| 18                           | 13.5                       |                      |
| 26                           | 16.0                       |                      |
| 34                           | 22.0                       |                      |
| 38                           | 29.0                       | -180                 |
| 40                           | 35.2                       |                      |
| 42                           | 43.5                       |                      |
| 44                           | 60.2                       |                      |
| 55                           | Set                        | -180                 |

TABLE XVI

Mixture as follows:

|  |          |
|--|----------|
| 55 c.c. HAc                              | pH - 4.7 |
| 50 c.c. Na <sub>2</sub> SiO <sub>3</sub> |          |
| 30 c.c. H <sub>2</sub> O                 |          |
| 20 c.c. 2.325 M NaNO <sub>3</sub>        |          |

| <u>Time after Mix (min.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------|
| 10                           | -184                 |
| 30                           | -178                 |
| 60                           | -179                 |
| 72 Set                       | -178                 |

TABLE XVII

Mixture as follows:

|   |          |
|---|----------|
| 55 c.c. HAc                                     | pH - 4.6 |
| 50 c.c. Na <sub>2</sub> SiO <sub>3</sub>        |          |
| 30 c.c. H <sub>2</sub> O                        |          |
| 20 c.c. 2.325 M NH <sub>4</sub> NO <sub>3</sub> |          |

| <u>Time after Mix (min.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------|
| 6                            | -180                 |
| 19                           | -180                 |
| 39                           | -180                 |
| 60 Set                       | -180                 |

TABLE XVIII

Mixture as follows:

|  |          |
|--|----------|
| 55 c.c. HAc                              | pH - 4.7 |
| 50 c.c. Na <sub>2</sub> SiO <sub>3</sub> |          |
| 30 c.c. H <sub>2</sub> O                 |          |
| 20 c.c. 2.325 M KNO <sub>3</sub>         |          |

| <u>Time after Mix (min.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------|
| 10                           | -176                 |
| 30                           | -176                 |
| 54                           | -176                 |
| 58 Set                       | -176                 |

TABLE XIX

Mixture as follows:

|  |          |
|--|----------|
| 55 c.c. HAc                              | pH - 4.8 |
| 50 c.c. Na <sub>2</sub> SiO <sub>3</sub> |          |
| 30 c.c. H <sub>2</sub> O                 |          |
| 20 c.c. 2.325 M NaCNS                    |          |

| <u>Time after Mix (min.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------|
| 11                           | -167                 |
| 45                           | -168                 |
| 63 Set                       | -170                 |

TABLE XX

Mixture as follows:

|  |          |
|--|----------|
| 55 c.c. HAc                              | pH - 4.8 |
| 50 c.c. Na <sub>2</sub> SiO <sub>3</sub> |          |
| 30 c.c. H <sub>2</sub> O                 |          |
| 20 c.c. 2.325 M KCNS                     |          |

| <u>Time after Mix (min.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------|
| 10                           | -156                 |
| 32                           | -159                 |
| 48                           | -166                 |
| 52 Set                       | -168                 |

TABLE XXI

Mixture as follows:

|  |          |
|--|----------|
| 55 c.c. HAc                              | pH - 4.8 |
| 50 c.c. Na <sub>2</sub> SiO <sub>3</sub> |          |
| 30 c.c. H <sub>2</sub> O                 |          |
| 20 c.c. 2.325 M NH <sub>4</sub> CNS      |          |

| <u>Time after Mix (min.)</u> | <u>Potentiometer</u> |
|------------------------------|----------------------|
| 13                           | -152                 |
| 21                           | -158                 |
| 32                           | -169                 |
| 47                           | -169                 |
| 56 Set                       | -169                 |





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