STUDIES ON "LUDOX" SILICA SOL

by

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I. General Introduction

"Ludox" silica sol is an opalescent, aqueous solution which contains approximately 30% SiO_2 plus a small amount of alkali acting as a stabilizing agent. It is made by the du Pont Company, and the physical properties and approximate chemical composition which they give it are as follows:

SiO_2	29-31%
Na_2O	.31-.41%
Sulfates as Na_2SO_4	.15% Max.
PH	(9.5-10.5
Specific Gravity $^{\circ}$Be	24.7-26.4
Viscosity at 25° C	2-3.5 cps.
Freezing Point	32°F

The "Ludox" is produced by an ion exchange technique which involves the passing of sodium silicate through the ion exchange material to replace sodium ion by hydrogen ion and adjusting the effluent to 30% silica. Some sodium is retained in the final product giving an SiO_2:Na_2O ratio of 75-90:1, whereas the starting material, commercial sodium silicate has a ratio of 3.3:1.

The work presented in this paper is an extension of the work done by Petrucci in 1950 (3). For those interested and for future workers in "Ludox" Petrucci's comprehensive paper should be consulted, as he suggests many other avenues of experimentation in addition to what he himself has done. Here we wish to consider the process of gelation of "Ludox" by measuring the change in surface tension during gelation and also the possibility of treating SiO_2-H_2O- alcohol systems on a triangular phase diagram.

II. Surface Tension During Gelation

The surface tension of solutions of "Ludox" and varying concentrations of the electrolytes potassium chloride and magnesium chloride was measured during the process of gelation. These measurements were made by means of a du Noüy surface tension balance with an evaporating dish containing about fifty cubic centimeters of solution being substituted for the watch glass ordinarily used. In this manner more fresh surface area was made available to the ring.

The results of this series of measurements are contained in tables I and II. The data have been plotted on curves with the scale readings as ordinates against the time in minutes as abscissas (figures 1 and 2). Multiplication of the scale readings by .776 gives the surface tension in dynes per centimeter. Temperature was maintained at or as near as possible to 25° C.

All of the curves are of essentially the same shape. As the concentration of the electrolytes decreases there is a further displacement of the surface tension increase to the right i.e. an increase in time. Also with greater time there appears to be a much slower increase in the surface tension along the middle portions of the curve. When the gel has set, no more readings can be taken, of course, as the ring would become incorporated into the gel. Since the gels made with magnesium chloride were much softer than those of potassium chloride, we find a more gradual increase in the surface tension near the point of gelation and not as high a surface tension at gelation.

Thus we have a fairly good indication of the course of gelation with "Ludox" sols. Present day theory considers aqueous inorganic gels as myriads of highly hydrous or gelatinous particles enmeshed into a network or sponge structure that entrains all the liquid phase. It has been found that a slow rate of precipitation favors the formation of a uniform gel structure and that time be allowed for the building up of an enmeshing network (7). This explains the rather gradual and uniform change in surface tension in the early portions of the curves. Once this network has been built up, gelation takes place, and there is correspondingly a large increase in the surface tension. It should be noted that similar surface tension curves have been obtained by Hurd and Letteron as a result of their work on silicic acid gels. (2)

Table I

Surface Tension "Ludox"- KCl mixtures

A. Concentration in final mixture

KCl- 250 millimoles/liter

SiO_2- 16.4%

Time Minutes	Scale Reading	Time Minutes	Scale Reading
2:35	15.5	34:10	48.0
5:00	17.5	37:30	49.0
7:35	22.0	40:20	52.0
10:10	24.5	44:00	60.0
12:40	26.0	47:50	64.5
15:25	32.0	48:20	69.0
18:25	36.0	50:00	70.0
22:00	38.0	51:45	77.5
25:00	41.5	53:10	85.0
27:40	45.0	55:20	150.0
30:35	47.0	57:15	180.0

B. KCl- 227 millimoles/liter SiO_2- 16.4%

Time Minutes	Scale Reading	Time Minutes	Scale Reading
1:35	11.0	20:25	33.0
4:10	18.0	26:15	36.0
7:25	20.0	29:25	38.5
9:40	23.5	34:00	40.0
11:30	27.0	37:00	40.0
13:40	29.5	39:30	41.0
16:35	33.0	43:00	41.5

B. Continued

Time Minutes	Scale Reading	Time Minutes	Scale Reading
46:00	42.0	79:35	52.0
49:20	43.0	82:20	55.5
55:20	48.5	85:00	62.0
58:10	49.5	88:00	72.0
62:00	45.0	91:00	90.0
67:00	49.0	93:00	110.0
70:15	46.0	94:30	135.0
74:30	51.5	96:00	165.0

C. HCl- 216 millimoles/liter SiO_2- 16.46

Time Minutes	Scale Reading	Time Minutes	Scale Reading
2:15	35	65:30	50
5:00	38	72:00	48
8:30	47	76:15	48
11:55	47	85:15	51
14:40	48	90:45	51
18:15	48	98:00	48
25:00	50	110:00	50
30:35	48	121:00	50
34:35	48	129:00	54
39:15	47	135:00	66
44:00	48	140:00	90
49:45	48	145:20	115
54:35	50	146:05	145
59:55	49	148:00	166

Table II

Surface Tension "Ludox-$MgCl_2$" $6H_2O$ mixtures

A. Concentration in final mixture

$MgCl_2 \cdot 6 H_2O$ - 18.5 millimoles/liter

SiO_2- 16.4%

Time Minutes	Scale Reading	Time Minutes	Scale Reading
1:50	15	42:30	69
4:00	28	45:10	65
5:45	34	46:30	72
7:30	35	48:25	70
10:00	39	50:40	69
12:45	41	53:00	68
15:55	48	55:00	75
19:15	50	57:00	76
22:15	55	59:30	85
25:45	57	61:25	92
29:55	58	64:00	101
33:55	59	66:00	122
37:30	64	66:30	140
40:30	68		

B. $MgCl_2 \cdot 6 H_2O$ - 17.5 millimoles/liter SiO_2 - 16.4%

Time Minutes	Scale Reading	Time Minutes	Scale Reading
2:30	17	16:05	40
5:45	30	21:00	41
9:00	32	27:00	42
12:15	36	31:15	45

B. Continued

Time Minutes	Scale Reading	Time Minutes	Scale Reading
41:15	45	78:15	69
53:30	45	81:30	67
62:40	50	84:35	75
65:00	51	86:25	80
68:30	55	88:00	92
72:45	64	89:00	104
74:15	64	89:35	117
76:15	69		

C. $MgCl_2 - 6\ H_2O$ - 16.7 millimoles/liter SiO_2 - 16.46

Time Minutes	Scale Reading	Time Minutes	Scale Reading
5:10	35	75:25	49
7:05	41	82:35	50
10:30	44	89:35	47
16:45	45	95:00	49
23:00	45	101:00	50
27:00	47	105:00	50
32:05	47	108:30	54
36:25	46	111:25	55
42:00	47	114:00	60
48:20	48	116:00	64
54:40	48	120:00	72
60:35	47	123:00	80
65:15	46	124:00	89
71:00	48		

SURFACE TENSION KCL - "LUDOX"

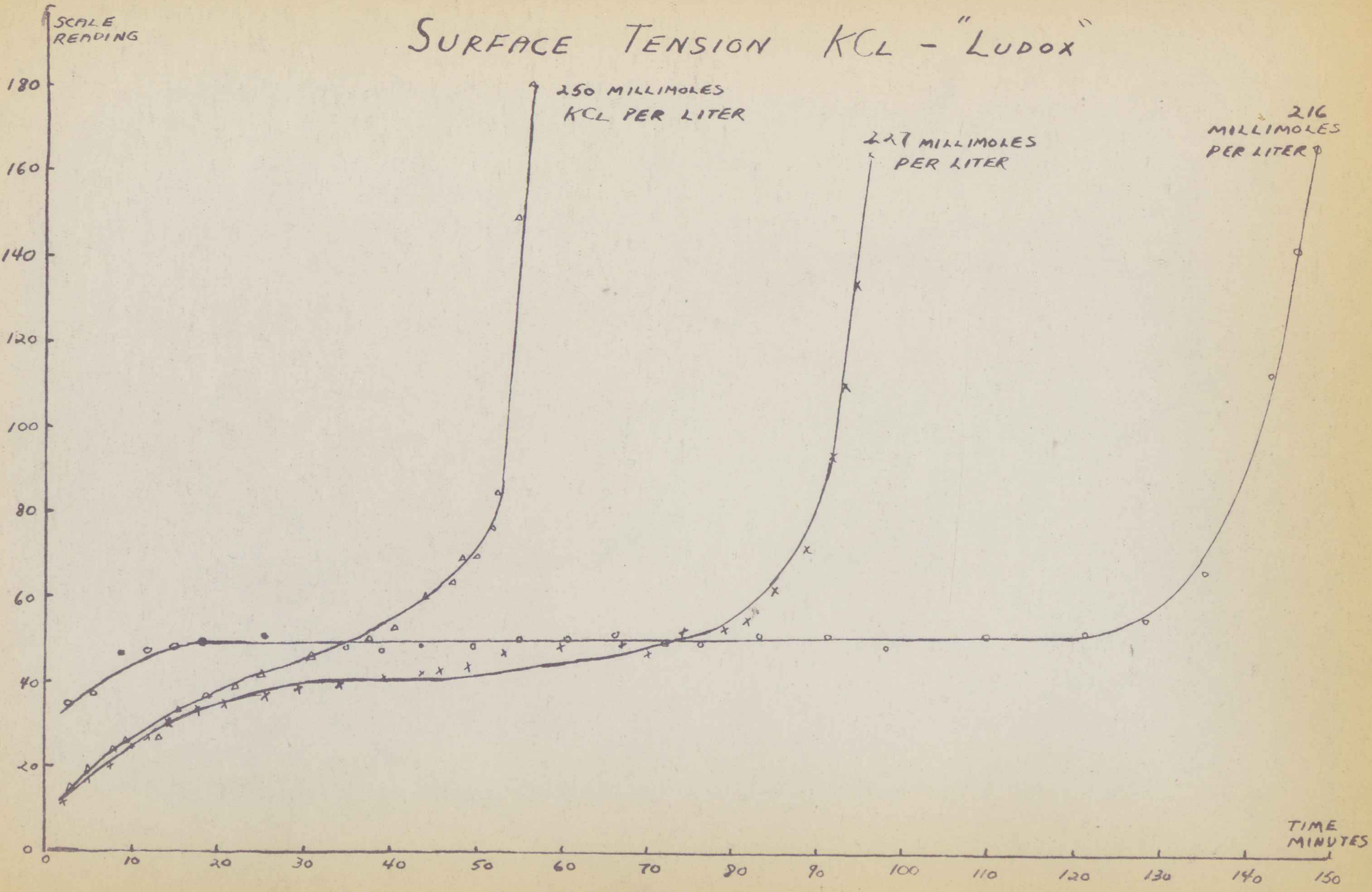


FIG. 1

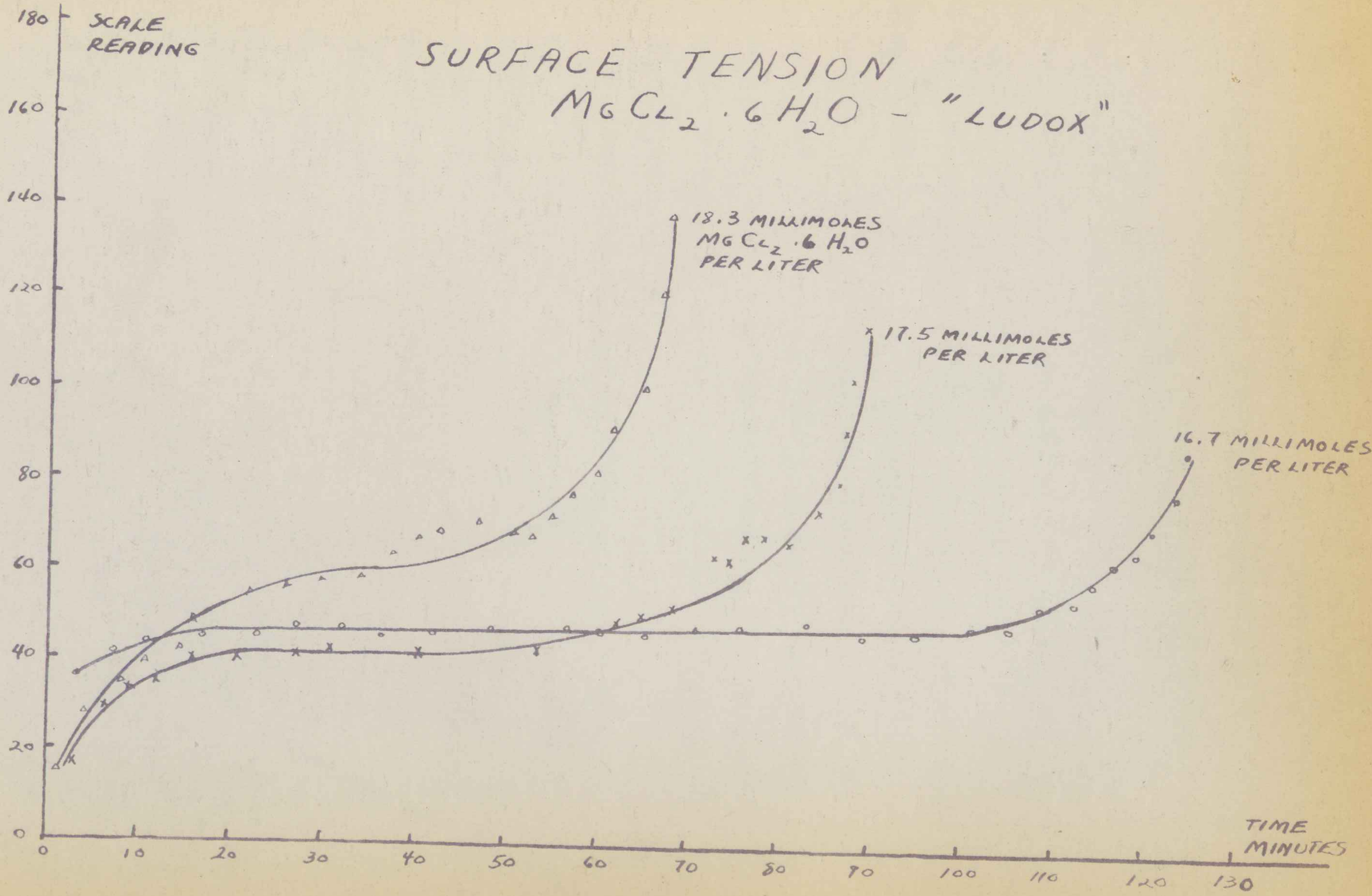

SURFACE TENSION
MgCl2 · 6H2O - "LUDOX"
SCALE READING
180
160
140
120
100
80
60
40
20
0
TIME MINUTES
10
20
30
40
50
60
70
80
90
100
110
120
130
18.3 MILLIMOLES MgCl2 · 6 H2O PER LITER
17.5 MILLIMOLES PER LITER
16.7 MILLIMOLES PER LITER

FIG. 2

III. Three Phase Systems: SiO_2 - H_2O - Alcohols

It was suggested by Petrucci that the three-component system: SiO_2 - H_2O - C_2H_5OH might be treated on a triangular phase diagram. He based his suggestion on an observation he had made while working on the gelation and dehydration of "Ludox" by ethyl alcohol (4). Petrucci noticed that with various mixtures of ethyl alcohol and "Ludox" there were some concentrations which produced gels , others which resulted in silica precipitation, and still others where solutions were the end product. As a result of Petrucci's suggestion, an attempt has been made here to construct a phase diagram of the SiO_2 - H_2O - C_2H_5OH system as well as the systems: SiO_2 - H_2O - CH_3OH and $CH_3CH_2CH_2OH$- H_2O - SiO_2.

The method employed was to vary the concentration of each of the three components so that mixtures falling over the entire diagram could be obtained which would indicate the regions of gelation, silica precipitation, and solution. The mixtures were made up in the following manner. Test tubes were used for the mixtures, and varying volumes of each of the components were added to the test tubes. For example: one might pipette 5 ml. "Ludox", 1 ml. of water, and 4 ml. of ethyl alcohol into one test tube and 6 ml. of "Ludox" , no water, and 4 ml. of ethyl alcohol into another test tube. The mixtures were then shaken and observed from time to time for a week at the end of which the results were recorded.

From the volumes of the original components the weight percentages of silica , water, and the alcohol added were computed. For example: silica was taken as 30% by weight of the "Ludox" added, the water included water from the "Ludox" (about 70%) as well as a small amount of water from the alcohol in addition to the original water, and, of course, the volume of alcohol had to be multiplied by its density to obtain its weight. To better illustrate the procedure used a sample calculation is given in the appendix.

The results obtained have been plotted in figures 3, 4, and 5, and the regions of gelation, solution, and silica precipitation are indicated. The percentage of silica, of course, does not exceed 30% since it was added in the form of "Ludox". Representing the relation between "Ludox" and the particular alcohol is the boundary line on the right, and it shows from bottom to top what happens as alcohol alone is added to the "Ludox".

Note that the area of silica precipitation increases as we move from methyl to n- propyl alcohol and also that the amount of water in the gels increases inthe same relation. In methyl alcohol gels the range of water and alcohol percentages is greater whereas the range of silica percentage is less than in ethyl alcohol gels which in turn bear the same relationship to the n-propyl alcohol gels. Now silica precipitation results from the dehydration of the "Ludox" by the alcohol. Petrucci showed (4), and these results also show that the principal condition necessary for the precipitation of silica rather than gelation of the sol is that the alcohol must be present in a large excess. The previous results can then be explained on a basis of dehydration power i.e. n-propyl is greater than ethyl which is greater than methyl as to power of dehydration.

An interesting point which turned up during the experimentation was the formation of both immediate gels and also gels requiring minutes, hours , and in a few cases days to gel. The only place where the longer setting gels seemed to appear was along or close to the boundary between the gel region and solution region. When the silica precipitation region was reached,gels of longer setting times were no longer obtained. This observation was noted in all three alcohol systems. All the other gels formed immediately. Although many slow-setting gels were obtained, no definite relationship between the components could be established. Perhaps further exploration of the subject would be worthwhile in clarifying the situation.

Table III- Data for SiO_2- H_2O - CH_3OH system

A. Solutions

Milliliters			Weight Percentages		
"Ludox"	Water	95% Methanol	Silica	Water	Methanol
1	2.5	6.5	4.0	40.6	55.4
3	5	3	12.1	62.7	25.2
3	3	5	10.3	55.9	33.8
3	5	2	13.2	67.1	19.7
3	2	5	11.3	49.3	39.4
2	2	6	7.9	42.6	49.5
4	3	3	14.2	63.6	22.2
7	2	1	22.5	70.8	6.7
5	1	4	17.7	52.7	29.6
4	2	4	14.5	55.1	30.4
1	2	7	4.1	35.4	60.5
6.5	0	3.5	22.3	53.5	24.2
.5	1.5	8	2.1	26.4	71.5
1.5	2.5	6	6.0	44.0	50.0
2	2.5	5.5	7.8	47.3	44.9
.5	1	8.5	2.1	21.1	76.8
3.5	1.5	5	12.6	47.4	40.0
1	1.5	7.5	4.1	30.4	65.5
4	1.5	4.5	14.6	50.9	34.5
2	8	1	6.5	86.4	7.1
1	6	3	3.7	72.6	23.7
3	6	1	10.4	82.3	7.3

A. Continued

Milliliters			Weight Percentages		
"Ludox"	Water	95% Methanol	Silica	Water	Methanol
1	4	5	3.9	54.8	41.3
5	2	3	17.4	60.8	21.8
5	4	1	16.8	76.2	7.0
2.5	4.5	3	9.7	66.1	24.2
B. Silica Precipitation					
2	.5	7.5	8.2	27.8	64.0
1	0	5	6.9	20.1	73.0
1.5	0	5	9.5	25.2	65.3
1	0	9	4.3	14.3	81.3
.5	.5	9	2.2	15.4	82.4
1	1	8	4.2	25.2	70.5
1.5	.5	8	6.3	23.9	69.8
.5	0	9.5	2.2	9.7	88.1
1	.5	8.5	4.2	20.0	75.8
C. Gels Setting in More than 10 Minutes					
2.5	1.8	5.7	9.7	44.2	46.1
6	0	4	20.8	50.1	29.1
4	1	5	14.7	46.6	38.7
1.5	1.5	7	6.5	53.8	39.7
3	1.5	5.5	11.4	44.8	43.8
5.5	.5	4	19.3	51.4	29.3
4.5	1	4.5	16.3	49.5	34.2
2	2	6	7.9	42.6	49.5
1.5	2	6.5	6.8	38.3	54.9

D. Gels Setting in Less than 10 Minutes

Milliliters			Weight Percentages		
"Ludox"	Water	95% Methanol	Silica	Water	Methanol
5	.5	4.5	17.9	48.4	33.7
2.5	1.5	6	8.8	42.2	49.0
2	1.5	6.5	8.0	37.8	54.2
3.5	1	5.5	12.8	42.8	44.4
2.5	.5	7	10.0	31.8	58.2
2.5	0	7.5	11.1	25.6	63.3
1.5	1	7.5	7.3	28.1	64.6
2	1	7	8.1	32.9	59.0
3	1	6	11.6	40.0	48.4
4	.5	5.5	14.9	42.2	42.9
4.5	.5	5	15.9	45.9	38.2
2.5	1	6.5	9.9	36.5	53.6
3	3	6.5	11.7	33.3	55.0
5	0	5	18.1	44.0	37.9
2	0	5	6.8	34.1	59.1
3	0	5	14.3	35.9	49.8
3	0	7	11.8	30.4	57.8

Table IV- Data for SiO_2- H_2O - C_2H_5OH system

A. Solutions

Milliliters			Weight Percentages		
"Ludox"	Water	95% Ethanol	Silica	Water	Ethanol
6	0	2	23.7	60.8	15.5
1	8	1	3.6	88.8	7.6
4.5	2.5	3	15.8	62.2	22.0
3	6	1	10.5	82.2	7.3
.5	3.5	6	1.7	56.5	41.8
1	6	3	3.7	72.6	23.7
5	3	3	15.9	64.2	19.9
5	4	1	16.8	76.2	7.0
5	0	1	26.8	62.1	11.1
3	5	2	10.6	74.6	14.8
5	5	5	12.1	62.7	25.2
1	5	4	3.8	63.9	32.3
5	1	2	21.0	61.4	17.6
7	2	1	22.5	70.8	6.7
.5	4	5.5	2.2	51.4	46.4
.5	3	6.5	2.0	41.9	56.1
2	4	4	7.6	60.9	31.5
2.5	3.5	4	9.4	59.3	31.3
5.5	2	2.5	18.6	63.6	17.8
1.5	4	4.5	5.8	57.9	36.3
7.5	.5	2	24.3	62.1	13.6

B. Silica Precipitation

Milliliters			Weight Percentages		
"Ludox"	Water	95% Ethanol	Silica	Water	Ethanol
1	2	7	4.1	35.5	60.4
1	1	10	3.5	22.3	74.2
2	0	5	11.4	29.5	59.1
3	0	5	14.3	35.9	49.8
3	1	6	11.6	39.0	49.4
2	2	6	7.7	44.3	48.0
4.5	0	5.5	16.6	41.0	42.4
4	.5	5.5	14.9	42.2	42.9
.5	0	10	2.1	9.8	88.1
3	1.5	5.5	11.4	44.8	43.8
2	1	7	8.1	32.9	59.0
1	2.5	6.5	4.0	40.5	55.5
1.5	2.5	6	6.0	44.0	50.0
2.5	2	5.5	9.7	45.9	44.4
4.5	.5	5	16.4	45.3	38.3
2	2.5	5.5	7.8	47.3	44.9
.5	2.5	7	2.1	36.8	61.1
.5	1.5	8	2.1	26.4	71.5

C. Gels Setting in Less than 10 Minutes

5	1	3	19.2	56.7	24.1
5	0	5	18.1	44.1	37.8
4	1	5	14.8	46.6	38.6
5	0	2	23.8	55.0	21.2

C. Continued

Milliliters			Weight Percentages		
"Ludox"	Water	95% Ethanol	Silica	Water	Ethanol
2	3	5	7.6	51.9	40.5
6.5	.5	3	21.9	56.9	21.2
3	2	5	11.3	49.2	39.5
2.5	2.5	5	9.6	50.6	39.8
3.5	1.5	5	12.6	47.4	40.0
3	2.5	4.5	11.3	53.1	35.6
4	2	4	14.5	55.1	30.4
5	1	4	17.7	52.7	29.6
6	0	4	20.9	50.0	29.1
1.5	3	5.5	6.0	48.7	45.3

D. Gels Setting in More than 10 Minutes

7	.5	2.5	23.1	59.5	17.4
1	3.5	5.5	3.9	50.3	45.8
2	3.5	4.5	7.7	56.4	35.9
5	4	5	12.9	60.1	27.0
5	2	3	17.4	60.8	21.8
3	4	5	9.4	57.0	32.6
6	1	3	20.4	58.2	21.4
1.5	3.5	5	5.9	53.7	40.8
3	3	4	11.1	57.9	31.0

Table V- Data for SiO_2 - H_2O - $CH_3CH_2CH_2OH$ system

A. Solutions

Milliliters			Weight Percentages		
"Ludox"	Water	99% Propanol	Silica	Water	Propanol
5	0	1	26.6	61.9	11.5
7	1	2	22.9	62.8	14.3
6	2	2	20.1	65.3	14.6
4	3	3	14.2	62.6	23.2
4.5	3	2.5	15.6	65.4	19.0
5	2.5	2.5	17.2	64.1	18.7
2	4.5	3.5	7.5	63.9	28.6
.5	4.5	5	2.0	54.5	43.5
1	4.5	4.5	3.9	57.9	38.2
3.5	3.5	3	12.1	63.9	24.0
3.5	4.5	2	11.9	72.5	15.6
2.5	4	3.5	9.3	62.4	28.3
3	4	3	10.9	63.3	25.8
2	5	3	7.4	68.4	24.2
1.5	4.5	4	5.8	60.9	33.3
1	8	1	3.6	88.5	7.9
1	6	3	3.7	71.5	24.8
3	6	1	10.4	82.0	7.6
5	4	1	16.8	75.9	7.3
7	2	1	22.5	70.5	7.0

B. Silica Precipitation

1	3	6	4.0	43.3	52.7
5	0	5	18.0	42.8	39.2
2	2	6	7.9	40.6	51.5

B. Continued

Milliliters			Weight Percentages		
"Ludox"	Water	99% Propanol	Silica	Water	Propanol
5.5	.5	4	19.3	50.4	30.6
2	3.5	4.5	7.7	54.9	37.4
3	2.5	4.5	11.2	52.1	36.7
1	2	7	4.1	33.0	62.9
3.5	2	4.5	12.5	50.4	37.1
.5	1.5	8	2.1	23.5	74.4
4	1.5	4.5	14.6	49.5	35.9
1	1	8	4.2	22.3	73.5
4.5	.5	5	16.4	43.7	39.9
5	1	4	17.7	51.4	30.9
1	0	5	6.9	17.1	76.0
6	0	4	20.9	48.8	30.3
3	0	5	14.3	33.7	52.0
3	2	5	11.3	47.6	41.1
3.5	0	5	15.0	35.5	49.5
2	3	5	7.8	50.2	42.0
4	1	5	14.8	44.9	40.3
3.5	1.5	5	12.6	45.7	41.7
4.5	1	4.5	16.3	48.1	35.6
5	.5	4.5	17.9	47.0	35.1
.5	.5	9	2.2	11.9	85.9
2.5	1	6.5	9.9	34.3	55.8
1.5	4	4.5	5.8	57.4	37.8
1	4	5	3.9	53.1	43.0
.5	4	5.5	2.0	49.5	48.5

C. Gels Setting in Less than 10 Minutes

Milliliters			Weight Percentages		
"Ludox"	Water	99% Propanol	Silica	Water	Propanol
5	1.5	3.5	17.6	55.5	26.9
4.5	2	3.5	16.0	56.9	27.1
3	3	4	11.1	56.6	32.3
6.5	0	3.5	22.0	51.9	26.1
2.5	3	4.5	9.5	53.5	37.0
5	0	2	25.8	53.5	20.7
4	2	4	14.5	53.8	31.7
6	1	3	20.4	57.3	22.3
5	1	4	17.7	51.4	30.9
3.5	2.5	4	12.4	54.9	32.7
7	.5	2.5	23.1	58.9	18.0
6	.5	3.5	20.6	53.1	26.3

D. Gels Setting in More than 10 Minutes

2	4	4	7.6	59.4	32.9
6.5	1	2.5	21.6	60.2	18.2
4.5	2.5	3	15.8	61.2	23.0
5	2	3	17.4	59.8	22.8
4	2.5	3.5	14.3	58.3	27.4
6	1.5	2.5	20.3	61.3	18.4
3.5	3	3.5	12.2	59.4	28.4
5.5	2	2.5	18.8	62.6	18.6
3	3.5	3.5	11.0	61.0	28.0
2.5	3.5	4	9.4	58.1	32.5
7.5	.5	2	24.3	61.6	14.1

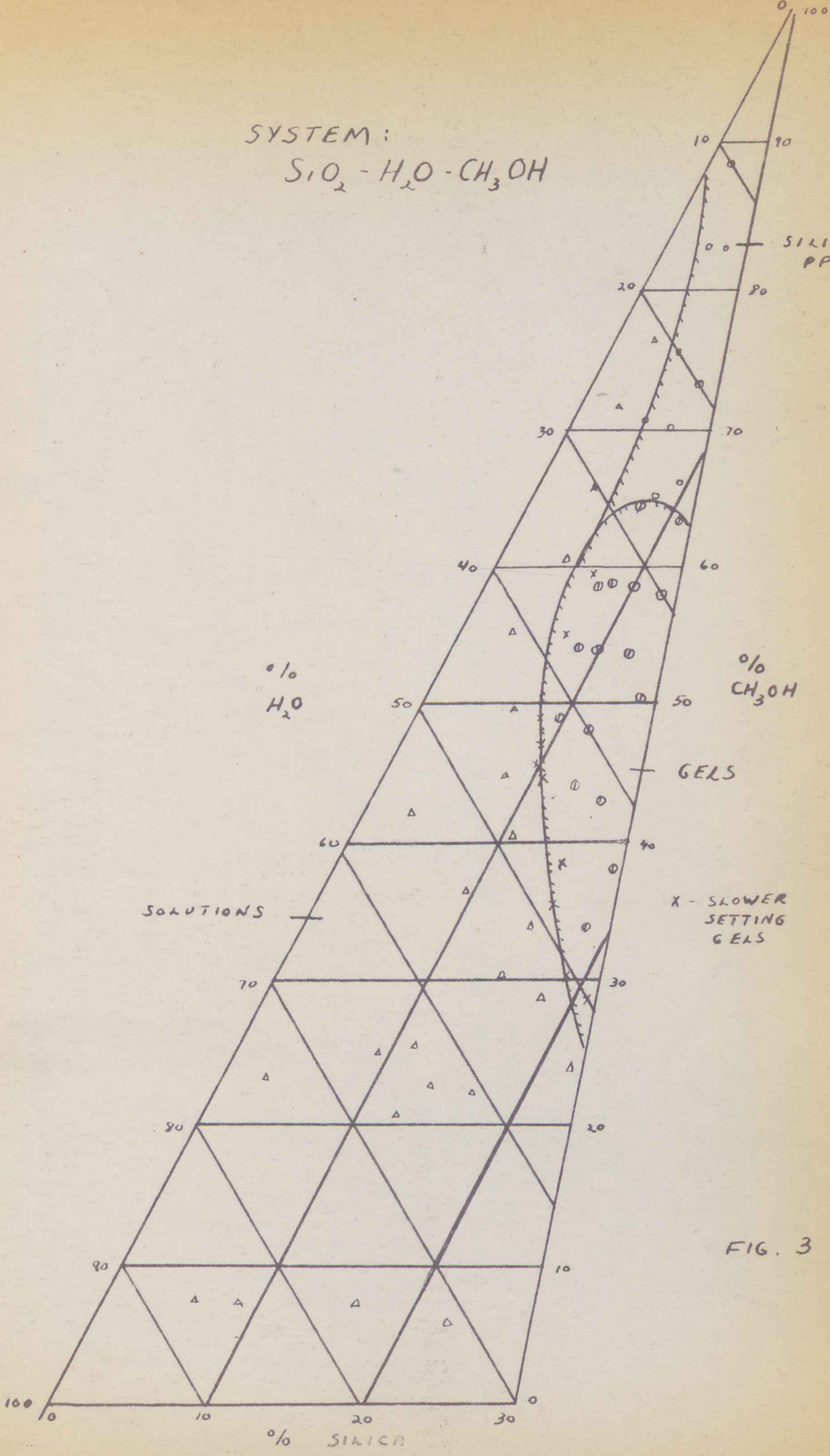

SYSTEM:
$SiO_2 - H_2O - CH_3OH$
% H_2O
% CH_3OH
% SILICA
SILICA PPT.
GELS
SOLUTIONS
X - SLOWER SETTING GELS
0
10
20
30
40
50
60
70
80
90
100

FIG. 3

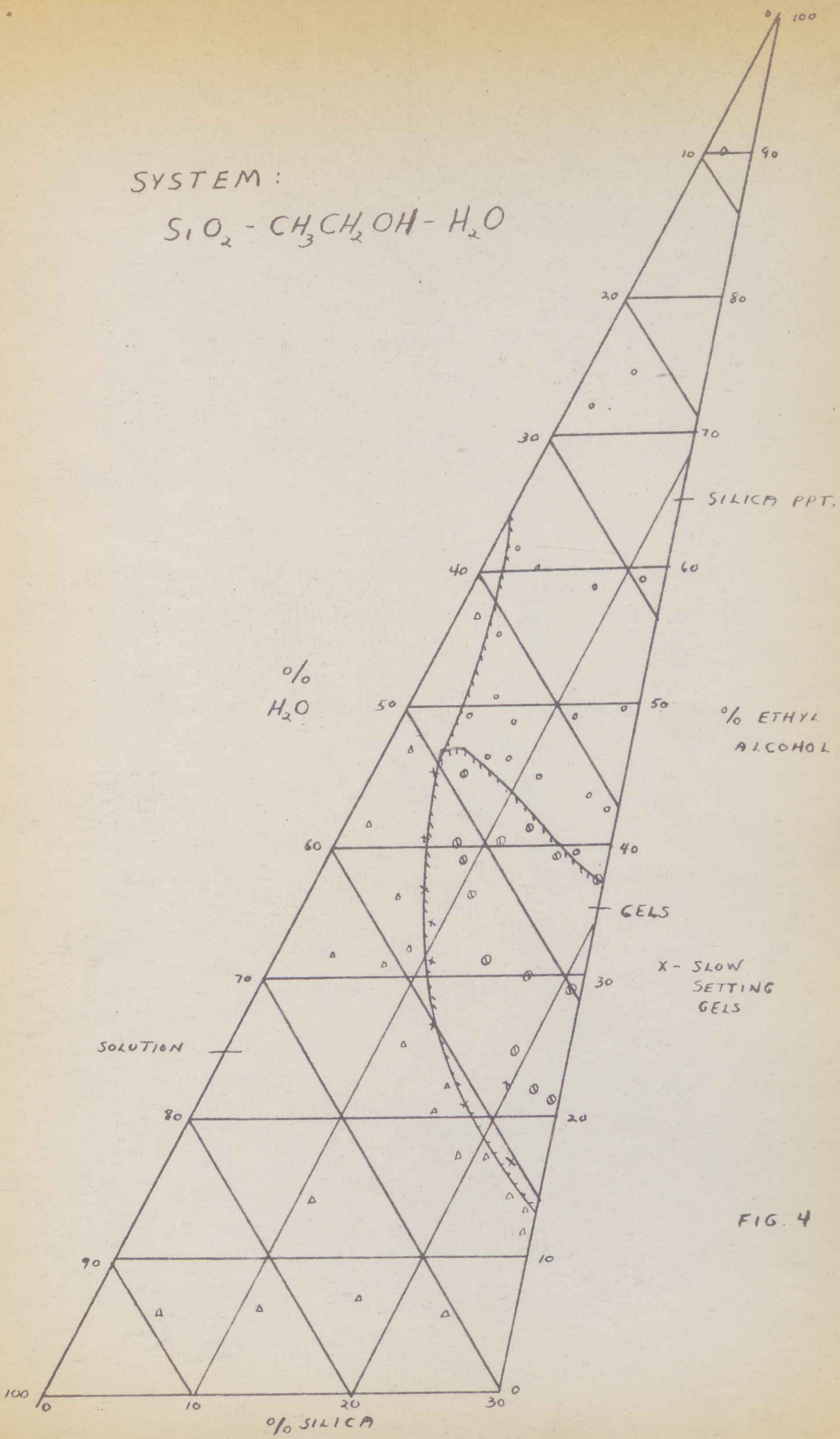
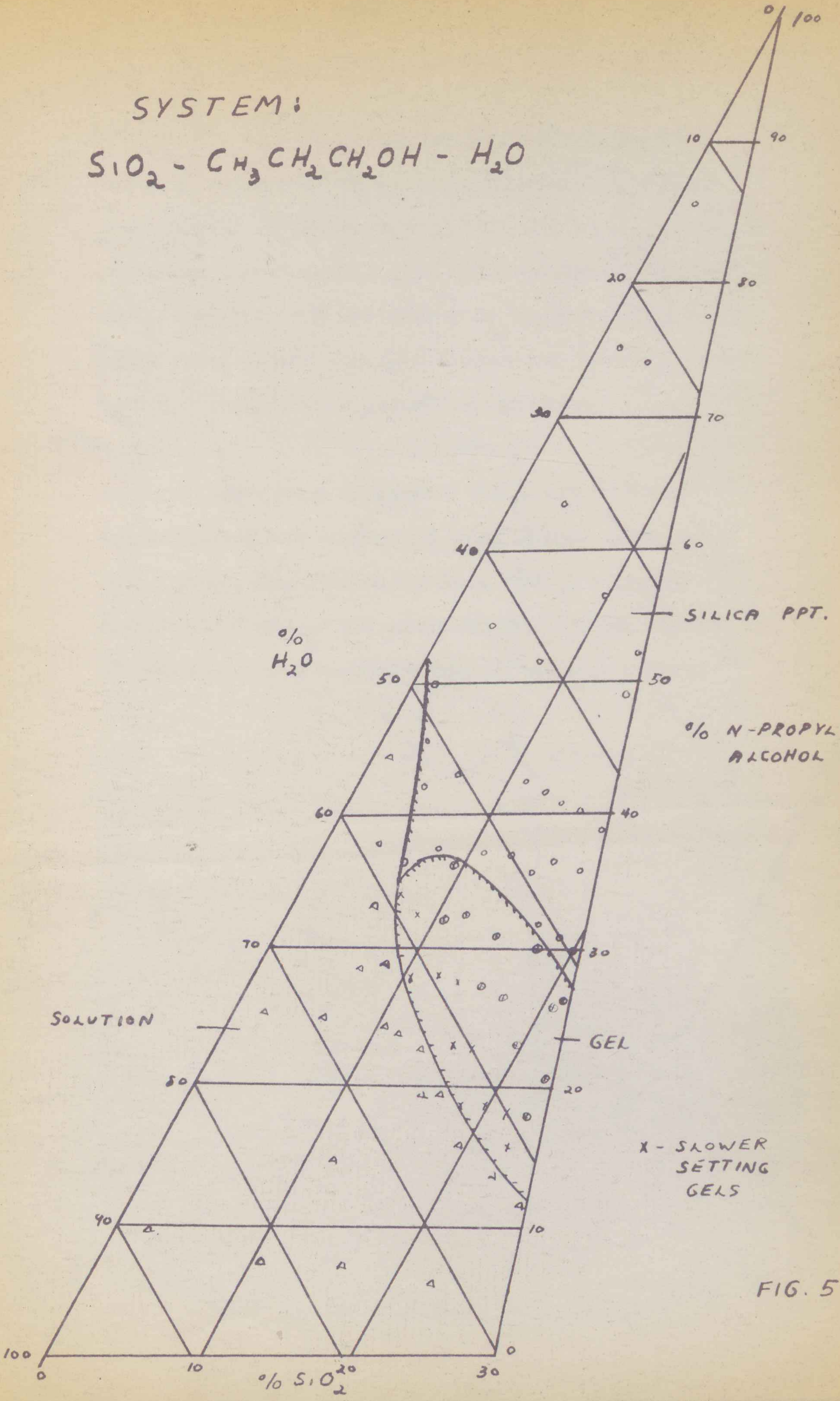

SYSTEM:
$SiO_2 - CH_3CH_2OH - H_2O$
% H_2O
% ETHYL ALCOHOL
% SILICA
SILICA PPT.
GELS
X - SLOW SETTING GELS
SOLUTION
100
90
80
70
60
50
40
30
20
10
0

FIG. 4



SYSTEM:
$SiO_2 - CH_3CH_2CH_2OH - H_2O$
% H_2O
% N-PROPYL ALCOHOL
% SiO_2
SILICA PPT.
SOLUTION
GEL
X - SLOWER SETTING GELS
0
10
20
30
40
50
60
70
80
90
100

FIG. 5

IV. Summary

The variation of surface tension of a mixture of "Ludox and each of the electrolytes potassium chloride and magnesium chloride ($MgCl_2 \cdot 6 H_2O$) was studied during the process of gelation. The surface tension readings remained fairly constant until the mixture was about to set at which point they rose very rapidly. This behavior appears consistent with present day theory of gel formation.

Three phase diagrams of the system; silica-water- alcohol were constructed using methyl, ethyl, and normal propyl alcohols. It was found that by obtaining mixtures fallinf over the entire diagram definite regions of gelation, silica precipitation, and solution existed.

Appendix I - Calculation of weight percentages for three phase diagrams: sample calculation

To illustrate the computation of the weight percentages for the phase diagrams a sample calculation of the first mixture under ethyl alcohol will be given (p.17) i.e. the mixture containing 8 ml. "Ludox", 0 ml. water, and 2 ml. of 95% ethyl alcohol. First we find the total weight of the mixture. Assuming the density of "Ludox" to be 1.21 gm./ml. we have in 8 ml. 8 x 1.21 or 9.68 gms, and assuming .80 gm./ml. as the density of 95% ethyl alcohol its weight is 2 x .80 or 1.60 gms. giving a total weight of 11.28 gm.

Now since silica is 30% by weight of "Ludox" its weight is .3 x 9.68= 2.90 gm. giving a silica weight percentage of 2.90/11.28 = 25.7%. The ethyl alcohol weight will be .95 x 1.60 = 1.52 gm.; weight percentage 1.52/11.28 = 13.5%. Water weight comes from both the "Ludox and alcohol i.e. (9.68- 2.90) plus (1.60- 1.52) or 6.86 gm.; weight percentage 6.86/11.28 or 60.8%. This calculation, of course, neglects the small amounts of other materials present.

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