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INVESTIGATIONS IN COLLOIDAL PHENOL

by

CHARLES JAMES MILLAR

A THESIS SUBMITTED TO THE FACULTY OF THE  
MISSOURI SCHOOL OF MINES AND METALLURGY, IN  
PARTIAL FULLFILLMENT OF THE WORK REQUIRED FOR  
THE DEGREE OF MASTER OF SCIENCE IN GENERAL  
SCIENCE.

Rolla Missouri

1 9 2 2 .

Approved by *Frederick W Shaw.*

Foreword

The author is indebted to Dr. F. W. Shaw for suggestions and advice in regards this work.

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## Investigations in Colloidal Phenol.

### Introduction.

In making some experiments with various colloids it was noticed that phenol and water seemingly formed a colloid, so it was deemed advisable to experiment farther and see if this fact could be established.

The references on this subject vary widely and for this reason some of them are given here. Ostwald recognised the colloid state of a phenol-water mixture at the critical temperature but evidently failed to see the possibility of the phenol-water colloid at all temperatures.

In the following article I will try to show that the phenol-water mixture forms a colloid, not only at the critical temperature, but at all temperatures and in all concentrations.

Seidell gives the solubility of phenol in water as follows,

T°	Grams phenol per 100 grams.	
	Aqueous layer	Phenol layer
10	7.5	75.0
20	8.3	72.1
30	8.8	69.8
40	9.6	66.9
50	12.0	62.7
55	14.1	59.5
60	16.7	55.4
65	25.9	49.2
68.3 (crit. temp.)	33.4	

The handbook of Chemistry and Physics gives the solubility of phenol as six parts in one hundred. The Chemiker Kalender gives the solubility as infinity at 67.5 degrees centigrade. The Chemical Dictionary gives phenol as soluble in alcohol, water, ether, chloroform, glycerine and the alkalies.

On page eighty eight of his book on Theoretical and Applied chemistry, Ostwald says,

" I have in this flask two liquids which at ordinary temperatures are only partially soluble in each other, namely, phenol containing some water, and water containing a little phenol. Even at a distance you observe that they scarcely dissolve in each other, for as soon as I shake the flask the mass of liquid shows the white color of an emulsion ( demonstration ). The solubility of phenol and water in each other increases greatly with increase in temperature. To prove this I heat the mixture while continuously shaking it ( demonstration ). As soon as I have warmed the mixture to a temperature of 70 C. the emulsion clears; in other words, the amounts of phenol and water with which I started dissolve completely in each other. I could actually add any amount more of either of the two, at this temperature, without their separating out.

" Above the 'critical' temperature, phenol and water are miscible in all proportions. For this experiment I have, of course, not chosen indifferent amounts of phenol and water, but a concentration of about 36 percent phenol. this represents the 'critical' concentration of phenol in water, the significance of which I cannot discuss further here.

"While I have been talking, the white emulsion of phenol and water has given way to a clear solution. We have before us now a molecularly dispersed solution of the phenol in water. But the phenomenon to which I wish to direct your particular attention is to be observed when we begin to cool this system, either by shaking the flask in the air or by placing it under the water tap. You can see in advance that as I reduce the temperature a separation of the system must again occur, for the solution phenomena that you have observed is entirely reversible. The solution has now cooled somewhat, but if you observe closely you note at the same time that it has changed in appearance. What was formerly a completely colorless liquid shows now a bluish yellow opalescence entirely identical with the opalescence of an egg white solution, or a highly dispersed mastic colloid. The opalescence increases as the mixture cools. The similarity between the opalescence in our phenol - water mixture and the opalescence of typical colloids compels the conclusion that we have before us a separation of the phenol in the water in colloid form. In fact, consideration of the problem compels the conclusion that such a colloid state must be passed



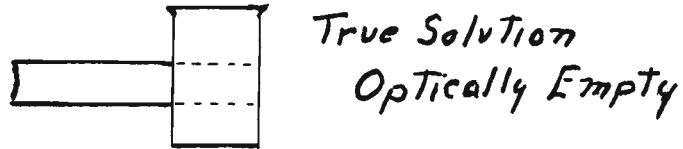
through in the course of the separation in such a system; and it only becomes a question as to whether we are able experimentally to maintain or stabilize this colloid state, when reached, long enough to examine it carefully. We begin with a temperature at which we have to do with a coarsely dispersed colloid, or even a non-dispersed mixture of phenol and water. Evidently somewhere between these two extremes we pass through the colloid realm and the opalescence of the mixture before you renders it probable that we have it before us now. Ultra-microscopic investigation furnishes direct proof that this opalescent critical fluid mixture is really one in which the divided phase exists in colloid dimensions!

With Ostwald's statement for a basis the writer will try to show that phenol and water not only form a colloid through the opalescent stage but in any mixture of phenol and water a colloid is formed.

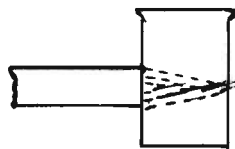
The problem that then arises is to distinguish the colloid from the true solution. This can be done by two methods:-

1. By the Tyndall phenomena. The Tyndall phenomena gives a very delicate test for observing very slightly turbid solutions. The experiments are best carried on in a darkened room with a small arc-light and condenser using a thin walled beaker for the solution to be tested. In making Tyndall experiments it is not the presence of many more or less evident particles that distinguish a colloid from a true solution. It is the intensity of the unbroken light cone passing through the solution that shows the true state of the liquid. The authorities agree that practically all colloid solutions give a Tyndall effect and that the liquids that show no definite Tyndall effect, or show it only in a highly concentrated state, are true solutions.

The Tyndall effect can best be illustrated by the following sketch.



*True Solution  
Optically Empty*



*Colloid  
Tyndall Phenomena*

2. By the Ultramicroscope. You obtain a good idea of the principles of ultramicroscopy if you imagine yourself looking at a Tyndall cone with a microscope. If the brightest spot of the Tyndall cone is thrown just below the objective of the microscope one sees innumerable brilliant points showing beautiful Brownian movement.

The limits of ordinary microscopic visibility are set by the length of the light waves. With the ultramicroscope we can obtain a diffraction picture of particles which are smaller than the length of the light wave.

The methods of testing for colloids have been fully described, and now it remains only to give the results of the actual experiments. These results are given in tabular form so that they may easily be compared.

No.	Parts		Colloid	
	Phenol	Water	Ultra.	Tyndall.
1.	00	20	-	-
2.	.02	20	+	+
3.	.06	20	+	+
4.	.08	20	+	+
5.	.10	20	+	+
6.	.50	20	+	+
7.	1.00	20	+	+
8.	1.50	20	+	+
9.	2.00	20	+	+
10.	2.50	20	+	+
11.	3.00	20	+	+
12.	5.00	20	+	+
13.	6.00	20	+	+
14.	33.40	33.60	+	+
15.	36.00	64.00	+	+
16.	95.00	5	+	+
17.	90.00	10	+	+

In case fourteen the entire volume of the solution is a colloid above 68 degrees centigrade and both layers show colloidal form below 68 degrees centigrade.

It is noticed that both the ultramicroscope and the tyndall cone check each other in all cases.

The effect of temperature is shown in the table given below. A concentration of 33.4 per cent phenol was used.

Temperature	Aqueous layer		Phenol layer	
	U.	T.	U.	T.
	+	+	+	+
20	+	+	+	+
40	+	+	+	+
60	+	+	+	+
65	+	+	+	+
68		Critical Temperature		
		U.	T.	
70		+	+	
75		+	+	
80		+	+	
85		+	+	

The temperatures for the tyndall method in this experiment are accurate, the temperatures for the ultramicroscope are approximate.

The addition of phenol to water until a concentration of 23% was reached gave only a very slight change in the PH of the solution. The following table shows the results of the determination.

c.c. Phenol	c.c Water	PH	E
00	20	4.4	5.4
.02	"	4.2	5.2
.06	"	4.2	5.2
.08	"	4.5	5.5
.10	"	4.5	5.5
.50	"	4.4	5.4
1.00	"	4.4	5.4
1.50	"	4.4	5.4
2.00	"	4.3	5.3
2.50	"	4.2	5.2
3.00	"	4.2	5.2
5.00	"	3.8	4.9
6.00	"	3.8	4.9

The photographs on pages seventeen and eighteen give a very comprehensive idea of the apparatus used in these experiments. The water in the viscosity apparatus was heated to 100 degrees C., and the viscosity readings were taken as the water cooled.

The graphs of the viscosity on pages fifteen and sixteen show the relative viscosity of phenol and water.

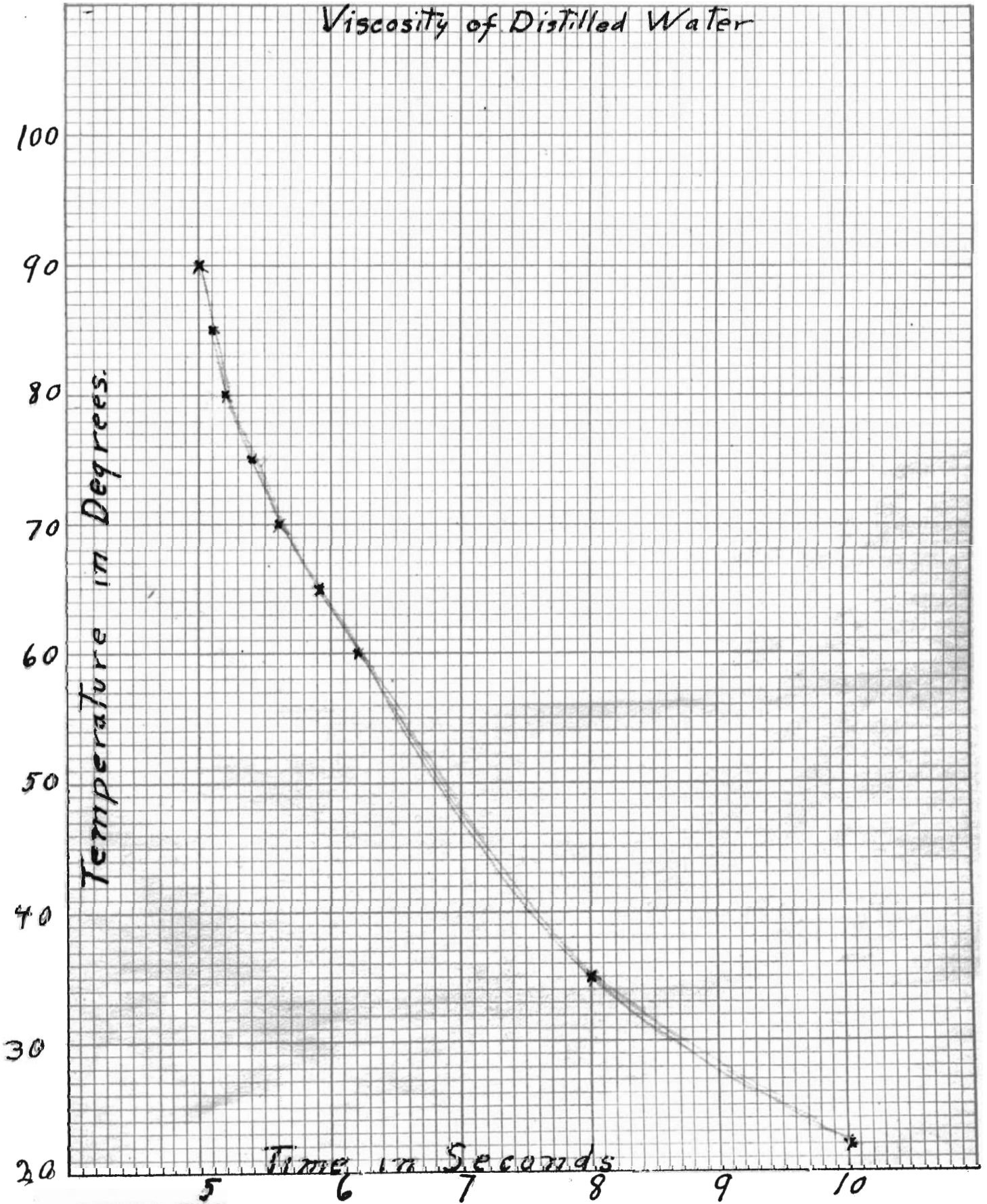
### Conclusion

It seems reasonable to conclude from the foregoing observations; that not only does phenol form a colloid, in the opalescent stage, the concentrations varying from six to eighty per cent, in aqueous solution, but so far as the writer was able to show, forms a colloid in all concentrations and at the temperatures, - between twenty and eighty degrees centigrade.



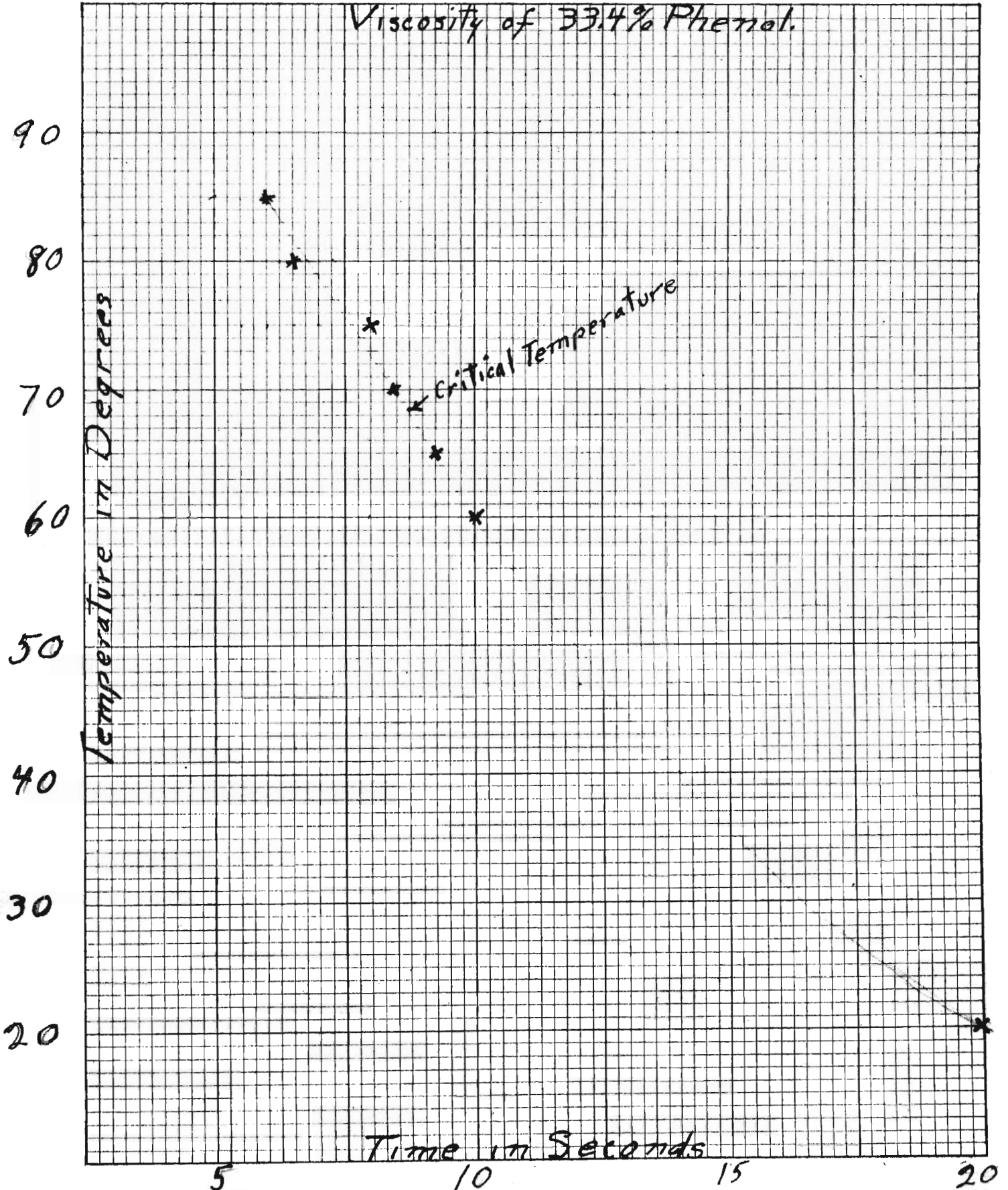
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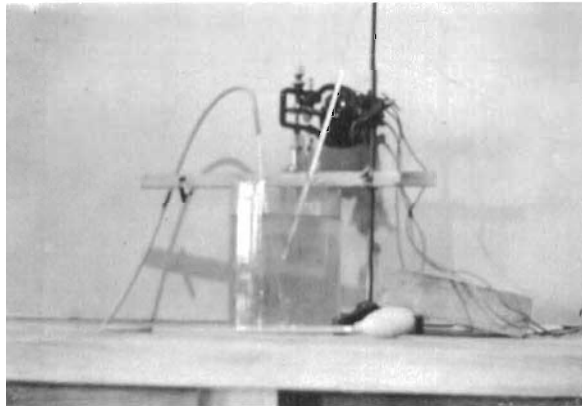
Viscosity of Distilled Water



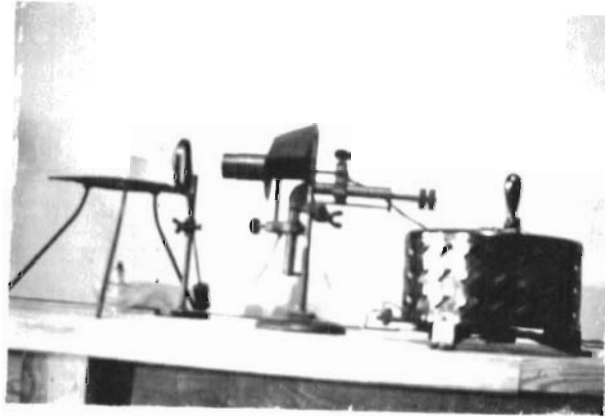
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Viscosity of 33.4% Phenol.

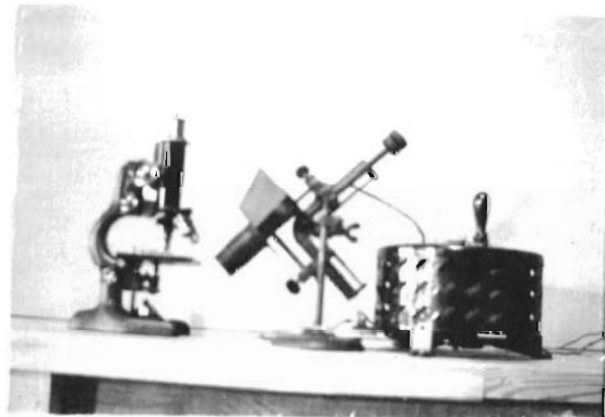




Apparatus used in making  
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Apparatus used to obtain  
Tyndall effects.



Apparatus used with the  
Ultramicroscope.

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