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Effect on Starch on Pigment Particle Packing and Its Effect on the Sheet Properties

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EFFECT OF STARCH
ON
PIGMENT PARTICLE PACKING
AND
ITS EFFECT ON THE SHEET PROPERTIES

by
Michael L. Armstrong

A Thesis Submitted to the
Faculty of the Department of Paper Technology
in partial fulfillment
of the
Degree of Bachelor of Science

Western Michigan University
Kalamazoo, Michigan

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ABSTRACT

The starch-clay suspension is the basis for the coating industry. This study shows that cationic and anionic starch at levels of addition below 10 parts per hundred can disperse the clay to obtain maximum particle packing or minimum void volume. The particle packing effect showed a relatively small effect on the sheet properties. But due to the large variation in coat weight and the physical effects of the adhesive only very general trends could be realized on these sheet properties.

TABLE OF CONTENTS

	PAGE
HISTORICAL BACKGROUND AND DEVELOPMENT	
OF THE PROBLEM	1
Clay	1
Starch	3
Starch-Clay Relationships	5
Relative Sediment Volume	6
Viscosity	7
Sheet Properties	9
PROCEDURE	10
Clay Preparation	10
Starch Preparation	10
R. S. V.	11
Hercules Hi-Shear Viscometer	11
Brookfield Viscometer	12
Drawdown Preparation and Evaluation	12
DISCUSSION OF RESULTS	13
Pigment Dispersion on Sheet Properties	23
CONCLUSIONS	30
BIBLIOGRAPHY	31
APPENDIX	33

HISTORICAL BACKGROUND ON THE
DEVELOPMENT OF THE PROBLEM

Clay is utilized as the primary pigment and starch as the basic adhesive in coating formulations. An improvement upon the starch-clay coating suspension could be a step toward higher quality coatings.

The primary purpose of this study was to determine the effect a starch adhesive has on the particle packing of clay and how this might affect the sheet properties. The most accurate means for measuring the particle packing in a slurry form, without an adhesive, has been the relative sediment volume.

To explore the problem background, the following subjects will be discussed; clay, starch, their effect upon one another in respect to dispersion, viscosity, and sheet properties.

CLAY

The clay used was a Georgia Kaolinite clay which is a hexagonal plate type of hydrous aluminum silicate with the formula $Al_2O_3 \cdot 2.5H_2O$. This structure is essentially neutral but if the structure is broken on the edges, it gives rise to anionic and cationic active areas. Thus, the splitting off of the layers of a clay particle will not give rise to a charge but an edge wise fracture will.

The Kaolin clay is the most widely used clay in paper coating because of its modest cost and its availability in different particle shapes and sizes. Its almost complete insolubility in water makes it less prone to agglomeration and, compared to other coating pigments, clay is the most easily dispersed in aqueous suspensions². Also, these clays are compatible with aqueous solutions of practically all adhesives due to their insolubility and chemical inactivity.

The addition of water to the clay gives a complex system of secondary charges. One of the basic factors with respect to viscosity in the aqueous-clay suspension is the existence of unsaturated or free valence bonds at the crystal edges of the Kaolinite particles³.

Even with a water-clay suspension, agglomeration exists. A dispersing agent is thus needed to overcome this attraction that the clay particles have for one another. The dispersion of clay was very well explained by Asdell in his discussion of the "Double Layer Theory"⁴. The theory basically states that the clay particle has an adsorbed water layer bearing a net negative charge and reacts as a negative ion. In water, the hydrogen ion reacts with the clay to balance the clay charge forming a water hull around the clay particles. Because of the small equilibrium distance that the hydrogen ion has, clay remains in a flocculated state. The hydrogen ions can be replaced by other positive ions through a base exchange reaction. The hydrogen ions can also remain and when a dispersant is added, some of the ions of a certain charge

are preferentially adsorbed onto the pigment surface. These attract the oppositely charged ions or counter-ions. These counter-ions are the ones which are the direct cause of the repulsion between the particles of a suspended pigment stabilized by a dispersant.

A good dispersant creates a layer of counter-ions that have a certain thickness and concentration of electrostatic charge. The clay particles coming together will not force the fields of repulsion to merge completely due to this cloud of counter-ions. Thus, the particles will not make closer contact⁵.

Pigment dispersion can follow two paths. The first path is to disaggregate or disrupt the clusters held together by deposits at their point of contact and is irreversible. The second path is the deflocculation process which is the disruption of particles held in a more loose manner like secondary bonds. It follows that the chemical action of the dispersant must be preceded by mechanical disruption of the particle clusters⁶.

STARCH

Natural starch, a polymer, has too high a viscosity to be used as a binding agent. For this reason, modified starches have been made by acid, oxidation, and enzyme conversion.

Starch is a carbohydrate composed of a 20 percent water soluble fraction called amylose and an 80 percent fraction, which is water insoluble, called amylopectin.

A polymer of glucose units, starch has alpha 1-4 couplings with the oxygen bridge on the opposite side of the glucose unit. This linkage imparts flexibility to the polymer⁷.

By substituting groups which contain a charge into their lattice, another modification of starch is attained.

The starch used in this study was an oxidized starch which is anionic in nature and a chemically modified cationic starch.

The three most common starch products traditionally used in coating are dextrin, enzyme modified, and oxidized. The first two are fragmented to a much smaller dimension than the oxidized starch. Thus, the oxidized starch gives a stronger coating⁸. Because of the charges on the starch used, there should be a definite interaction between them and the clay particles. This should be of a beneficiary nature with respect to dispersion of the clay clusters.

The cationic starch has a definite attraction for both the anionically charged fiber and pigment particle⁹. These starches also improve coating rheology and produce high strength coatings which allows a reduction of binder while maintaining the strength¹⁰.

When starch is used for an adhesive in a clay suspension, optimum results are obtained by the fragmentation of the starch molecules progress only far enough to produce the required viscosity at the desired concentration. The starch should also have its linearity warped enough to overcome the effects of retrogradation, an aligning of the starch molecules to give an insoluble gel or precipitate¹¹.

STARCH-CLAY RELATIONSHIPS

In studying the effect of an adhesive on the particle packing, variables have to be controlled and defined. Particle packing is the manner in which a particle arranges itself with respect to another particle and the proximity of the particles from the standpoint of distance and geometric configuration¹².

The clay used was an undispersed Kaolin. Undispersed clay was used to more effectively measure the result that an adhesive has on particle packing. A dispersed clay tends to overshadow the effect of the starch on the degree of dispersion.

A high percent solids suspension was made with the fluid being water. Keeping a constant solids concentration is very important as it affects the packing and dispersing characteristics of the mixture¹³. This suspension was run at a constant mixing intensity in a high speed mixer for a specified length of time. The degree of dispersion improves as mixing intensity increases. By increasing the speed of the equipment or by increasing the concentration of the pigment, this can be done. Time is also a factor to be controlled because time is required to cause all particles to flow through the zone of highest energy density¹⁴.

After sufficient mechanical deflocculation has occurred, the starch was added to the clay suspension. The amount of starch added was varied in the attempt to get the optimum condition for disper-

sion. This was to be determined by measuring the viscosity and relative sediment volume and obtaining a minimum value when optimum dispersion occurred¹⁵.

It was imperative for the starch to be prepared in the same manner because starch molecules differ when the cooking varies.

RELATIVE SEDIMENT VOLUME

The relative sediment volume (R.S.V.) was measured on the slurry. The R.S.V. is a method of predicting pigment particle agglomeration.

A concept of importance is the fact that an adhesive and all other dissolved materials are a part of the fluid in which the pigment is suspended. By reducing the void volume, dispersing the pigment particles to avoid flocs, a formerly insufficient amount of fluid can become sufficient to fill the void volume furnishing a lubricating film between the particles, thus allowing a lower R.S.V. In this pigment-liquid mixture, the void volume must be filled with liquid. By this concept, the sediment volume is related to the flow behavior of the mixture, and the process of dispersion may be considered to be the process of reduction of sediment volume¹⁶.

The sediment volume (S.V.) is the volume which the pigment occupies when the particles are resting against one another. If the particles are clustered, the S.V. is large, but if the particles

are separate, a minimum of empty space is attained and the S.V. is small.

The relative sediment volume (R.S.V.) is the ratio of the sediment volume of the pigment slurry to the volume of the solids in the slurry. Technically, the S.V. is the volume of pigment (V_p) in a slurry plus the amount of fluid (V_w) just necessary to fill the voids between the pigment particles.

$$\text{S.V.} = V_p + V_w \quad (1.)$$

$$\text{R.S.V.} = (V_p + V_w)/V_p \quad (2.)$$

The volume of the pigment (V_p) is obtained by dividing the pigment weight by the specific density of the clay which is $2.583/\text{cm}^3$. The water volume (V_w) is the difference between the original amount of water added and the amount of supernatant left after centrifugation.

VISCOSITY

Viscosity, the proportionality constant relating the flow of a material to the shear rate, was next measured. In this study, Brookfield and Hercules high shear viscosity were measured at different shear rates. Viscosity is very dependent upon the rate of shear at which they are measured that it is not enough to

measure the viscosity at a single convenient rate of shear¹⁷. High Hercules viscosity which is taken at the maximum shear rate shows an excellent correlation with high sediment volume and closely related to the high void volume¹⁸.

If we have a force of attraction between pigment particles, their ability to flow is restricted. Conversely, if the dispersant prevents particles from attracting one another, the particles attribute to viscosity simply by the volume they occupy and their collisions. Thus, even at the best dispersion possible, there is a minimum to which the viscosity can be lowered.

It was concluded by Hemstock and Bergman¹⁹ that when a clay slip is deflocculated to minimum viscosity, the dried films prepared from the slip can be expected to exhibit minimum porosity, pore size, and scattering coefficient, but maximum gloss. Also, when the particles are deflocculated to minimum viscosity, they have maximum freedom to behave as individuals rather than as agglomerates. The capillary forces exerted on the particles as the film dries results in maximum packing efficiency.

It thus appears that the changes in the film properties which are observed when drying conditions are changed are attributable to the binder rather than to the migration of the fine pigment particles. Their study was made without the use of an adhesive and an absorptive surface for the application of the clay film.

SHEET PROPERTIES

Coat weights were kept as constant as possible because this is imperative to the properties being tested. The paper stock upon which the coating was laid was as uniform as possible as this affects the properties being tested. The drying rate must be constant otherwise the adhesive migration to the surface would greatly influence the test results.

A paper coating may be visualized as a spatial configuration of pigment particles of definite size, shape, and distribution in a matrix composed of air and adhesive, deposited on an irregular web of fiber. Irrespective of adhesive migration into the paper, the dried coating consists of distinct volumes of pigment, adhesive, and air or void²⁰.

As the void volume increases giving more interfaces, the brightness and opacity should increase and the gloss should decrease. As the adhesive level is increased, the brightness, and hiding power of the coating has been observed to go down and gloss development upon calendering becomes harder to accomplish.

Finding the adhesive and clay interaction, especially with respect to pigment particle packing and its related sheet properties, is very important to the coating industry.

PROCEDURE

CLAY PREPARATION

An undispersed clay was chosen as the pigment so as to be able to control the dispersion of the clay. The dry clay, an H.T. Lump, was screened through a #30 screen with 590 micron openings to eliminate large aggregates. A suspension of 50 percent solids was made using just distilled water. The suspension was subjected to a high speed mixer (Eppenbach Model L146525) for a period of 15 minutes. 400 gram slurry samples were made up.

STARCH PREPARATION

Anionic starch, Stayco M, was prepared using 3 parts distilled water to one part starch. The temperature was raised to 200°F. and held there for 15 minutes while under constant agitation. After the 15 minutes, the starch was allowed to cool to 150°F. and then added to the clay water suspension.

The cationic starch, Cato-Kote 1378, was prepared the same way as the anionic starch except the pH was adjusted to 3.5 in order to fully develop the cationic charge.

The starches were added at 0, 1, 2, 3, 5, and 10 parts per hundred based on the weight of the clay in the slurry. The testing of these samples was according to the appropriate following procedure for the R.S.V., Hercules Hi-Shear Viscometer, and the Brook-

field Viscometer.

R.S.V.

Part of each sample was weighed into graduated centrifuge tubes with the graduation marks perpendicular to the axis of rotation so that the sediment volume could be read directly. The samples were then run at 2500 r.p.m. for 30 minutes in an International Centrifuge (size 2, Model V). The total volume and sediment volume were recorded. Relative sediment volume was then calculated as previously shown in equation 2.

HERCULES HI-SHEAR VISCOMETER

The rheogram for each sample was obtained by running the viscometer according to instructions issued with the instrument. All samples were run using the "E" bob and a 25,000 dyne-cm/cm spring set. The maximum r.p.m. was 1100 which corresponds to 11,475 sec.⁻¹ for the shear rate. Viscosity at 11,475 sec.⁻¹, 5610 sec.⁻¹, and 1530 sec.⁻¹ were calculated using the formula below.

$$\eta = \frac{\text{Torque X Bob Constant}}{\text{Shear Rate}} \quad (3.)$$

"E" Bob Constant = .00008

BROOKFIELD VISCOMETER

The Brookfield viscosities were obtained using different spindles at 6 and 12 r.p.m. The instrument was run according to the instructions and viscosities calculated according to the chart with the instrument.

DRAWDOWN PREPARATION AND EVALUATION

Drawdowns were made with each coating using a #11 Meyer rod. The sheets were allowed to air dry in TAPPI standard conditions.

The tests run on the sheets were coat weight, I.P.C. Brightness, Hunter 75^o gloss, Bausch and Laub opacity, and K and N ink absorption. The sheets were also supercalendered using the Western Michigan University lab supercalender using 40 p.s.i. and retested for Hunter 75^o gloss.

DISCUSSION OF RESULTS

Clay in a water suspension has preferentially adsorbed hydroxyl ions on its surface and a counter-ion layer of hydrogen ions. This makes the clay particle in water negatively charged because of the small equilibrium distance. Thus, the point of zero charge lies in the water hull and strong attraction results in large agglomerates.

By adding a cationic starch, a positive ion is introduced and some of the hydrogen ions could be replaced by the positively charged starch. This action could lead to the starch dispersing the pigment. The Brookfield viscosity data shown in Figures 1, 2, 3, and 4 show that the cationic starch containing coatings reach a minimum viscosity at 3 parts per hundred. The Hercules viscosity also reached a minimum in the range of one to 3 parts per hundred of starch addition, as shown in Figure 5. The yield value from the Hercules rheograms exhibited a minimum value at 2 parts per hundred cationic addition as shown in Figure 6. A minimum R.S.V. which wears a minimum void volume or maximum dispersion occurred for cationic starch at one part per hundred. Figure 7 displays the relative sediment volume range. This data shows the starch to act as a dispersing agent. Due to the size of the cationic starch, between one and 3 parts per hundred starch level a maximum equilibrium distance was attained. At this distance,

some of the starch is outside the water hull and a net increase of charge results on the clay particle. This charge because of repulsion disperses the slurry. As more cationic starch is added, the increased concentration of starch in the water medium forces more cationic charges into the water hull by the law of mass effect. This tends to decrease the cloud size resulting in less repulsion or more flocculation.

The anionic starch is believed to draw the counter ion layer of hydrogen ions away from the preferentially absorbed hydroxyl ions. When the concentration of the anionic starch reaches 5 parts per hundred, a maximum equilibrium distance is achieved between the counter ion layer of remaining hydrogen ions and the absorbed hydroxyl ions. The Brookfield viscosity data shown in Figures 1, 2, 3, and 4 support this trend as they reach a minimum viscosity at 5 parts per hundred level of anionic starch addition. A minimum viscosity exhibited by Figure 5 for the Hercules viscosity also occurred at 5 parts per hundred. The yield value illustrated in Figure 6 also shows its minimum value at 5 parts per hundred level of anionic starch. Figure 7 exhibits a maximum dispersion or minimum R.S.V. at 5 parts per hundred for anionic starch. As more anionic starch is added, the concentration increases, the starch molecules due to the law of mass effect move in closer to the clay particle. This forces the equilibrium distance to decrease. Thus, flocculation is on the increase. These trends for the anionic

starch are illustrated in Figures 1 - 7.

In Figures 1, 2, 3, and 4, it has been shown that the minimum Brookfield viscosity of the cationic starch was lower than that of the anionic starch but above 5 parts per hundred level of starch addition, the anionic starch yields a lower viscosity. Figure 5 parallels the trends exhibited by the Brookfield data showing the Hercules viscosity for the cationic starch to be lower than the anionic starch until 5 parts per hundred of starch addition was reached. Above this level anionic starch has a lower viscosity. R.S.V. which correlates with viscosity as shown in Figure 7 shows that above 2 parts per hundred of starch addition, the anionic starch shows a lower R.S.V. value. The dispersion characteristics of cationic starch are most effective around 2 parts per hundred level of addition while the anionic starch does not reach its potential for dispersion until 5 parts per hundred of starch was added.

FIG. 1

EFFECT OF STARCH LEVEL ON
BROOKFIELD VISCOSITY SPINDLE 3, 6RPM

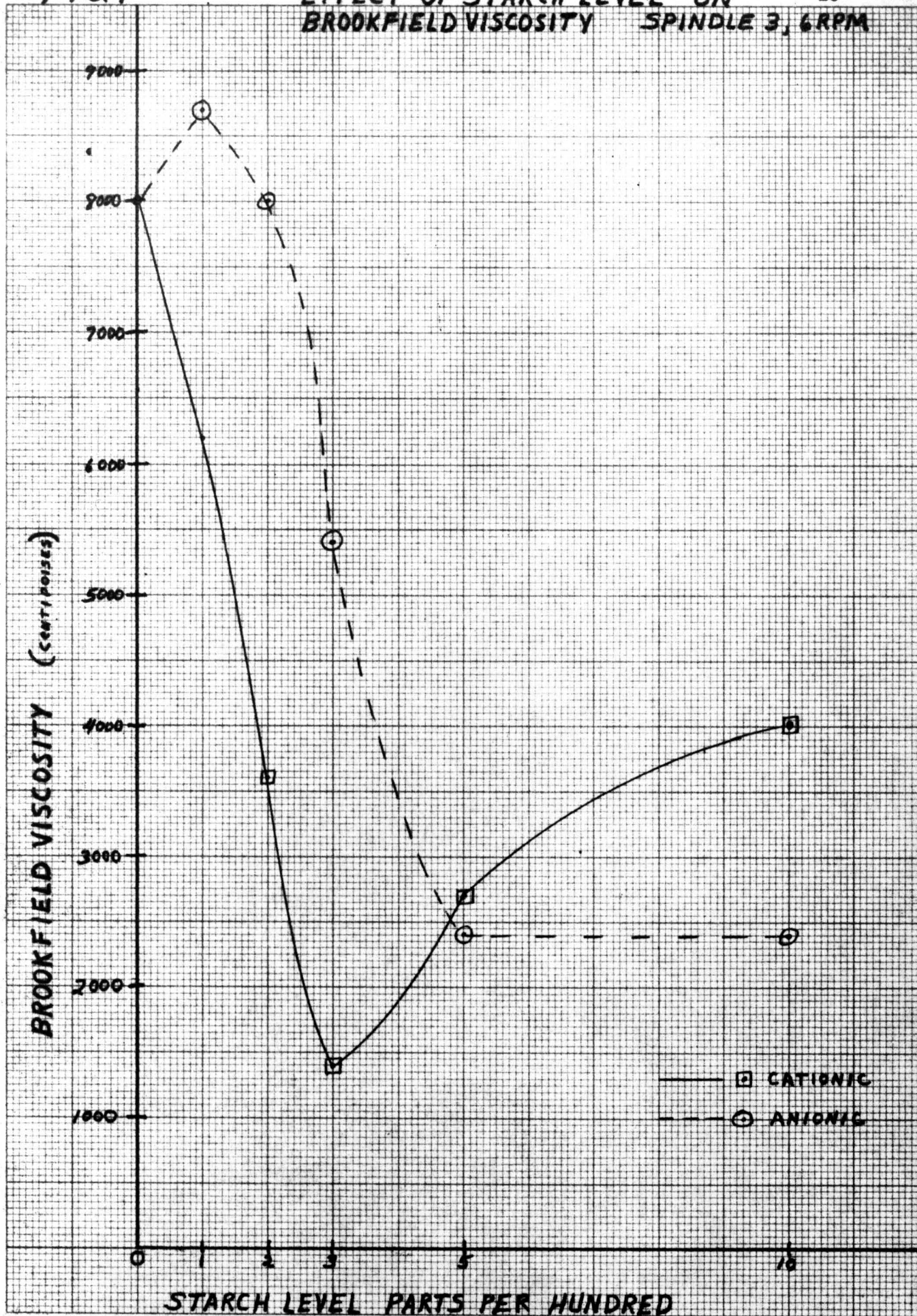


FIG. 2

EFFECT OF STARCH LEVEL ON
BROOKFIELD VISCOSITY SPINDLE 3, 12 R.P.M.

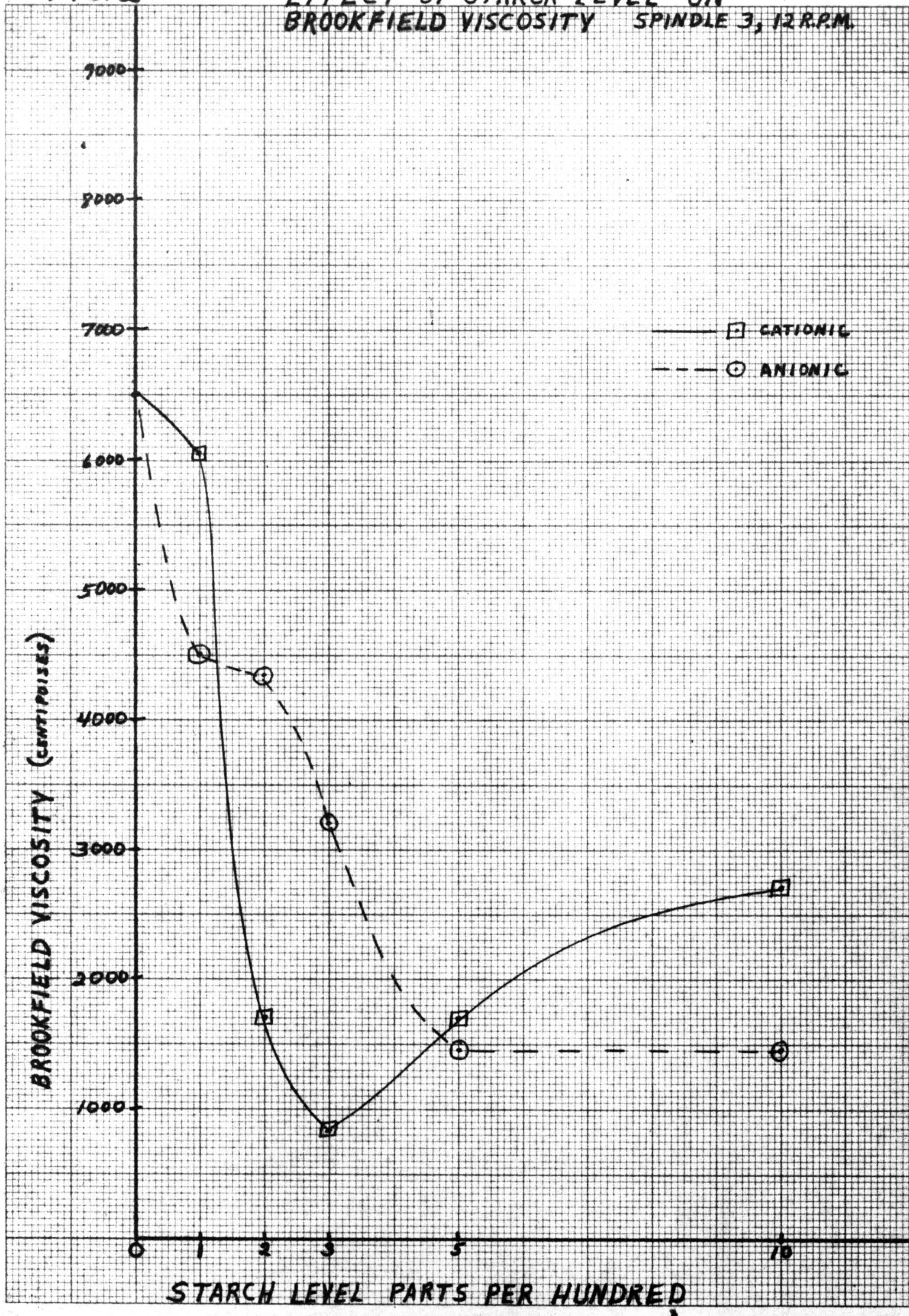


FIG. 3

EFFECT OF STARCH LEVEL ON
BROOKFIELD VISCOSITY SPINDLE 4, 6R.P.M.

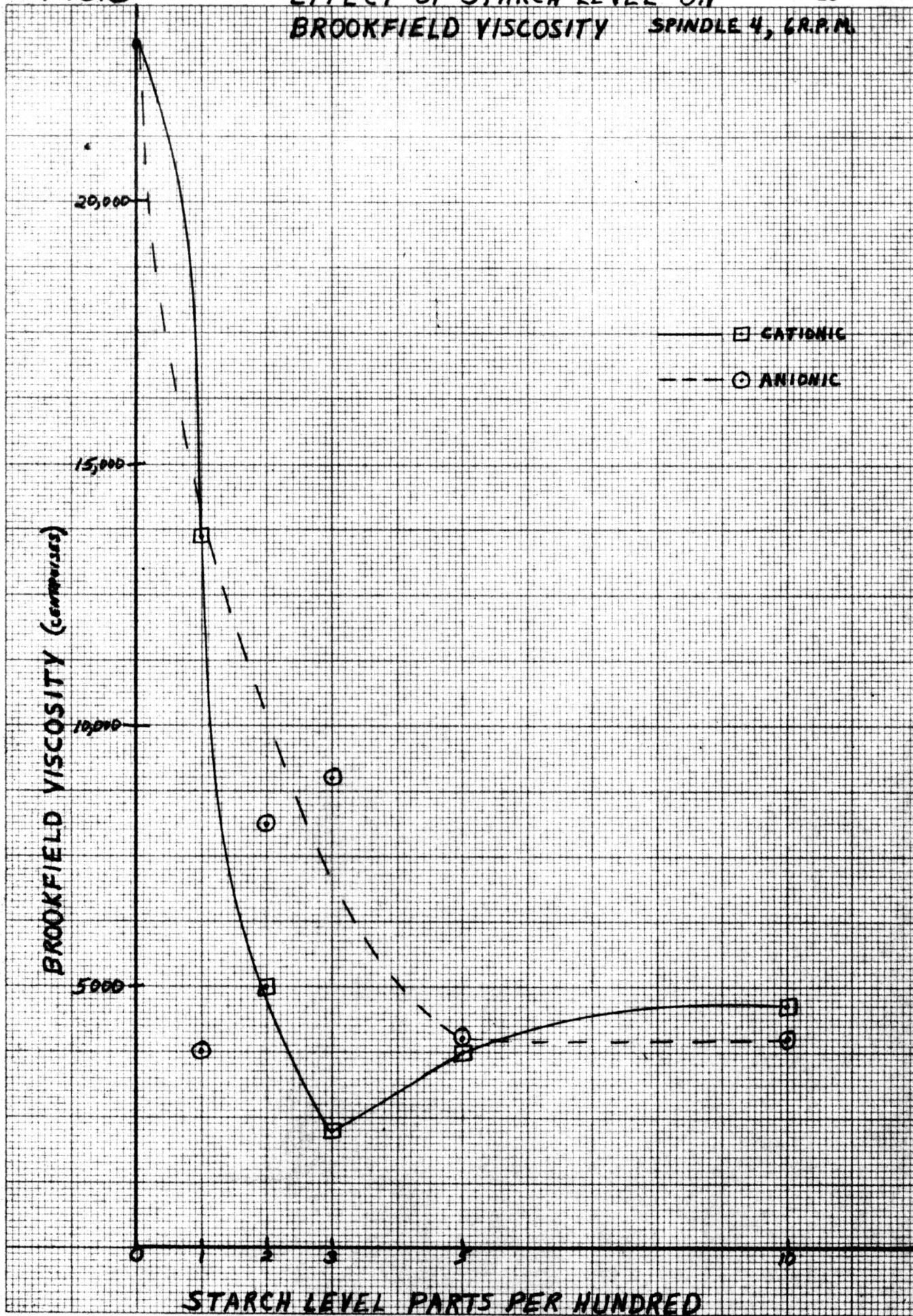


FIG. 4

EFFECT OF STARCH LEVEL ON
BROOKFIELD VISCOSITY SPINDLE 4, 12 R.P.M.

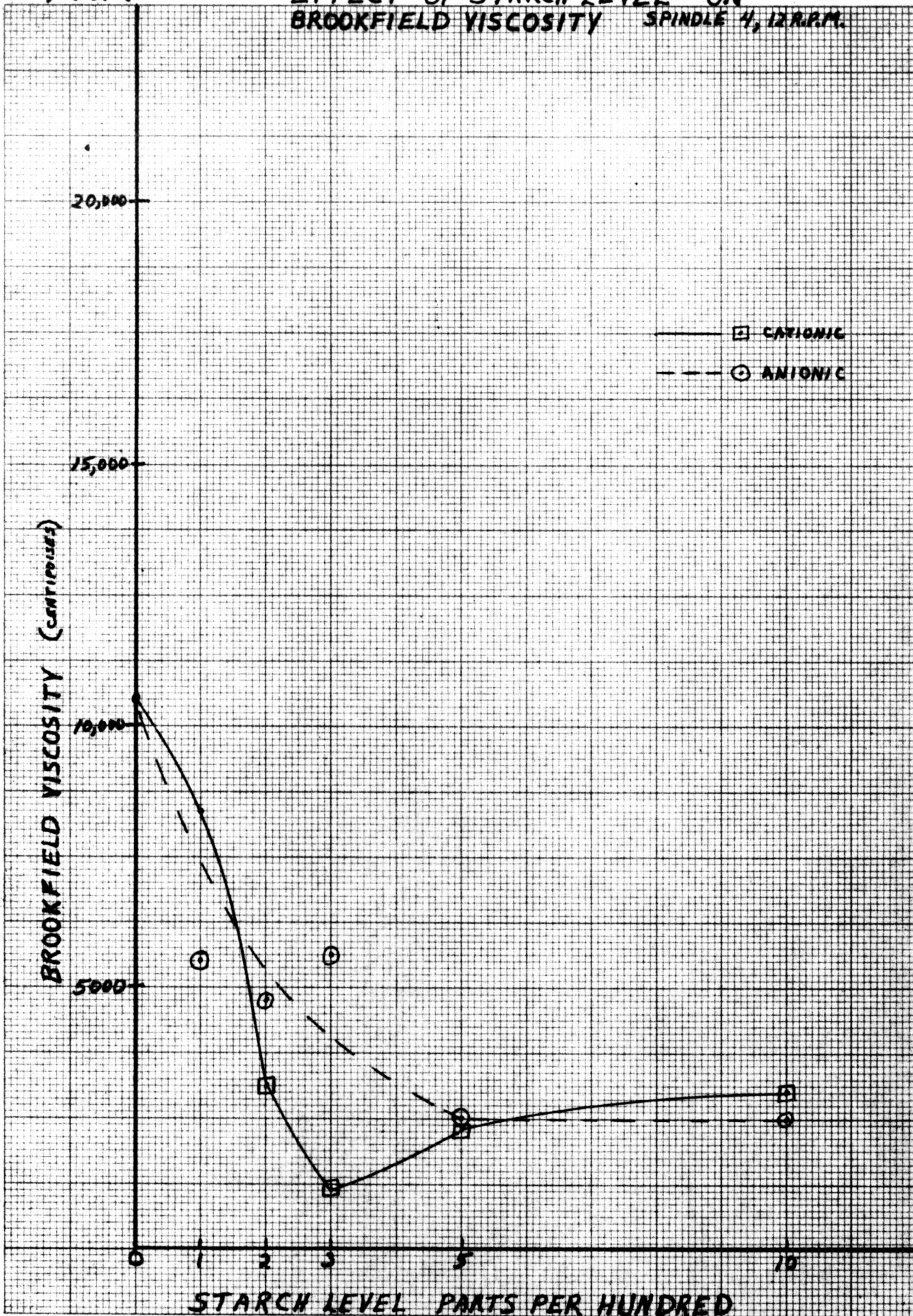


FIG. 5

EFFECT OF STARCH LEVEL ON HERCULES VISCOSITY

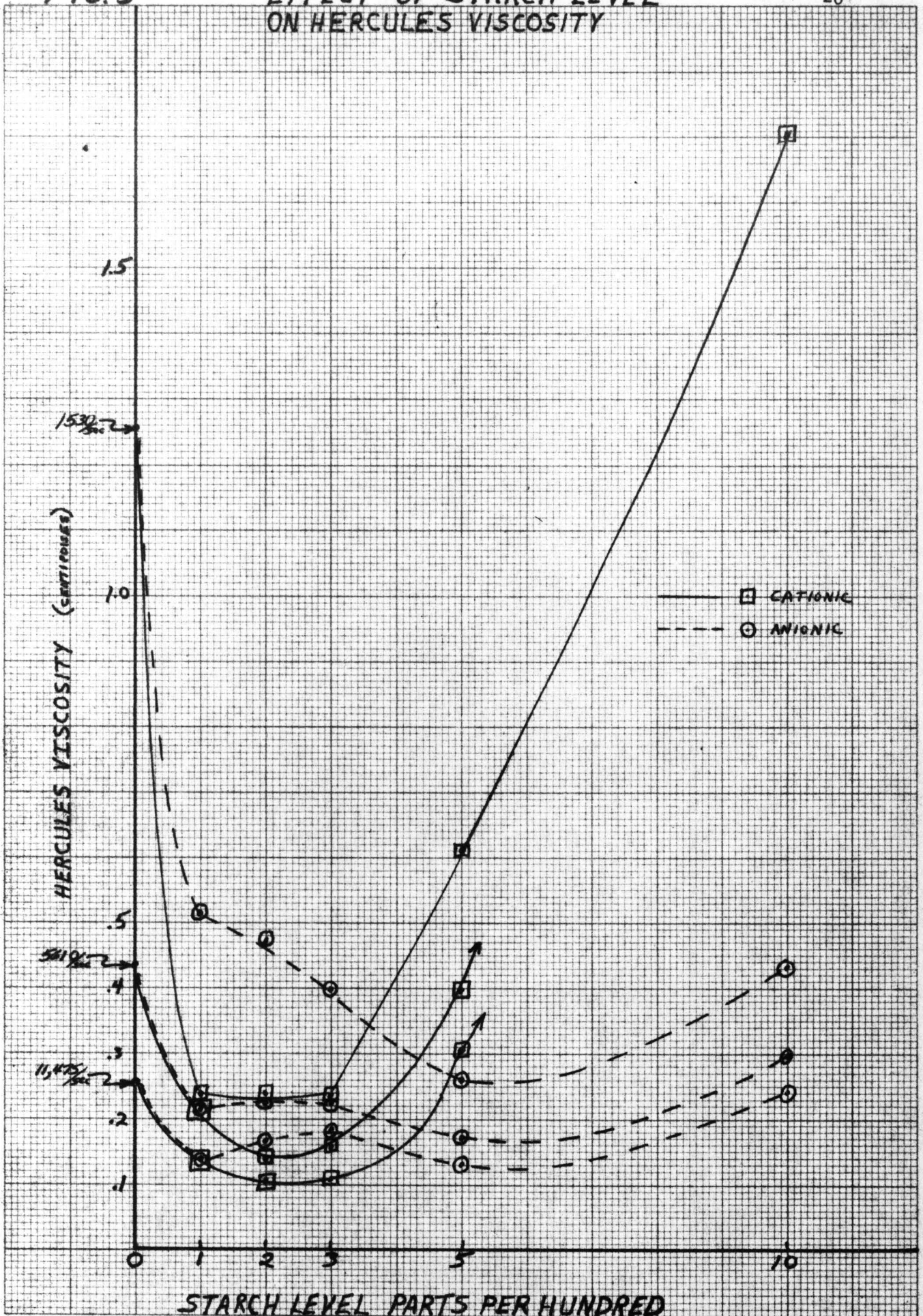
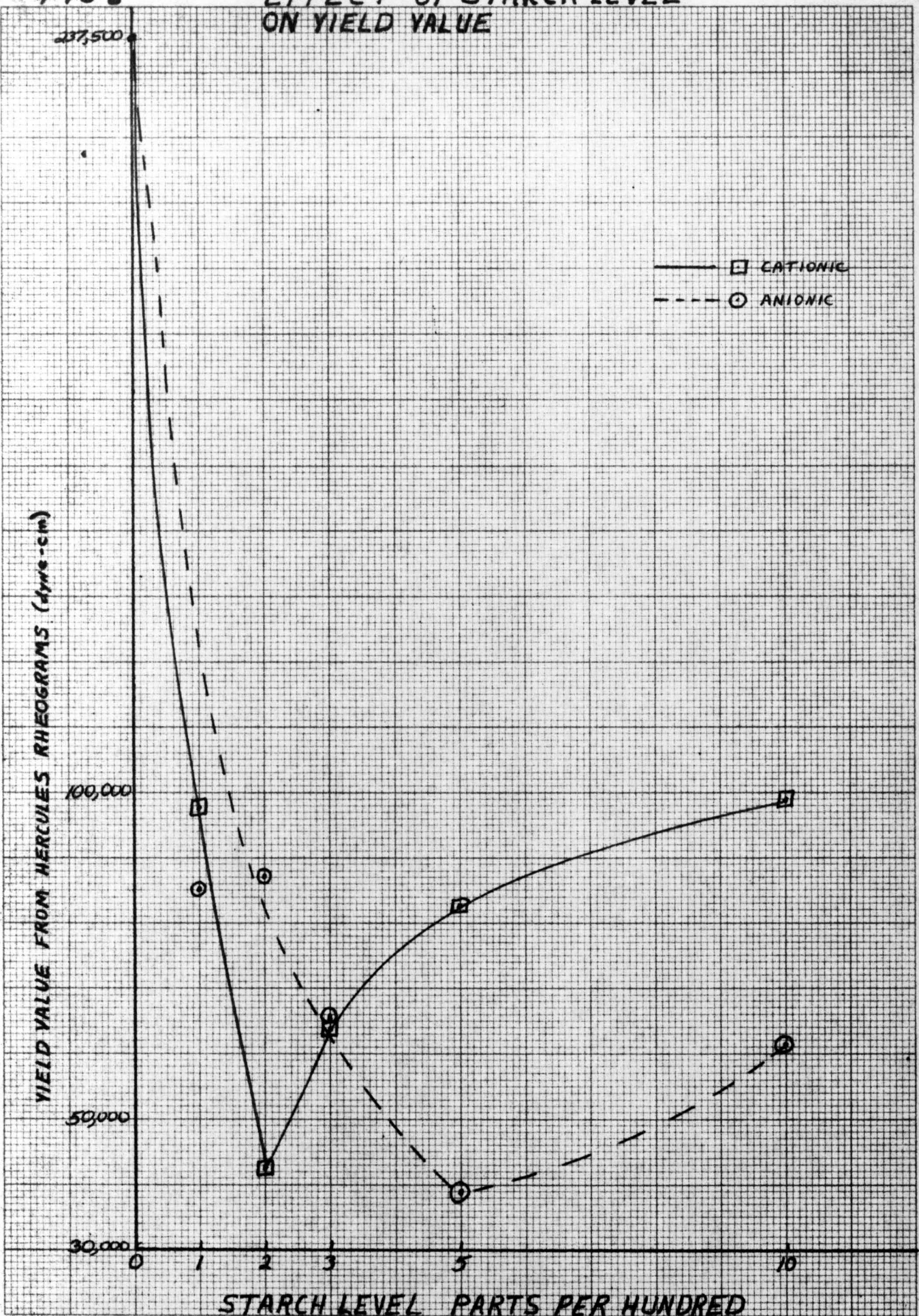
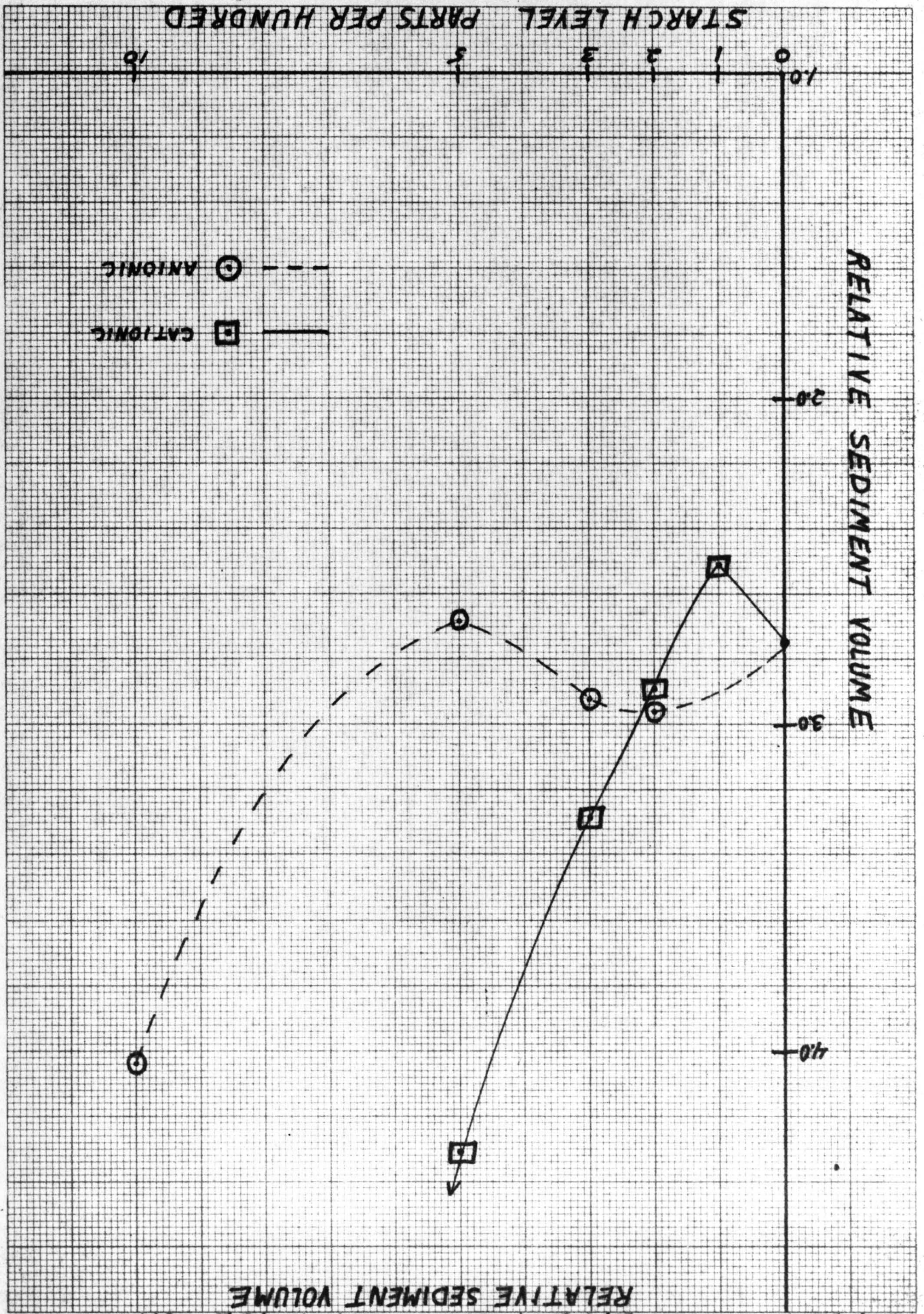


FIG 6

EFFECT OF STARCH LEVEL ON YIELD VALUE





PIGMENT DISPERSION ON SHEET PROPERTIES

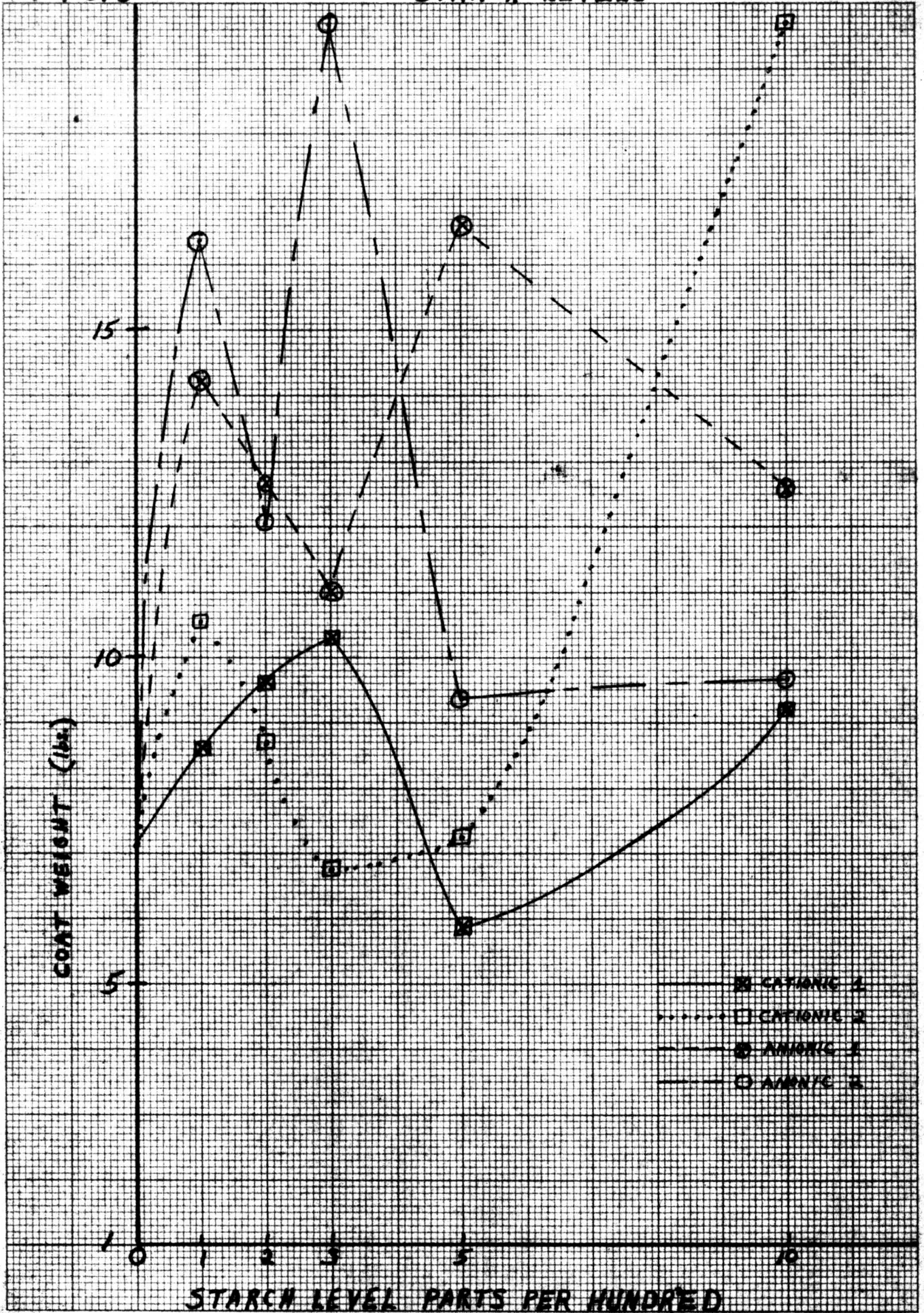
Due to the large variation in coat weight as shown in Figure 8 only very general trends can be obtained for brightness and opacity. Brightness decreased with increasing starch level. Overall, the cationic starch brightness was lower than the anionic brightness. Brightness trends are shown in Figure 9. The opacity as exhibited in Figure 10 decreased with increasing starch level of addition.

On the uncalendered sheet, the cationic starch increases the gloss from the control while anionic starch decreases the gloss. This trend is shown in Figure 11. After calendering, the cationic starch showed a much higher gloss improvement than the anionic starch before the 3 parts per hundred addition. After 3 parts per hundred addition, the cationic starch due to its great increase in void volume exhibits a much faster decrease in gloss improvement than the anionic starch. This curve is shown in Figure 12.

K and N ink values should increase with increasing void volume in the coating. However due to the increasing starch level and variation in coat weight, the only deduction that can be made was that the cationic starch exhibited a higher K and N ink value than the anionic starch. Figure 13 exhibits this trend.

COAT WEIGHT APPLIED AT DIFFERENT STARCH LEVELS

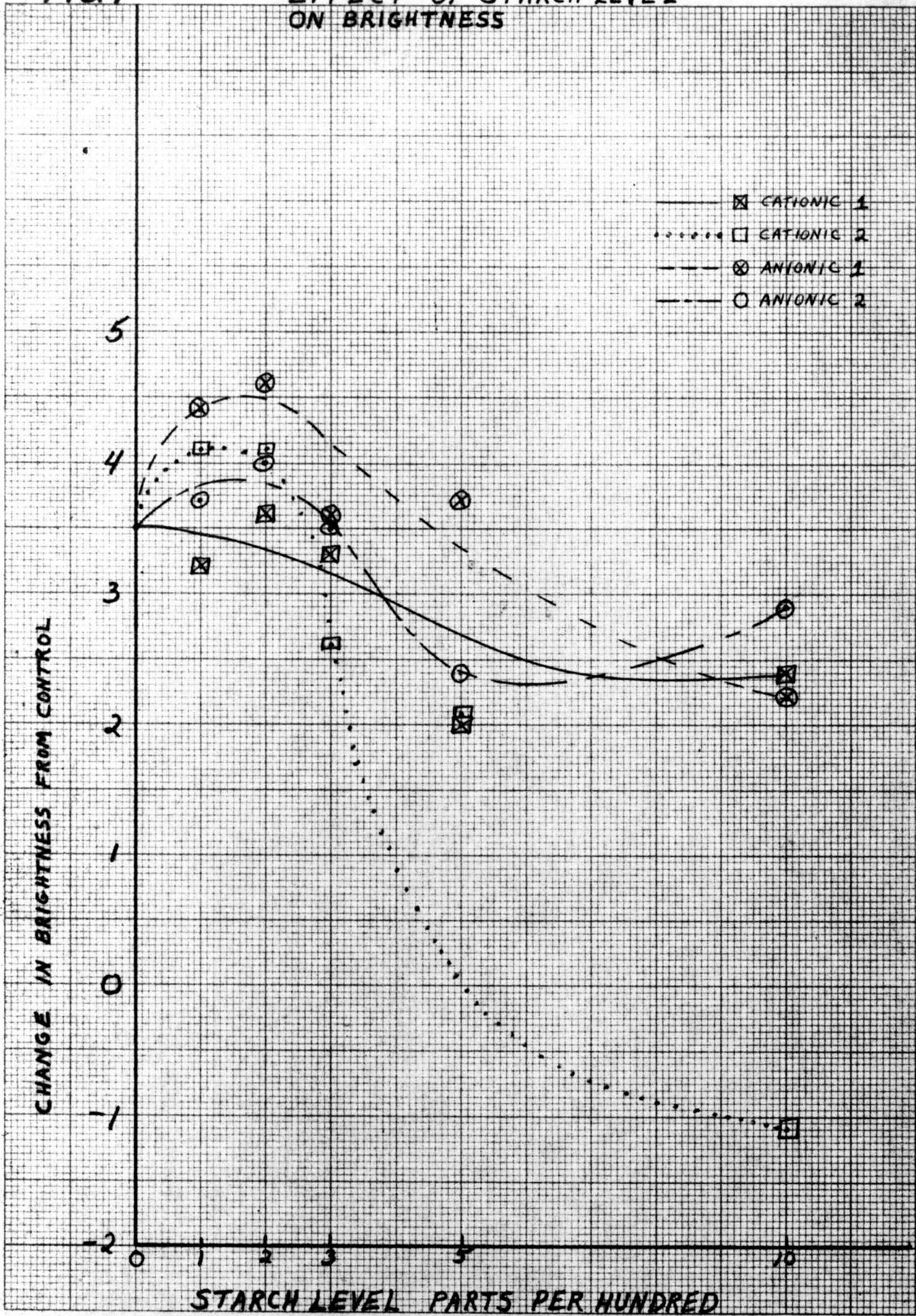
FIG. 8

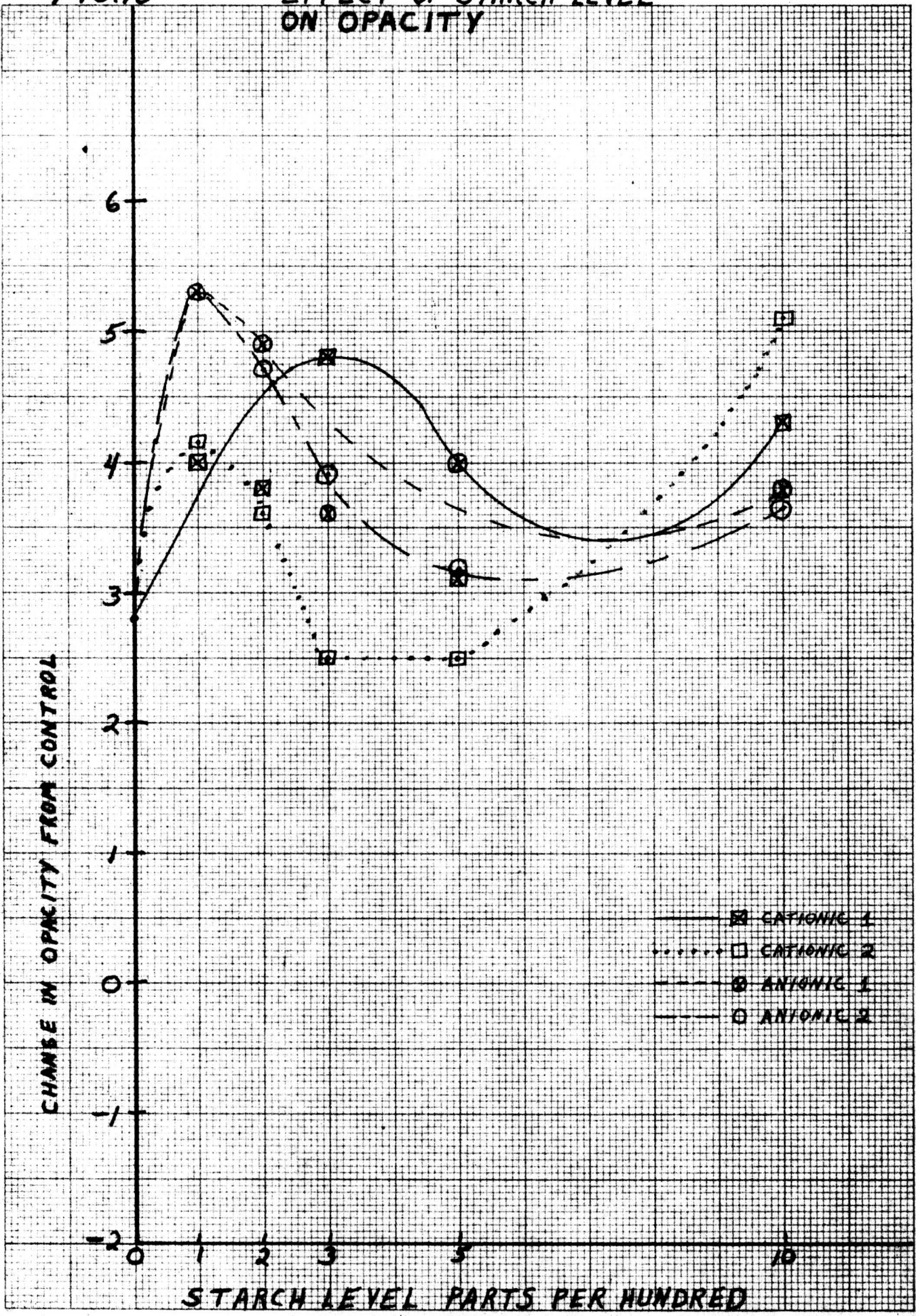


20 50 100 PARTS PER HUNDRED

FIG. 9

EFFECT OF STARCH LEVEL ON BRIGHTNESS





CHANGE IN OPACITY FROM CONTROL

—■— CATIONIC 1
□..... CATIONIC 2
 - - - - - ⊙ - - - - - ANIONIC 1
 —○— ANIONIC 2

STARCH LEVEL PARTS PER HUNDRED

FIG. 11

EFFECT OF STARCH LEVEL ON NONCALENDERED GLOSS

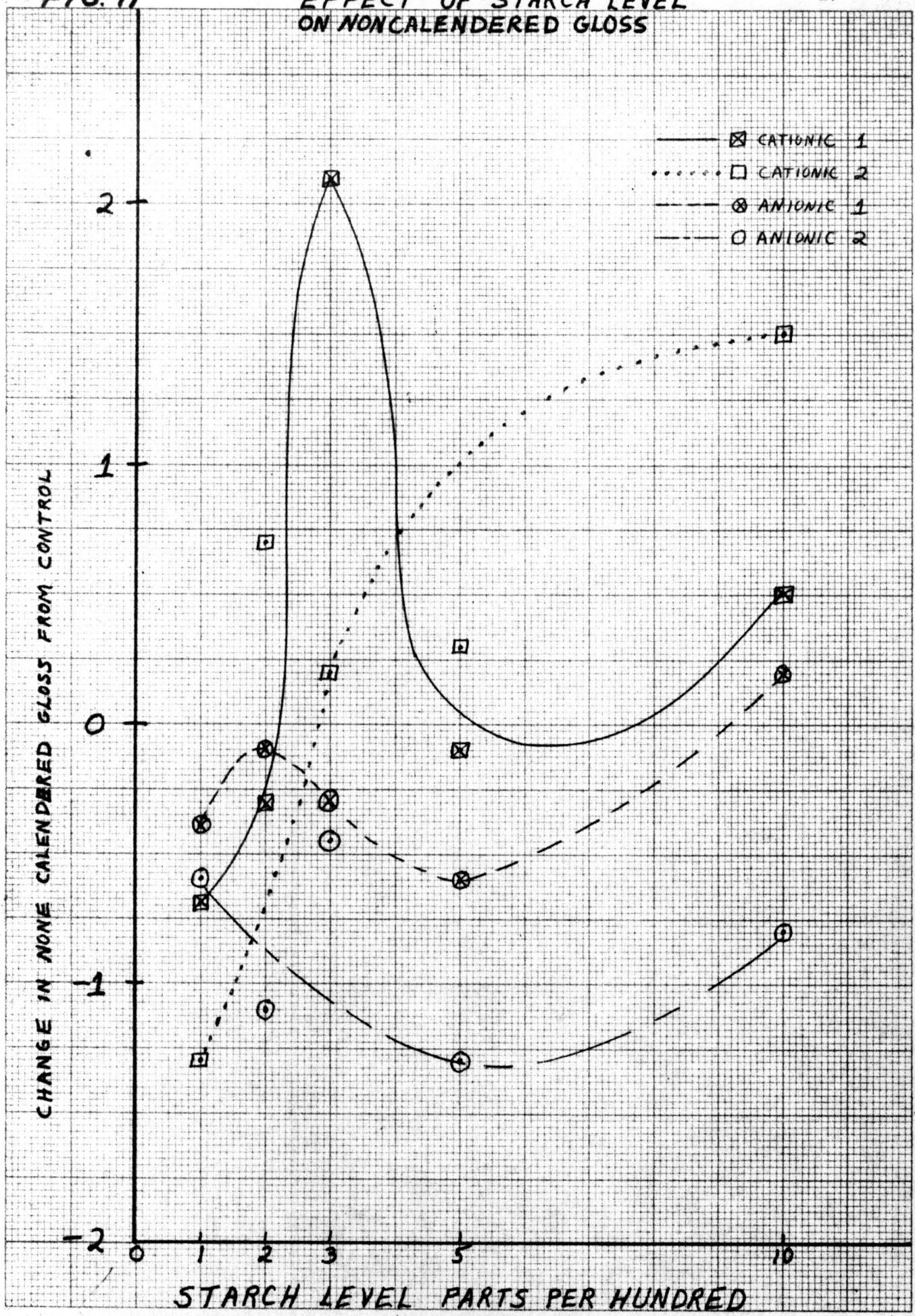
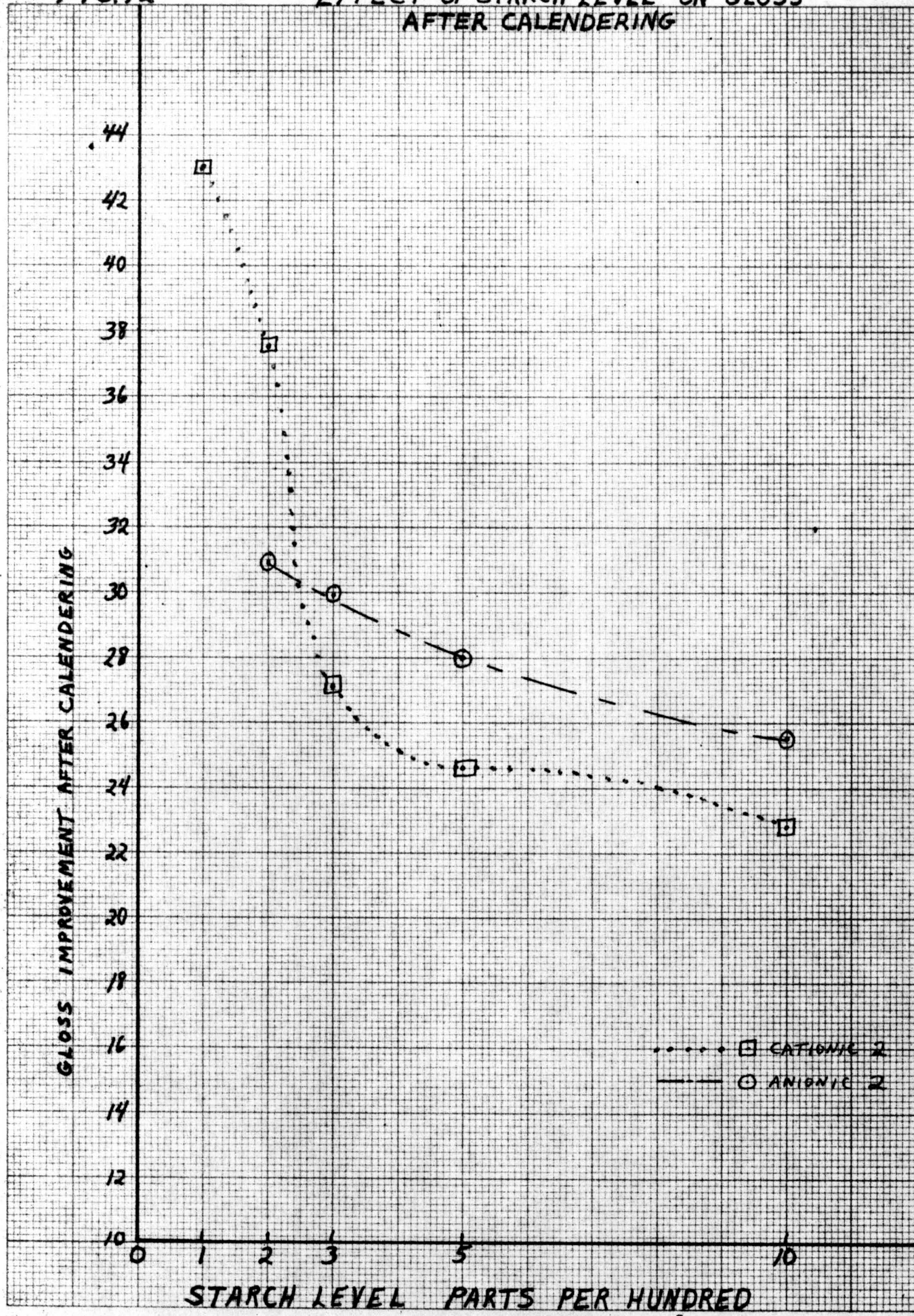


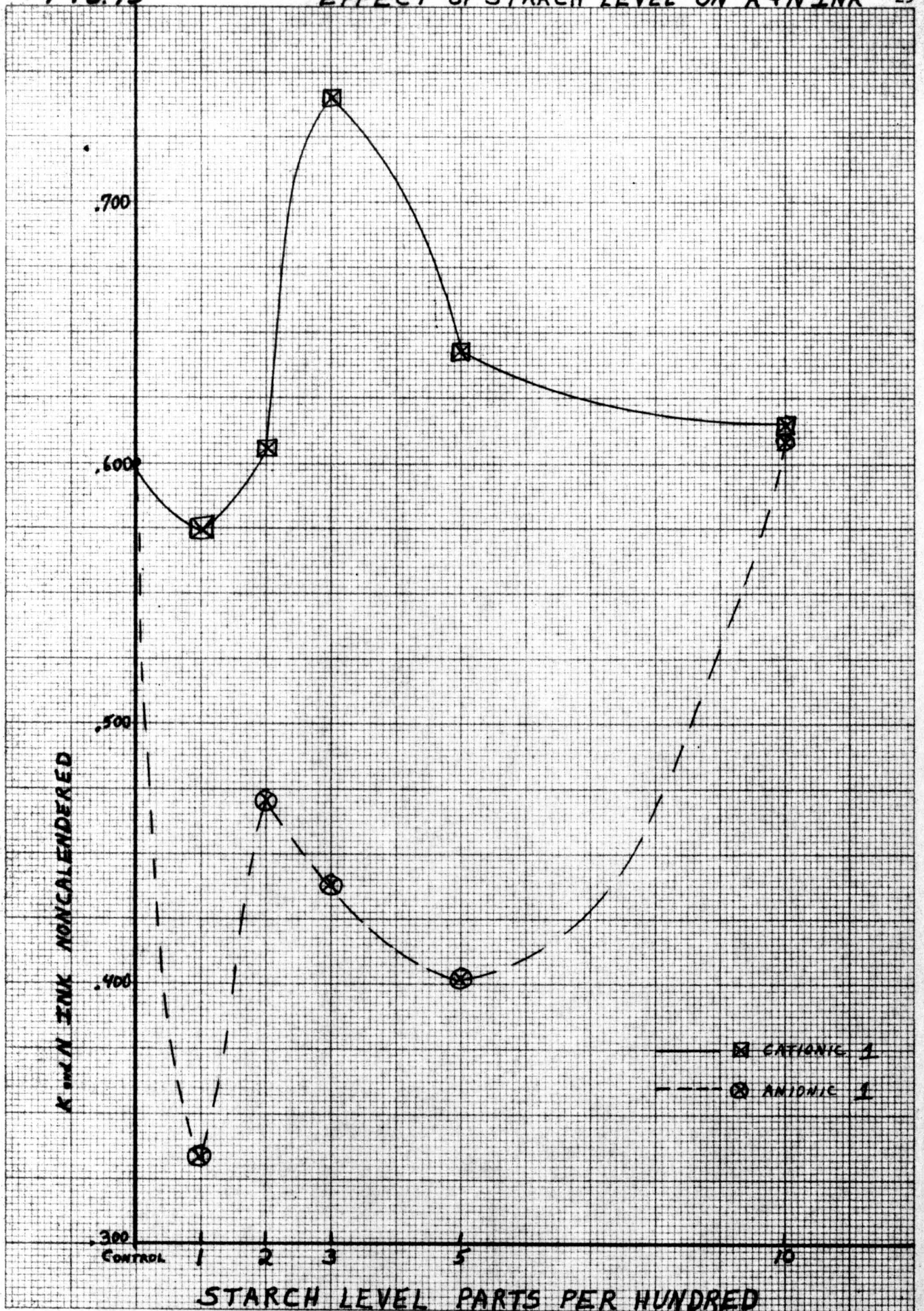
FIG. 12

EFFECT OF STARCH LEVEL ON GLOSS
AFTER CALENDERING



PAPER BY MANUFACTURE
 OF THE UNITED STATES OF AMERICA

FIG. 13



CONCLUSIONS

The dispersion characteristics of cationic starch are most effective around 2 parts per hundred level of addition. At this level, viscosity, relative sediment volume, and yield value all show a minimum value. This indicates a low void volume, effective dispersing action and maximum and uniform particle packing. The anionic starch didn't reach its potential for dispersion until 5 parts per hundred level of starch was added.

Due to the large variation in coat weight and the effect of a non-white adhesive, no real results were obtained from the data on sheet properties.

Starch can be used to disperse an undispersed clay water system but upon addition of more of this starch, the void volume increases rapidly and less particle packing is achieved.

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APPENDIX

Starch Level	Slurry Conc. % Solids	R. S. V.	Hercules -1 11,475 Sec Centipoises	Hercules -1 5610 Sec Centipoises	Hercules -1 1530 Sec Centipoises	Yield Value dyne-cm
OppH	39.6	2.7343	.254	.434	1.256	237,500
<u>Cationic</u>						
1ppH	38.8	2.5307	.139	.214	.235	97,500
2ppH	38.9	2.8902	.105	.139	.235	42,400
3ppH	39.8	3.2861	.107	.157	.229	63,800
5ppH	41.4	4.3185	.305	.394	.614	82,500
10ppH	43.2	(1)	(2)	(2)	1.704	98,900
<u>Anionic</u>						
1ppH	38.9	(3)	.141	.214	.516	85,000
2ppH	40.3	2.9650	.165	.228	.471	87,500
3ppH	40.7	2.9207	.181	.230	.398	64,900
5ppH	39.5	2.6774	.130	.171	.261	38,800
10ppH	40.5	4.0443	.241	.296	.431	61,250

(1) Would not settle out.

(2) Too high to calculate.

(3) Two runs were made and the centrifuge tubes broke both times, duplicates could not be run due to shortage of coating.

Starch Level	Slurry Conc. % Solids	R. S. V.	Brookfield Viscosity #3 6 RPM Centipoises	Brookfield Viscosity #3 12 RPM Centipoises	Brookfield Viscosity #4 6 RPM Centipoises	Brookfield Viscosity #4 12 RPM Centipoises
OppH	39.6	2.7343	8000	6500	23,000	10,000
<u>Cationic</u>						
1ppH	38.8	2.5307	6200	6050	14,000	8200
2ppH	38.9	2.8902	3600	1700	5,000	3100
3ppH	39.8	3.2861	1400	830	2,200	1200
5ppH	41.4	4.3185	2700	1700	3,800	2300
10ppH	43.2	(1)	4010	2700	4,500	3200
<u>Anionic</u>						
1ppH	38.9	(3)	8700	4500	3,800	5500
2ppH	40.3	2.9650	8000	4350	8,000	4600
3ppH	40.7	2.9207	5400	3200	9,000	5600
5ppH	39.5	2.6774	2400	1450	4,000	2500
10ppH	40.5	4.0443	2400	1450	4,000	2500

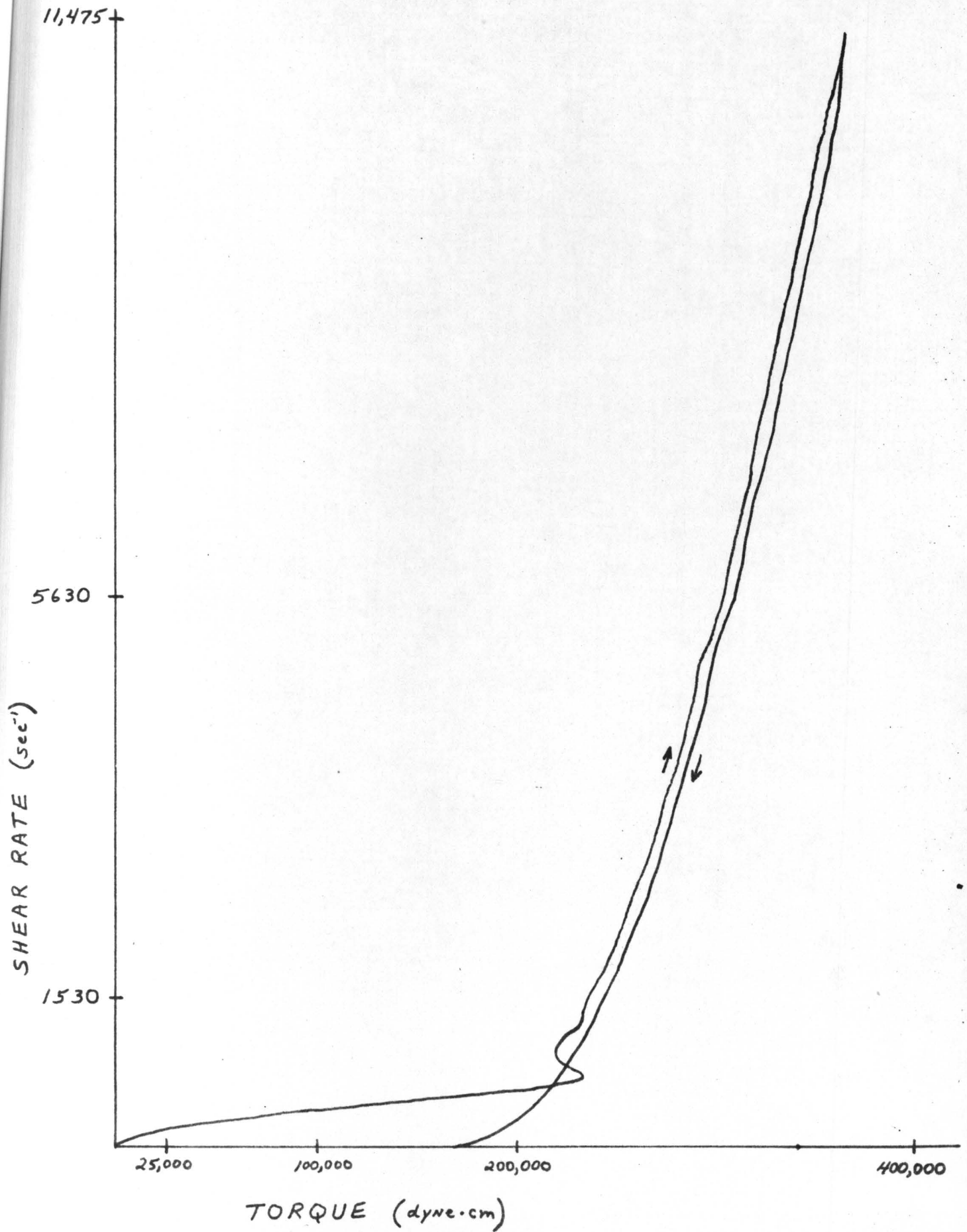
(1) Would not settle out.

(2) Too high to calculate.

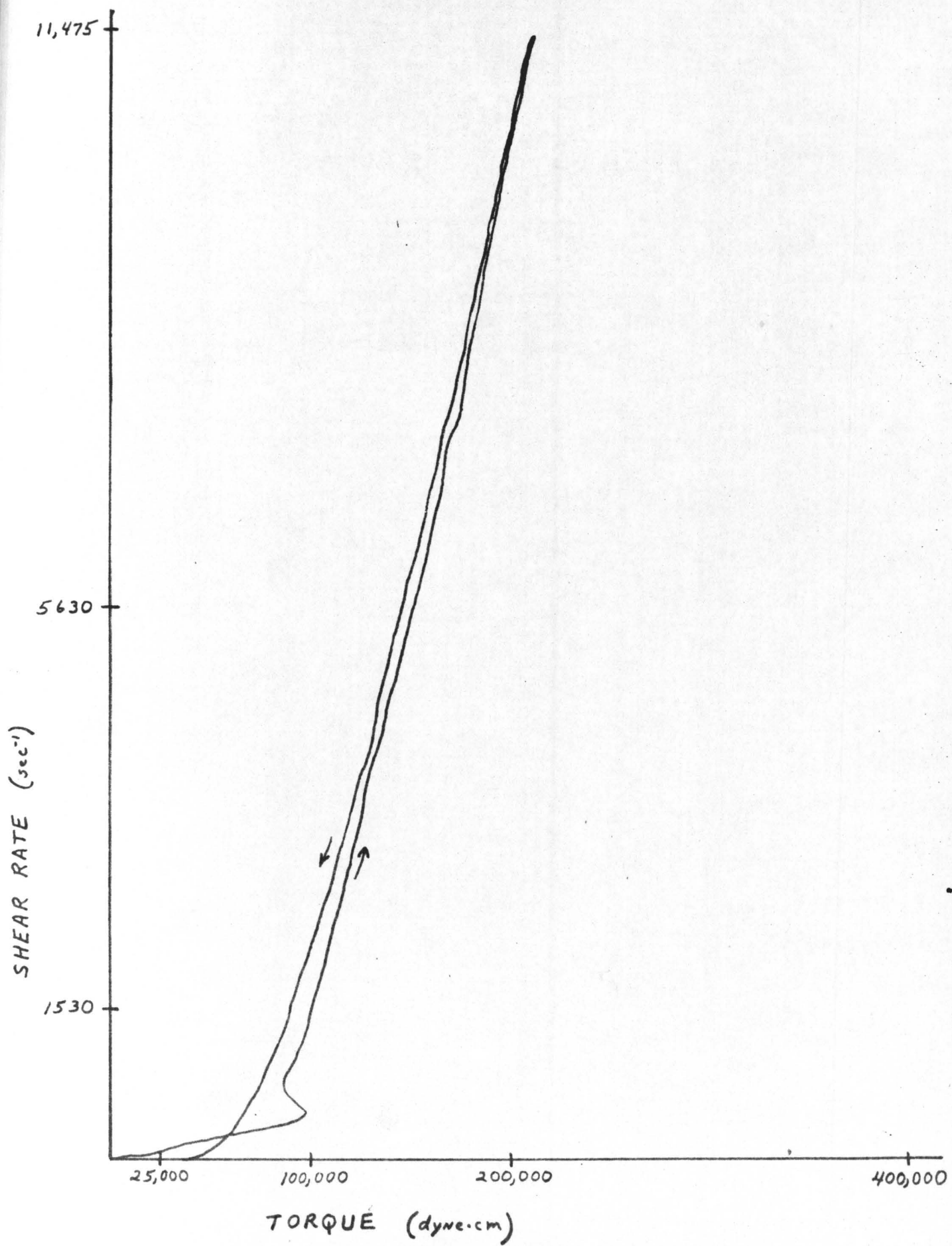
(3) Two runs were made and the centrifuge tubes broke both times, duplicates could not be run due to shortage of coating.

Starch Level	Coat Weight (lbs.)	Change in Brightness from Control	Change in Opacity from Control	Change in Uncalendered Gloss from Control	Gloss Improvement by Calendering	K & N Ink Uncalendered
Control	0	0	0	0		.600
OppH	7.09	3.5	2.8			
<u>Cationic 1</u>						
1ppH	8.60	3.2	4.0	- .7		.574
2ppH	9.62	3.6	3.8	- .3		.606
3ppH	10.33	3.3	4.8	+2.1		.741
5ppH	5.85	2.0	3.1	- .1		.643
10ppH	9.22	2.4	4.3	+ .5		.615
<u>Cationic 2</u>						
1ppH	10.55	4.1	4.2	-1.3	+43.0	
2ppH	8.67	4.1	3.6	+ .7	37.5	
3ppH	6.75	2.6	2.5	+ .2	27.1	
5ppH	7.23	2.1	2.5	+ .3	24.6	
10ppH	19.69	-1.1	5.1	+1.5	22.8	
<u>Anionic 1</u>						
1ppH	14.36	4.4	5.3	- .4		.332
2ppH	12.63	4.6	4.9	- .1		.470
3ppH	10.99	3.6	3.6	- .3		.438
5ppH	16.61	3.7	4.0	- .6		.403
10ppH	12.58	2.2	3.8	+ .2		.609
<u>Anionic 2</u>						
1ppH	16.35	3.7	5.3	- .6		
2ppH	12.04	4.0	4.7	-1.1	30.9	
3ppH	19.69	3.5	3.9	- .4	29.9	
5ppH	9.34	2.4	3.2	-1.3	28.0	
10ppH	9.67	2.9	3.7	- .8	25.5	

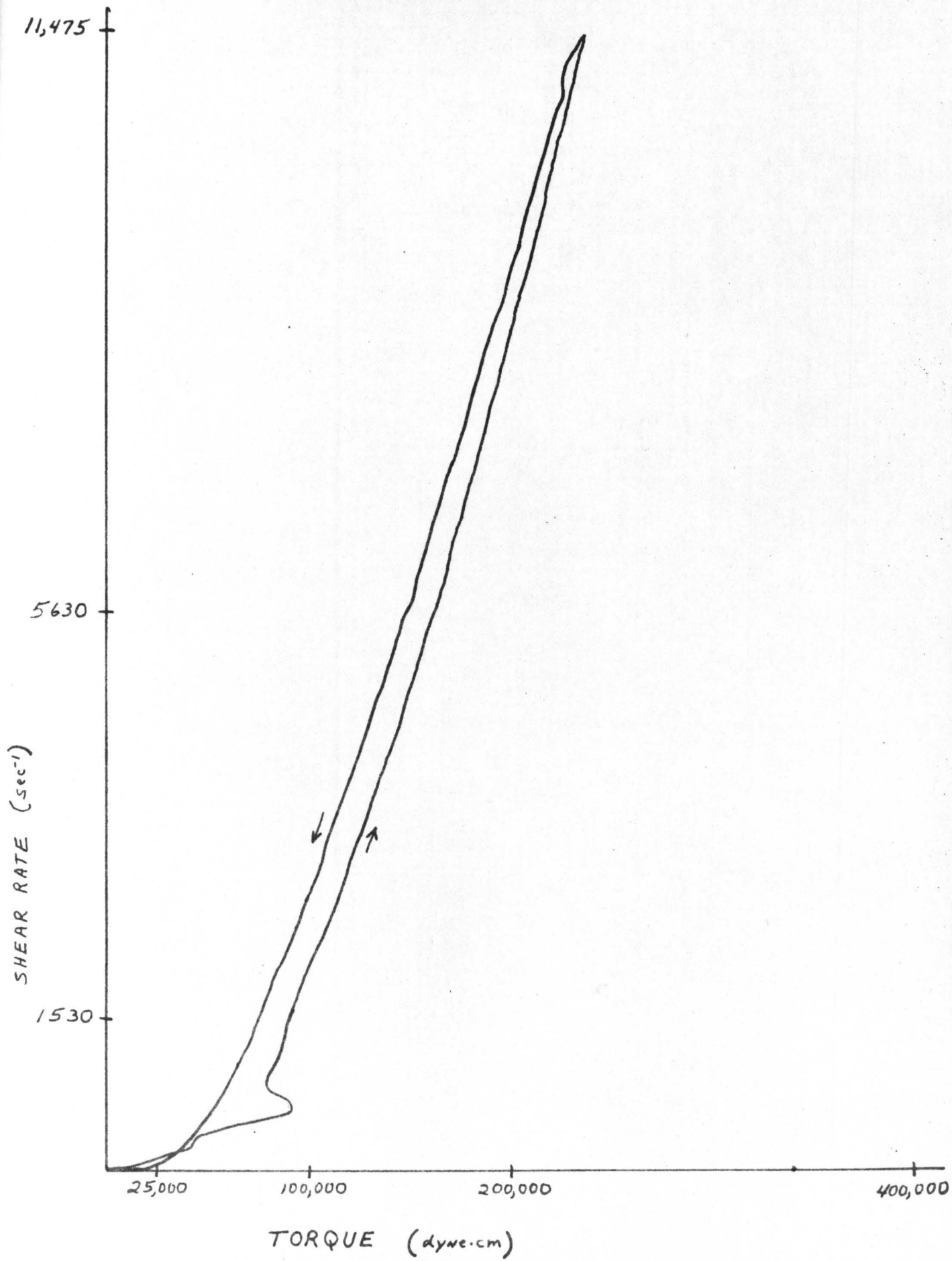
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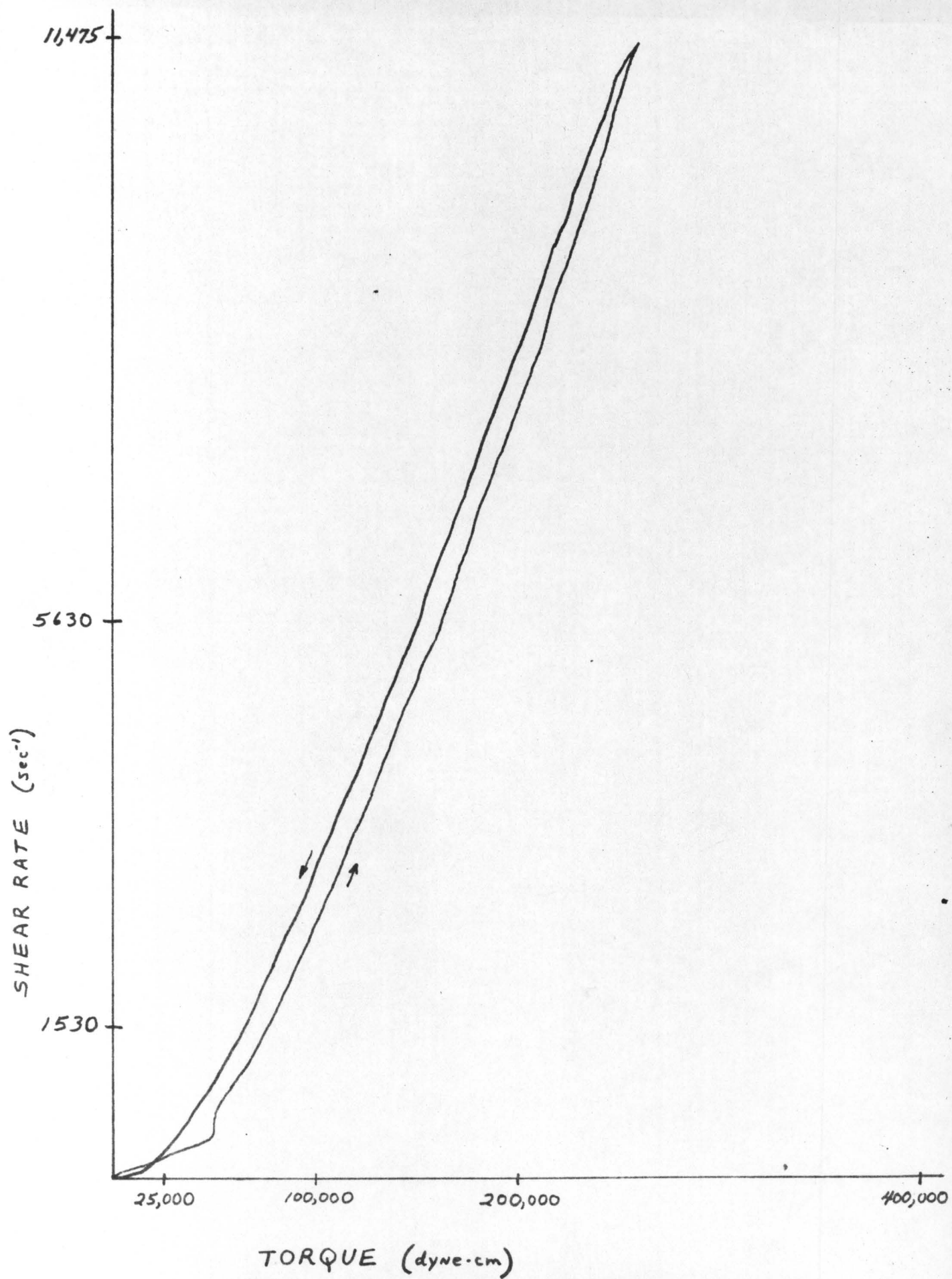
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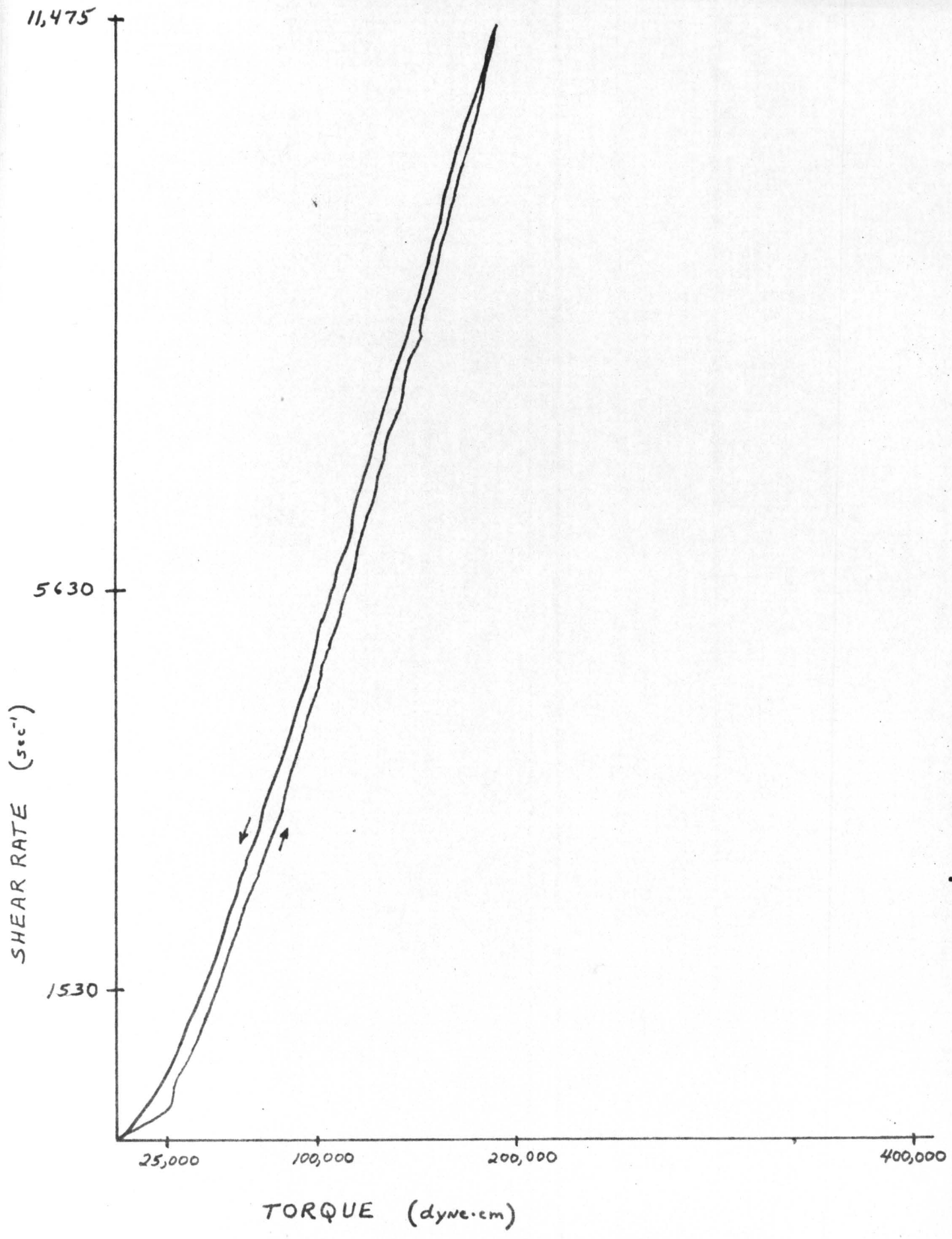
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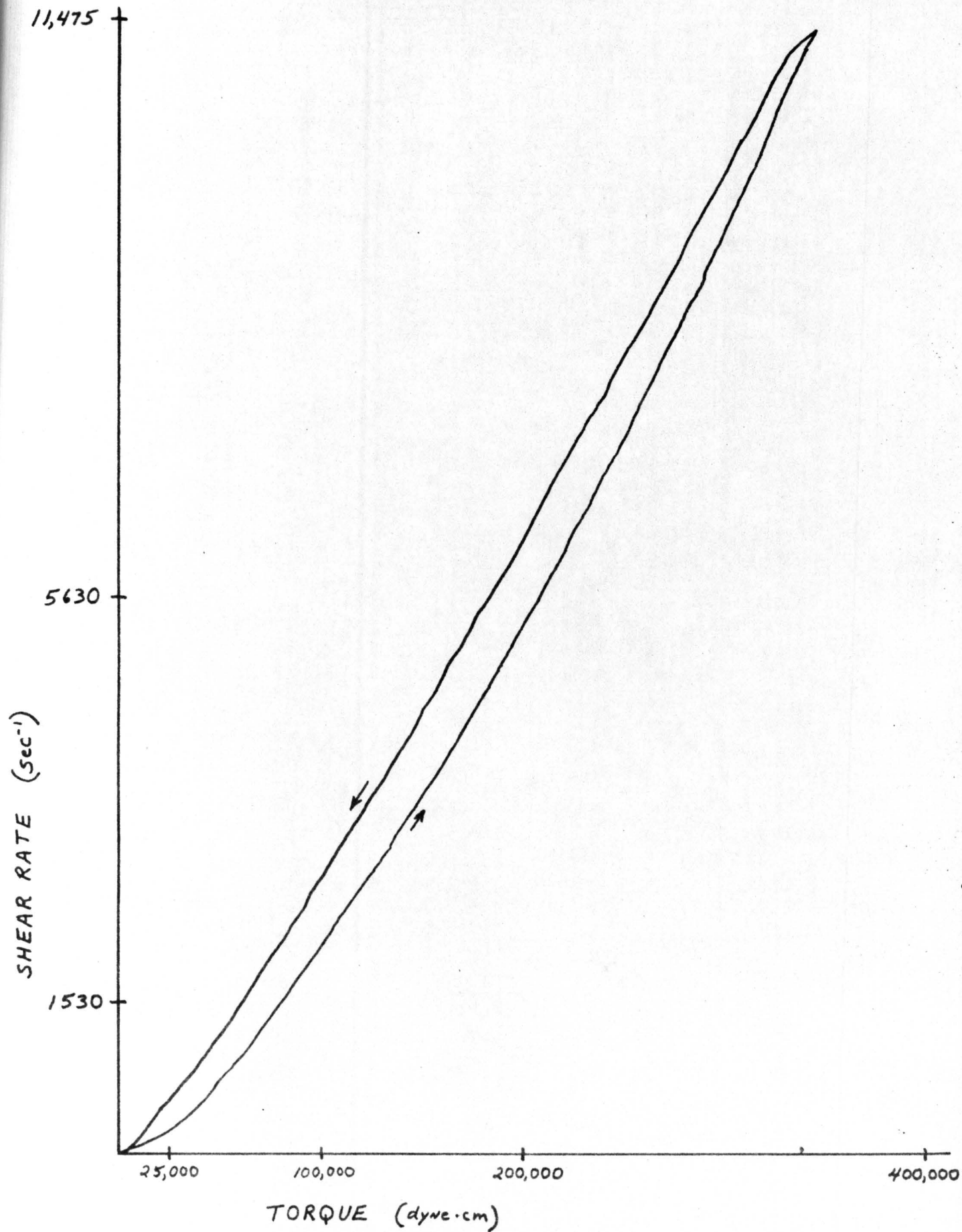
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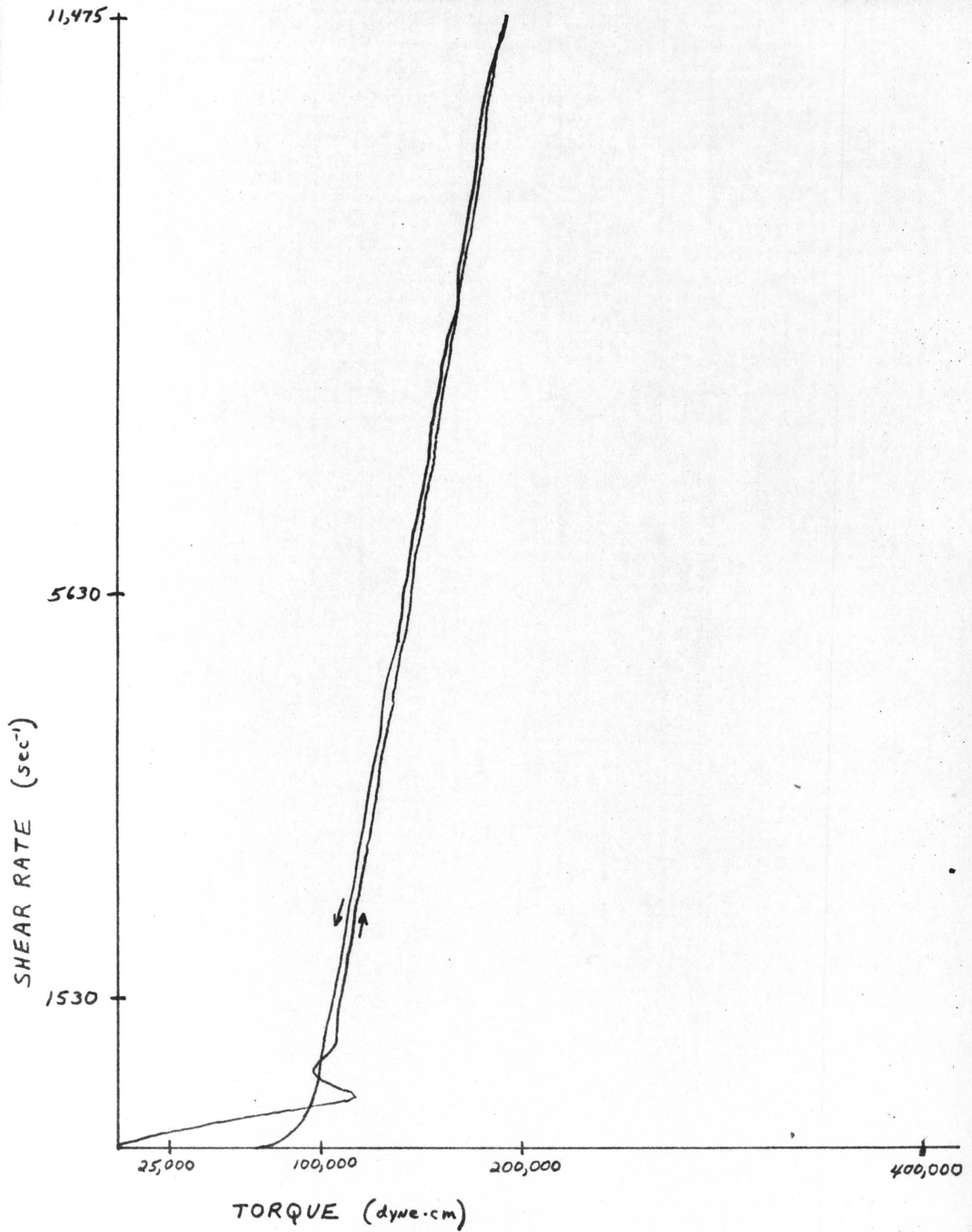
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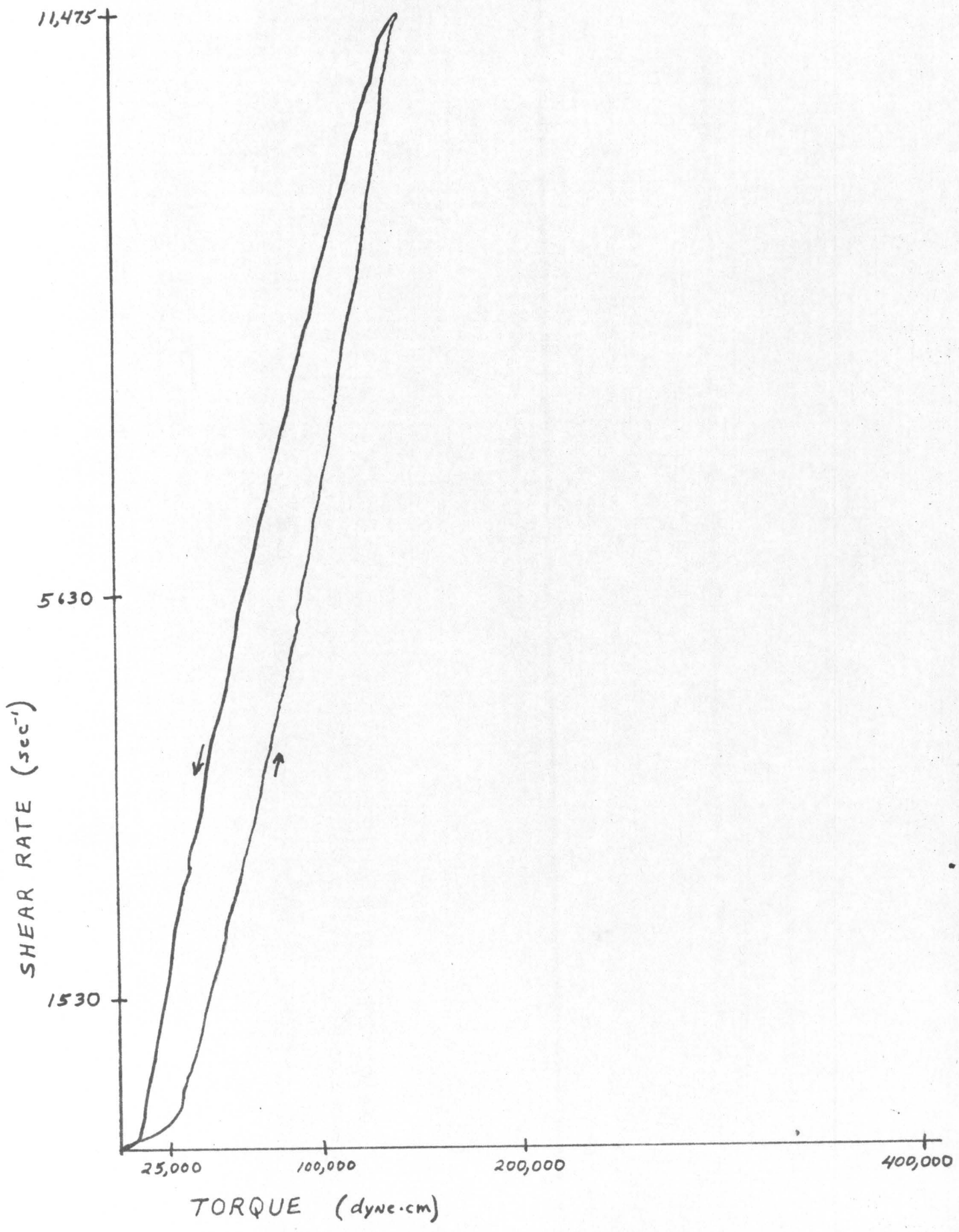
10 PARTS PER HUNDRED ANIONIC STARCH



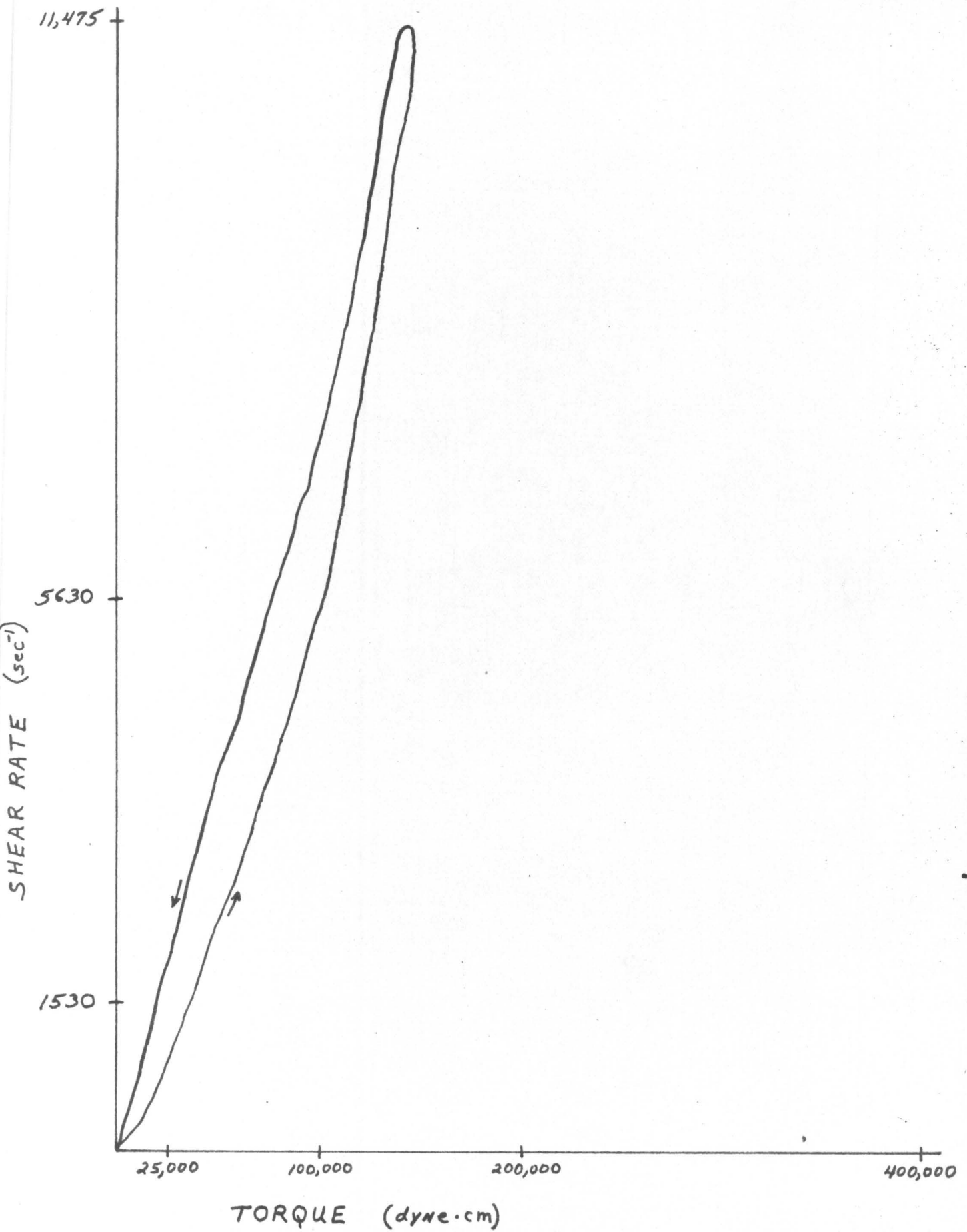
1 PART PER HUNDRED CATIONIC STARCH



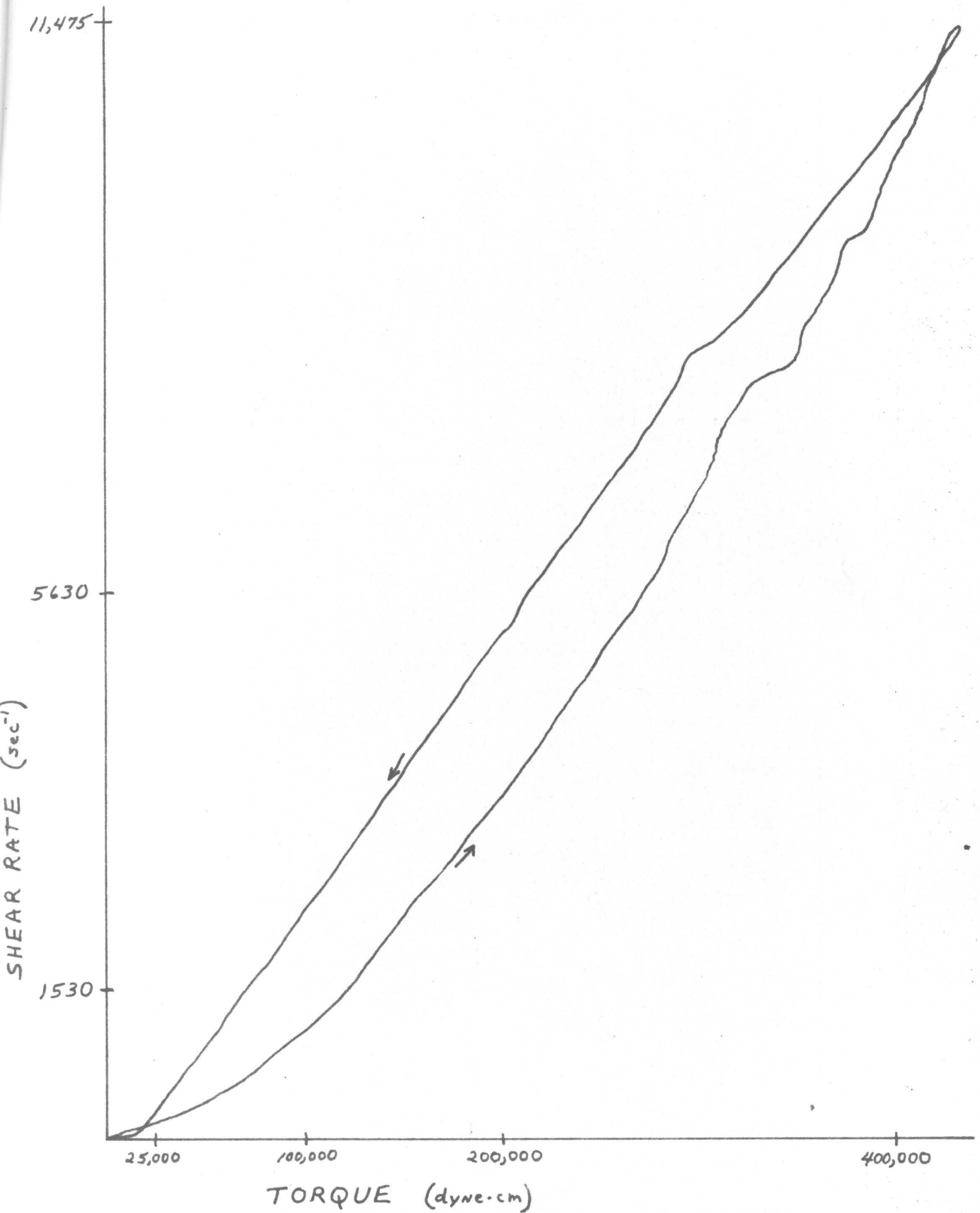
2 PARTS PER HUNDRED CATIONIC STARCH



3 PARTS PER HUNDRED CATIONIC STARCH



5 PARTS PER HUNDRED CATIONIC STARCH



10 PARTS PER HUNDRED CATIONIC STARCH

