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THE INFLUENCE OF SOAP
ON STARCH-PIGMENT COATED PAPER!

Submitted to
the faculty of Western Michigan University
in partial fulfillment of the requirements
for the degree of Bachelor of Science

by
Paul W. Keck
June 12, 1958

Abstract

A literature survey is presented concerning the effects of soap on the rheological properties of coatings, and upon the physical properties of the coated paper. The influence of sodium, ammonium, and calcium stearates is investigated, and it is found that soaps produce desirable effects upon both the flow properties of coating colors and the physical properties of the coated paper.

The experimental results indicate that sodium stearate is superior to either ammonium or calcium stearate, in that all of the properties tested for are either enhanced or remain about the same when sodium soap is added to the coating, whereas the ammonium and calcium soaps produce both desirable and deleterious effects.

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LITERATURE SURVEY

THE INFLUENCE OF SOAP ON STARCH-PIGMENT COATED PAPERS

Introduction

Starch as an adhesive for machine coated papers has become more and more important in recent years. For this reason it has been decided to make a literature survey of the effect on starch-pigment coatings of an important additive, soap. This survey has been divided into three basic parts:

1. The influence of soap on the finished coated paper.
2. Suggested amounts of soap and methods of coating color formulation.
3. The influence of soap on rheological properties of coating colors.

Soap and the Finished Product

Soaps have been included in coating mixtures since the very beginning of modern coating practice. One of the first uses of soap was in the reduction of dusting during supercalendering. (1,2,3,4,5) Available information indicates that dusting is reduced because soap increases the pliability of the coating (1), or acts as a lubricant for the clay particles. (2,3,5) Water soluble soaps such as ammonium stearate (1,2,6), sodium stearate (6), ammonium oleate (6), and sodium oleate (1), as well as insoluble soaps such as calcium stearate (6) are said to impart an anti-dust quality to coatings.

In addition to anti-dust qualities, however, it has been discovered that soap also affects other desirable properties of starch-pigment coatings. Soaps have been found to improve the brightness and finish of coated papers, i.e., brightness, smoothness, and gloss of the finished product are enhanced when soap is included in coating formulations. (1, 3,5,6,7,8) Specifically, calcium stearate has been recommended in this capacity. (1,5,6)

Several other favorable soap effects have also been reported. Casey (1) states that calcium stearate prevents excessive absorption of printing inks, although too much soap can cause poor drying of the ink film. Natwick, Luch, and Halluck (2) claim that ammonium stearate greatly reduces wear of calender doctor blades. McGinn (8) reports that soap may be used as a foam reducer in coating colors.

The above information shows that even though soap is, percentage-wise, a minor component of coating colors, it nevertheless plays an important part from a "benefits obtained" viewpoint.

Amounts and Formulation

Although the correct amount has been agreed to be between two and ten percent on the basis of starch (1,3,9), there has been some disagreement as to when soap should be added to the coating mixture. Ritson and Ward (3), and TAPPI monograph 3 (6) recommend that soap be dissolved in water and then added to the color, either before or after the starch sol is added. Kesler and Rankin (10) have patented a process in which they claim that the soap may be added to the dry starch, the starch suspension, or the gelatinized starch. Evidently each coating mill must determine for itself which method is best, as is the case with so many other papermaking variables.

Several workable coating formulas have been proposed in the literature; three of them follow:

Ingredient	lbs.	Brush coating (6)	Machine coating (11)	Magazine paper coating (12)
Clay		900	1200	1000
Calcium carbonate		300	---	200
Dispersing agent	1*		2.4***	1*
Alkali	2**		pH 9-10***	2**
Starch		230	168	250
Water		920	396	1600
Soap		3	7-12	3

* Sodium tetraphosphate

** Sodium carbonate

*** Not specified

The Influence of Soap on Coating Color Rheology

Although the effect of soap on the qualities of finished coated papers is duly recognized, the effect of soap on the rheological properties of starch-pigment coatings is of equal or perhaps greater importance. Soap has definitely been found to enhance the flow characteristics of coatings, i.e., a minimum of pattern is produced under useful flow conditions if soap is added to the coating.

One investigator, W.C. Gallagher (4), has written, "Originally soap was added to minimize dusting during supercalendering, but more important is the effect which soap has on the plastic viscosity and leveling properties of high-solids coating mixtures." Gallagher made a study of the effect of sodium base and ammonium stearate soaps on the thixotropic and leveling qualities of starch coating mixtures, and determined that the addition of one to five percent soap greatly

improved these properties, in some cases raising the coating from an inoperable to a pattern-free mixture. Gallagher found that sodium base soaps produced greater changes in flow characteristics than did ammonium stearate. Casey (1), and Ritson and Ward (3) also note the desirable rheological properties imparted by soap, and a table indicating the effect of soap on the leveling index, yield value, viscosity, and performance has been prepared by Majani and Crane (13)

In an attempt to explain why soap alters the flow properties of starch coatings, there is a direct conflict between investigators. Ritson and Ward (3), and Culp (14), state that soap forms a thixotropic complex with amylose, the greater amylose content producing a greater thickening upon the addition of soap. A.M. Heald (15), however, has reported that the amylopectin content of starch controls the viscosity increase produced by soap.

Regardless of whether or not Heald is correct about soap's reaction with amylopectin, his research has produced some very interesting results. He found that soaps first lower the viscosity of starch solutions, then cause a rapid rise in viscosity upon further addition. Still further addition again causes the viscosity to fall. The point at which viscosity is a maximum was determined to be where twelve percent soap is added to the starch. Heald theorizes that the starch-soap viscosity effect is the result of 1) An electroviscous effect due to attraction between starch and soap molecules, and 2) Structural viscosity as a result of gel formation.

Another investigator, H.H. Houtz (16), states the existence of the starch-soap complex without attempting to explain it, and Kesler and Black (17) report the complex with the statement that it can be completely precipitated from solution by insolubilizing the soap.

The starch-soap complex is beyond the scope of this paper, but it is evident that further work is required along these lines in order to clarify the existing situation.

Conclusion

Inasmuch as soaps have been incorporated in coatings for many years, one would expect accurate and complete data concerning its effects on coatings. Just the reverse has been found to be true, however; insufficient and conflicting data exists in the literature. Thus, it has been decided to conduct experiments in which soaps are included in color formulations, in an effort to add to the existing information.

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EXPERIMENTAL PART

Experimental Design

In order to determine the effects of soap on coatings, it has been decided to evaluate three different soaps, sodium, ammonium, and calcium stearates, and to compare the results with each other and with a control coating using no soap. In addition to variation of type of soap, the influence of soap concentration will be studied. Coatings with soap concentrations of 0.75, 1.5, and 3.0 percent soap based on adhesive will be evaluated. The other ingredients to be used in all coatings are: predispersed clay, 370.4 parts; oxidized starch, 100 parts; and Dowicide G, two parts. All other variables are to be held constant.

Coating colors are to be formulated from the starch, clay, soaps, Dowicide G, and enough water to produce a coating color of 50% solids. Rheograms are to be made on the Hercules High Shear Viscometer at this consistency, and leveling index and high shear viscosity will be calculated from the rheograms.

The coatings are then to be adjusted to a standard Brookfield viscosity of 300 ± 50 cps and pH 8.0 ± 0.5 . Sheets for physical testing will be prepared by coating a standard coating base to 14 ± 1.5 lb (25 X 38-500), using wire wound doctor rods for the draw downs. Coated sheets are to be dried while suspended at 200 F for 4 minutes, conditioned at 73 F and 50% R.H., then calendered at 20 lb gauge pressure and a roll surface temperature of 100 F.

Calendered sheets are to be evaluated for brightness, gloss, opacity, smoothness, Fade-O-Meter stability, wet rub resistance, Dennison wax pick, I.G.T. pick, and the K & N ink receptivity test.

Experimental Procedure

The experimental procedure actually followed was essentially that given under Experimental Design, however a more detailed account will be given here.

Materials and Equipment Used

Materials Used. The basic materials used for all coating colors were the same, in order to determine the effect of soap alone. These ingredients are listed below:

1. Pigment-Predispersed clay with 80% of particles finer than two microns.
2. Adhesive- Oxidized starch.
3. Preservative- Dowicide G.
4. Coating Base- Commercial coating base weighing 43.5 lb per ream. (25 x 38-500)

In addition to the basic ingredients, commercial grades of sodium, ammonium, and calcium stearate were used.

Equipment Used. The equipment used in the experimental work is listed below:

1. The high shear viscosity and leveling index were calculated from rheograms made on the Hercules High Shear Viscometer; a rotational viscometer capable of rates of shear up to 4540 reciprocal seconds, and equipped with a continuous recording mechanism which plots torque vs rpm.
2. Clay slips were prepared in a sigma blade "dough" mixer.
3. Coatings were centrifuged in a macro centrifuge to remove entrapped air.

4. A pH meter was used for determining pH.
5. Starch was cooked in a double boiler.
6. Coating viscosity was adjusted with a Brookfield viscometer.
7. Beakers and spatulas were used for mixing ingredients.
8. Standard testing equipment was used for physical testing of the coated paper.

Coating Color Formulation

All coating colors were prepared by the slurry technique, that is, the clay slip and binder were prepared separately and mixed just before application to the base sheet. Actual coating formulations tested are given in Table I.

Preparation of Individual Components. The individual components, clay slip, starch cook, preservative, and soaps, were prepared as listed below:

1. Clay Slip.- The clay slips were prepared by charging dry clay to the sigma blade mixer, then adding distilled water slowly until the clay "wet out". After the clay wet out, the solids content of the clay slip was adjusted to 75%, and mixing was continued for thirty minutes.
2. Starch Cook.- Starch used for coatings was cooked in a double boiler in order to prevent caramelizing or burning of the starch. Dry starch and distilled water sufficient to give a dispersion of 30% solids were mixed together until smooth. The dispersion was then heated to 195 F, and held at that temperature for fifteen minutes, with constant agitation

to prevent local overheating. After the starch had been cooked, the solution was covered to prevent skin formation, and allowed to cool to room temperature. Distilled water was added to compensate for evaporation loss.

3. Preservative.- In order to facilitate even mixing of the preservative, a 20% water solution of Dowicide G was prepared.

4. Soaps.- The sodium stearate (Ivory Flakes) was made into a 10% water dispersion. The ammonium and calcium soaps were obtained from the manufacturer as 25% and 50% dispersions, respectively, and were used as such.

Mixing of Components. Due to the high viscosity of the clay slip, all mixing was done by hand; however, a small sigma blade mixer would serve very well. The steps followed in mixing the separate ingredients are listed below. All amounts given are on a dry basis.

1. Clay equivalent to 370.4 grams was weighed into a metal beaker.
2. Starch in excess of required 100 grams was weighed into a separate beaker. To this was added 2% Dowicide G and required percentage of soap in dispersion form.
3. The starch-soap-preservative mixture was added to the clay slip in small portions, with sufficient mixing between additions to provide a smooth dispersion.

4. The total solids content of each coating was adjusted to 50% with distilled water.
5. The pH of the finished coating color was adjusted to $\text{pH } 8.0 \pm 0.5$.

Testing of Coating Colors

Rheograms for all coatings were made on the Hercules High Shear Viscometer at 50% solids and $\text{pH } 8.0 \pm 0.5$. In order to insure smooth rheograms, the coatings were centrifuged for five minutes at 2400 rpm. Centrifuging removes entrapped air and also settles any agglomerates which may then be stirred back into the coating.

Calculations of rheological properties were made from the original rheograms which were then traced as smooth curves for inclusion in this paper.

The rheograms were interpreted according to equations given by Smith, Trelfa, and Ware^{*}, who have done pioneering work on leveling index calculations. A sample calculation follows:

In order to utilize the equations of Smith, Trelfa, and Ware, instrument constants are required. A constant for all rotational viscometers is 9.55, but S and C depend upon the bob employed. For the Hercules High Shear with bob A, $S = 2.066 \times 10^{-4}$ and $C = 8.172 \times 10^{-3}$.

The numerical values used in the example are from the control coating.

* Smith, J.W., Trelfa, R.T., and Ware, H.O., Tappi 33, no 5: 217, (May 1950)

Torque @ 1000 rpm $T_2 = 480,000$ dyne-cm

Torque @ 500 rpm $T_1 = 390,000$ dyne-cm

Extrapolated torque intercept of downcurve $T_0 = 20,000$ dyne-cm

Apparant viscosity @ 500 rpm $N = 9.55 T_1 \text{ S/rpm} = 1.54$ poises

Plastic viscosity @ 1000 rpm $U_2 = 9.55(T_2 - T_0) \text{ S/rpm} = 0.91$ poises

Plastic viscosity @ 500 rpm $U_1 = 9.55(T_1 - T_0) \text{ S/rpm} = 1.46$ poises

Yield Stress $F = T_2 C = 163$ gm/cm-sec

Coefficient of Thixotropy $M = U_1 - U_2 / \ln(\text{rpm}_2 / \text{rpm}_1) = 0.793$ dyne/cm-sec

Leveling Index $I = M / U_2 = 0.87$

The leveling index is a dimensionless ratio of the coefficient of shear thixotropy to plastic viscosity, and is an important measure of the suitability of coating mixtures for high shear roll coating, since it indicates the ease with which the coatings will level after leaving the nip.

Trelfa has set forth empirical values concerning leveling index. If I is less than 0.25, inoperability is to be expected; if I equals 0.25 - 0.35, borderline performance is to be expected; and if I is greater than 0.35, operability is to be expected.

Application to Base Sheets

After rheograms were made, the coating colors were adjusted to a Brookfield viscosity of 300 ± 50 cp at 80 F. The pH was then readjusted to 8.0 ± 0.5 , and the coatings were centrifuged to remove air bubbles.

A commercial coating base weighing 43.5 lb (25 x 38-500) was prepared for coating application by cutting sheets 9" x 12", with the long dimension in the machine direction. A 14 lb ± 1.0 coating was applied to the wire side of this base, using R.D. Specialties wire wound rods for the drawdowns, and a plate glass base to insure smooth, even application. Drawdowns were made in the machine direction. Coated sheets were suspended in the oven at 200 F, and left to dry for 4 minutes. After drying, the sheets were trimmed to 7" x 9", and placed in the constant temperature and humidity room to condition prior to supercalendering.

Calendering

Conditioned sheets were calendered to produce maximum increase in gloss with minimum loss in brightness. This condition was satisfied by calendering four passes on the laboratory calender at a roll surface temperature of 100 F and a pressure of 20 psig. After calendering, the sheets were again conditioned prior to testing.

Evaluation of Coated Paper

Conditioned sheets were evaluated for brightness, gloss, opacity, smoothness, fading resistance, wet rub resistance, Dennison wax pick, I.G.T. pick, and the K & N receptivity test. Instruments used for the various tests are listed below:

1. Brightness- I.P.C. (G.E.) Brightness meter.
2. Gloss- Photovolt.
3. Opacity- Bausch and Lomb opacity meter.

4. Smoothness- Bekk tester.
5. Fading resistance- Atlas Fade-O-Meter.
6. I.G.T. pick- I.G.T. printability tester.

All optical tests, the smoothness test, and Dennison wax pick test, were conducted according to Tappi Standards. K & N ink absorption was determined by applying an excess of K & N ink, leaving it on the sheet for two minutes, and then wiping off the ink with a soft cloth. Reflectance at 587 mμ was taken as an indication of ink absorption. Light at 587 mμ is complementary to the ink color; thus, the lower reflectances denote more ink absorption. Fading resistance was determined by exposing the sheets to ultra violet light at 100 F in the Fade-O-Meter for 4 and 8 hours, and then measuring papermaker's brightness.

The I.G.T. pick yest was conducted according to manufacturer's specification. A specified amount of no. 4 tack ink was applied to the impression disc, and speed at which coating pick began was read by a comparison with a chart that shows the speed of the sector at varying distances along its length.

Wet rub resistance was determined by the "finger" method; that is, two drops of water were placed on the coated sheet, allowed to remain there for one minute, then rubbed with the finger onto a sheet of black construction paper. Six rubs were made over the same spot; however, the black paper was moved each time, thus producing six streaks of coating on the paper. Visual observation was used to determine the relative wet rub resistance of the coatings.

Presentation of Data

The coating formulations tested, rheological properties, and physical properties of the coatings are presented in Table I.

Physical properties of the coated paper are recorded in Table II, and presented graphically in Figures 11 and 12. Rheograms of the coatings are included in Figures 1-10. The numbers assigned to coatings in all tables and figures correspond to the numbers given in Table I.

Discussion of Results

As seen from Table I, the addition of soap to coatings produced pronounced variations in apparent viscosity, yield stress, and leveling index in all instances. Sodium stearate increased the apparent viscosity and yield stress of the coatings, whereas ammonium stearate reduced these values. The change produced by calcium stearate fell between the changes produced by sodium and ammonium stearates. The leveling indices of all coatings except that with 0.75% ammonium stearate were in the operable range. In all cases, apparent viscosity was increased with increasing level of soap concentration, but yield stress reached a minimum value at 1.5% soap concentration.

Figures 1-10 illustrate the changes in flow properties graphically. On these graphs, torque is plotted against rate of shear, and the area of the loop of the curves indicates the amount of thixotropy present in each coating. From the rheograms it can be seen that sodium stearate greatly increased thixotropy, and ammonium stearate reduced thixotropy, in one case to the level of Newtonian flow. Calcium stearate did not produce any significant change in thixotropy.

It is apparent from Figures 11 and 12 and Table II that physical properties of coated paper are markedly altered by soap. In all cases gloss, brightness, and opacity were either increased or remained about the same.

Soaps did not produce any significant change in fading resistance. Smoothness was increased by both sodium and calcium stearates, but was reduced by ammonium stearate. Wax pick was altered only by ammonium stearate, in which case picking resistance was drastically reduced. The I.G.T. pick test results show that sodium stearate increases picking resistance, while ammonium and calcium stearates reduce the speed at which picking occurs. Ink receptivity was increased by both sodium and ammonium soap, and slightly reduced by calcium stearate. The wet rub tests did not show any appreciable change in wet rub resistance; thus, the results are omitted from this paper.

Summary and Conclusions

This investigation was conducted with the intent of determining the effects of sodium, ammonium, and calcium stearates when incorporated in starch-pigment coatings. From the course and methods of this work, the following conclusions have been drawn:

1. The rheological properties of coatings may be altered by the addition of soaps, perhaps even to the extent of fitting a coating to any given machine conditions.
2. Coatings incorporating any of the soaps will show higher brightness as compared with coatings using no soap. Ammonium stearate produces the greatest brightness increase.
3. Gloss of coated paper may be increased as much as 7 points with sodium or ammonium stearate.
4. There is a gain in opacity of coated paper with the

addition of any of the soaps, indicating that lower coat weights may be used to obtain the same hiding power.

5. Soap does not significantly affect the fading resistance of coated paper.
6. Smoothness of coated paper is greatly increased by sodium stearate, somewhat increased by calcium stearate, and lowered by ammonium stearate. Thus, print quality may be improved by sodium or calcium stearate.
7. Picking resistance is increased by sodium stearate, remains the same with calcium stearate, and is reduced with ammonium stearate. This indicates that printability of coated paper may be enhanced by sodium stearate, and decreased by ammonium stearate.
8. Ink receptivity is increased by sodium and ammonium stearates, and reduced by calcium stearate. Thus, ink absorption or gloss ink holdout of printing papers may be altered with soap.
9. The level of soap concentration does not significantly vary the physical properties of coated paper, but the concentration of soap is important to the rheological properties of coating colors.
10. From an overall viewpoint, sodium stearate is superior to the other soaps, in that all properties tested for were enhanced or remained about the same, whereas both desirable and deleterious effects were produced by calcium and ammonium stearates.

FORMULATION AND CHARACTERISTICS

OF COATING COLORS

Coating Number	1	2	3	4	5	6
<u>Formulation</u>						
Pigment						
Clay - Parts	370.4	370.4	370.4	370.4	370.4	370.4
Adhesive						
Starch - Parts	100	100	100	100	100	100
Dowicide G - Parts	2.0	2.0	2.0	2.0	2.0	2.0
Sodium Stearate - Parts	0	0.75	1.5	3.0	0	0
Ammonium Stearate - Parts	0	0	0	0	0.75	1.5
Calcium Stearate - Parts	0	0	0	0	0	0
<u>Characteristics</u>						
Total Solids - Percent	42.7	39.5	38.1	37.8	45.2	43.8
Brookfield Viscosity - cps	350	320	322	325	318	320
pH of Coating Color	8.4	8.5	8.4	8.3	8.4	8.5
Apparant Viscosity @ 500 rpm - poises	1.54	2.03	2.27	2.62	0.59	0.79
Yield Stress - grams/sq cm	163	776	695	1062	0	0
Leveling Index - Dimensionless	0.87	0.67	0.64	0.81	0.22	0.37

Figure 1

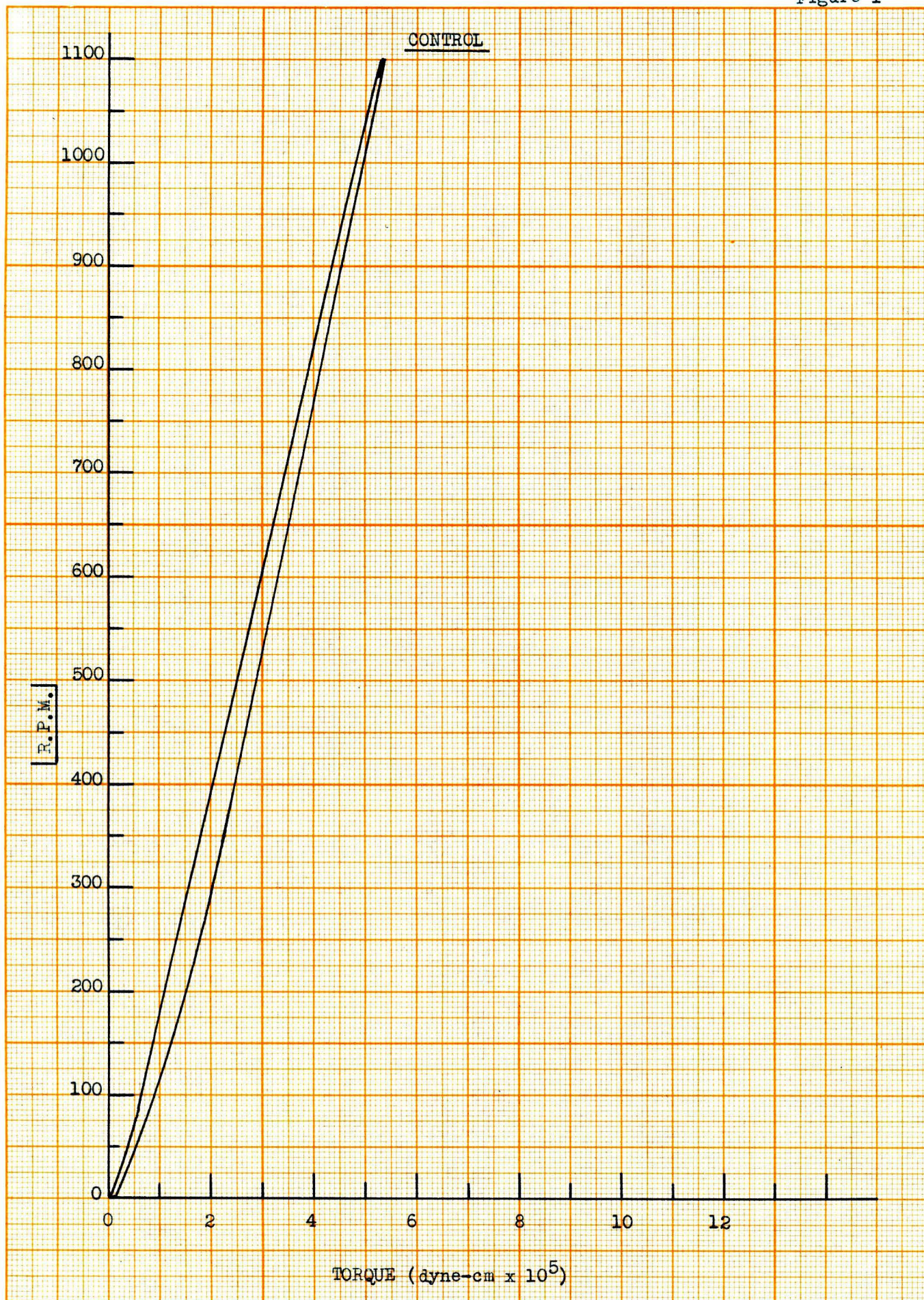


Figure 2

0.75% SODIUM STEARATE

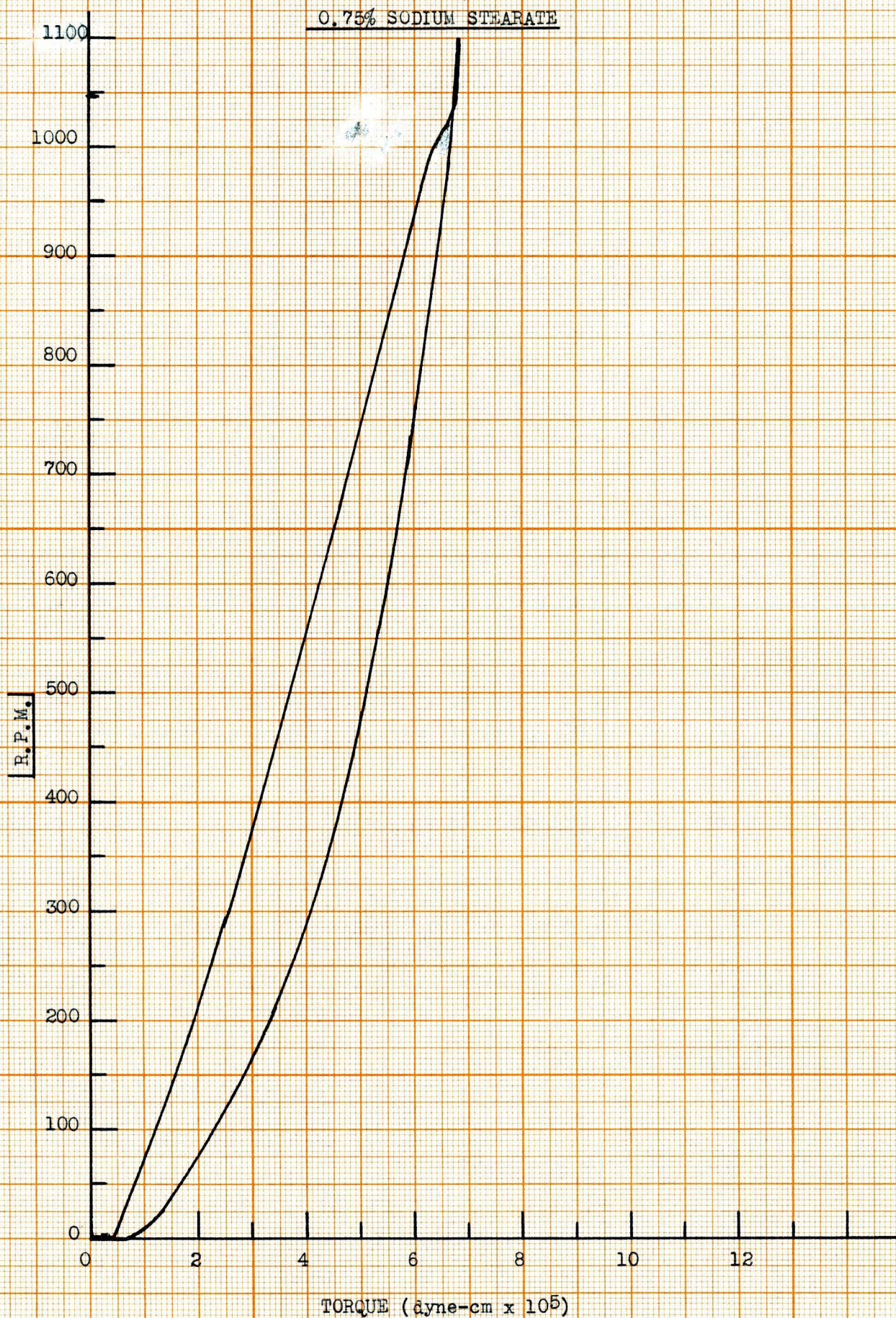


Figure 3

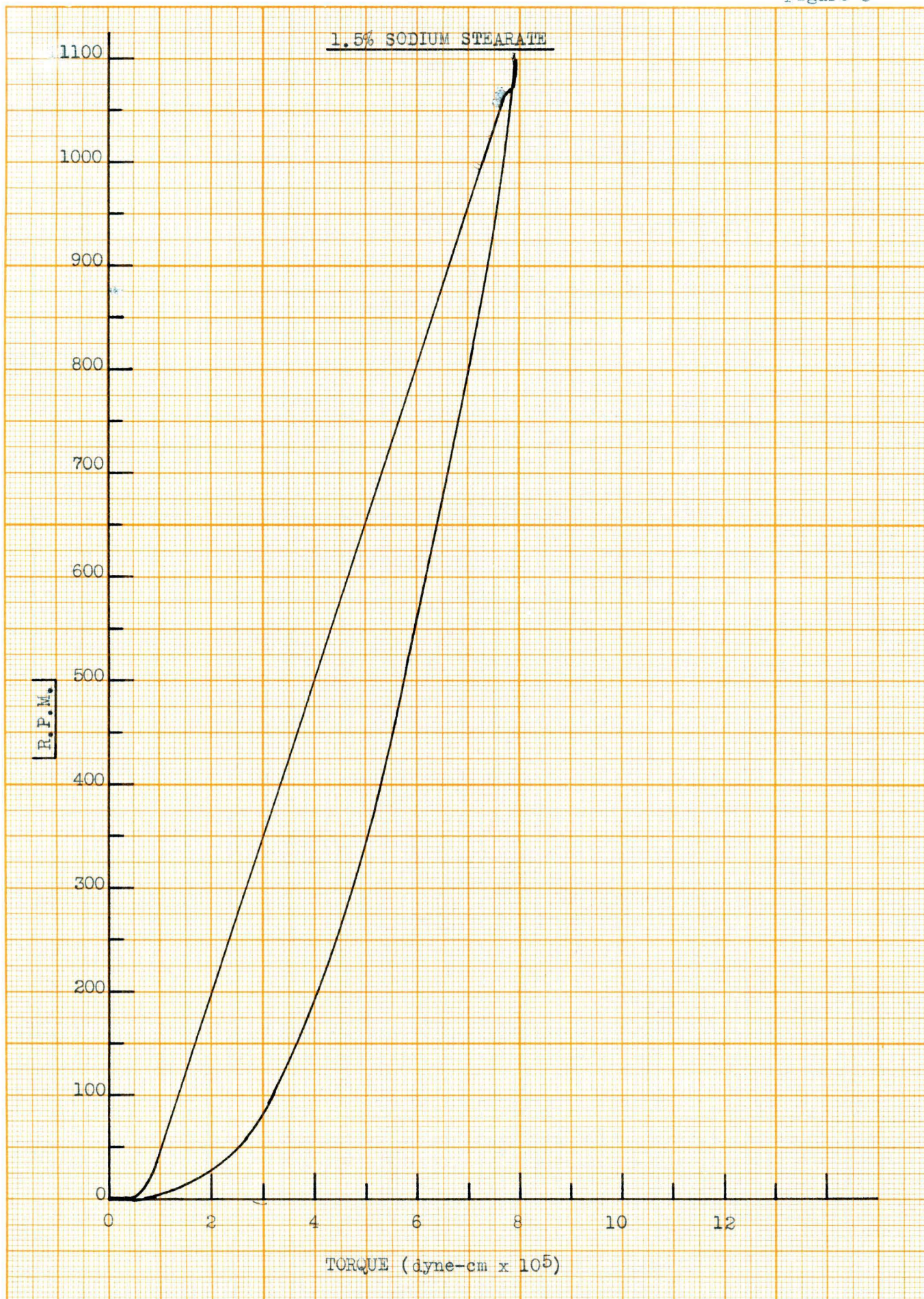


Figure 4

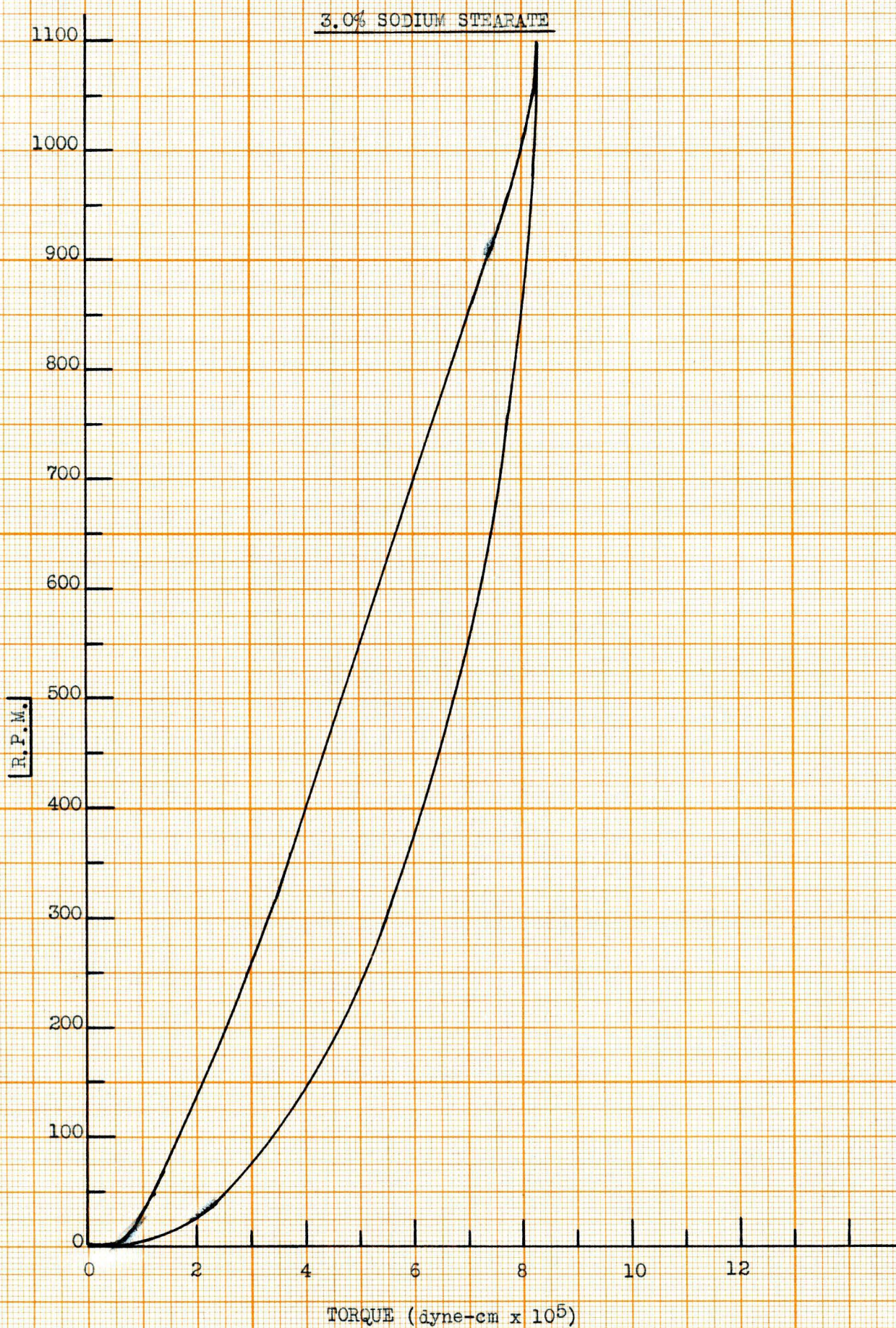


Figure 5

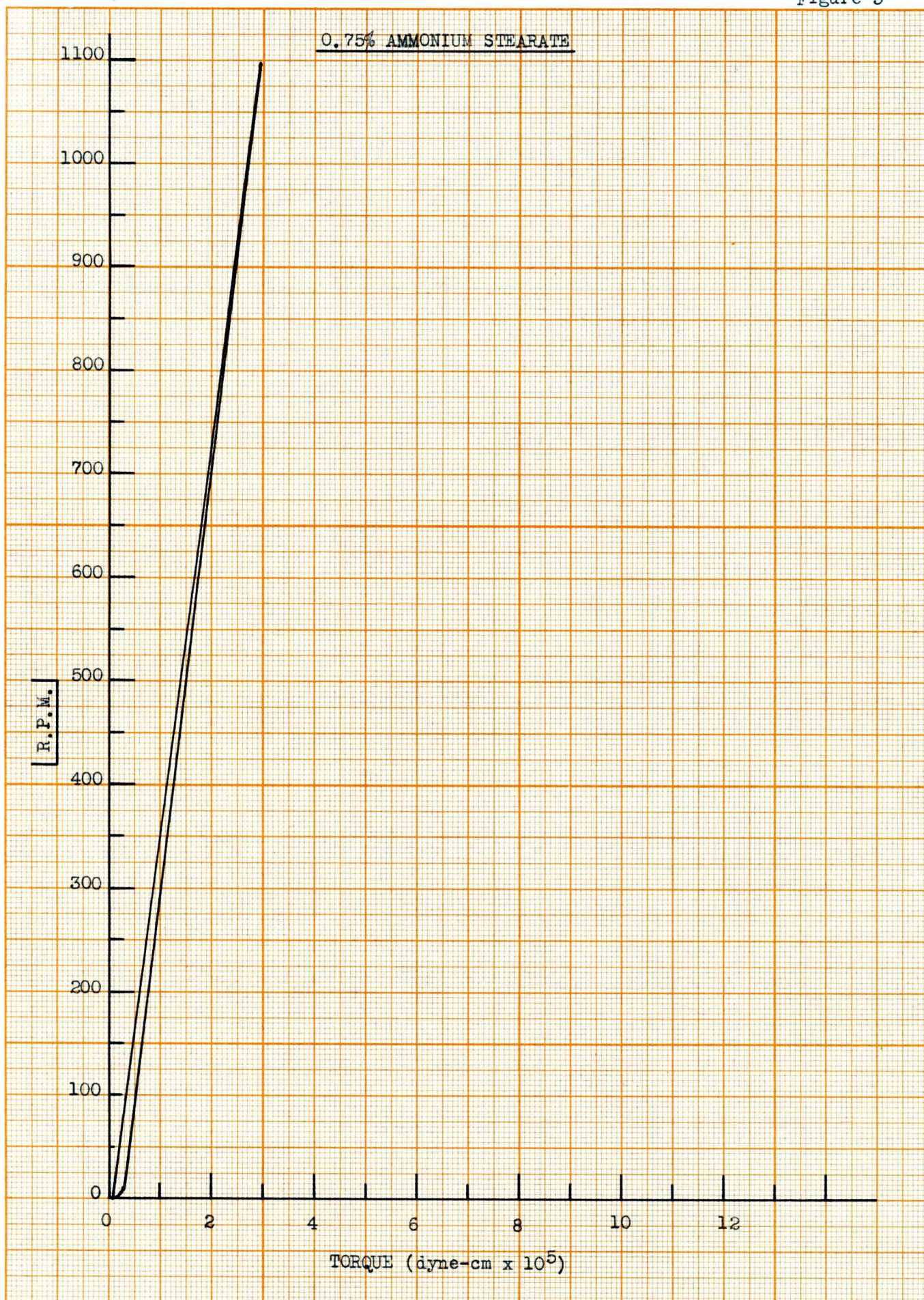


Figure 6

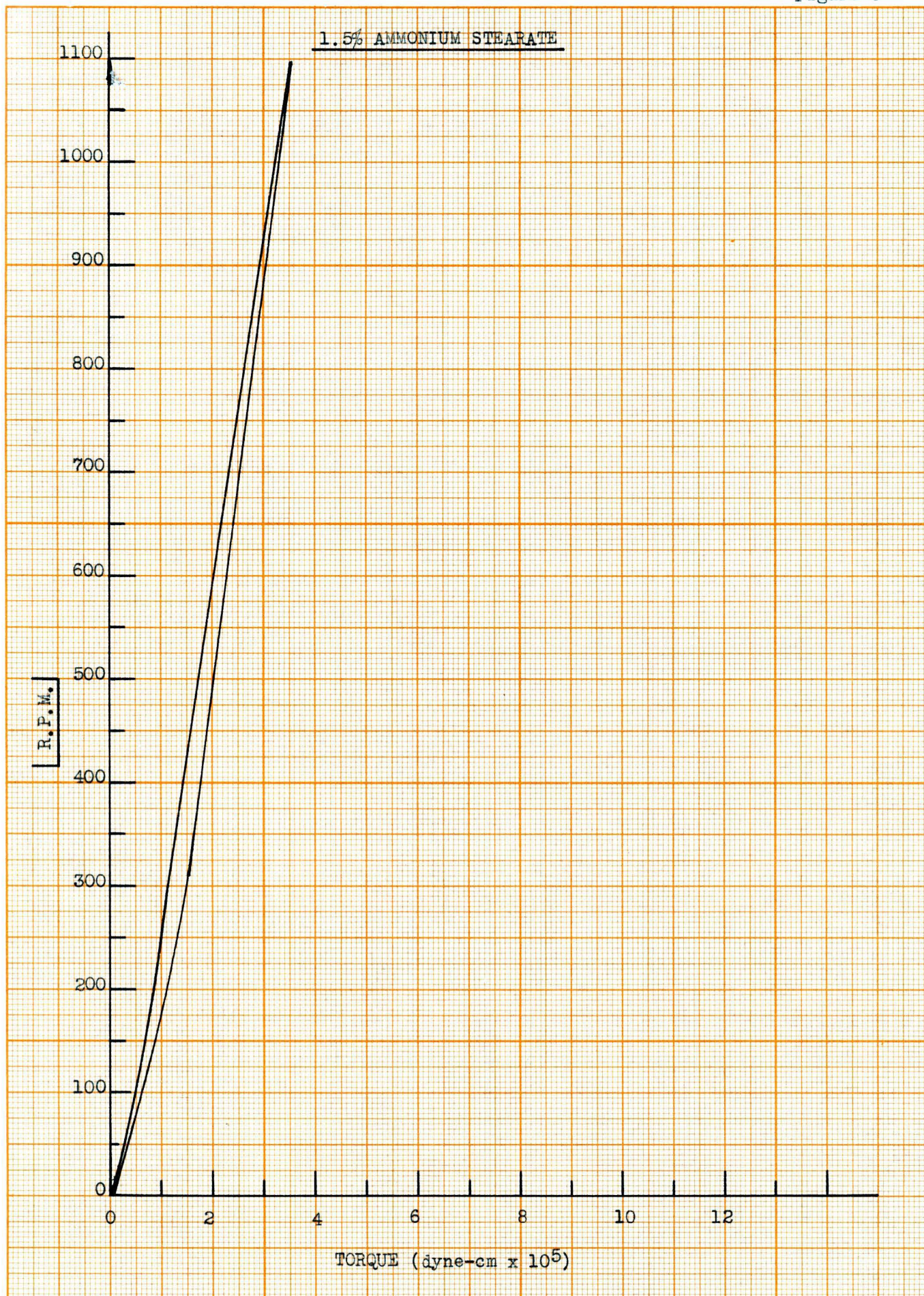


Figure 7

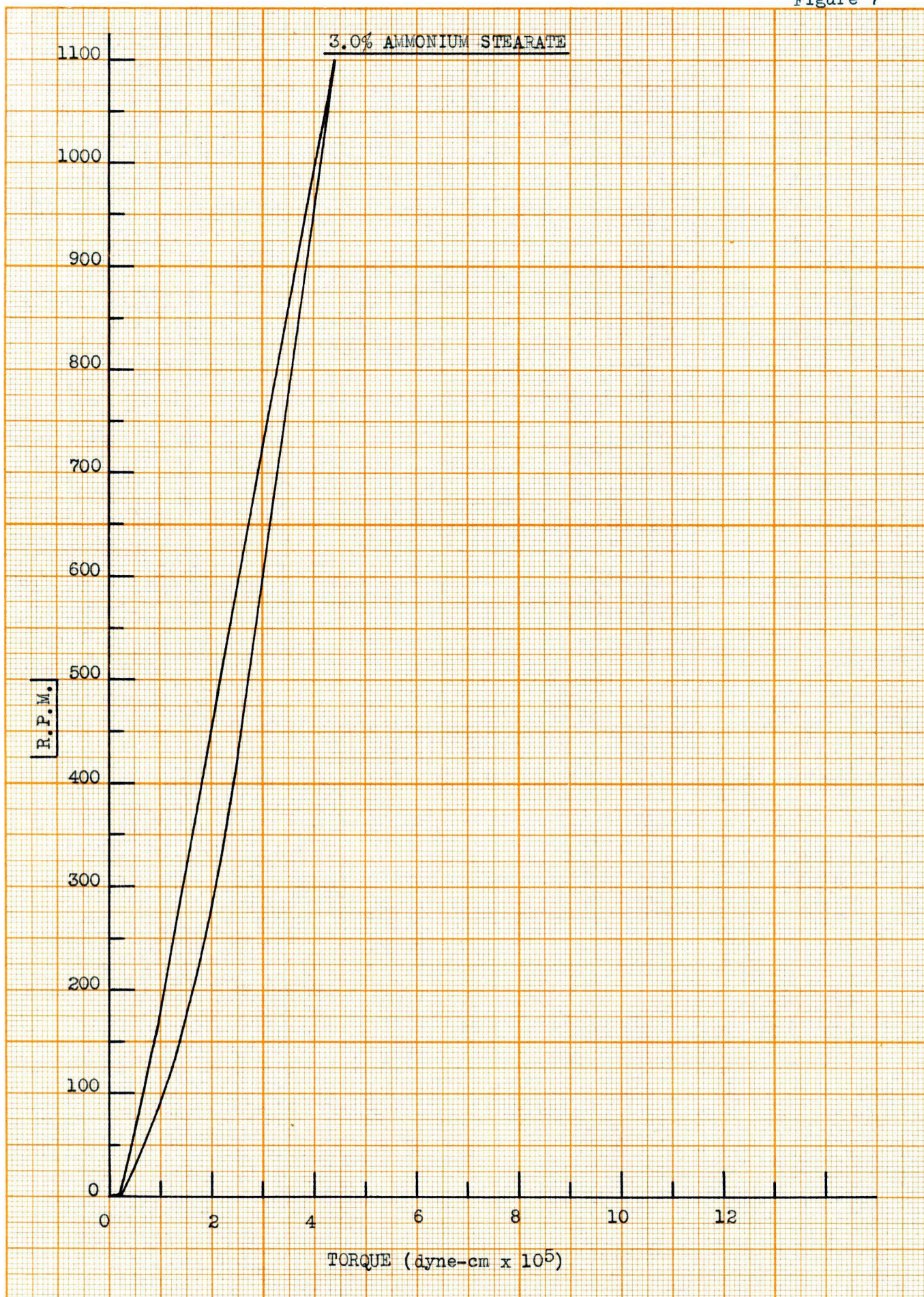


Figure 8

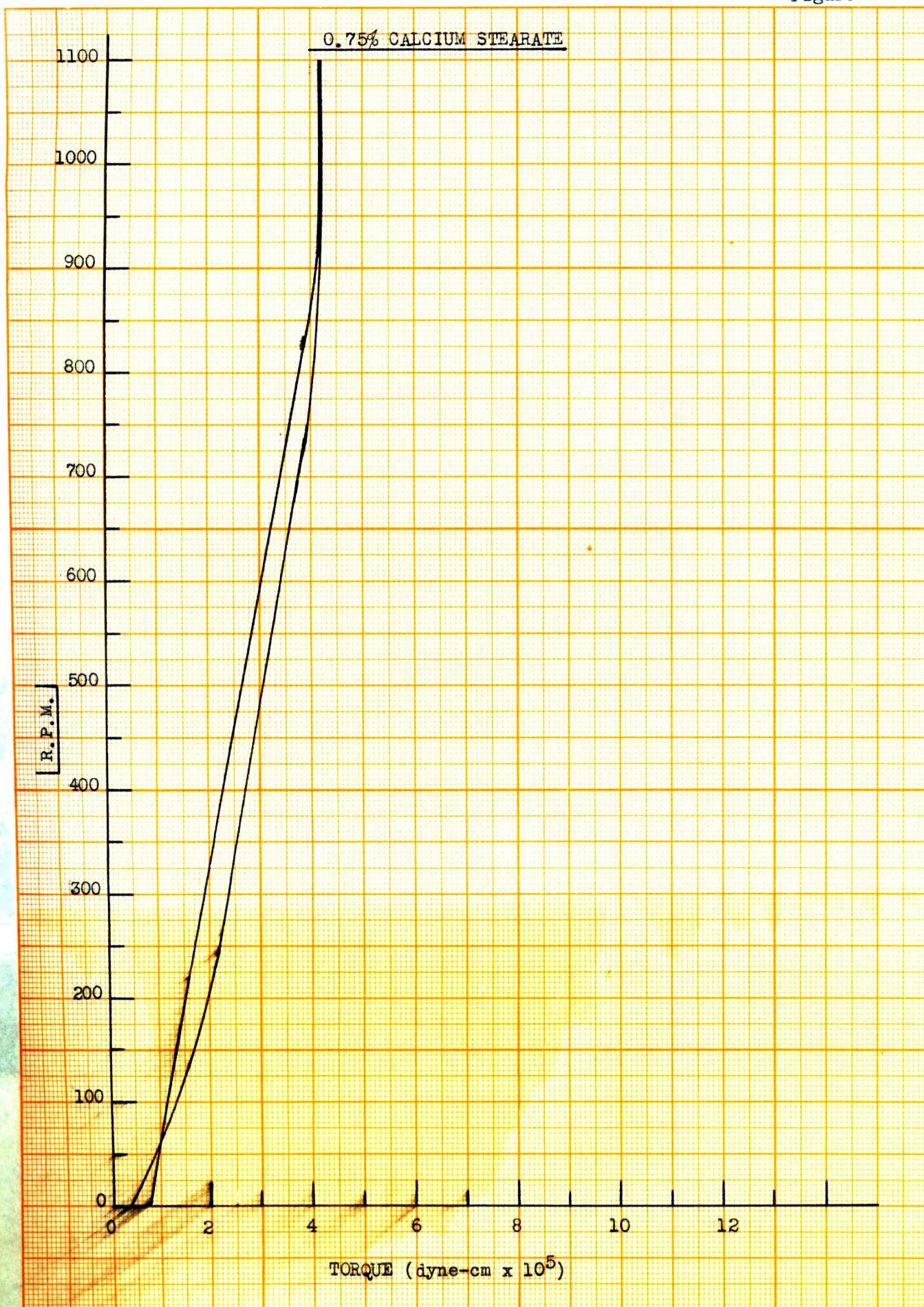


Figure 9

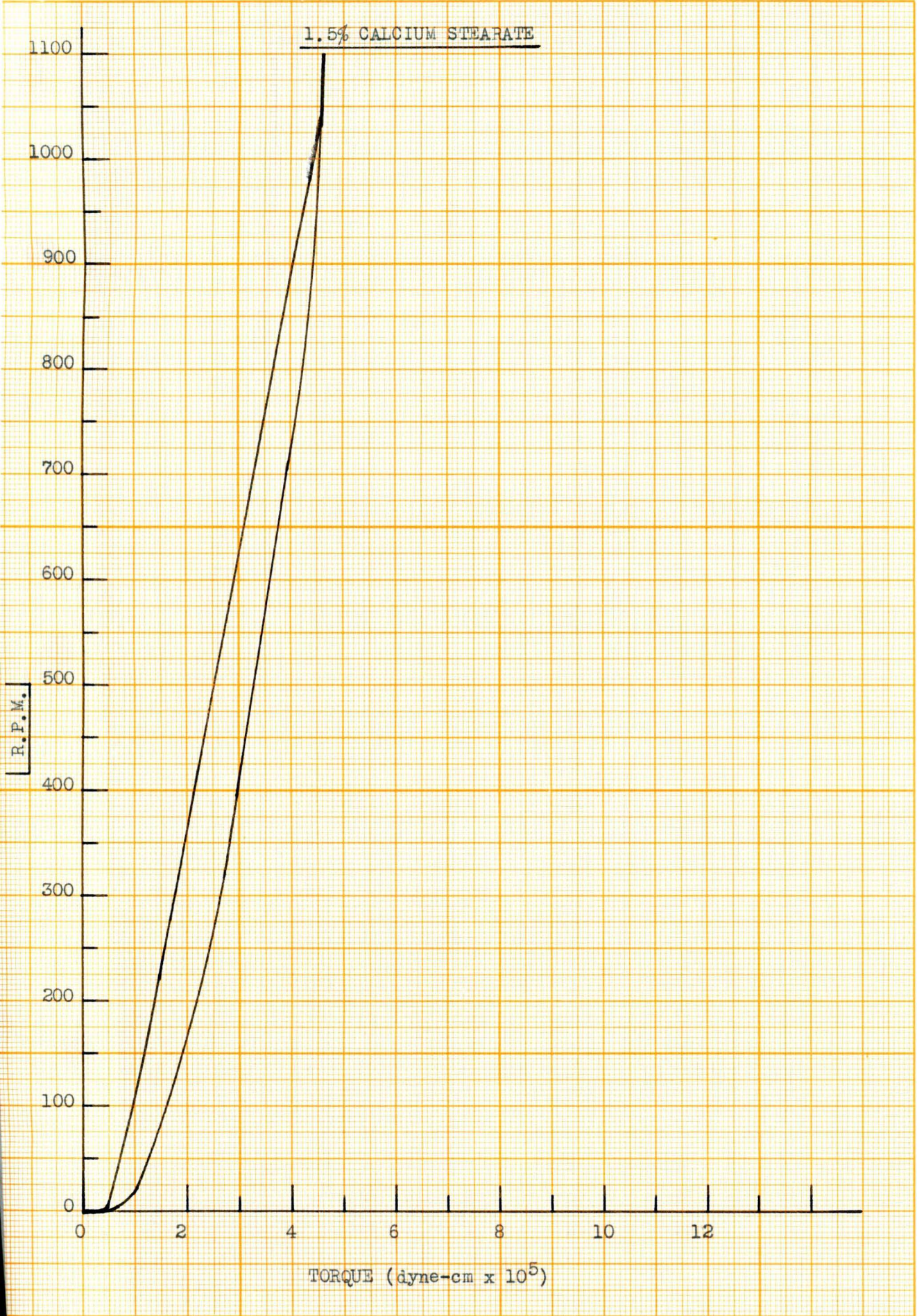


Figure 10

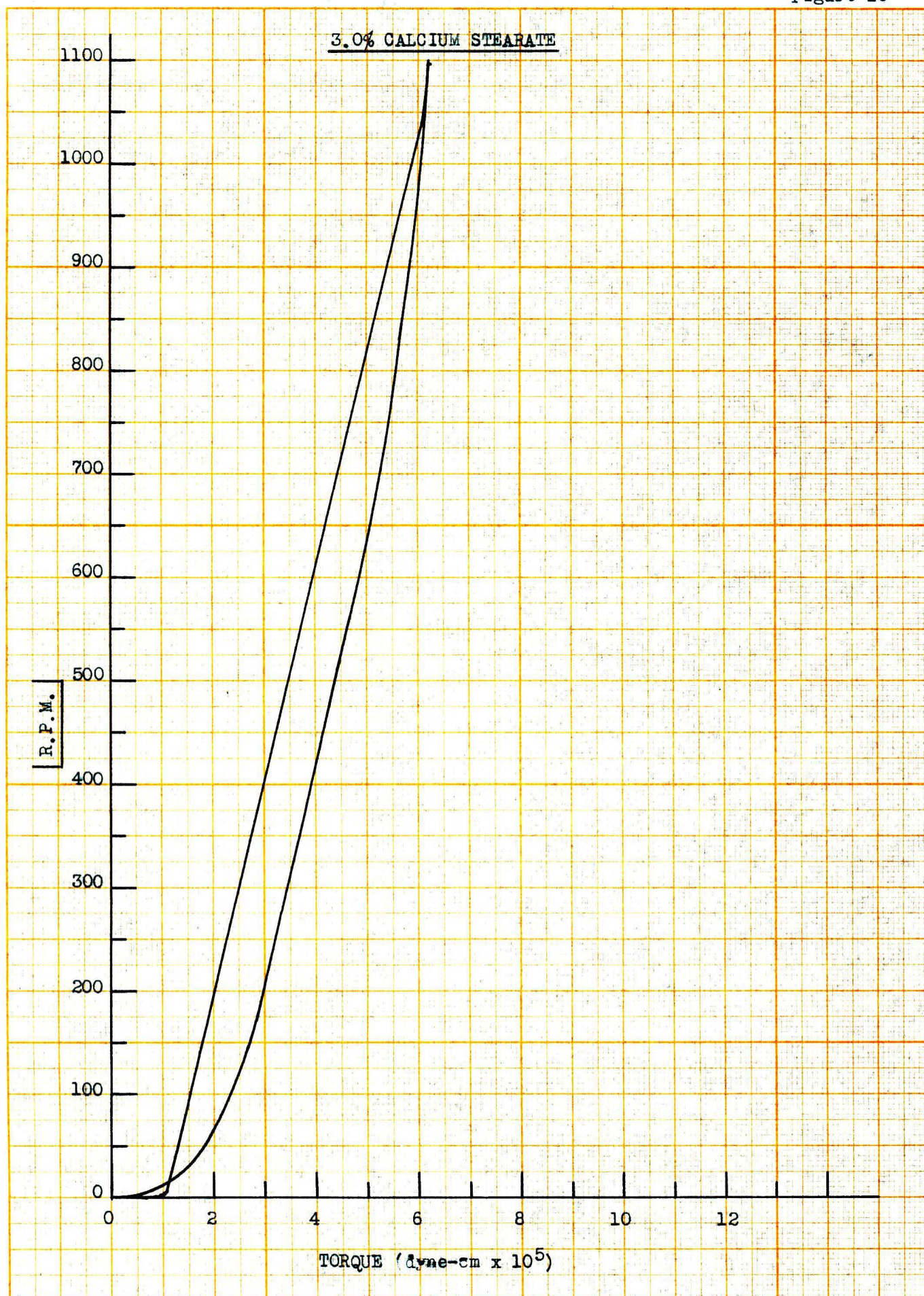


Table I

7	8	9	10
370.4	370.4	370.4	370.4
100	100	100	100
2.0	2.0	2.0	2.0
0	0	0	0
3.0	0	0	0
0	0.75	1.5	3.0
42.7	38.5	37.7	40.6
330	328	330	340
8.3	8.5	8.2	8.4
1.05	1.1	1.22	1.68
245	409	286	817
0.39	0.52	0.52	0.47

Table II

7	8	9	10
57.9	58.0	57.3	57.7
43.5	43.5	43.5	43.5
14.4	14.5	13.8	14.2
35.1	27.7	28.2	27.6
90.4	87.4	87.1	88.3
75.6	75.0	75.4	75.0
74.4	72.8	73.2	73.4
74.7	72.6	73.0	72.7
292	378	400	373
6.6	9.0	9.0	9.8
---	10.0	10.0	---
8.0	11.0	11.0	11.0
51	110	101	113
49.0	60.4	59.8	60.8

PHYSICAL PROPERTIES OF COATED PAPER

Coating Number	1	2	3	4	5	6
Basis Weight - 25 x 38 - 500						
Coated Paper - a.d.	58.2	57.9	57.5	58.0	58.0	57.4
Coating Base - a.d.	43.5	43.5	43.5	43.5	43.5	43.5
Coating Applied - lb/ream - a.d.	14.7	14.4	14.0	14.5	14.5	13.9
Gloss - Photovolt - Percent	28.2	33.7	33.8	34.8	35.1	34.0
Opacity - Bausch & Lomb - Ratio	86.6	88.0	87.0	87.4	90.9	91.1
Brightness - IPC - Percent	74.9	73.9	75.0	75.4	75.8	76.7
Fading Resistance - Brightness						
After 4 hour exposure	73.2	71.3	72.1	72.6	74.3	74.8
After 8 hour exposure	72.9	71.1	71.9	72.3	74.2	74.9
Smoothness - Bekk - Seconds	339	527	566	572	309	272
Dennison Wax Pick Test						
Pass Number	9.0	9.0	9.0	8.8	6.8	6.4
Slight Pick	10.0	10.0	10.0	---	7.8	7.4
Body Stock Pick	11.0	11.0	11.0	10.6	9.0	8.6
IGT Pick Test						
Start of Pick - cm/sec	143	185	181	203	55	61
K & N Ink Receptivity						
Brightness at 571 mu	59.8	54.7	50.4	52.7	52.1	47.3

Figure 11

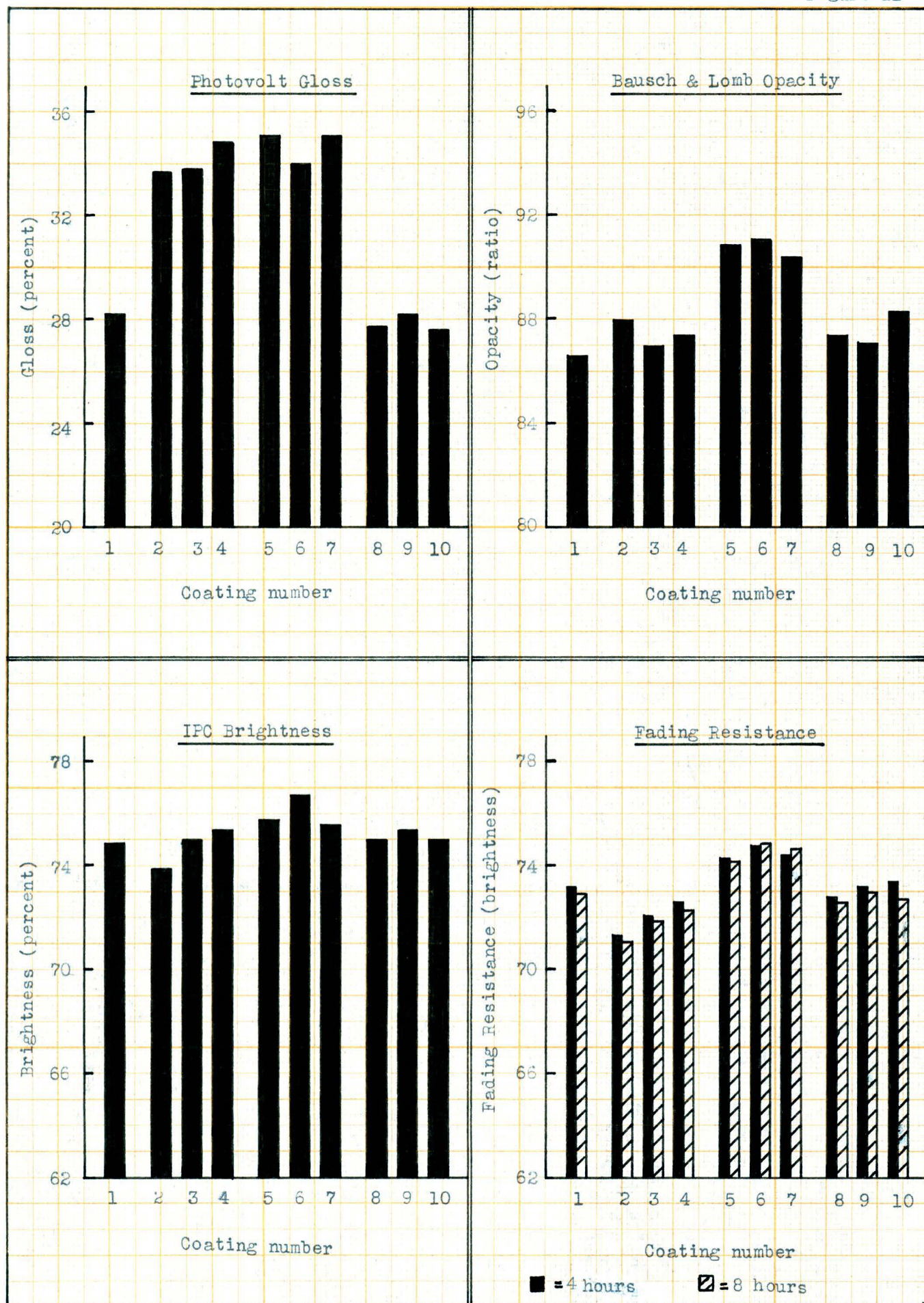


Figure 12

