THE EFFECT OF THE DEGREE OF POLYMERIZATION OF THE CELLULOSE AND THE DRAFT IN SPINNING ON THE MICELLE ORIENTATION AND THE PHYSICAL PROPERTIES OF THE RAYON

A Thesis

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ABSTRACT

Rayon, the degree of polymerization of which was varied by varying the aging time of the alkali cellulose, was spun under drafts ranging from 1 to 4. One series of batches was spun without tension and another under thirty grams tension. A variation in draft during spinning without tension produced very little change in the serimetric properties of the yarn. The tensile strength of yarn spun under 30 grams tension showed an even increase, and the elongation a proportional decrease for each increase in the draft. The value of degree of polymerization which gave the best tensile properties of yarn spun at 30 grams tension was 371 (alkali cellulose aged $2\frac{1}{2}$ days at 18°C.) The tensile properties decreased gradually with an increase in the degree of polymerization above the optimum value of 371. A decrease in degree of polymerization below the optimum gave a sharp decrease in the tensile strength of the yarn.

PURPOSE

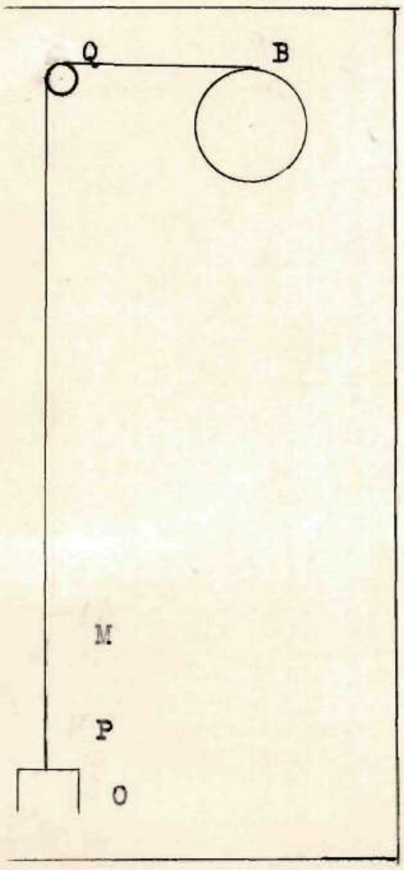
It has been known for a long time that there is a relationship between the serimetric properties of rayon and its chemical and physical properties. However, the investigations so far have been either qualitative or incomplete. The purpose of this thesis is to make a systematic study of the relations between micelle length of cellulose, and the spinning draft and effect on the strength, elongation, and micelle orientation. This investigation should give a fairly complete picture of this portion of rayon production and will enable conditions of optimum draft to be defined.

INTRODUCTION

The physical properties and inner structure of a viscose rayon filament are dependent on many factors. By causing one or more of these factors to be varied at definite intervals within proper limits it is possible to get an idea of the type and magnitude of influence exercised by these factors on the properties of the yarn. Preston (1), in an article on the skin effect in viscose rayon, gives a clear description of the functions of each of the factors which tend to orient the cellulose micelles during spinning. "In the production of viscose rayon, the cellulose xanthate solution in dilute caustic soda (viscose) is extruded under pressure through small orifices into a coagulating bath. The viscose consists of an unoriented swarm of cellulose micelles. As these pass through the orifice in the spinneret, surface friction tends to orient the micelles at the outside of the stream of viscose, while having little or no influence on those in the center. Independent of the orientation effect of the flowing viscose, there is a second orienting influence imposed on the filament due to the draft applied to the filament. The draft is independent of the absolute speed of spinning, and of the total number of filaments in the thread, and equals the ratio of the velocity of collection of the filament to the velocity of extrusion of the viscose solution. The effect of the draft depends on the stage at which it is applied to the filaments.

(1) J. M. Preston, J. Soc. Chem. Ind. 1931, 50, 199T

In the diagram shown, OPQB represents the track of the filaments during the spinning process. The viscose emerges from the spinneret at O and immediately forms a "skin" of oriented micelles which progressively thickens until by the point P the whole filament has set to a plastic solid though still containing alkali and undecomposed cellulose xanthate. Between "O" and "M" the filament travels through the coagulating bath, being progressively converted into cellulose. The point at which the cellulose xanthate is completely converted is earlier the higher the concentration and temperature of the coagulating bath, and the slower the speed at which the filament is traveling. During its passage



through the bath the filament is subject to hydrodynamic friction which varies with the viscosity of the coagulating bath liquor and the length of immersion. At the point "Q" the filament passes around a guide which subjects the filament to mechanical friction. The filament is then wound on a bobbin "B". Now the draft is applied to the filament between "O" and "B", but the position at which it is applied, and thus the state of coagulation of the filament at which it is applied, depends on the relative values of the frictional factors enumerated above. If the guide "Q" is of a frictionless type, the draft will mostly occur in the coagulating bath, which is generally referred to as spinning without tension, whilst if in addition to the former (i. e., little friction at the guide) there is a short immersion and a very fluid coagulating bath liquor, then the draft will occur near the spinneret, and much of this draft will take place between "O" and "P" while the centre of the filament is still liquid; it will thus increase the "skin" effect without orienting the central portion to the same extent. On the other hand, if hydrodynamical and mechanical friction is applied to the filament, the draft will occur between "P" and "Q" and "B" and will thus orient the "skin" and the central portion of the filament equally."

Schorger (2) states that the spinning operation, for the sake of an analogy, may be likened to driving logs in a stream; here by controlling the number of logs, their size and the speed of the water, it is possible to drive them largely in parallel.

Rogorin and Chernaya (3) concluded from the results of a comparative study of the effect of the degree of cellulose destruction, obtained by varying the time of pre-aging, on the serimetric properties of viscose rayon that with increasing cellulose destruction above a definite limit, the strength of rayon decreases somewhat and the stretching considerably. It is probable that short chains are better oriented than long ones. Hence, at the same stretching in spinning a greater orientation results for the filaments formed from cellulose of high destruction. Nakashima, Murakami and Ohara (4) state that cellulose which forms cuprammonium

(2) A. W. Schorger, J. Society of Chem. Ind. 1930, 49, 154T
(3) Z. A. Rogorin and F. Chernaya, Org. Chem. Ind. (U.S.S.R.) 1, 405-409 (1936)
(4) T. Nakashima, J. Murakami and S. Ohara. J. Soc. Chem. Ind., Japan 33, suppl. binding 44 (1930)

solutions of low viscosity forms viscose of low viscosity and the fibers spun from it are weaker, the lower the viscosity. Under certain conditions, however, strong fibers can be spun from the low viscosity solutions.

Kita, Tomihisa, Sakusada, Nakamura and Karo (5) in an investigation of the influence of aging and ripening on the spinning find that the tenacity and elongation increase with the time of aging of the alkali cellulose and the ripening of the viscose up to a certain point, beyond which they remain constant and decrease slightly.

(5) G. Kita, R. Tomihisa, K. Sakusada, Y. Nakamura and H. Karo. Cellulose Ind. (Tokyo) 3, 117-125 (1927)

PROCEDURE

1. Method

A batch of 780 grams of spruce sulfite sheets was placed in a water-jacketed steeping press containing 18% NaOH. After steeping for one hour at 20 °C., the caustic was drained off and the swollen sheets pressed by a ram. The ratio of the weight of pressed alkali cellulose to weight of the charge of cellulose sheets was 3. The sheets of alkali cellulose were then fed into a water-jacketed shredder, where the alkali cellulose was reduced to soft crumbs. The temperature was kept below 20 °C. during the 2-1/4 hours of shredding. After the crumbs had been aged at 18 °C. for a time period which varied with each batch, the crumbs were charged into the xanthating drum. The calculated amount of carbon disulfide (31% of the weight of cellulose) was added in two portions, one-half at the start, and the remainder one hour later. The total time for xanthation was three hours and the temperature was kept close to 25 °C. by cooling water in the jacket. A viscose solution containing 7% cellulose and 6.5% NaOH was obtained by mixing the xanthated crumbs in a calculated amount of dilute caustic soda for a dissolving period of three hours at a temperature ranging from 15-18 °C. The viscose was filtered twice during the ripening period of three days at 18.0 °C. and exposed to vacuum throughout the ripening time to remove air bubbles.

The viscose was spun on a five-jet spinning machine, but only one jet was used in order to keep the conditions constant. The spinning rate was 70.5 meters per minute. Each bobbin was spun for 15 minutes. The spinning bath of the following composition was kept at 45 °C.:

H_2SO_4 - 9.8%
Na_2SO_4-18.1%
$MgSO_4$ - 3.8%
$ZnSO_4$ - 1.0%

A series of four batches (aging time of alkali cellulose 2, 3, 4 and 5 days) was spun with a minimum spinning tension averaging around ten grams. Spinning at such a low tension is generally referred to as spinning without tension. Each batch consisted of six bobbins, each spun at a different draft. The draft ranged from a value of 1.1 (the rate of withdrawal slightly greater than the rate of expulsion of the viscose from the nozzles) to a value of four. The variations in draft were produced by decreasing the rate of expulsion of the viscose while maintaining a constant rate of withdrawal. In this way it was possible to keep the immersion time constant.

Spinning at drafts in excess of four would have been impossible with the spinnerets available. A second series of batches (aging time of alkali cellulose ranged from one to seven days at half day intervals) was spun at a tension of thirty grams. The tension was applied at point "Q" (see above diagram) by means of an adjustable glass thread guide. The variation in draft for the second series was similar to the first. The rayon was washed by placing the bobbins in a large pan of running water and allowing them to remain there for twenty-four hours. The rayon was then allowed to air-dry.

2. Physical Testing of the Rayon

The rayon was twisted to 2.5-3.0 turns per inch. The twisted rayon was then reeled into four skeins of 225 meters length. Each skein was weighed for denier determination. The third skein was used for the breaking strength determinations. The yarn was conditioned at 65% relative humidity and the breaking and weighing were done in that atmosphere. A Suter serimeter was used for the breaking strength tests. The number of breaks made on each skein varied with the uniformity of the yarn. Twenty breaks were made on yarn whose tensile properties varied within a small range. The number of breaks was increased to thirty or more when the variation exceeded set limits. The average deviation from the mean breaking strength of the thread allowed for the strength test was 1.5 grams. The number of breaks necessary to produce this accuracy of results was calculated by means of statistical analysis. For wet strengths, the strands were tied in the clamps and the yarn wetted by a brush with water containing a little pine oil. Desulfurization of the yarn under alkaline conditions would produce changes in the properties of the yarn. Since slight variations in the magnitude of these changes from one bobbin to another would tend to introduce an error in the results, it was decided to make all tests on undesulfurized yarn.

3. Micelle Length Determination

Staudinger (6) states that the relation between solution viscosity of cellulose in cuprammonium solution and its molecular weight (chain length) is of a quantitative nature. In general, this theory claims that viscosity in solution increases proportionally with increasing chain length, and vice versa, and that in solution the single chain molecules ("macro-molecules","thread molecules") exist.

(6) H. Staudinger, Ber., 1930, 63, 222

It is important that the concentration of cellulose in cuprammonium be very low so that the macro-molecules do not interfere with each other. The upper limit of concentration which may be used for artificial fibers is 0.37%. The concentration of cellulose used was 0.20%.

The fluidity method of viscosity measurement of Clibbens and Geake was employed. A set of 15 viscometers of suitable dimensions were calibrated with a glycerine-water mixture and with pure water. The cuprammonium solution was made up by bubbling air through strong ammonia contained in a cylinder packed with copper turnings.

It was found that in spite of strict adherence to the usual precautions outlined in the Clibbens and Geake method that it was impossible to remove all air bubbles from the tube when filling with cuprammonium solution. This difficulty was overcome by sweeping the tubes clear of all air by alternately filling with hydrogen and evacuating.

In order to connect viscosity and molecular weight, the relative viscosities of solutions were not compared, but rather the specific viscosity, i. e., the increase in viscosity which is produced in a solvent by the dissolved substance. The specific viscosity is then $n_r - 1$, and between the specific viscosity, n_{sp}, of equally concentrated solutions and the molecular weight, M, of the cellulose there exists a simple relation:

$$n_{sp}/c = K_m M$$

where "c" is the concentration of a primary molar solution. The value of "K_m" was calculated by Staudinger (7) to be 5×10^{-4} for cellulose.

A sample calculation of the molecular weight and the D. P. of a sample of rayon will clarify the steps necessary for the determination of D. P.

The viscosity of the solutions was obtained in terms of fluidity which is the reciprocal of viscosity.

The fluidity of cuprammonium solvent = 70 reciprocal poises.

The fluidity of 0.20% solution of an average sample = 55.0

$$n_{sp} \text{ (specific viscosity)} = \frac{70.0}{55.0} - 1 = 0.273$$

The concentration of solution in primary mols

$$= \frac{\text{grams cellulose per liter}}{\text{molecular weight of glucose anhydride}} = \frac{2}{162} = 0.01234$$

$$n_{sp} = K\ M$$

$$\frac{0.273}{0.1234} = 5 \times 10^{-4}\ M$$

$$M = 44{,}300$$

$$D.P. = \frac{44{,}300}{162} = 273$$

(7) H. Staudinger, Ann. 1937, 529, 219.

The composition of the cuprammonium solution used was:

Copper:	15 $\pm$ 0.2 grams per liter
NH_3:	240 $\pm$ 10 grams per liter
Glucose:	3 grams per liter
HNO_2:	Less than 1 gram per liter

The copper content was found by evaporation of a known volume to dryness and conversion of the residue to copper oxide. The ammonia content was found by adding 2 cc, to 25 cc. of 2N sulfuric acid and titrating back with 0.5N alkali, using methyl red as an indicator, allowance being made for the copper present. The allowance consisted of a subtraction of 0.536 "C" grams per liter of ammonia where "C" is the number of grams of copper per liter. The nitrous acid content was found by the volume of cuprammonium solution necessary to decolorize 10 cc of 0.1N potassium permanganate in the presence of excess dilute sulfuric acid. The HNO_2 concentration was calculated in the usual way.

The analytical procedures described above are used in standard practice and may be found in most books on the chemistry of cellulose.

4. Micelle Orientation Measurement

The micellar orientation of the rayon fiber was measured by the method of Morey (8). This method is based on the polarization of fluorescence of dye molecules attached to the cellulose micelles of the fiber. The dye molecules are assumed to be attached alongside the micelles.

(8) D. R. Morey. Textile Research, 3, 325 - 345 (1933)

The instrument consists of a microscope with a rotatable polarizing prism mounted behind the objective. A tiny mirror is mounted in the focal plane of the eyepiece and reflects the comparison light coming in at right angles to the body tube. The photometer arrangement consists of a fixed and a rotatable Nicol prism. A graduated disc makes it possible to measure the angle of the rotating prism from the position of extinction.

The measurements are made by placing the fiber on the stage and adjusting so that the mirror is superimposed on the image of the fiber. The photometer prism is rotated and adjusted until the intensity of the light from the fiber and the intensity from the mirror are matched. Two measurements are made, one with the plane of vibration perpendicular to the fiber and the other with the plane of vibration parallel to the fiber. These measurements give the minimum and maximum intensities respectively. The intensity is calculated from the formula:

$$I = \sin^2 \theta$$

where I = intensity
θ = the angle the photometer makes with position of extinction.

The degree of orientation or percent orientation is given by:

$$\% \text{ Orientation} = \frac{I_{max} - I_{min}}{I_{max} - 2I_{min}} \times 100$$

EXPERIMENTAL RESULTS

All values for strengths are given in grams per denier. The values for elongation are in percentages of original length.

The draft is the ratio of the velocity of collection of the filament to the velocity of extrusion of the viscose solution.

The denier of yarn is the weight in grams of 9,000 meters of the yarn.

Batches E-C-4, E-C-2, E-C-5, and E-C-3 were spun at minimum spinning tension of 10 grams. The remainder of the batches were spun at 30 grams tension.

D. P. is an abbreviation of degree of polymerization. The D.P. multiplied by the molecular weight of a glucose anhydride residue, the primary molecule of cellulose, equals the molecular weight of cellulose.

Table No. 1

Composite Data Sheet

Draft	Denier	Strength Dry	Strength Wet	Elongation Dry	Elongation Wet	Percent Orientation
			Batch E-C-4			
			Age of crumbs: 43 hours			
			D. P. of rayon: 411			
1.14	217	1.53	0.63	17.9	33.6	21.5
1.52	164	1.50	0.64	18.1	33.0	23.2
2.02	126	1.49	0.64	17.8	32.8	26.2
2.46	101	1.44	0.64	14.5	28.3	29.8
3.15	80	1.54	0.68	17.3	29.2	28.5
			Batch E-C-2			
			Age of crumbs: 67 hours			
			D. P. of rayon: 380			
1.18	219	1.42	0.56	17.1	31.7	17.7
1.56	165	1.48	0.59	17.8	29.4	20.0
2.02	125	1.39	0.59	16.0	29.4	18.7
2.54	102	1.42	0.60	14.7	27.1	21.1
3.18	80	1.45	0.64	15.8	29.4	19.9
			Batch E-C-5			
			Age of crumbs: 91 hours			
			D. P. of rayon: 347			
1.15	221	1.37	0.49	18.8	30.5	21.1
1.56	168	1.37	0.52	15.8	27.6	22.2
1.98	130	1.42	0.59	16.8	30.2	22.8
2.50	102	1.39	0.60	14.7	28.6	25.7
3.12	82	1.40	0.57	14.2	26.0	25.0
			Batch E-C-3			
			Age of crumbs: 115 hours			
			D. P. of rayon: 320			
1.15	221	1.46	0.54	15.6	27.2	16.9
1.53	167	1.52	0.60	16.1	28.8	16.6
1.98	128	1.52	0.57	14.8	25.2	19.8
2.42	104	1.55	0.64	16.2	28.0	20.4
3.09	82	1.56	0.61	15.5	26.9	21.1

Table No. 1 (Cont.)

Draft	Denier	Strength Dry	Strength Wet	Elongation Dry	Elongation Wet	Percent Orientation
			Batch E-C-13			
			Age of crumbs: 19 hours D. P. of crumbs: 605 D. P. of rayon: 540			
1.16	210	1.67	0.65	12.4	14.0	
1.55	160	1.80	0.69	10.8	13.4	
2.04	122	1.83	0.75	9.3	12.5	
2.43	99	1.89	0.78	7.5	10.5	
3.12	76	2.02	0.90	7.0	7.9	
			Batch E-C-20			
			Age of crumbs: 36 hours D. P. of crumbs: 584 D. P. of rayon: 570			
1.25	201	1.72	0.71	13.4	20.4	
1.63	156	1.72	0.74	10.3	17.7	
2.18	121	1.82	0.77	10.0	15.9	
2.58	98	1.94	0.84	8.7	13.3	
3.31	77	1.94	0.90	7.2	9.3	
4.27	61	1.85	0.89	6.2	8.3	
			Batch E-C-12			
			Age of crumbs: 43 hours D. P. of crumbs: 498 D. P. of rayon: 470			
1.17	212	1.66	0.87	14.0	16.2	28.7
1.51	158	1.77	0.73	9.6	14.2	31.4
1.98	123	1.87	0.78	9.3	12.0	33.7
2.48	99	1.96	0.82	7.8	11.8	35.2
3.14	76	2.12	0.94	7.1	8.5	37.1
			Batch E-C-16			
			Age of crumbs: 61 hours D. P. of crumbs: 457 D. P. of rayon: 372			
1.17	216	1.78	0.74	11.5	18.6	
1.57	161	1.92	0.82	10.2	16.9	
2.02	122	1.98	0.87	9.5	15.5	
2.52	99	2.07	0.90	8.4	11.0	
3.20	79	2.05	0.94	7.8	10.7	
4.35	59	2.09	0.97	7.2	9.3	

Table No. 1 (Cont.)

Draft	Denier	Strength Dry	Strength Wet	Elongation Dry	Elongation Wet	Percent Orientation
		Batch E-C-14				
		Age of crumbs: 67 hours; D. P. of crumbs: 440; D. P. of rayon: 365				
1.16	218	1.68	0.74	12.8	18.3	29.8
1.53	163	1.75	0.72	10.6	16.5	34.7
2.00	125	1.88	0.78	8.2	10.2	39.8
2.52	99	2.02	0.88	7.3	10.8	38.0
3.18	78	2.08	0.92	6.4	8.7	39.4
4.22	62	2.12	0.95	6.5	8.3	40.2
		Batch E-C-17				
		Age of crumbs: 84 hours; D. P. of crumbs: 408; D. P. of rayon: 340				
1.16	208	1.78	0.74	11.6	20.1	
1.52	160	1.86	0.79	10.2	19.9	
1.99	124	1.89	0.80	10.2	15.8	
2.48	98	2.02	0.88	8.8	13.3	
3.06	80	1.87	0.82	6.6	9.5	
4.07	61	2.03	0.95	7.3	10.1	
		Batch E-C-9				
		Age of crumbs: 92 hours; D.P. of crumbs: 358; D. P. of rayon: 351				
1.17	214	1.64	0.62	11.1	13.7	27.4
1.56	164	1.70	0.66	8.5	10.9	36.8
1.98	125	1.89	0.75	7.1	9.3	39.8
2.46	100	1.94	0.82	6.8	8.8	43.7
3.13	76	2.06	0.86	6.4	7.9	47.6
4.15	62	2.06	0.92	5.4	6.8	42.2
		Batch E-C-21				
		Age of crumbs: 108 hours; D. P. of crumbs: 387; D. P. of rayon: 330				
1.13	214	1.76	0.71	10.8	15.5	
1.52	161	1.85	0.75	10.8	17.7	
1.94	125	1.94	0.84	10.0	14.8	
2.42	99	1.98	0.86	7.3	11.0	
2.92	81	2.02	0.89	8.0	11.5	

Table No. 1 (Cont.)

Draft	Denier	Strength Dry	Strength Wet	Elongation Dry	Elongation Wet	Percent Orientation
			Batch E-C-7			
			Age of crumbs: 115 hours			
			D. P. of crumbs: 334			
			D. P. of rayon: 312			
1.13	218	1.59	0.60	10.8	15.7	25.6
1.50	164	1.75	0.68	9.3	12.7	31.3
1.98	124	1.84	0.73	8.1	10.7	32.1
2.50	100	1.92	0.78	7.1	9.8	38.3
3.00	80	1.84	0.79	6.9	7.8	39.5
			Batch E-C-18			
			Age of crumbs: 132 hours			
			D. P. of crumbs: 390			
			D. P. of rayon: 300			
1.14	213	1.81	0.73	11.0	17.7	
1.51	162	1.80	0.72	8.9	14.0	
1.91	128	1.87	0.81	9.0	14.1	
2.34	106	1.96	0.84	8.0	13.0	
2.88	83	1.92	0.95	7.2	11.5	
3.64	68	2.04	0.95	7.1	9.9	
			Batch E-C-10			
			Age of crumbs: 139 hours			
			D. P. of crumbs: 313			
			D. P. of rayon: 248			
1.13	217	1.59	0.62	8.7	8.9	31.1
1.50	163	1.75	0.68	8.6	9.7	35.1
1.92	125	1.85	0.70	6.3	7.7	38.6
2.46	102	1.93	0.78	6.2	7.3	37.7
3.09	80	1.98	0.84	5.5	6.3	41.1
			Batch E-C-19			
			Age of crumbs: 156 hours			
			D. P. of crumbs: 330			
			D. P. of rayon: 283			
1.08	221	1.67	0.64	9.6	15.2	
1.42	170	1.72	0.62	8.8	12.8	
1.76	137	1.79	0.72	8.2	12.8	
2.26	114	1.77	0.73	7.3	10.3	
2.81	93	1.87	0.78	6.7	9.0	
3.54	76	1.85	0.77	6.1	8.3	

Table No. 1 (Cont.)

Draft	Denier	Strength		Elongation		Percent Orientation
		Dry	Wet	Dry	Wet	

Batch E-C-11

Age of Crumbs: 163 hours
D. P. of crumbs: 280
D. P. of rayon: 222

1.14	222	1.51	0.54	8.7	9.6	
1.50	167	1.60	0.57	7.2	8.9	
1.92	126	1.69	0.64	7.4	9.1	
2.36	106	1.68	0.62	6.2	7.5	
3.02	86	1.73	0.59	5.6	6.3	

Table No. 2

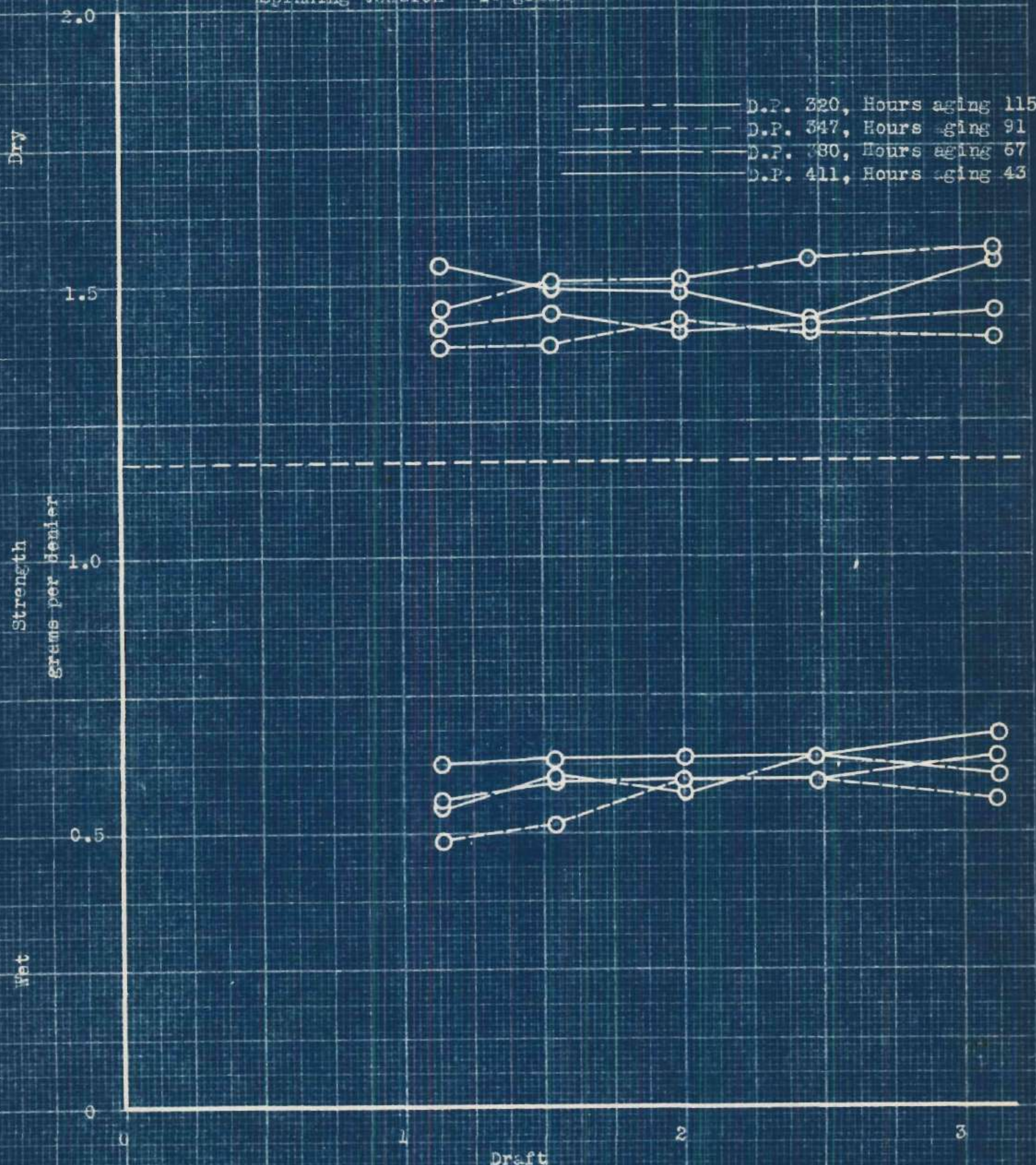
Composition and Properties of Viscose

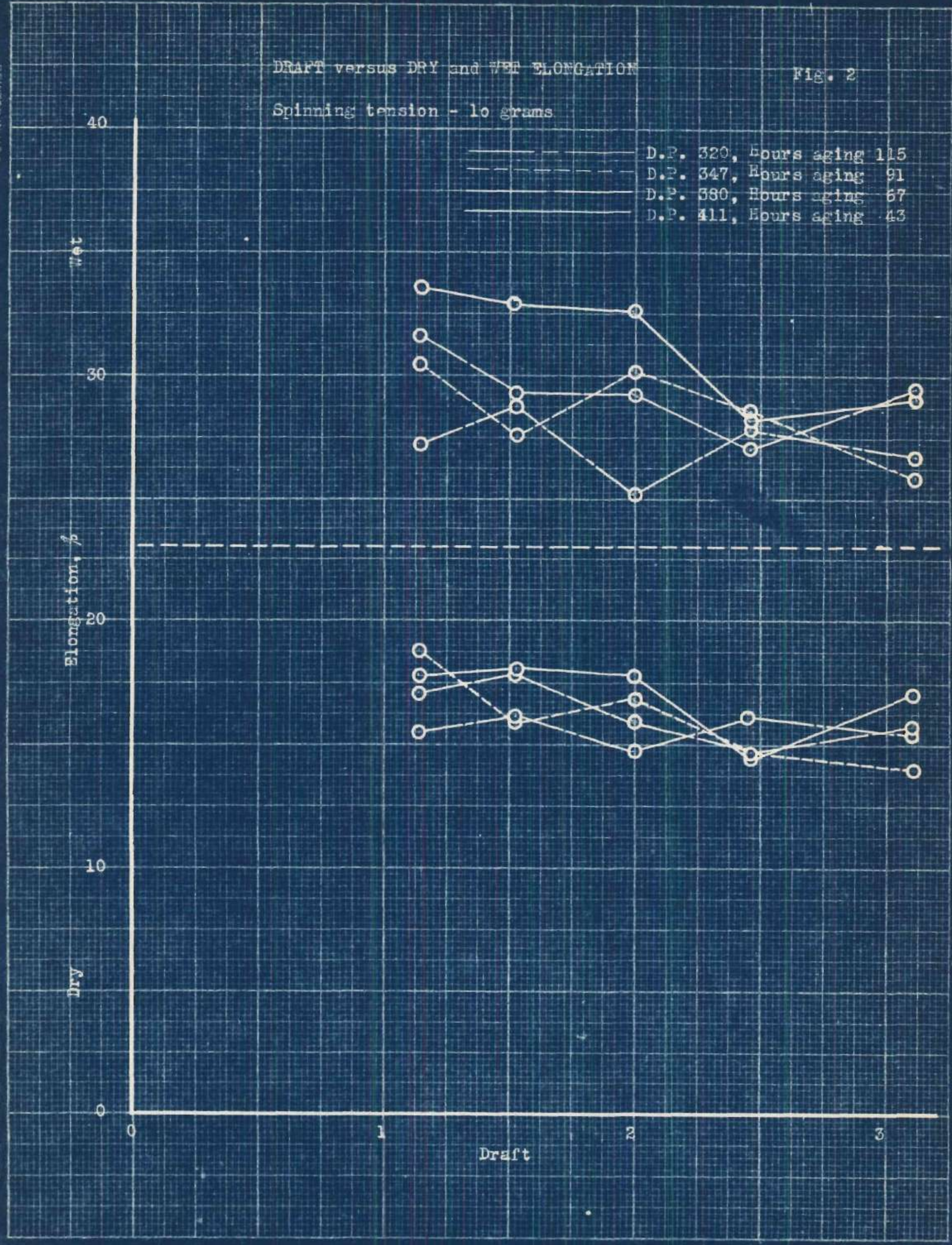
Batch No.	NaOH, %	Cellulose %	Viscosity, Seconds	Maturity, cc.10% NH_4Cl
E-C-4	6.53	6.99	78.0	7.9
E-C-2	6.42	7.04	30.0	7.8
E-C-5	6.45	7.06	24.0	7.7
E-C-3	6.49	6.95	13.5	8.1
E-C-13	6.73	7.07	302.0	7.2
E-C-20	6.58	7.31	233.7	8.05
E-C-12	6.45	6.94	81.5	7.8
E-C-16	6.30	7.05	41.8	8.0
E-C-14	6.42	6.98	37.8	8.4
E-C-17	6.31	6.86	30.8	8.4
E-C-9	6.42	7.03	24.2	7.4
E-C-21	6.53	6.79	20.5	9.3
E-C-7	6.40	6.91	16.0	8.3
E-C-18	6.34	7.10	12.5	9.1
E-C-10	6.48	6.95	8.6	8.4
E-C-19	6.54	6.86	11.0	8.85
E-C-11	6.65	6.98	7.3	8.2

DRAFT versus DRY and WET TENSILE STRENGTH

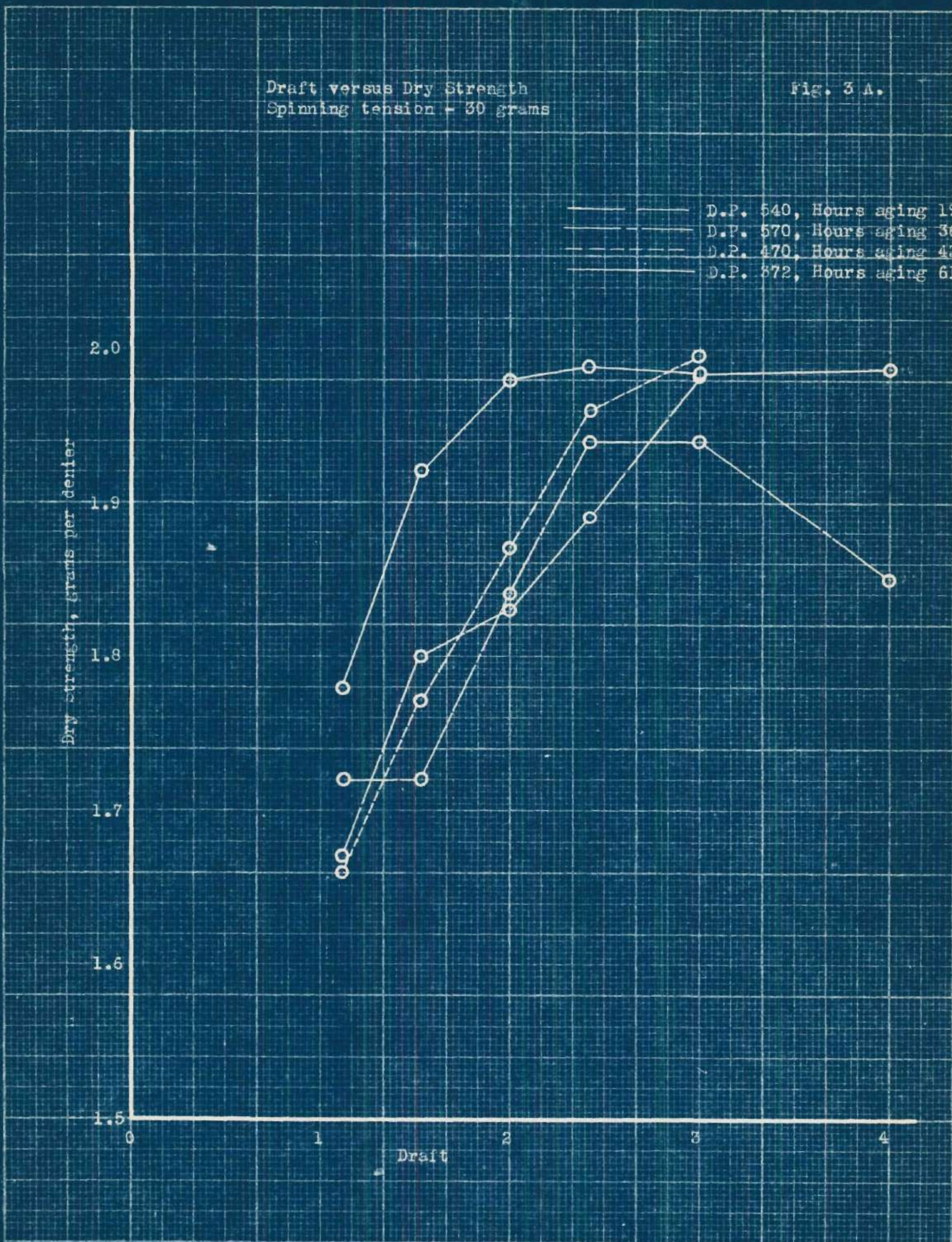
Fig. 1.

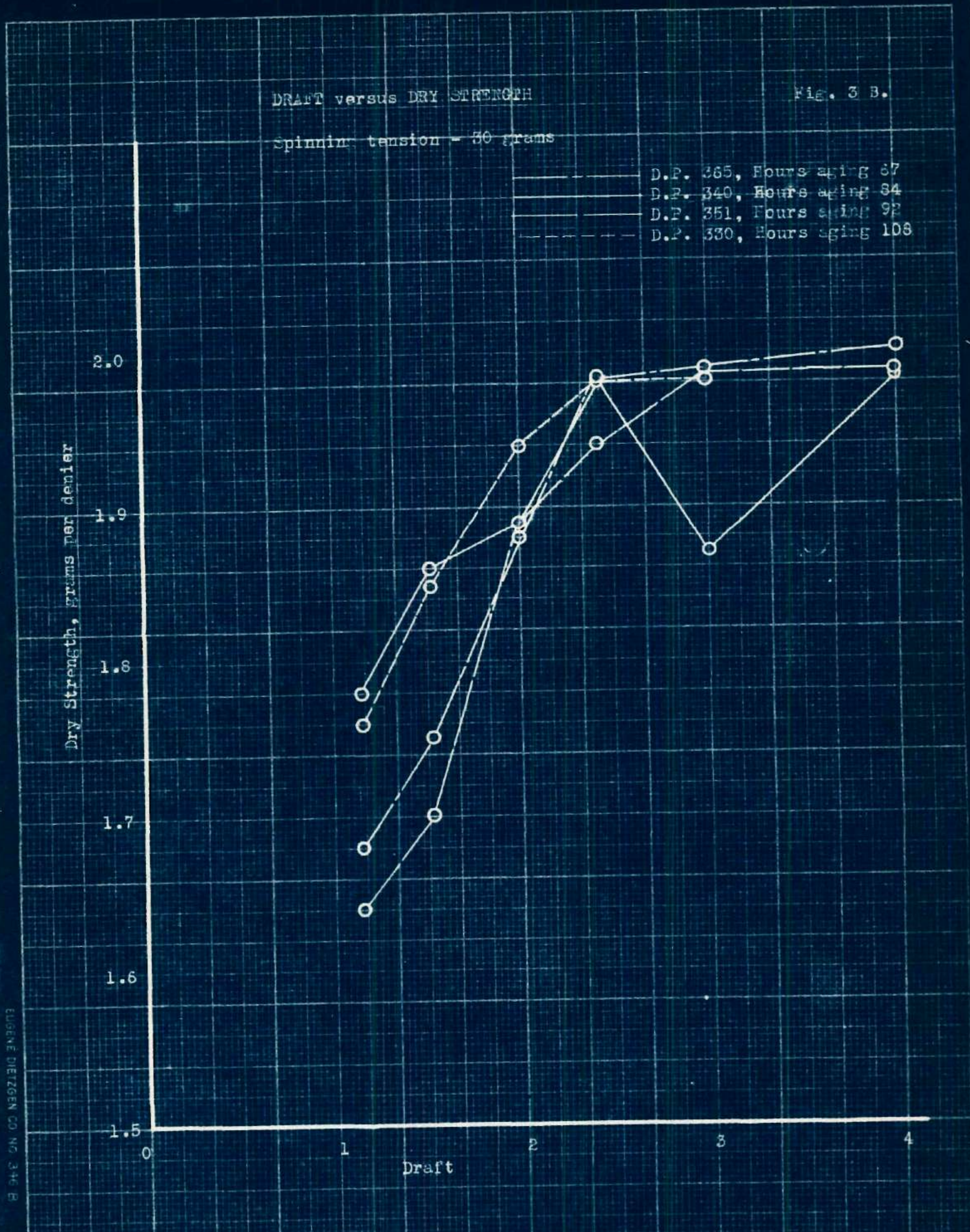
Spinning tension - 10 grams





DRAFT versus DRY and WET ELONGATION
Fig. 2
Spinning tension - 10 grams
D.P. 320, Hours aging 115
D.P. 347, Hours aging 91
D.P. 380, Hours aging 67
D.P. 411, Hours aging 43
Wet
Elongation, %
Dry
40
30
20
10
0
0
1
2
3
Draft

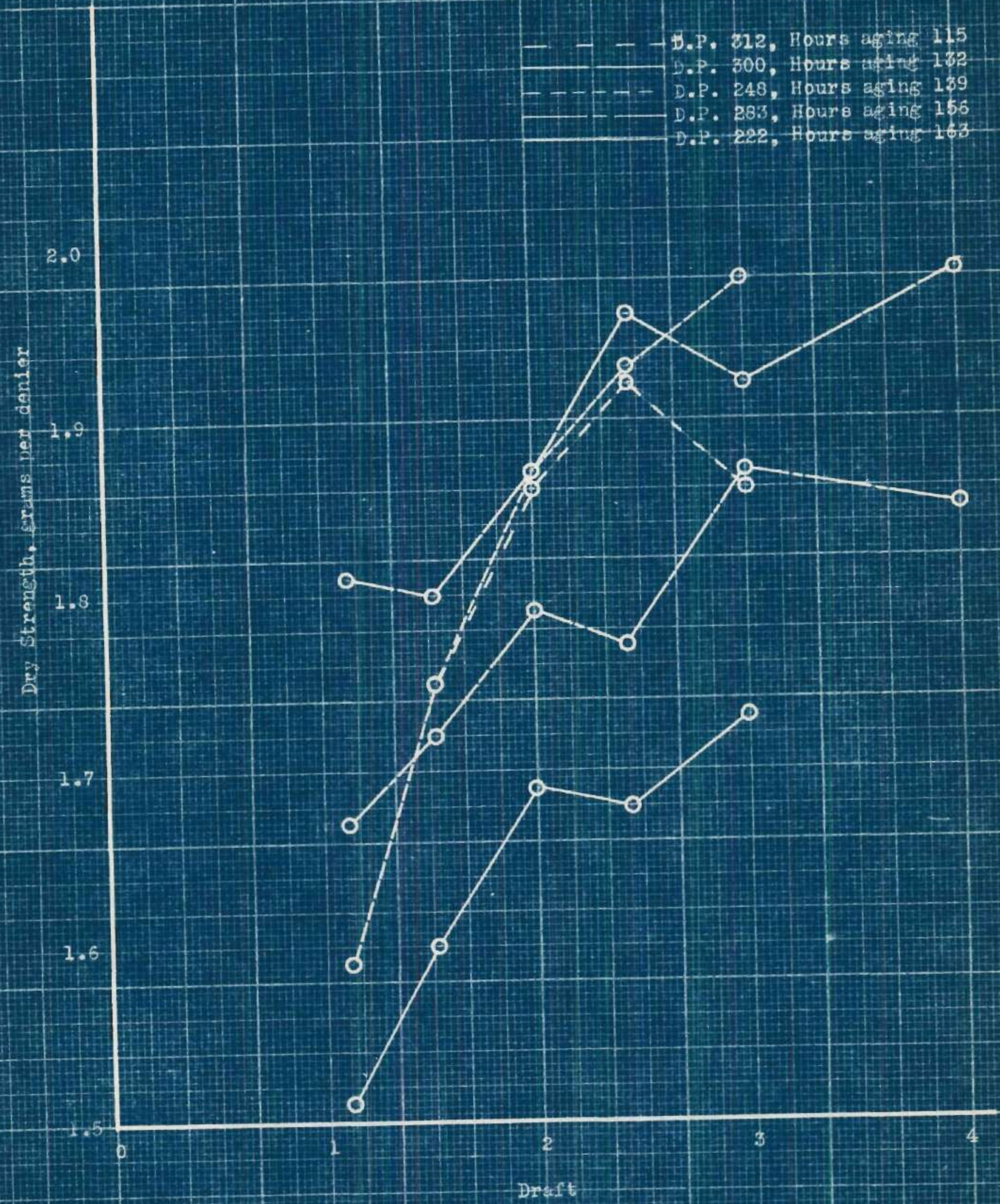

Draft versus Dry Strength
Spinning tension - 30 grams
Fig. 3 A.
D.P. 540, Hours aging 19
D.P. 570, Hours aging 36
D.P. 470, Hours aging 43
D.P. 872, Hours aging 61
Dry strength, grams per denier
2.0
1.9
1.8
1.7
1.6
1.5
0
1
2
3
4
Draft

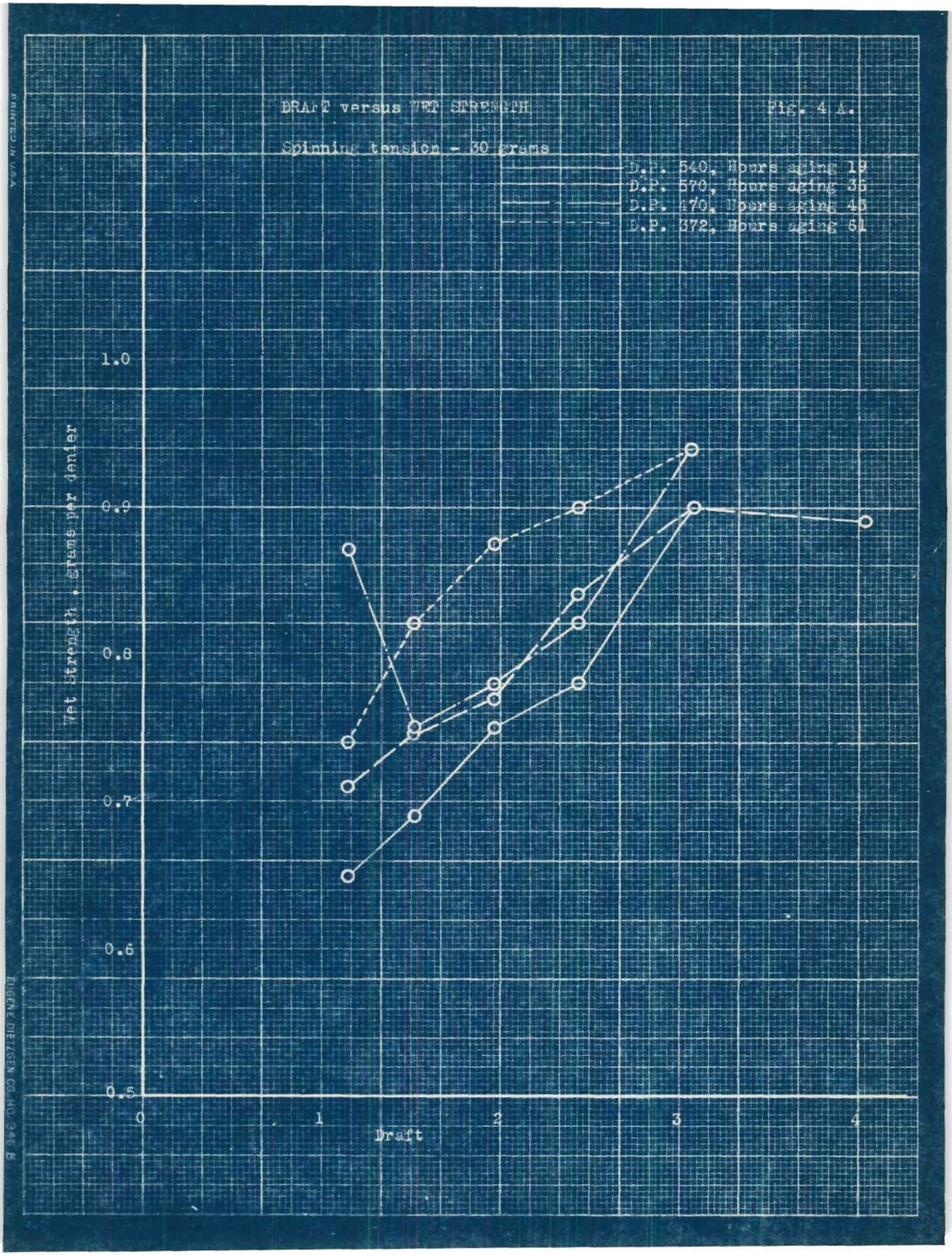

DRAFT versus DRY STRENGTH
Fig. 3 B.
Spinning tension - 30 grams
D.P. 365, Hours aging 67
D.P. 340, Hours aging 84
D.P. 351, Hours aging 92
D.P. 330, Hours aging 108
Dry Strength, grams per denier
2.0
1.9
1.8
1.7
1.6
1.5
0
1
2
3
4
Draft

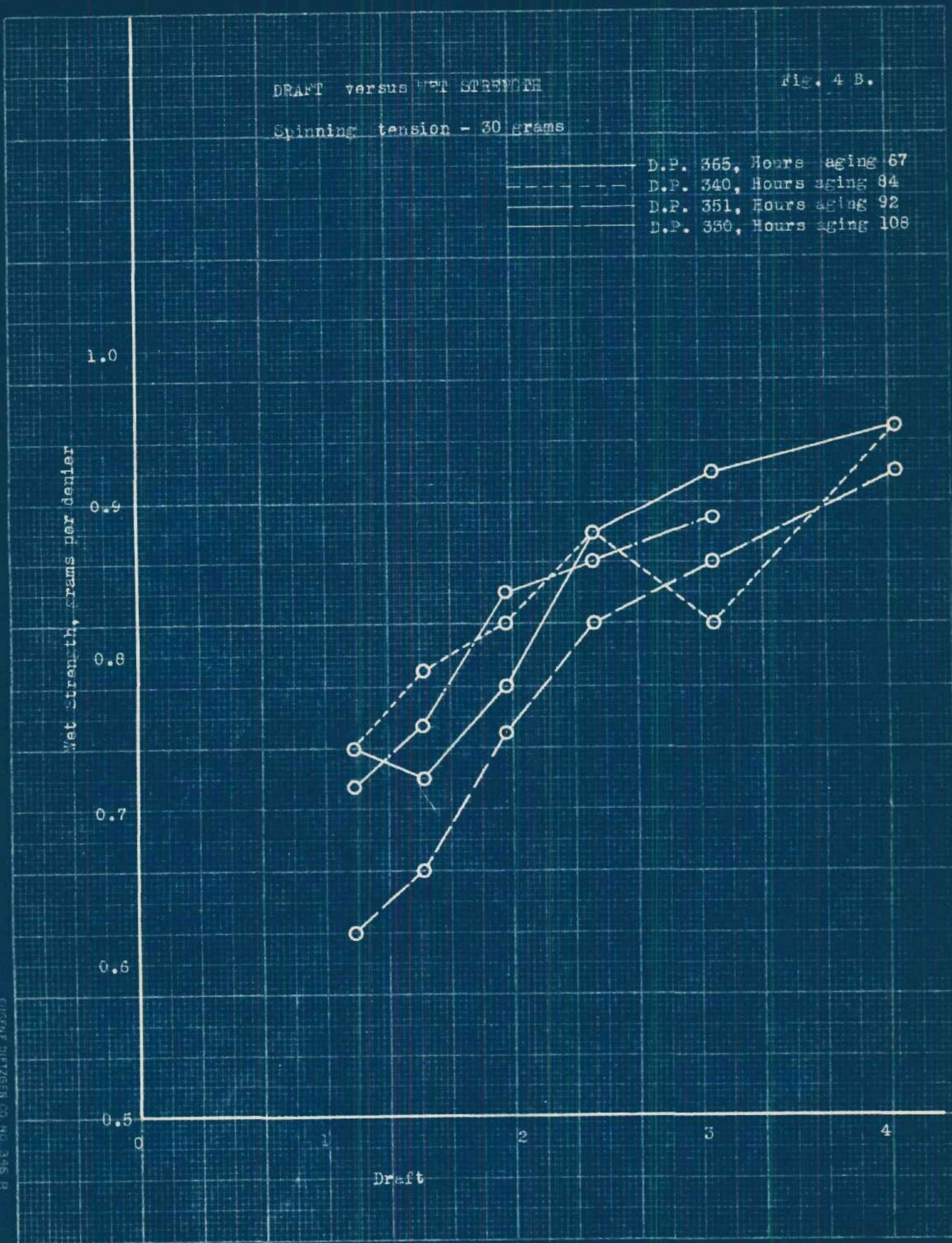
DRAFT versus DRY STRENGTH

Fig. 3 C.

Spinning tension - 30 grams

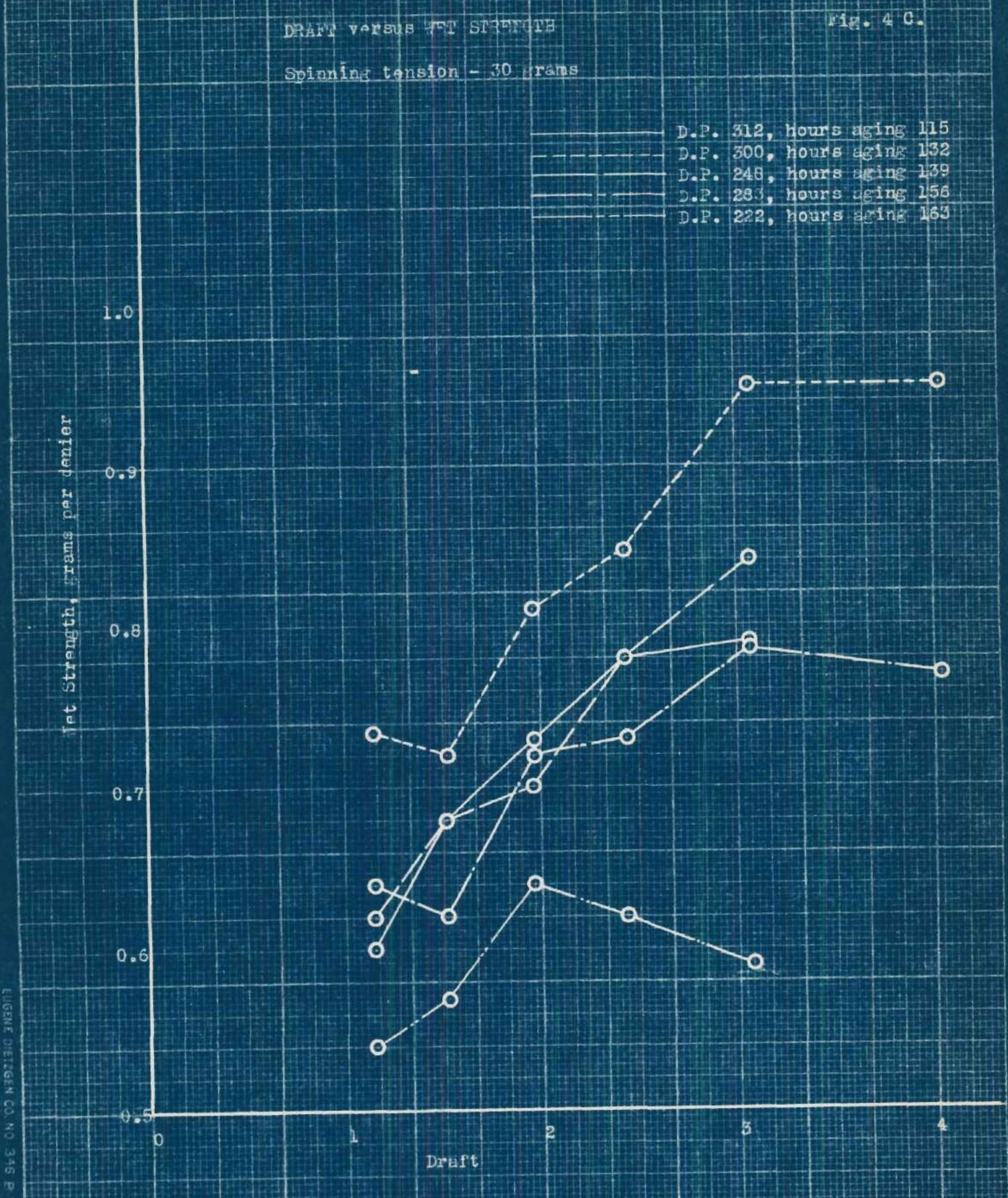


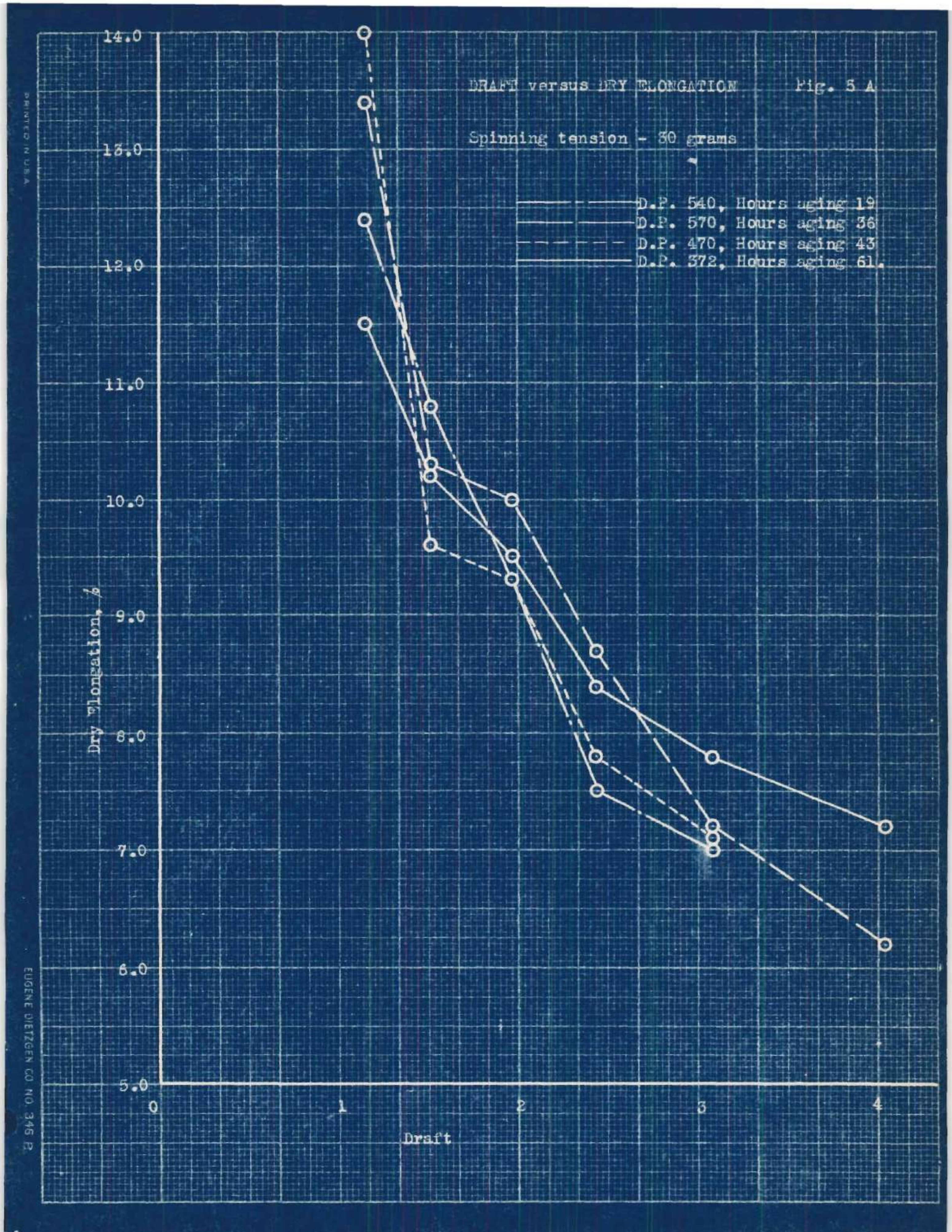

DRAFT versus WET STRENGTH
Fig. 4 A.
Spinning tension - 30 grams
D.P. 540, Hours aging 19
D.P. 570, Hours aging 35
D.P. 470, Hours aging 45
D.P. 372, Hours aging 61
Wet Strength, grams per denier
1.0
0.9
0.8
0.7
0.6
0.5
0
1
2
3
4
Draft



DRAFT versus WET STRENGTH
Spinning tension - 30 grams
Fig. 4 B.
D.P. 365, Hours aging 67
D.P. 340, Hours aging 84
D.P. 351, Hours aging 92
D.P. 350, Hours aging 108
Wet strength, grams per denier
1.0
0.9
0.8
0.7
0.6
0.5
0
1
2
3
4
Draft

Fig. 4 C.


DRAFT versus WET STRENGTH
Spinning tension - 30 grams
D.P. 312, hours aging 115
D.P. 300, hours aging 132
D.P. 248, hours aging 139
D.P. 283, hours aging 156
D.P. 222, hours aging 163
1.0
0.9
0.8
0.7
0.6
0.5
Wet Strength, grams per denier
0
1
2
3
4
Draft



DRAFT versus DRY ELONGATION
Fig. 5 A
Spinning tension - 30 grams
D.P. 540, Hours aging 19
D.P. 570, Hours aging 36
D.P. 470, Hours aging 43
D.P. 372, Hours aging 61.
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
Dry Elongation, %
0
1
2
3
4
Draft
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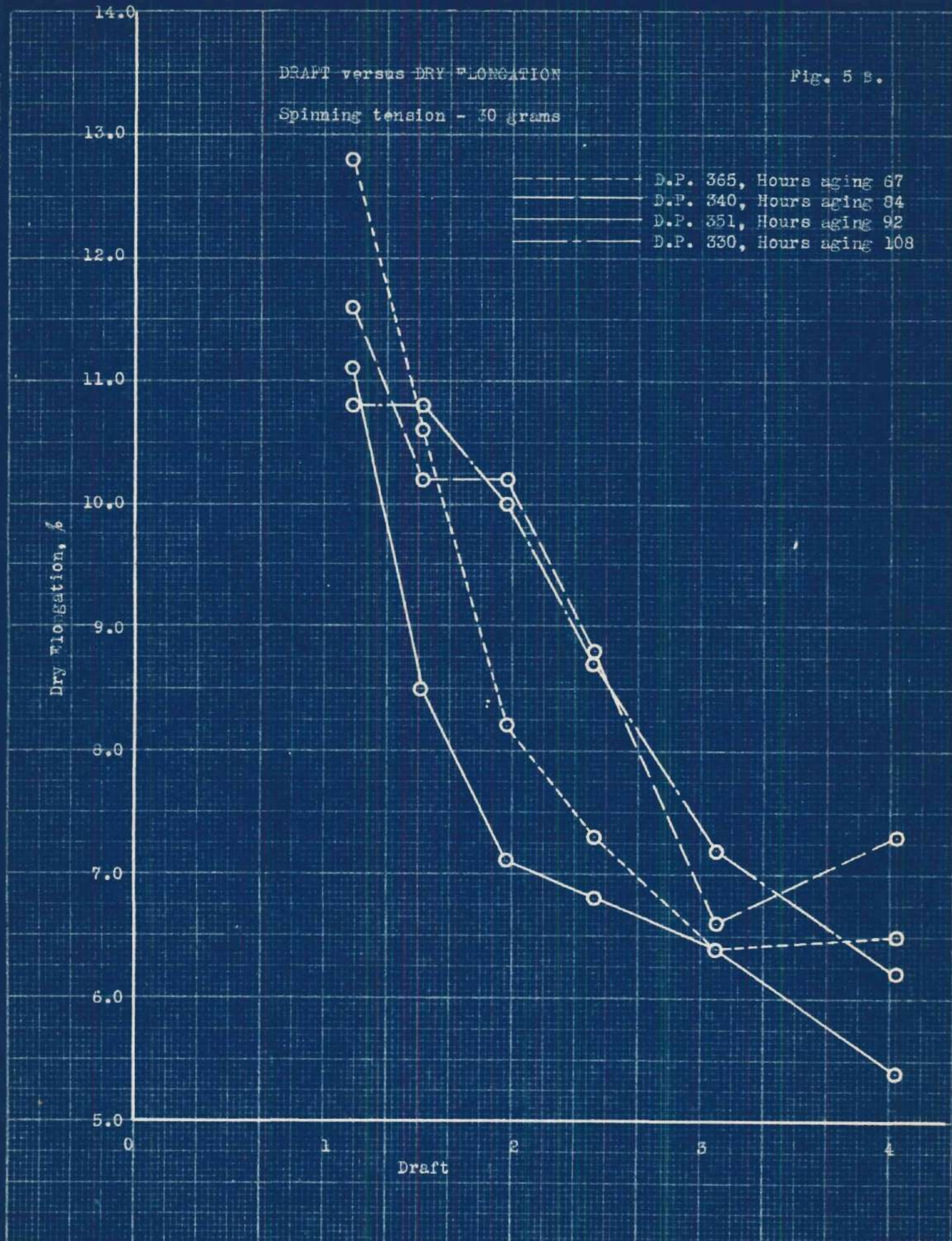
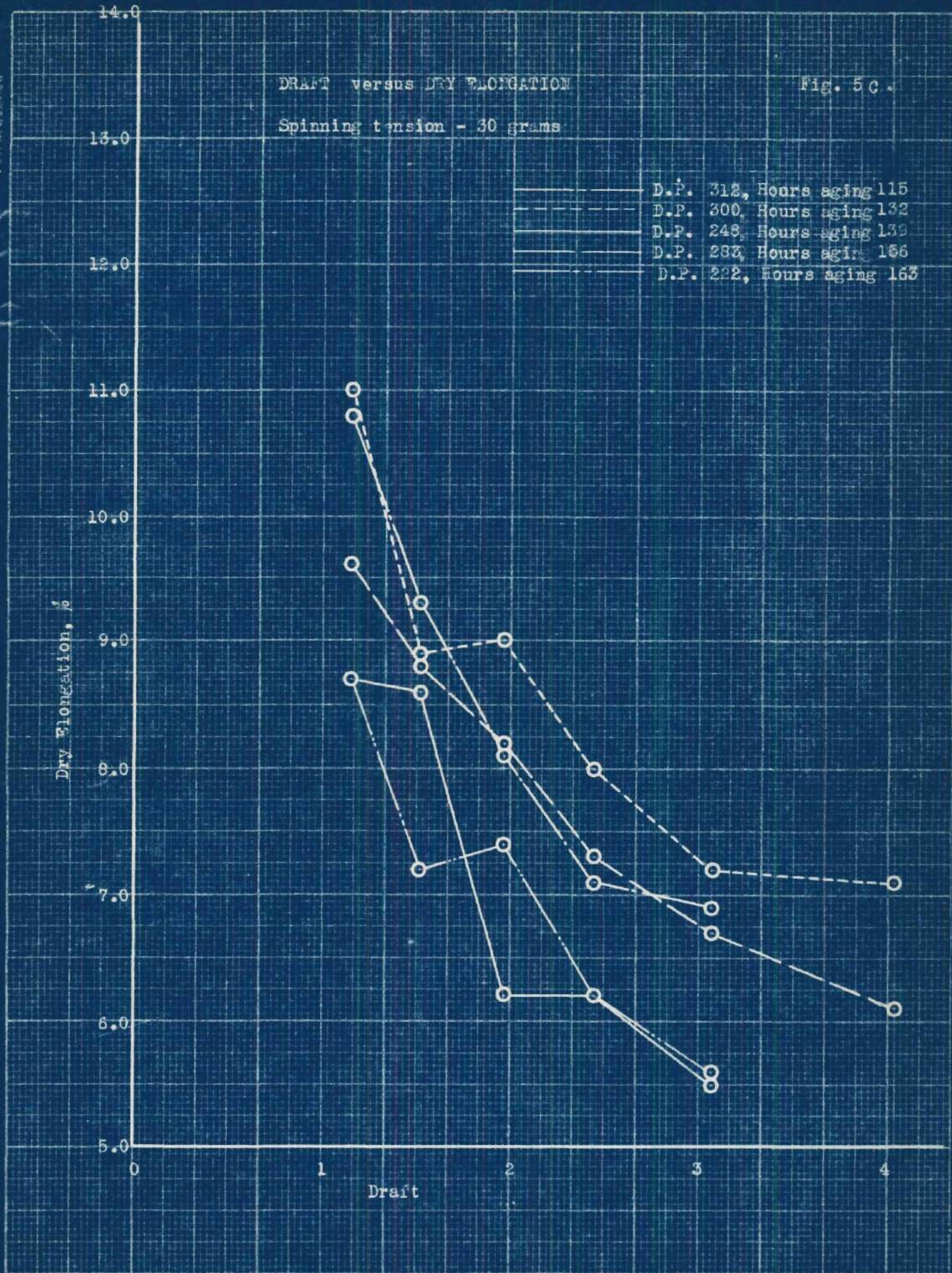
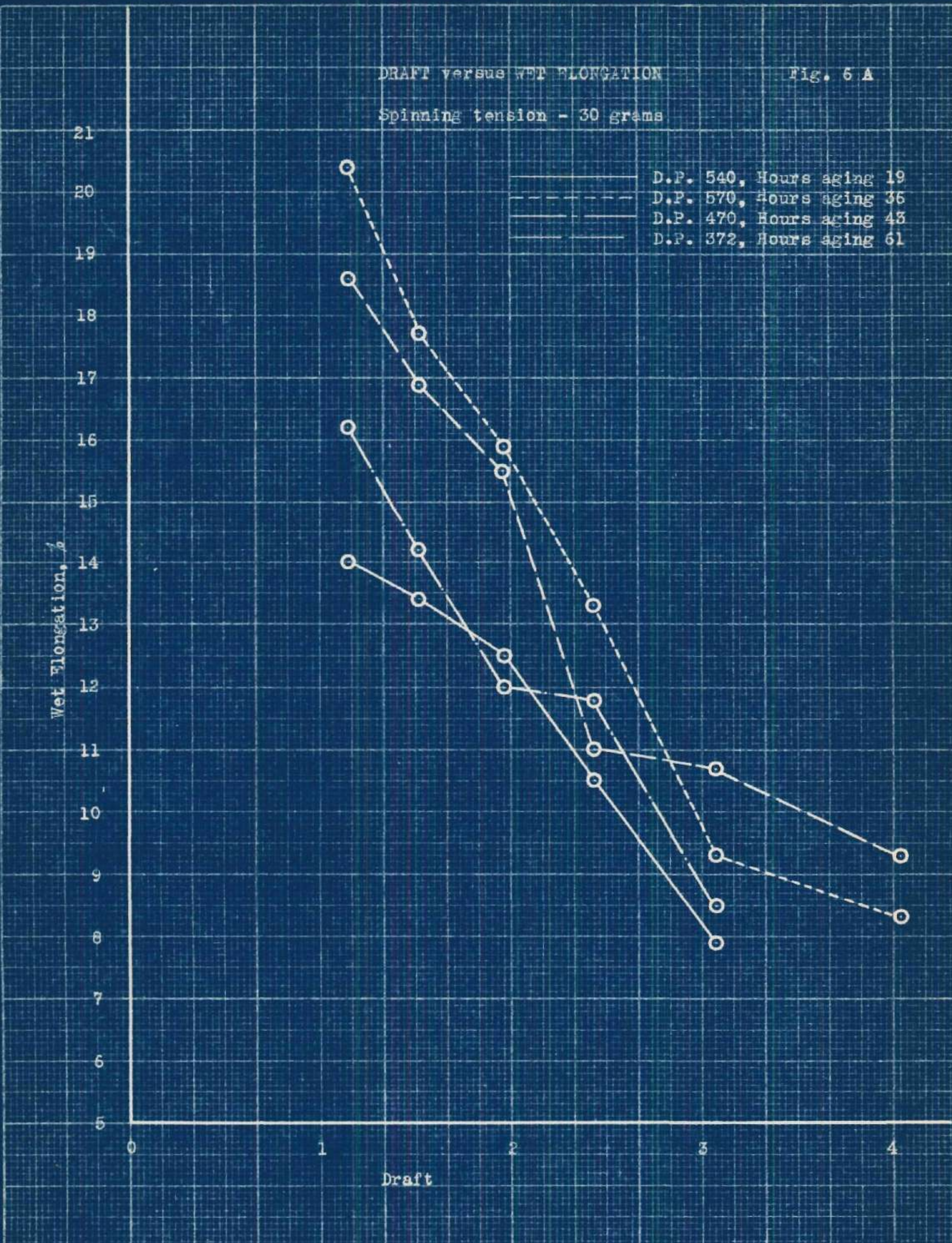
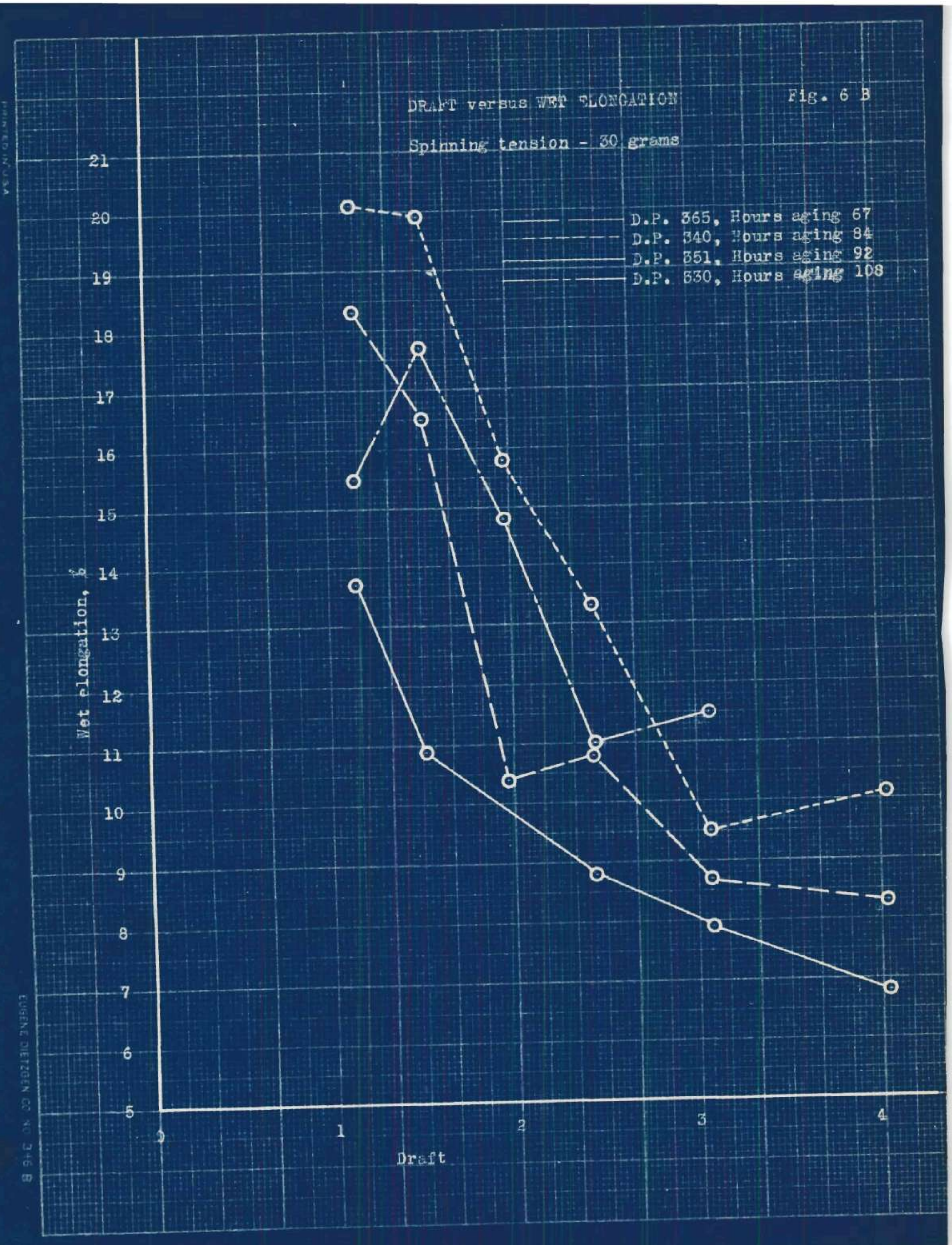


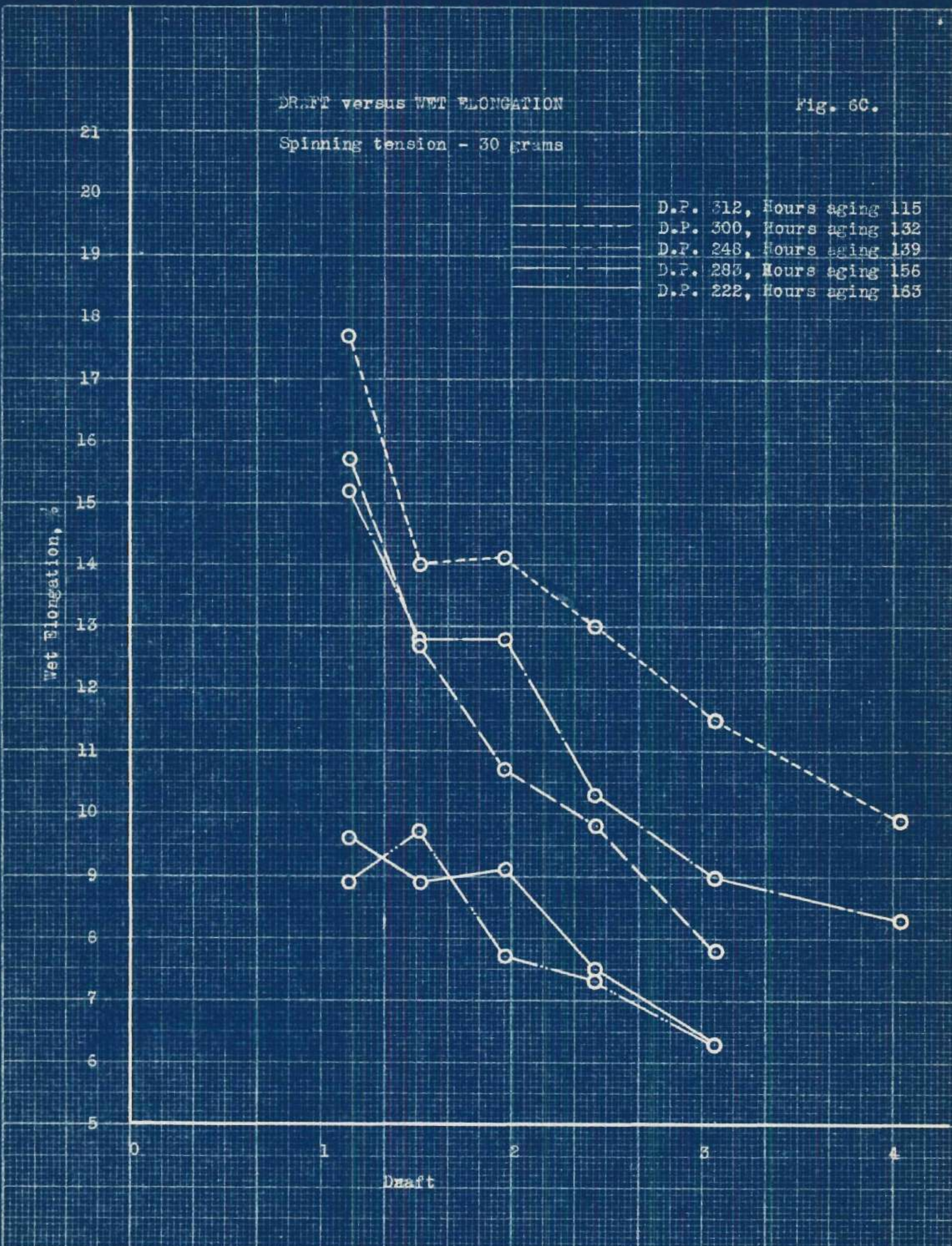
DRAFT versus DRY ELONGATION
Spinning tension - 30 grams
Fig. 5 B.
D.P. 365, Hours aging 67
D.P. 340, Hours aging 84
D.P. 351, Hours aging 92
D.P. 330, Hours aging 108
Dry Elongation, %
Draft
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
0
1
2
3
4


DRAFT versus DRY ELONGATION
Fig. 5C.
Spinning tension - 30 grams
D.P. 312, Hours aging 115
D.P. 300, Hours aging 132
D.P. 248, Hours aging 139
D.P. 283, Hours aging 156
D.P. 222, Hours aging 163
Dry Elongation, %
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
0
1
2
3
4
Draft

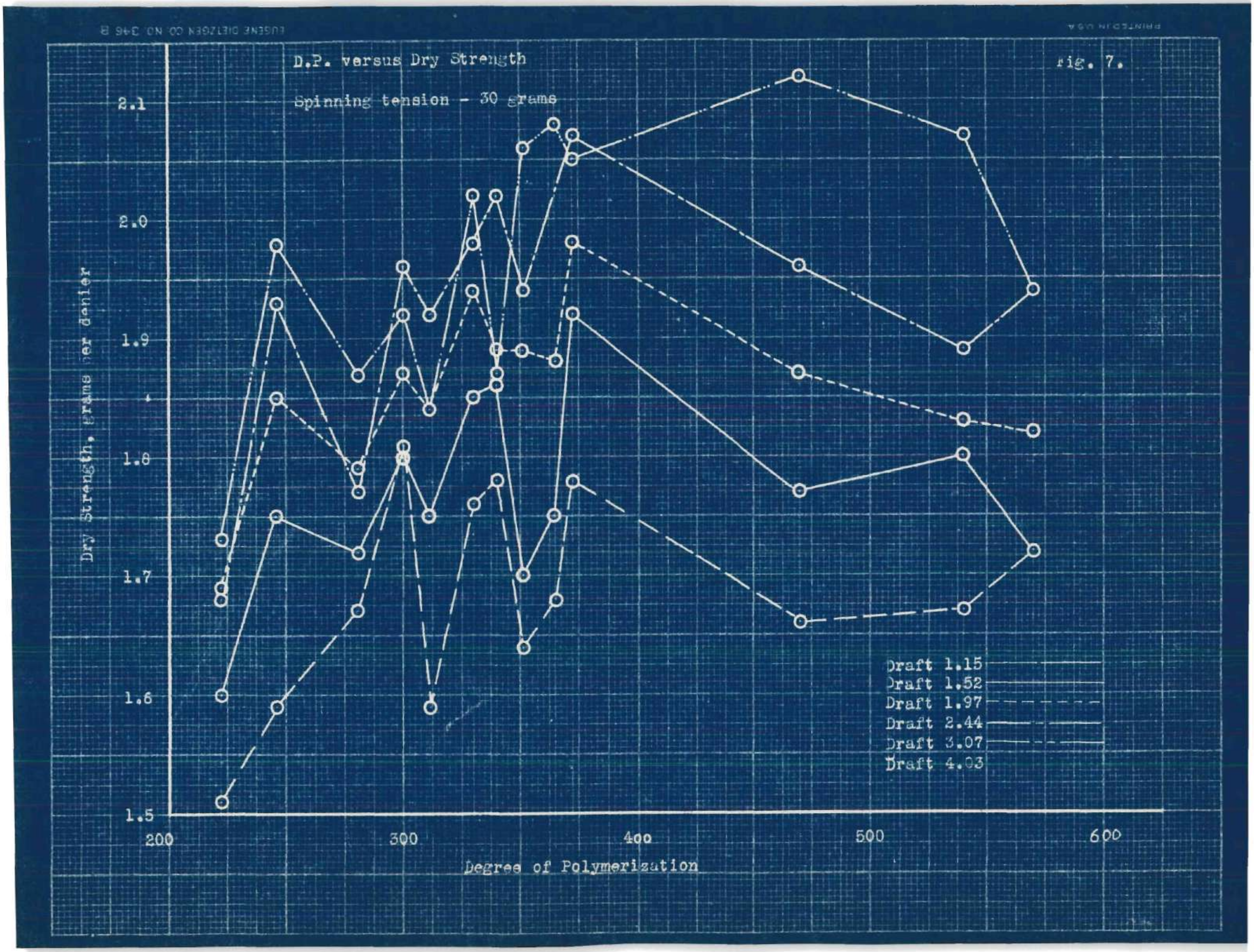


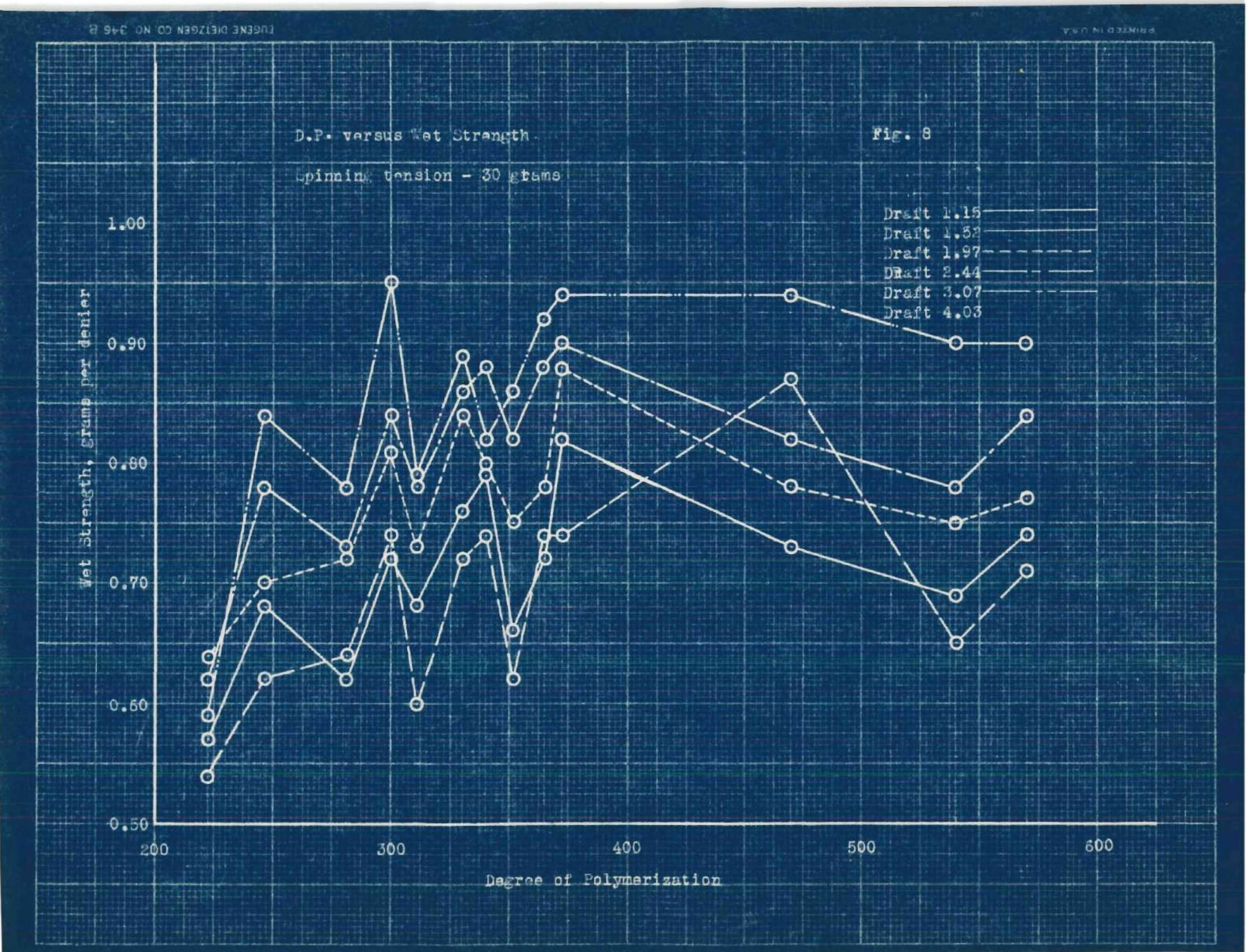
DRAFT versus WET ELONGATION
Fig. 6 A
Spinning tension - 30 grams
D.P. 540, Hours aging 19
D.P. 570, Hours aging 36
D.P. 470, Hours aging 43
D.P. 372, Hours aging 61
Wet Elongation, %
Draft
21
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
0
1
2
3
4

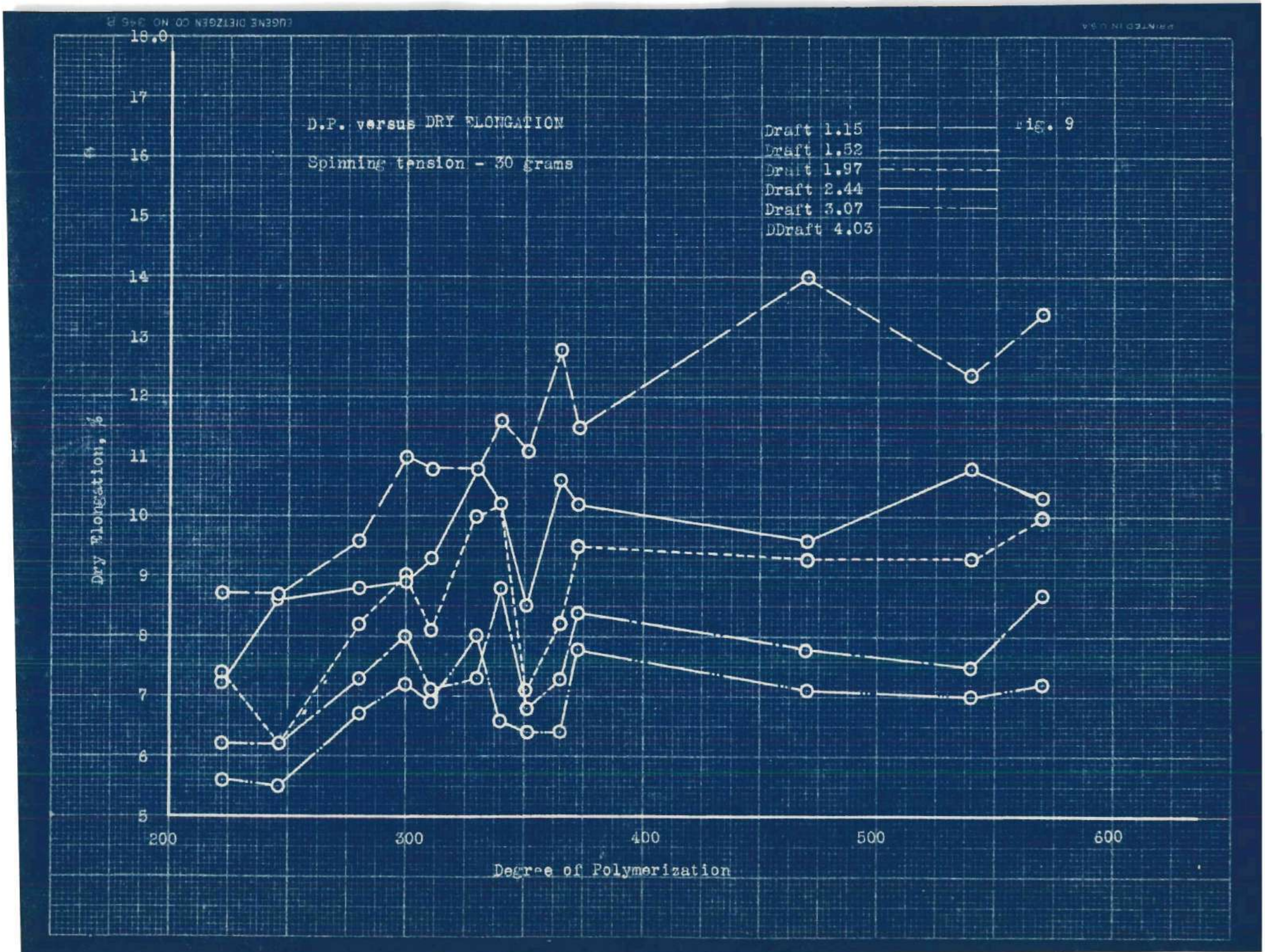

DRAFT versus WET ELONGATION
Fig. 6 B
Spinning tension - 30 grams
D.P. 365, Hours aging 67
D.P. 340, Hours aging 84
D.P. 351, Hours aging 92
D.P. 330, Hours aging 108
Wet elongation, %
Draft
21
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
0
1
2
3
4



DRAFT versus WET ELONGATION
Fig. 6C.
Spinning tension - 30 grams
D.P. 312, Hours aging 115
D.P. 300, Hours aging 132
D.P. 248, Hours aging 139
D.P. 283, Hours aging 156
D.P. 222, Hours aging 163
Wet Elongation, %
Draft
21
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
0
1
2
3
4


D.P. versus Dry Strength
Spinning tension - 30 grams
Fig. 7.
Dry Strength, grams per denier
2.1
2.0
1.9
1.8
1.7
1.6
1.5
200
300
400
500
600
Degree of Polymerization
Draft 1.15
Draft 1.52
Draft 1.97
Draft 2.44
Draft 3.07
Draft 4.03


D.P. versus Wet Strength
Fig. 8
Spinning tension - 30 grams
Draft 1.15
Draft 1.52
Draft 1.97
Draft 2.44
Draft 3.07
Draft 4.03
Wet Strength, grams per denier
1.00
0.90
0.80
0.70
0.60
0.50
200
300
400
500
600
Degree of Polymerization



D.P. versus DRY ELONGATION
Spinning tension - 30 grams
Fig. 9
Draft 1.15
Draft 1.52
Draft 1.97
Draft 2.44
Draft 3.07
DDraft 4.03
Dry Elongation, %
Degree of Polymerization
18.0
17
16
15
14
13
12
11
10
9
8
7
6
5
200
300
400
500
600
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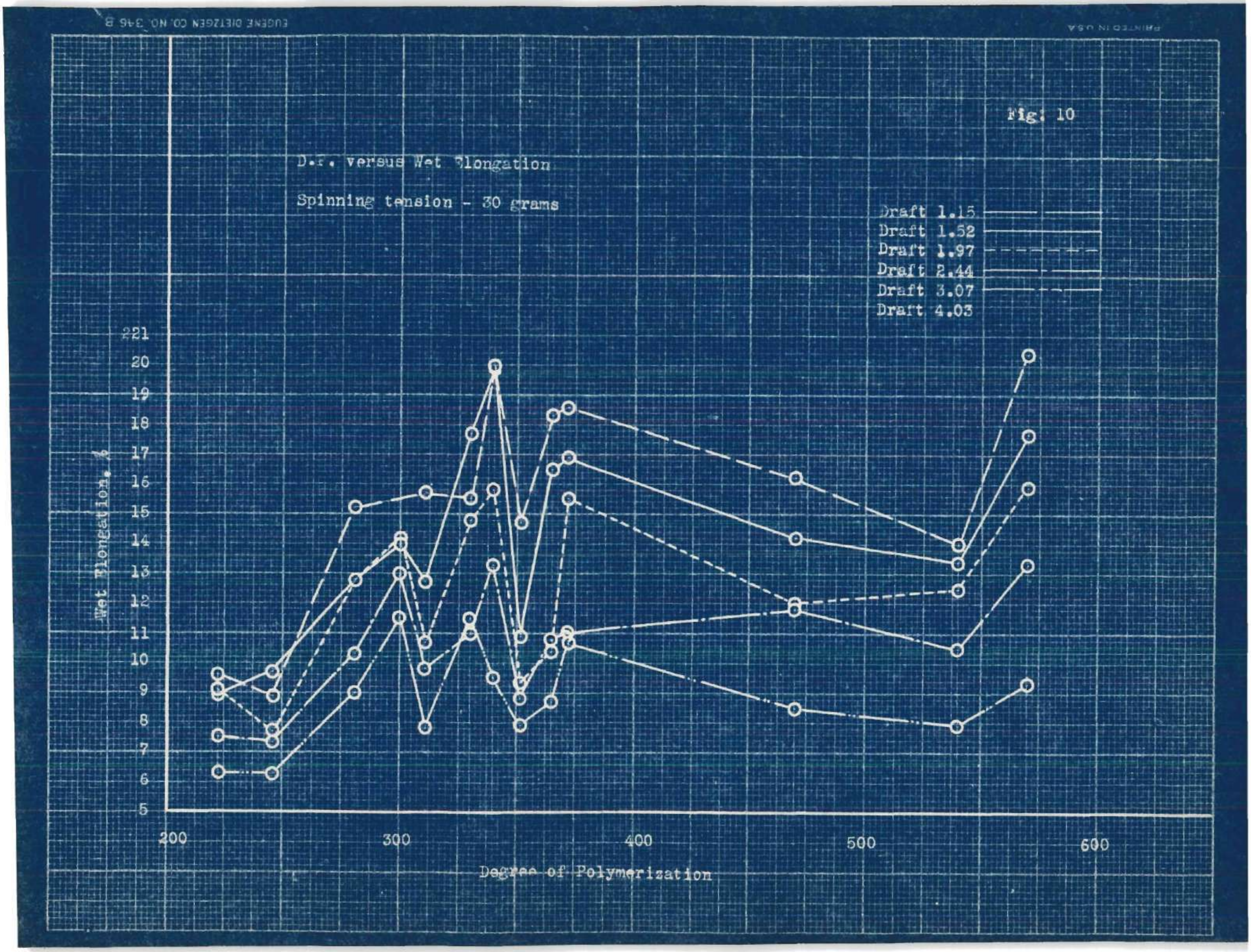

Fig. 10
D.P. versus Wet Elongation
Spinning tension - 30 grams
Draft 1.15
Draft 1.52
Draft 1.97
Draft 2.44
Draft 3.07
Draft 4.03
Wet Elongation, %
221
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
200
300
400
500
600
Degree of Polymerization

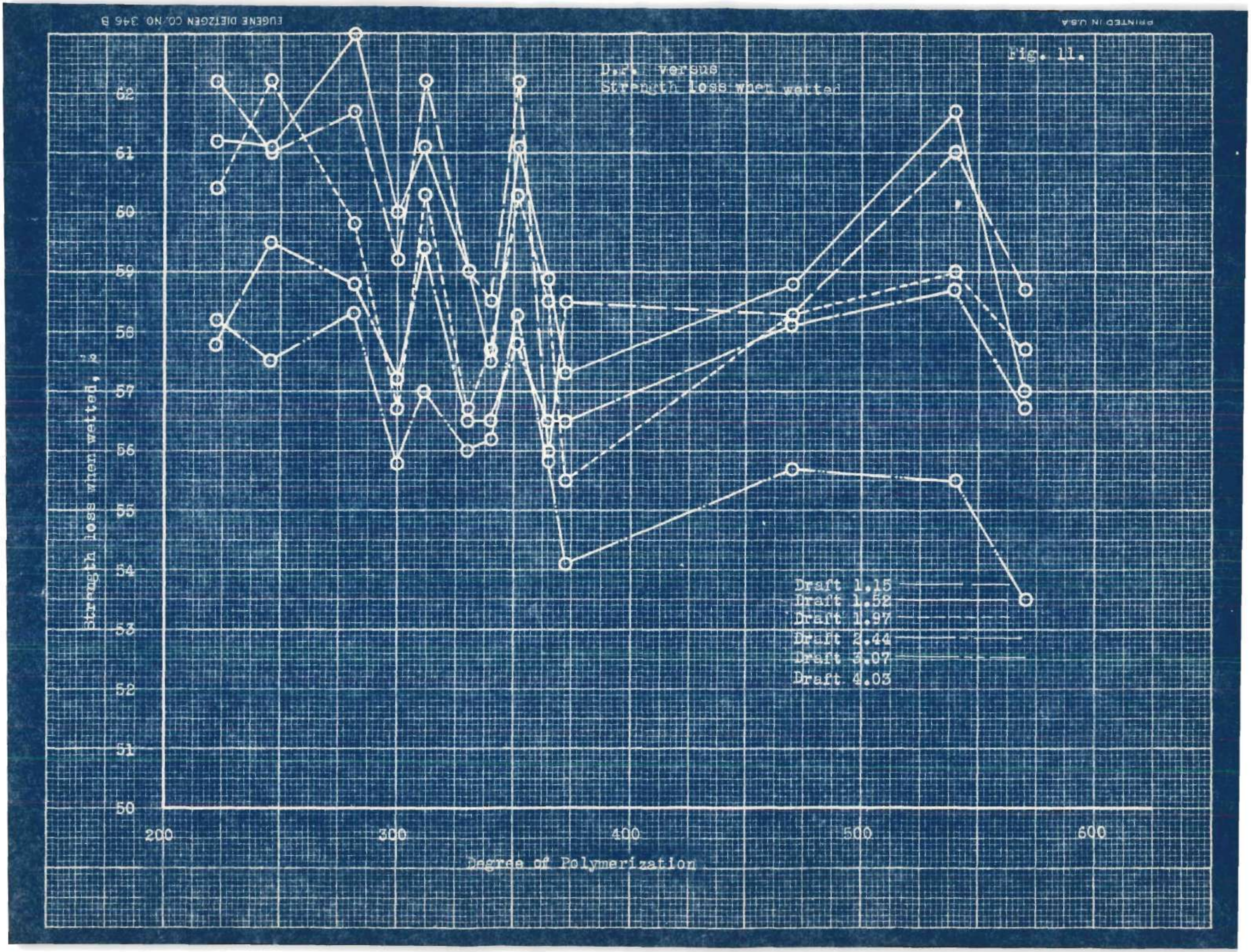

Fig. 11.
D.P. versus
Strength loss when wetted
Strength loss when wetted, %
62
61
60
59
58
57
56
55
54
53
52
51
50
200
300
400
500
600
Degree of Polymerization
Draft 1.15
Draft 1.52
Draft 1.97
Draft 2.44
Draft 3.07
Draft 4.03
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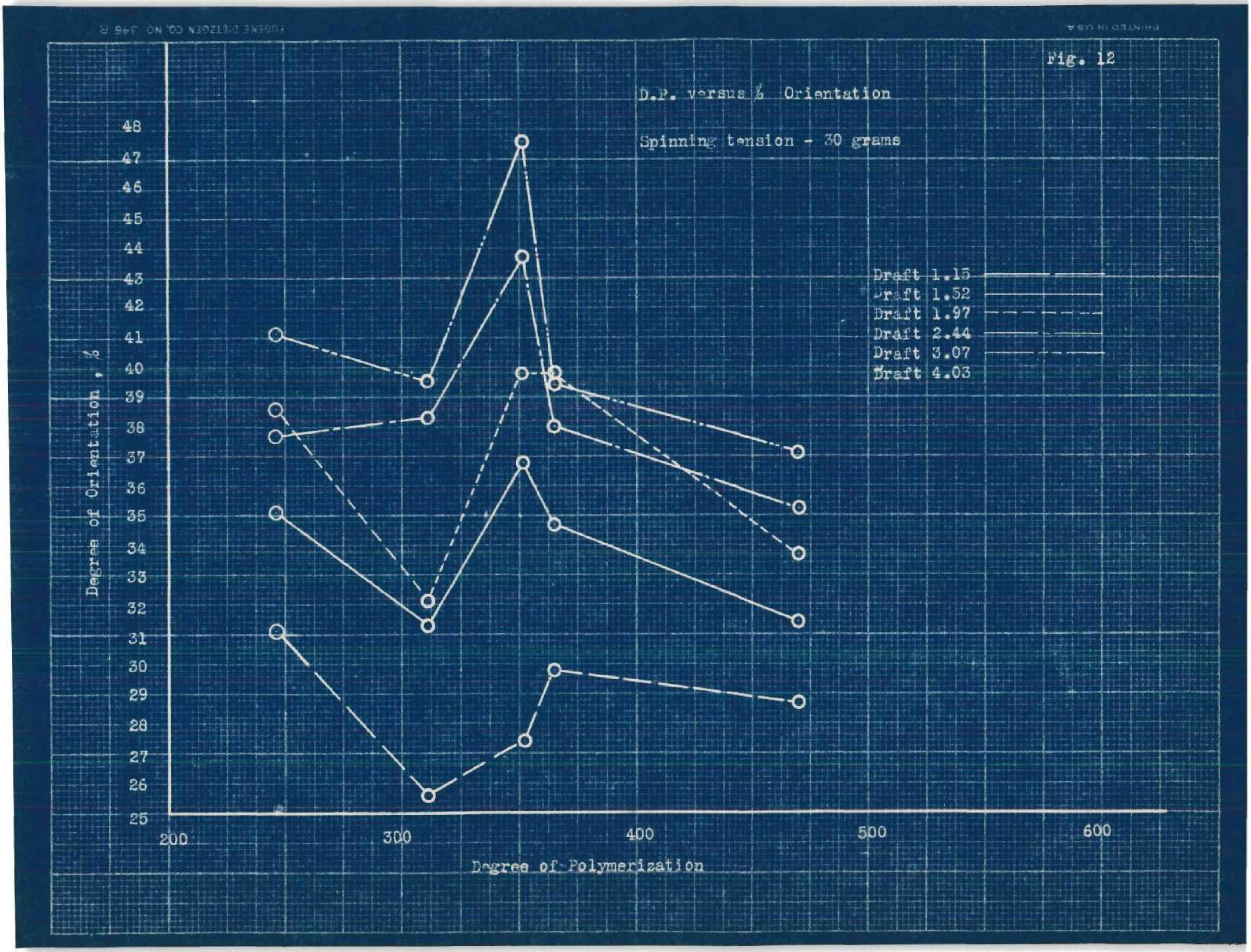

Fig. 12
D.P. versus % Orientation
Spinning tension - 30 grams
Draft 1.15
Draft 1.52
Draft 1.97
Draft 2.44
Draft 3.07
Draft 4.03
Degree of Orientation, %
48
47
46
45
44
43
42
41
40
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
200
300
400
500
600
Degree of Polymerization

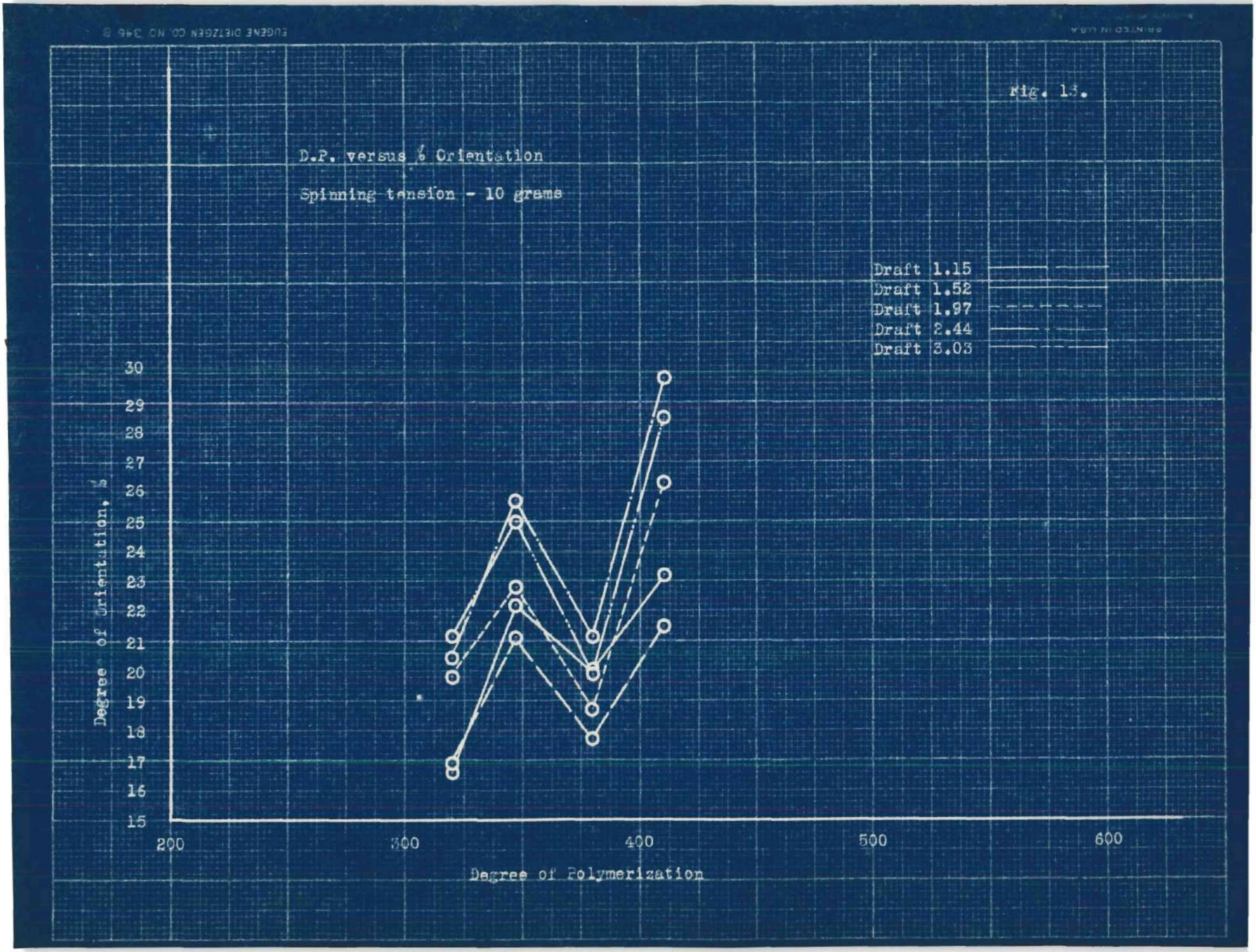
Fig. 13.
D.P. versus % Orientation
Spinning tension - 10 grams
Draft 1.15
Draft 1.52
Draft 1.97
Draft 2.44
Draft 3.03
Degree of Orientation, %
30
29
28
27
26
25
24
23
22
21
20
19
18
17
16
15
200
300
400
500
600
Degree of Polymerization
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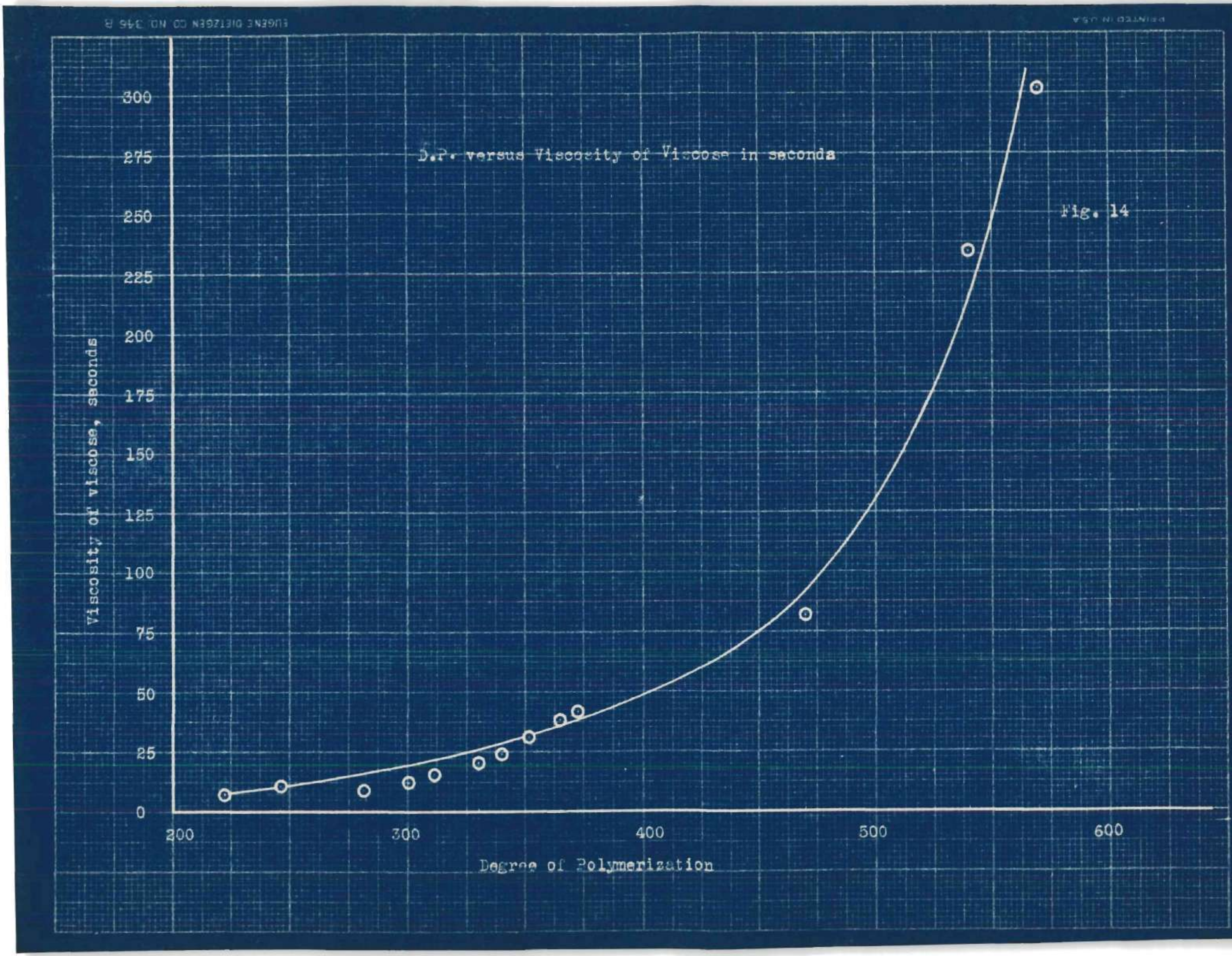
D.P. versus Viscosity of Viscose in seconds
Viscosity of viscose, seconds
Degree of Polymerization
0
25
50
75
100
125
150
175
200
225
250
275
300
200
300
400
500
600

Fig. 14

CONCLUSION

One observes from draft versus serimetric properties curves (Figs. 1 & 2) plotted from data on yarn spun without tension that the serimetric properties of the yarn do not vary appreciably with changes in spinning draft from 1 to 3. In the series of batches spun under a tension of 30 grams an increase in the values of spinning draft produces a very definite effect on the serimetric properties of the yarn. It may be concluded therefore, that the point of application of the draft or stretch is a very important factor in spinning. In spinning without tension the draft is applied mostly in the coagulation bath while the center of the filament is still liquid. The draft will tend to increase the "skin" effect without orienting the central portion of the filament. These results merely substantiate statements of the same facts by Preston (9).

The tensile strength increases at an even rate with each increase in draft for all batches spun under 30 grams tension (Figs. 3A, 3B, 3C, 4A, 4B, 4C). The percent elongation shows a corresponding regular decrease with each increase in draft (Figs. 5A, 5B, 5C, 6A, 6B, 6C). It may be concluded that the maximum draft which can be applied successfully will give a maximum tensile strength and a minimum percent elongation. However, the qualities of rayon with too small an elongation (under 10%) are liable to cause difficulties when treated by textile machinery. So the amount of draft which can be profitably

(9) See Ref. (1), p 2.

applied in the production of commercial rayon is definitely limited by this minimum value of percent elongation.

The value of degree of polymerization which gives the best tensile properties for yarn spun at 30 grams tension is found to be 371. This is for a batch with crumbs aged for $2\frac{1}{2}$ days at 18°C., the conditions used in making up regular standard batches of viscose (D. P. versus serimetric properties curves, Figs. 7, 8, 9 and 10). The serimetric properties decrease gradually with an increase in the degree of polymerization above the optimum. A decrease in degree of polymerization below the optimum gives a sharp decrease in the serimetric properties of the yarn.

There is a rather diversified opinion as to the effect of the degree of polymerization on the serimetric properties of rayon. One book on the chemistry of cellulose states (10), "It would appear that pronounced improvements could be made in rayon manufacture by increasing the degree of polymerization from 500, its present maximum, to 700-800, which represents degraded cellulose of good strength. This might be accomplished by using the highest quality of starting material, but the spinning solution would be highly viscous and perhaps difficult to spin." In direct contrast to the above suggestion for producing rayon of improved quality we have the statement (11) of the results of an

(10) J. T. Marsh & F. C. Wood, An Introduction to the Chemistry of Cellulose, p-366.
(11) See Ref. (5), p. 5.

investigation of the influence of aging on the spinning, which points out that the tensile strength and elongation increase with the time of aging (depolymerization) of the alkali cellulose up to a certain point, beyond which they remain constant or decrease slightly.

The results of this investigation definitely oppose the almost universally accepted idea that an increase in the degree of polymerization of rayon above the present maximum of 500 for commercial yarns would be accompanied by an increase in the tensile properties. An increase in the degree of polymerization above the optimum value of 371 was found to give a gradual decrease in the tensile properties of the yarn.

The percent strength loss when wetted is less for higher drafts and decreases with increasing degree of polymerization to a minimum at a D. P. of 372, beyond which there is a rise to a D. P. of 540 and again a decrease (Fig.11). A possible explanation for the variation of the strength loss of the yarn when wetted is that there are more interstices in a given volume with short chains than with long chains, thereby allowing more water to enter and produce swelling. As the chain length increases, the amount of space is decreased. The micelles are still well oriented. As the chain length increases, it may be more difficult to orient them, and it would be possible for more water to enter. On further increase of chain length, the effects of orientation and swelling decrease, and the greater lengths of the chains exert more influence.

The work on the determination of the percent orientation is incomplete. No definite conclusions can be drawn from the data available. The work on determination of percent orientation will be continued, and in addition will be checked by X-ray examination of the samples. It seems likely that this further work will show that a medium size chain length (D. P. 370) is more easily oriented than either a smaller or larger chain length.

It was unfortunate that only one size spinneret was available. If a set of spinnerets with varying size holes had been available it would have been possible to vary the draft over a much wider range, and to keep the denierage constant throughout the investigation. A constant denierage would be very desirable because a number of small related variations would be eliminated.

An interesting problem for future investigation is one which is now receiving considerable attention in the rayon industry, that of determining the optimum stage at which the draft or stretching should be applied.