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PIGMENT COLOR RESEARCH REPORTS

JANUARY - 1936

PIGMENT COLOR RESEARCH REPORTS

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PIGMENT COLOR RESEARCH REPORTLIGHT CHROME YELLOWS

SUBMITTED BY: *L.C. Horning*
APPROVED BY: *amb*

FILE: CHROME YELLOWS
DATE: JANUARY 1936

INTRODUCTION

In several recently developed ~~developed~~ processes, such as K-8021, an essential part of the process is heating the pigment in aqueous suspension. Laboratory work has been undertaken with the object of obtaining similar products without including the heating step, since such a modification may be necessary to avoid infringement of U.S. 2,023,928.

SUMMARY AND CONCLUSION

Several experiments were made striking at an elevated temperature and omitting the development after the strike, other conditions being similar to K-8021. The best product, that struck at 180°F, was slightly inferior to material made by the regular process. This study is being continued.

EXPERIMENTAL DETAILS

N.B. #481.

481-36. Study effect of high temperature strike in connection with U.S. patent #2,023,928.

In running the CS-7903 procedure at increased temperatures (110°F, 140°F, 160°F, 180°F) the cleanness and redness of the pigment increased in direct proportion to the increase in temperature.

481-38. Study treatment before washing on high and low temperature strikes; also heat one (struck at 80°F) to 160°F after the addition of soda ash.

It appears from these results that treatments before washing offer no merit towards obtaining the desired properties. D, which was heated to 160°F before washing, gave a clean product.

PIGMENT COLOR RESEARCH REPORT

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STUDY OF U.S. 2,023,928

SUBMITTED BY: *L.C. Hanning*
APPROVED BY: *amc*

FILE: CHROME YELLOW
DATE: JANUARY 1936

INTRODUCTION

An intensive study of U.S. patent 2,023,928 and of prior art references possibly related to it has been started. Some preliminary work indicates that the products disclosed in this patent are not greatly different from products obtained by some of the early processes recorded in the literature. This study is being continued.

SUMMARY AND CONCLUSION

A number of pigments were prepared by the processes described in U.S. 2,023,928. They were similar to a previous series, 389-89, run 7-23-34, following some of the examples in the corresponding British Patent 403,762. A search is being made of the patent and scientific literature for processes likely to give similar production. Several samples have been prepared not greatly different from the products disclosed in U.S. 2,023,928. The literature survey and laboratory study are being continued with the object of obtaining products equal to the disclosed pigments by methods old to the art.

EXPERIMENTAL DETAILS

N.B. #481.

481-37. Check examples 1, 2 and 6 of U.S. patent # 2,023,928.

These products although they were weaker and dirtier than those of the 389-89 series, were of the same type.

481-39. Study examples from the Chemical Journal article. See notebook for clearer details.

481-40. Continue study on 381-39B (example 3 from table I) by increasing the bichromate at the expense of the sulphate content.

Increasing the bichromate at the expense of the sodium sulphate improved the cleanness, greenness and strength of the pigment. Substituting sulphuric acid for sodium sulphate tends to decrease the strength even after increasing the bichromate from 76# to 82# per mol.

PIGMENT COLOR RESEARCH REPORT

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CANARY YELLOW

SUBMITTED BY:
APPROVED BY:

J. J. Allouew
H. Chalupski

FILE: CHROME YELLOWS
DATE: JANUARY 1936

INTRODUCTION

This titanyl-sulfate treated Y-180-D was given an initial trial in the plant, with subsequent manufacture of sufficient material for standardization as K-8153.

SUMMARY AND CONCLUSION

Six batches of this yellow were made during the month on formulae based on those used in the semi-works study. On testing in rubouts, all of this material was a little on the dark side of Y-180-D standard. On reading the dry rubouts, however, the plant material is markedly superior to the standard, indicating instability of the color, a property possessed in all probability by the standard. Light fastness of the plant material was much better than that of Y-180-D.

The strength of the color was found to be increased as the amount of salt in the strike was reduced. The reduction in salt, together with a long period of washing gives strong, green and clean tints.

Ink mill tests on two of the plant batches indicate that the color is superior to Y-180-D in resistance to lithographic breakdown, bleed and to livering. The inks were softer, and exhibited shorter mixing time and longer flow than the standard.

A laboratory mix of Y-403-D and K-8146, the new primrose and excelsior yellows, respectively, was made to Y-180-D depth and tested on the ink mill against Y-180-D standard. The mix was stronger than the standard and showed better stability. In ink tests it was superior to Y-180-D except for the fact that it livered badly.

A mix of four batches of plant material has been made and set up as K-8153. Against standard Y-180-D the mix is dark in masstone and somewhat strong, red and clean on extension with slightly improved light fastness.

EXPERIMENTAL DETAILS

For Experimental Details see N.B. #492 p. 45-49.

DATE	BATCH	FORMULATION	CONTROL	MASESTONE	UNDERSTONE	STRENGTH	TINT	LICEN STABILITY
1/2/36	47024	Check SW 7324 on a five mol charge	Y-180-D	vs gr. & dty	s dk, str & dty	vvs wk	vvs red & dty	better
1/7/36	47096	Check 47024 but reduce amount of salt by 50%	Y-180-D 47024	s dry vs dk & dty	s dk, str & dty vs str & dty	s str s str	vs red & dty dk	better =
1/14/36	47187	Check 47024 but reduce salt 25%	Y-180-D	s dk, vs brt	vs red	vs str	vs red	vs better
1/15/36	47208	Check 47024 but wash overnight	Y-180-D 47096	s dk, vs gr vs gr; vvs cl	vs red & str vs gr & wk	vs wk s wk	red & dty o k	better vs worse
1/24/36	47349	Check 47096 but wash overnight. Decrease T102 from 50# to 32#	Y-180-D	vs dk, s red	s red, vs str	str	gr & cl	
1/24/36	47350	Check 47096 but wash overnight	Y-180-D	vs dk & cl	s str, vs red	str	gr & cl	
1/30/36	16672	Mix of 47024 47096 47349 47350	Y-180-D	dk	s str & red	s str	s red, vs cl	s better

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PIGMENT COLOR RESEARCH REPORT

EXCELSIOR YELLOW

K-8146 (K-8021)

SUBMITTED BY:
APPROVED BY:

J. J. McQuinn
V. Chalupot

FILE: CHROME YELLOWS
DATE: JANUARY 1936.

INTRODUCTION

The study of this color was continued in the semi-works and plant during the month. A mix of plant material has been made and standardized under the K-8146.

code

SUMMARY AND CONCLUSIONS

Semi-Works -

In one batch the heating was omitted during the period of development, resulting in an off batch. In the two following batches the additional sodium sulfate was added before the development and in the strike, respectively. Both batches were dirty and had poor light fastness as compared to K-8146. It is evident, therefore that the additional sodium sulfate, used to increase the yield, must be added; just before washing the color.

Plant -

Two batches of this yellow were made during the month on the K-8021 code. The first was washed overnight. Its masstone was v.v.s. light but somewhat flat and changeable with satisfactory light fastness. The tint was a little strong and green and clean compared to K-8021. It was stronger and redder than Y-309-D with better light stability.

The second batch was made with an increase in the amount of titanyl sulfate. This batch was a little darker and greener than the first, with small improvement in light stability.

These two batches have been tested on the ink mill and are quite similar and superior to the other material made on this code.

A mix has been made of the first five plant batches of K-8021 and recoded as K-8146. Against K-8021 this new mix is a little light, green, and flat in masstone, v.s. strong, green and clean in tint, and is inferior in stability to light.

EXPERIMENTAL DETAILS

See N.B. 492 - pp. 15
25.

Date	Batch	Formulation	Control	Massstone Dty. & Gr.	Undertone V.V.S.dty.	Strength	Tint	Light Stability
1/6/36	SW-7589	Check SW-7400 but omit heating.	K-8021	s.flat & chromy	dirty	-	-	v.s.worse
1/8/36	SW-7599	check SW-7400 but add the additional sulfate after the soda ash (i.e. before dev.)	K-8021 K-8146	chg. s.gr.dty.flat & chg.	v.s.lt.& cl.	D.K.	s.gr.	s.worse
1/9/36	SW-7603	check SW-7384 but increase sod.sulfate in strike 5#	K-8021 K-8146	dirty,chromy dirty,sred & chromy.	red v.s.red & cl.	-	-	s. worse v.s.dty. "
1/2/36	47027	check 46716 but wash overnight.	K-8021 Y-309-D	v.v.s.lt. s.flat & chg. v.v.s.red & cl.	v.s.gr.& cl. v.s.red	v.s.str. s.str.	Gr.& Ch. v.s.red	O.k. better
1/8/36	47114	check 46716 but use 50# ThO ₂ instead of 32#	K-8021	v.s.dk. s.gr.& dty.	v.s.gr.& cl.	v.s.str.	gr.& cl.	v.v.s. better.
	16526	Mix of 46190 46667 46716 46820 47027	Y-309-D K-8021	s.gr.& v.s.dty. v.v.s.lt.& gr. v.s.flat	v.s.dty. v.s.str.& dty.	str. v.s.str.	O.k. v.s.gr.& cl.	better worse.

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW

SUBMITTED BY: *J.C. Hopping*
APPROVED BY: *amk*

FILE: CHROME YELLOWS
DATE: JANUARY 1936

INTRODUCTION

Satisfactory results having been obtained in the plant with a new primrose process laboratory work has been resumed with the object of improving its resistance to livering. An improved product with respect to livering has been obtained and a complete evaluation of this product is being made.

SUMMARY AND CONCLUSIONS

In the development of a new primrose yellow K 7888 it had been found that stannous chloride gave a product slightly green and slightly dirty in masstone with better light fastness. It was found that by using both stannous chloride and disodium hydrogen phosphate at appropriate points in the K-7888 process a product equal to the Imperial primrose C-38349 in masstone, strength, light fastness, and livering resistance was obtained. Further work is being postponed pending more complete evaluation of this sample.

EXPERIMENTAL DETAIL

N.B.#481

481-34. Study of phosphate yellow to compete with C-38349, using the following variation: Add sodium phosphate to 481-24A procedure (present semi-works procedure), and replace the bicarbonate with soda ash; strike at 70°F instead of at 110°F; decrease alum.

None of these variations aided in improving the 481-24A product. Striking at 70°F instead of at 110°F. improved the strength slightly.

481-35. Study use of stannous chloride; further reduction of alum on the 34C type; reduction of sodium chloride in the strike.

It appears from this experiment with the use of both stannous chloride and sodium acid phosphate added at an optimum point of the process material equal to C-38349 can be made in the laboratory. The use of stannous chloride greatly improved the light stability.

481-41. Study on the 35B (regular 24A with the addition of tin and a reduction of alum) the use of sodium sulphate added after the tin and carbonate; lower temperature strike.

In this series no marked effect was observed by the addition of sodium sulphate after the tin and carbonate. Striking at 70°F instead of 110°F resulted in a trace stronger and greener product.

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481-42. Continue further the use of sodium sulphate; also vary the amount of tin used.

None of these variations had any appreciable effect on the present laboratory control (35B). The entire series showed an improvement over C-38349 both in strength, tint and light fastness.

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW

Y-403-D (K-8124)

SUBMITTED BY:
APPROVED BY:

J. L. Allen

FILE: CHROME YELLOWS
DATE: JANUARY 1936.

INTRODUCTION

This yellow has been inactive in the plant since the setting up of the new standard.

A review of yields obtained in the research, semi-works, and plant study of this color showed them to be much lower than desired. As a consequence, some work was done in the semi-works to improve the process in this respect.

SUMMARY AND CONCLUSIONS

Two batches of this yellow were made in the semi-works to determine the effect on the yield, and also on the properties of the color, of completely precipitating the lead which has previously been rejected in the wash waters. In the first batch an excess of sodium sulfate was added to the color just before washing. The resulting color was very light, clean, green and somewhat strong against Y-403-D with equal light fastness. It was even greener than K-7888 and the yield was materially increased over that obtained in former batches.

The second batch was similar to standard formula except that the amounts of the striking components were increased to insure more nearly complete precipitation of the lead. A distinct lead excess was present, however, at the end of the strike. The yield of this batch was equal to that of the first, and it was v.s. redder and v.v.s. stronger. Against K-7888 the masstone was still a little greener.

It has been recommended that Y-305-D be replaced by Y-403-D; and at such time that production is resumed in the factory, the variable made in the first batch mentioned herein will be tried on a larger scale.

EXPERIMENTAL DETAILS

See N.B. 492, pp. 66.

Date	Batch SW	Formulation	Control	Massstone	Undertone	Strength	Tint	Light Stability
1/19/36	7610	Check SW-7299 but add 5# Na ₂ SO ₄ before flooding.	Y-403-D K-7888	Gr. & Cl. V.S.Gr.	Lt.Gr. & Cl. -	V.S.Str. V.V.S.Str.	Gr. & Cl. V.S.Gr. & Cl.	- V.V.S. worse.
1/15/36	7616	Check SW-7299 but increase amounts of precipitating agents 3%.	Y-403-D K-7888 K-7610	Gr. & Cl. V.S.Gr. V.S.Red	Lt.Gr. & Cl. - V.V.S.Gr. & dirty	S.Str. V.S.Str. V.V.S.Str.	Gr. & Cl. V.S.Gr. & Cl. V.V.S. dirty	= V.V.S.Worse V.V.S.Worse

YIELD RESUME.

Semi-Works Yields

	Lump	Grind
SW-7610	152	150
SW-7616	152	152

Average of Laboratory - 310#/mol
 Average of previous S.W. work 265#/mol
 Average of plant work 292#/mol
 Average of improved S.W. process 304#/mol.

36-7

PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOWS OF IMPROVED STRENGTHSUBMITTED BY: *S.C. Manning*
APPROVED BY: *am*FILE: CHROME YELLOWS
DATE: JANUARY - 1936INTRODUCTION:

The preparation of Medium Yellows of high strength by striking in very dilute solution is difficult to carry out on a large scale because of the high volumes involved. It has been proposed to avoid this by developing a continuous process in which the lead nitrate and sodium chromate solutions are added simultaneously to a mixing tube and the pigment slurry is then pumped to a filter. The results indicate that products prepared by this proposed process are about equal in strength to those prepared by the normal precipitation process.

SUMMARY & CONCLUSIONS:

Several samples of Chrome Yellow were prepared by the simultaneous addition of .1M solution of lead nitrate and sodium chromate to a mixing tube both with and without the simultaneous addition of water to the mixing tube to obtain more efficient mixing. Without the addition of water the product obtained was approximately equal to that resulting from a reverse strike. The addition of water to the mixing tube produced a dirty mass and a decrease in light fastness but practically no change in strength.

EXPERIMENTAL DETAILS:

See Notebook 452

Experiment 87 - Simultaneous strike in a mixing tube.

Results - Preliminary results indicate the process can be used in place of the usual striking procedure.

PIGMENT COLOR RESEARCH REPORT

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MEDIUM YELLOWS OF IMPROVED STRENGTH

SUBMITTED BY: *L.C. Homing*
APPROVED BY: *and*

FILE: CHROME YELLOWS
DATE: JANUARY 1936

INTRODUCTION

The study of medium yellows of improved strength has been continued from the past month. The effect of a number of variables has been determined and the process seems ready for Semi-Works study.

SUMMARY AND CONCLUSION

The effect of the more important variables in the preparation of a strong medium yellow was discussed in the December report. A few additional variables were studied during the past month. Small amounts of barium chloride added after the strike increase the strength while larger amounts decrease the strength. Heating to 180°F after the strike results in a product equal to Y-328-D in cleanness of masstone and only slightly weaker than the pigment finished cold. Decreasing the volumes to equal plant Y-359-D resulted in only a slight decrease in strength. Several experiments using a Y tube mixing device resulted in products similar to those obtained by the regular procedure. The process seems ready for semi-works study.

EXPERIMENTAL DETAILS

N.B.# 452 and 497.

452-92. Study an increase of chromate and volumes on 452-84B procedure.

No marked effect was obtained by increasing the chromate two grams per mol. Increasing the volume of the chrome solution had no appreciable effect, however, a very, very slight dirtiness was observed.

452-94. Study a decrease in temperature; the addition of Barium chloride after the strike; the effect of heating after the strike.

A large decrease in temperature produces dirtiness, likewise the addition of Barium chloride produced dirtiness of masstone.

Heating after the strike to 180°F resulted in a red masstone with a marked improvement in light fastness.

452-95. Compare the effect of increase of volume against standard plant volume.

In this experiment increasing the volume of the lead nitrate resulted in an increase in dirtiness of masstone. Cutting

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the volume of the chrome solution gave a product dirty and red in masstone versus control, but somewhat stronger than B and C made with higher chromate volumes.

497-14. Study effect of adding Barium chloride and sodium chloride when added to higher strength yellow procedures. Using increased amount of Barium chloride tends to increase the greenness of the masstone and tint. Following the strike with sodium chloride had no marked effect tinctorially on the pigment obtained.

PIGMENT COLOR RESEARCH REPORTMEDIUM YELLOW Y -299- D

SUBMITTED BY:
APPROVED BY:

J. J. Hill
L. Chalupski

FILE: CHROME YELLOWS
FILE: JANUARY 1936

INTRODUCTION

The development section began a study of Y-299-D in this plant during the month to obtain a formulation which will give satisfactory material, especially from the viewpoint of light fastness.

SUMMARY AND CONCLUSIONS

Trouble has been encountered in the manufacture of this color for some time, the main difficulty lying in the inability to produce material of satisfactory light fastness.

Two lead nitrate formulae have been given a trial in the factory. The first was a check on the K-7031 formula (an old plant formula for Y-299-D) a mixture of sodium chromate and caustic soda being run in two portions into a lead nitrate solution. It is believed that this process should be satisfactory when pure lead nitrate is used. This batch was dark and red in masstone against the temporary standard (15115) with a strong green tint and slightly better light fastness. Compared to the real Y-299-D standard (13645) the temporary one is vs green, silky and changeable, vs clean and green, vvs weak, and vvs green with slightly worse light stability.

The second plant batch was made on a formula similar to that for Y-359-D except for the addition of caustic after strike instead of muriatic acid in the strike. The masstone of this batch was slightly light and flat and a little red against Lot 15115. The tint was strong, green and clean with slightly better fastness to light.

The study is being continued as production permits.

EXPERIMENTAL DETAILS

N.B. #448 p. 106.

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DATE	BATCH	FORMULATION	CONTROL	MASS TONE	UNDER TONE	SER.	TINT	LIGHT STABILITY
1/13/36	47161	Check K-7031 ie. Sod.Chromate and caustic soda struck in 2 portions into lead nitrate	Lot 15115	Dk.red	red,s str.	str.	gr	s better
1/15/36	47207	Lead nitrate struck into Sod. Chromate and caustic soda added (Check SW 7395)	Lot 15115	s lt & flat vvs & cl. red	s gr. vs gr. & cl.	str.	gr. & s better cl.	
		Lot 15115	Y-299-D Lot13645	vs gr, silky & change-able	vs cl. & gr.	vvs wk.	vvs gr.	s worse

PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW

Y-359-D

SUBMITTED BY:
APPROVED BY:

J. J. Allmon
J. Chalupski

FILE: CHROME YELLOWS
DATE: JANUARY 1936

INTRODUCTION

Production of this yellow was continued during the month in the plant. The development section tried several changes in formulation to obtain red, weak tint material for use in Y-366-D.

SUMMARY AND CONCLUSIONS

Seven batches were struck during January. On the first three, attempts were made to obtain material with red, weak tints by brushout test for Y-366-D mixes. In the case of the first, the strike was made at a higher temperature than ordinarily with a large increase in the amount of muriatic acid added to the chromate. It was a little dark and dirty in masstone and vva strong, vs green and clean in tint. Light fastness of all batches was slightly better than standard. The second batch was struck at the usual temperature with sodium chloride and muriatic acid added to the chromate. This material was a little redder and weaker than the first. Compared to standard the tint was equal in strength and vs red. The third batch was a check on the second except for an increase in temperature. The yellow was similar except for being a little dirtier.

A brushout on the first batch made (47028) showed it to be red against Y-366-D and strong, green and clean on extension.

The rest of the month's production was made on standard formula and ran a little flat in masstone, and strong, green and clean in tint. Light fastness was superior to that of standard.

EXPERIMENTAL DETAILS

See N. B. # 448 p. 76.

DATE	BATCH	FORMULATION	MASSSTONE	UNDERTONE	STRENGTH	TINT	LICHEP STABILITY
1/2/36	47028	35# HCl added to chromate 1 hour strike at 115°F	vvs dk, vs dty	vs gr & cl	vvs str	vs gr & cl	s better
1/7/36	47095	300# NaCl & 25# HCl added to chromate. Struck at 90°F	vvs red, dty	vs red	O.K.	vs red	s better
1/9/36	47138	Check 47095 but strike at 115°F	vs dty vvs dk, & red	vs gr & cl	O.K.	dty, s red	s better
1/16/36	47229	Std. formula - 6# HCl in chromate. 1 hour strike at 90°F	s dty, flat vs lt & gr	vs red	str	gr & cl	s better
1/20/36	47267 47268	Check 47229	vs lt & flat	vs gr & cl	str	gr & cl	better
1/26/36	47376	Check 47229	vs gr & dty	vs gr & cl	str	gr & cl	s better

PIGMENT COLOR RESEARCH REPORT

36-10

M

DARK MEDIUM YELLOW

SUBMITTED BY: *L. C. Hornung*
APPROVED BY: *W. E.*

FILE: CHROME YELLOWS
DATE: JANUARY 1936

INTRODUCTION

The dark medium yellows as represented by Y-216-D have not received laboratory attention for some time. A brief study is being made with the object of developing a product similar to Y-216-D but of improved strength and light fastness.

SUMMARY AND CONCLUSION

A number of strikes have been made in which three ingredients, lead nitrate, sodium chromate, and caustic have been mixed in different ways. It seems desirable in all cases to strike at 130°F or above and to heat to about 150°F to 180°F after the strike. The best results were obtained by adding sodium chromate plus caustic to lead nitrate and by simultaneously striking lead nitrate and a sodium chromate caustic mixture into water. The darker or redder shades are obtained by increasing the amount of caustic used. In the simultaneous strike a considerable excess of sodium chromate seems necessary to obtain maximum strength.

EXPERIMENTAL DETAILS

N.B. #497.

497-3. To obtain an improved dark medium yellow comparable to Y-216-D.

None of the variations studied in this experiment offered a lead towards improving the dark medium yellow Y-216-D.

497-5. Continue variations on 497-3B (simultaneous strike).

Nothing of any value was obtained from this series. B, the check on 497-3B gave a light, clean, strong pigment which was much too light to match Y-216-D. A and C made with an increase of Caustic soda in the one chrome solution (strike solution) were too red and dirty.

497-8. Study effects of striking simultaneously into varying amounts of chromate; add salt to chromate.

None of the variations made in chromate pertaining to the material in the strike jar showed an improvement. The addition of salt to the chromate gave a fair lead; this will be checked.

497-9. Study a reduction in chromate and caustic; also effect of adding molybdate to one chrome (strike solution) solution; study striking at 130°F.

In this series C made at 130°F, without any molybdate gave the most promising lead, being the greenest and cleanest of the series. The addition of molybdate resulted in a very red yellow.

497-19. Study a reduction of sodium hydroxide and the omission of sodium chloride; also a further increase of strike temperature. Study these same quantities struck in the regular striking manner (chromate, etc. - into the lead solution).

B made without salt resulted in a very red yellow. Increasing the strike temperature to 150°F resulted in a somewhat light product accompanied by an increase in strength. It appears from this series that the regular striking method gives the better product; this being illustrated by C.

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PIGMENT COLOR RESEARCH REPORT

CHROME ORANGE YO-38-D

SUBMITTED BY: *H.C. Mehring S.C. Worring*
APPROVED BY: *[Signature]*

FILE: CHROME ORANGE
DATE: JANUARY 1936

INTRODUCTION

The study of YO-38-D has been continued from the past month. The experimental work has consisted chiefly of a continuation of the study of the effect of possible impurities in the raw materials used and a study of the effect of various treating agents. This work is being continued.

SUMMARY AND CONCLUSIONS

Experimental work on the effect of various possible impurities was continued from the past month. The conclusion of the December report that impurities seem a minor factor still appears correct. The use of sodium acetate from crude acetic acid resulted in no improvement.

Experimental work on the effect of agents, such as gardinol, oleic acid, citric acid, H.T.P. alcohol, amyl alcohol, ammonium chloride and strontium nitrate gave no indication that these agents were of any value. A number of similar products will be tried during the next month.

Lead liquor from plant water and distilled water gave similar results.

Attempts to prepare a bright YO-38-D by the use of lime were unsuccessful. All products obtained seemed poor tinctorially. A few experiments on the use of lead nitrate gave negative results.

The effect of treating a washed slurry of YO-38-D with alum and acid was determined. The results are similar to those obtained several years ago. Either no effect is noted with small amounts of alum or acid or a dark yellow masstone and high finish results. The study of the effect of acid is being continued.

EXPERIMENTAL DETAILS

N.B. #486.

486-63. To study the effect of impurities from water. Lead liquors were made in the laboratory using the regular plant water and distilled water.

The lead liquor made using distilled water gave a product which is a little brighter than the other members of this series. This difference, however, may be due chiefly to experimental conditions and not to the difference in the type of water used.

486-65. Check results obtained in series 486-63.

It is quite apparent from the results obtained in the series that lead liquor made using distilled water has no greater tendency to produce brighter pigments than those made using tap and plant water.

486-66. Study the effect of dilute strike and of a long stirring period; also add bichromate during development to increase the CrO_4 excess.

No marked improvement was observed from the four hour stirring period after the development. Dilute volumes for the lead liquor resulted in light products. Adding bichromate during development did not improve the pigment.

486-67. Study the effect of the use of white lead, and the use of stannic chloride in the lead liquor.

There was a brightness observed in the pigment made using white lead that was considerably outstanding versus the control made from lead liquor. However, the product was very very dark and weak versus YO-38-D standard. It appears an increase in acid (in the white lead base) would tend to produce a lighter pigment. What effect this will have on the brightness cannot be estimated.

486-68. Study long stirring periods as in 66c, also study use of acetic acid in a dilute strike.

There is a slight indication that digesting for four hours at 205°F after development gives a product very very slightly cleaner than the control. Acetic acid in the bichromate resulted in a brighter and cleaner product than the control of this series. None were near the standard in brightness. These leads will be checked.

486-69. Study further the use of white lead.

Increasing the amount of lead nitrate used with the white lead, here, indicated a very very slight improvement in brightness.

486-70. Study the effect of H.T.P. alcohol, amyl alcohol and "Duponol" when added to the lead liquor.

Here, a noticeable improvement in brightness was obtained from using amyl alcohol in the lead liquor; this will be given further study. H.T.P. alcohol and "Duponol" added to the lead liquor gave dirty pigments.

486-71. Study concentrated strikes, with and without acetic acid, stirred for a long period at high temperatures. Here, a slight inconsistency was observed in the effect of holding for four hours at 205°F. A-1 and A-2 are very close in masstone, while B-1 and C-1 are somewhat cleaner than B-2 and C-2. None of the variations have shown any marked improvement over the control.

486-72. Study further the use of amyl alcohol in the strike and lead liquor; study a four hour development period on the 67A procedure.

In this experiment the four hour development period gave a very slight improvement in brightness. Here the use of amyl alcohol did not check the marked improvement obtained in the 486-69C experiment.

486-73. Study effect of purifying the lead liquor with feathered lead before making an orange; also study the use of amyl alcohol, and Ammonium chloride.

Here, the purified lead liquor gave a product considerably darker than the control, but no marked improvement in brightness. Following the strike with Ammonium chloride resulted in a light pigment, but with no improvement in brightness. Amyl alcohol added after the strike had no marked effect.

486-74. Study consistency of the present laboratory standard procedure; also add gloss white to the orange before developing.

The addition of gloss white resulted in a very high finish but did not add to the brightness and cleanness of the pigment. Very poor consistency was obtained.

486-75. Study again the use of purified lead liquor; also try using crude acetic acid in the lead liquor; study the effect from washing the pigment before development.

Here again the purified lead liquor (purified by passing over feathered lead) gave a dark weak product. Crude acetic acid resulted in a very dark weak product. Washing the pigment before developing resulted in a light dirty pigment.

486-76. Study further the process of washing the struck pigment before developing; add bichromate to the washed pigment before developing, also try the use of calcium chloride in the base.

The addition of bichromate to the washed pigment in its unbleached stage resulted in a dull dirty product. Treating the base with Calcium chloride gave a light masstone, but no improvement in brightness was obtained.

486-77. Study Calcium oxide procedures used in 1918.

None of the calcium oxide procedures gave a very deep orange. Another run will be made to study further conditions necessary to obtain greater depth from these methods.

486-78. Study the use of calcium oxide when added to Bicarbonate and struck at a boil; also study the effect of removing the carbon dioxide by means of a suction.

Heating the calcium oxide treated pigments tends to produce depth and strength. The addition of the calcium oxide to the lead liquor at very high temperatures gave a much darker pigment than was obtained in the 77 series. Developing by means of a vacuum did not give very satisfactory results.

486-79. Study striking at 140°F; also simultaneously striking sodium bicarbonate and sodium dichromate.

Simultaneous strikes gave very yellow and strong products. Striking at 140°F gave a very light orange.

486-80. Study the problem of development on the 67A procedure, (the regular laboratory standard procedure).

Here it appears that boiling until the foam breaks helps to brighten the product. Treating after the development period does not seem to be very effective. Striking at 140°F, and then bringing to a boil to develop the orange resulted in a light orange.

486-81. Study the part of the procedure pertaining to time of development and striking.

Striking in thirty minutes (81C) resulted in the brightest pigment of the series; this might only be due to experimental differences. Preparing the white base at a boil and boiling for 30 minutes did not have any effect on brightness. Stirring for 30 minutes at 200°F had no favorable effect on the brightness.

486-82. Check 81C, but use the regular procedure for preparing the lead-base; also study the effect of manganese salts.

Boiling the lead base, otherwise like 81C resulted in a dull dirty product.

Using manganese salts tends to produce very very brown pigments.

486-83. Study the use of Kadox, citric acid and strontium nitrate.

The addition of Kadox has little or no effect on the pigment. Citric acid used after the slurry was heated to 212°F gave no improvement in brightness.

Here strontium nitrate appears to give a slightly lighter and cleaner product. This may be only an experimental difference, therefore will be checked.

486-84. Study further the use of strontium nitrate; also molybdate.

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The position for adding the strontium nitrate affected the depth but had little effect on the brightness. Molybdate added at a boil during the development period gave a product vvs brighter than the control.

486-86. Study various K procedures in the laboratory (used in plant to make YO-38-D).

Two K procedures of YO-38-D made in the laboratory (K 5946 and K7536) gave dull products versus the standard material when rubbed out.

486-87. Study effects from Y.P.S. method of purifying the lead liquor; also study the use of a sulphuric acid treatment after washing the pigment four waters.

Using lead liquor purified by the Y.P.S. method gave a slightly darker pigment than the control but gave no improvement in neither brightness and cleanness of masstone.

Treating the washed pigment with sulphuric acid gave a brighter pigment with a yellowish cast, and a high finish undertone.

486-88. Study sulphuric acid, alum and oleic acid treatments on the washed pigment.

The use of oleic acid tends to increase the dirtiness of the pigment.

Alum gave a high finish, but no improvement in cleanness of masstone. Sulphuric acid treated pigments were quite yellow in undertone, with a very undesirable finish. No improvement was obtained from this series.

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PIGMENT COLOR RESEARCH REPORT

MOLYBDATE CHROME ORANGE

SUBMITTED BY: *J. C. Homing*
APPROVED BY: *all*

FILE: MOLYBDATE CHROME ORANGE
DATE: JANUARY 1936

INTRODUCTION

An intensive study of this color was undertaken in the laboratory during the past month. This work can be divided into two parts, first a study of the K-8074 process to determine the cause of instability on large scale operation, and second the development of a process giving a product from 20% to 40% stronger than the Rex Orange.

Laboratory work to date indicates that greater variations in tinctorial properties result from slight variations in operating conditions than from slight variations in the quantities of chemicals used.

A new laboratory process has been developed giving a product similar to the Red Orange C 38041 but more than 20% stronger. This study is being continued.

SUMMARY AND CONCLUSION

Semi-Works difficulties with the K 8074 process made it necessary for the laboratory to resume work on this process. Lead nitrate liquor and bichrome liquor were found equal to the corresponding crystals. Slight variations in molybdate had little effect but large variations influenced the shade and strength. There appears to be an optimum amount above which the tint is weak and yellow and below which the tint is weak and blue. Occasional strikes have come out flat and slightly weak versus a corresponding control indicating variations in operating conditions may be a more important factor in brightness of masstone than small variations in the quantities of ingredients used.

A new laboratory process has been obtained giving a product similar to the Red Orange but considerably stronger. The process involves the addition of a mixture of sodium chromate, sodium molybdate, and sodium sulphate to lead nitrate. Laboratory strikes are in progress to determine the optimum ratio; the optimum precipitant balance, and the quantity of nitric acid required after the strike to obtain conversion to the tetragonal form.

EXPERIMENTAL DETAILS

N.B. #452 and #497.

~~452-95~~. Compare laboratory molybdate with plant material and compare laboratory process with the semi-works procedure.

The plant molybdate gave a duller and very, very slightly weaker product than the laboratory material. Using plant lead liquor

in the K 8074 procedure gave a much stronger and yellower tint than using Crystals. All were worse in light fastness than K 8074. The Semi-Works procedure gave a stronger product than the laboratory formula. 25

497-1. Study variation in molybdate. In this experiment decreasing the amount of molybdate salts resulted in darker masstones, and bluer tints. However, in some cases no effect in strength was observed; the same was also true with brightness.

497-2. Study a more alkaline strike, making variations in acid after the strike, and study the use of sodium sulphate.

Changing from the use of Sodium Bichromate to Sodium Chromate gave a duller light product greatly improved in strength. A slight variation (in the chromate procedure) of nitric acid, and sodium sulphate had no appreciable effect on the products obtained.

497-6. In this experiment stirring overnight (chromate procedure) and heating to 160°F in 30' had a deleterious effect on the pigment. "D", stirred two hours after the strike, was the brightest of the series.

497-7. Continuation of 497-6 D. Study variation of chromate, sulphate, molybdate, ratio; study use of salt in the strike.

The control was a very poor check on 497-6D. A reduction in molybdate and sulphate resulted in a very poor orange. Salt used in the bichromate resulted in a light milky orange. None were very promising.

497-11. Study higher precipitant balance; neutral strike; low molybdate on the bichromate procedure.

Increasing the precipitant balance favored an increase in depth. Replacing the sulphuric acid with sodium sulphate gave a large increase in depth. However, an increase of acid together with a cut in molybdate gave a pigment somewhat lighter than the control - but not as bright.

497-12. Continuation of 497-6D. Balance strike to determine optimum chromate, sulphate and molybdate ratio to yield maximum strength and brightness.

None of the combinations of chromate-sulphate and molybdate studied gave an improvement over the control. All were on the red (top) weak and blue (tint) side of the control.

497-13. Study on 497-2C the stability of the formula, using a slight reduction in molybdate; lower precipitants.

Lowering the precipitant balance, here, resulted in a very, very slightly weaker and bluer tint.

Stirring 30 minutes before adding the alum had no marked effect on the pigment. Decreasing the molybdate from 10.5 to 10 gave a pigment very slightly stronger than the control.

PIGMENT COLOR RESEARCH REPORT

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MOLYBDATED ORANGE K-8074

SUBMITTED BY: *H. Rotherham*
APPROVED BY: *V. Chalupski*

FILE: CHROME ORANGE
DATE: JANUARY 1936

INTRODUCTION

A study of molybdated orange, (based on the formula for SW 7540), was made in the laboratory, to determine the factors producing light, flat, and weak semi-works material. Later plant and semi-works runs have been quite close, tinctorially, to the K number (8074) using some of the information developed in this laboratory study.

SUMMARY AND CONCLUSION

This series of experiments included the study of a number of variables. Factors such as alkalinity of strike, variation in amount of sodium molybdate, delayed addition of treating agents, and variation in the amount of sodium bichromate were studied. No single experiment gave results directly comparable to the previous semi-works batches. However the work done indicates that a combination of several of the variables studied would produce a light, flat and weak pigment; particularly, a large lead excess produces weakness; high acidity gives a weak, blue product; high molybdate results in a lighter, stronger, yellower product.

EXPERIMENTAL DETAILS

N.B. #440 p. 52

Study variation in amount of sodium molybdate

- A - Control-check SW 7540
- B - Increase molybdate 10%
- C - Decrease " 10%

Results

B vs A - vs yellow, vvs flat,-; vs str, s yellow - Light fastness -
vvs better

C vs A - vs red, vs flat,-; s wk, s blue - Light fastness - s better

p. 53

Observe the effect of using different amounts of soda ash in the lead base

- A - Control-check 440-52A
- B - Decrease soda ash 25%
- C - Increase soda ash 25%

Results

B vs A vs red, vvs flat,-; s wk, blue - Light fastness - vvs better
C vs A vvs light, vvs bright,-; vs str. vs yellow - Light fastness - equal

P. 54

Study variation in amount of sodium bichromate and excess sulphate in bichromate.

- A - Control - check 440- 52 A
- B - 5 grams extra sulphate in bichromate liquor
- C - Increase sodium bichromate 10%
- D - Decrease sodium bichromate 10%

Results

B vs A - O.K., vs flat, vs yellow; vvs wk, vs yellow - Light fastness - equal
C vs A - vs dk, vs flat, vs blue; equal, s blue - Light fastness - vs better
D vs A - s lt, vs flat, vs yellow, vs wk, yellow - Light fastness - equal

P. 55

The effect of delaying the addition of sodium bicarbonate.

Due to an error in weighing this series had a strong chromate excess.

Rubouts showed all of the series to be dark and muddy in masstone, poor in light fastness, but much superior to the K number in strength. This information has been passed along to the research group.

P. 56

The effect of delaying the addition of sodium bicarbonate.

- A - Control - check 440-52
- B - Sodium Bicarbonate added immediately after strike
- C - " " " 20 minutes " "
- D - " " " 20 " " "

Results

B vs A - O.K., O.K, O.K; vvs wk. O.K. - Light fastness - equal
C vs A vvs lt. vvs flat, o.k; vs wk, vs yellow - Light fastness - equal
D vs A vvs lt, vvs flat, O.K; vs wk, vs yellow - Light fastness - equal

P. 57

Observe the effect of delaying the addition of alum

- A - Control - Check 440-52 A
- B - Delay adding alum 10 minutes
- C - " " " 20 "

Results

B vs A O.K, O.K, O.K; O.K, vvs yellow - Light fastness - equal
C vs A O.K, vvs flat, O.K; vvs wk, vvs yellow - Light fastness - equal

PIGMENT COLOR RESEARCH REPORT

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MOLYBDATED CHROME ORANGE YO-8074-D

SUBMITTED BY:
APPROVED BY:

J. E. Allen
V. Chalupski

FILE: MOLYBDATED CHROME ORANGE
DATE: JANUARY 1936

INTRODUCTION

Following an unsuccessful plant trial of this color last month, the formula was returned to the semi-works for revision. A considerable amount of work has been done. Satisfactory material is now being made, on a slightly revised formula in both the semi-works and plant.

A new formula giving an orange of greatly increased strength is being tried in the semi-works.

SUMMARY AND CONCLUSIONS

Semi-Works

A total of eighteen small batches of this color were made in the semi-works during the month. The first six of these were run on variables in molybdate, different lots and quantities being used in an attempt to ascertain the cause of the yellow, weak material being produced. Results were inconclusive, all of the six batches being distinctly yellow flat and weak against K-8074.

The K-8074 formula was then checked using crystalline materials wherever possible, and a new lot of molybdate. This batch was close to the K-number, being a little on the dark, bright side, with satisfactory strength, and a somewhat yellowish tint. The substitution of lead nitrate liquor for crystals gave a little lighter product that was vs weaker. The further substitution of sodium bichromate liquor for crystals gave a little cleaner, weaker and bluer color than the previous batch. An increase of ten percent in the amount of molybdate increased the strength to that of the K-number and gave a somewhat yellowish masstone and tint. This batch was checked except for a five percent increase in bichromate, which gave a very faint bichromate excess after strike. The result was a vs dark, red and bright masstone with a vs strong and blue tint. Light fastness on all of the batches ran somewhat inferior to that of K-8074.

Two batches were made in which the addition of the treating agents, alum and sodium bichromate, was delayed twenty minutes. Both batches were a little weak and flat, but not nearly as much so as the material made at the first of the month.

A new laboratory formula was given a trial in the semi-works. It differs from the present formula in that the strike is made at a much higher pH which is subsequently lowered by the addition of acid. Material forty percent stronger than K-8074 was obtained in the laboratory using this formula. In the semi-works the first trial was blue, flat and weak. On a slightly revised formula, material about

twenty-five percent stronger than K-8074 was obtained, the masstone, however, being a little blue, dirty and chromy. A check batch was a little lighter and flatter.

Plant

Three 2-mol batches of this color were made in the factory. The first of these was a check on the K-8074 formula, - as was the off-batch made last month, except for a five percent increase in the amounts of sodium bichromate and molybdate. This batch was on the dark, red, bright side of the K-numbers, with a weak, red tint and vvs worse light stability. We have been unable to isolate the cause of the yellow, flat appearance of last month's batch except to conclude that it was a result of a combination of unfavorable conditions; ie., too high an excess of lead, not enough molybdate, and too high acidity. The first two items may have been the result of inaccurate analyses.

The second batch of the month was made with an increase in bichromate and molybdate over the former one. It was a little flat, dirty and strong against the former batch. An increase in soda ash gave a lighter, yellower product.

This study will be continued in the semi-works and plant.

EXPERIMENTAL DETAILS

For Experimental Details see N.B. #448, p. 193-195.
N.B. #492, p. 75-85

DATE	BATCH	FORMULATION	CONTROL	MASSTONE	UNDERTONE	STRENGTH	TINT	LIGHT STABILIT
12/30/35	SW 7571	Check K-8074 formula but use plant molyb- date	K-8074	Lt, yel & flat	yel & wk	-	-	worse
12/31/35	7576	Check SW-7571 but use Molybdate of new anal- ysis	K-8074 7571	yel & flat yel & wk	weak	yel.		s wors
1/2/36	7580	Check SW-7571 but use different lot of Molybdate	K-8074 7571	lt yel & flat				
1/3/36	7585	Check SW-7580 but de- crease Molybdate 5%	K-8074 7580	s lt & yel s yel & wk vs flat vs dk, s w b l & str & bl	wk	vs yel		vs wor
1/8/36	7595	Check SW-7462 using same molybdate as in that batch	K-8074 7585	s lt & yel & wk flat s lt, yel yel & wk & flat				equal
1/9/36	7600	Check SW-7595 but use lead nitrate crystals	K-8074	s lt, vs dull	wk			s wors
1/13/36	7608	Check SW-7462 using lead nitrate and Sod. Bichromate crystals and new Molybdate	K-8074	vs dk, vvs brt	0 K	vs yel, vvs dty		
1/14/36	7613	Check 7613 but use lead nitrate liquor	K-8074 7608	vvs lt & flat s lt	vs wk s yel	vs yel vs yel		

DATE	BATCH	FORMULATION	CONTROL	MASSTONE	UNDERTONE	STRENGTH	TINT	LIGHT STABIL
1/15/36	7617	Check SW-7613 but use Sod. Bichromate liq.	K-8074 7613	trace cl vvs cl	- vvs wk & dty	s wk vvs wk	vs bl vs bl	
1/16/36	7618	Check 7617 but increase molybdate 10%	K-8074	vvs lt, trace yel	s yellow	0 K	s yel	
1/20/36	7634	Check SW-7617 but delay Sod. bicarbonate addit- ion 20 min. Increase molybdate 5%	K-8074 7617	vvs fl & dty vvs dull & dty	vvs dty vs dty	vs wk vs str	- vvs yel	s wors vvs be
1/21/36	7635	Check SW-7617 but delay alum addition 20 min. Increase molybdate 5%	K-8074 7634	trace bl & flat trace lt	- -	vs wk vvs wk	+	s wcrs vs wor
1/22/36	7642	Check SW-7618 but in- crease bichromate 5% (trace Cr2O7 xs)	K-8074 7618	vvs dk, red & brt vs bl & brt	vs str & Bl s str & bl	vs str s str	vs bl bl	vs wor vs bett
1/23/36	7645	Check Lab. exp. 497-20 (neutral strike)	K-8074	s bl, flat & dty	wk	wk	bl	s wors
1/24/36	7652	Check 7595 using same equipment	K-8074	vvs brt	o k	o k	vs bl	
1/29/36	7666	Check Exp. 497-13 B for improved strength	K-8074	vs dty, bl & chromy	s str	str	-	
1/30/36	7671	Check SW-7666	K-8074 7666	vs dty, flat s yel & wk & chg vs lt & flat s yel & wk	vs yel & wk trace str	str trace str	yel -	
1/31/36	7672	Check SW-7666 but use lead nitrate liquor						

DATE	BATCH	FORMULATION	CONTROL	MASSTONE	UNDERTONE	STRENGTH	TINT	LIGHT STABILITY
1/27/36	* 47380	Check 46713 but increase molybdate and bichromate 5%	K-8074	dk, red, s brt	red	wk	red, s dty	vvs wvrs
1/29/36	47429	Check 47380 but in- crease bichromate & molybdate 2% & 5% resp.	K-8074 47380	s dk, & red s flat, vs dty	vs wk, s red vs dty & str	red vvs str.	red, s dty	red, s dty
1/31/36	* 47476	Check 47429 but in- crease soda ash 25%	K-8074 47429	vs lt, s flat s lt, vs yel	vs yel s yel, & wk	O.K.	Yel, vs cl	Yel, vs cl

* from control tests these batch numbers were evidently interchanged in 34 Bldg. Readings given here are probably correct.

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PIGMENT COLOR RESEARCH REPORT
LEAD NITRATE PURIFICATION -
FERROCYANIDE PRECIPITATION

SUBMITTED BY: *J. L. Kuchel*
APPROVED BY: *V. Chalupski*

FILE: LEAD & LEAD MATERIALS
DATE: JANUARY 1935.

INTRODUCTION

Work has been continued in the semi-works on the purification of lead nitrate by the precipitation of the copper with sodium ferrocyanide.

SUMMARY AND CONCLUSIONS

Twelve purifications have been carried out. The following variables have been found effective in increasing the filtration rate:

1. An increase in temperature - 150°F is the temperature now being used.
2. A change in filtering medium from single 4/0 duck cloths to double XX chain weave cloths.
3. The addition of filtercel to the slurry. One to three pounds of the filter aid were used.

The last variable was also effective in cutting down the time necessary to obtain a clear filtrate at the beginning of the filtration cycle. No difficulty was experienced in obtaining adequate purification as judged by the analysis of the resulting solutions and the quality of the Y-359-D made from them, in which, however, a slight dirtiness was observed, possibly due to a red dye bleed from the filter press used. A series is being run to check this matter.

On the basis of the results of one batch, it appears that "Lightnin" Agitation will be satisfactory.

EXPERIMENTAL DETAILS

See Notebook #492, pp. 99-137. Additional results are given on the batches mentioned in the December report.

Batch	#Pb(NO ₃) ₂	# Y.P.S.	# Filtercel in Slurry	# Filtercel in	Volume Gals.
SW-7549	265	5.84			120
7555	265	6.84			"
7560	259	5.34			"
7563	248	5.09			"
7565	257	5.29			"
7574	129	2.67			60
7578	196	3.69			100
7586	254	0.78			"
7620	250	4.84			"
7621	250	4.84			"
7624	200	3.87			"
7637	173	3.36			"
7640	196	3.80			"
7647	193	3.84			"
7649	195	3.78	1		"
7653	196	3.80	3	3.2	"
7660	185	3.55	3		"
7664	193	3.55			"
7667	184	3.55			"
7674	155	4.30			"

Batch	Precipitation Temp. OF	Time min.	% Copper in Feed Yield	#Y.P.S. #CU.	#Y.P.S. #Pb(NO3) ₂	Filter Area Sq.Ft.	
7549	60	5	.046	.0003	10.4	.022	16.3
7555	"	30	.046	.00011	12.2	.026	"
7560	110	5	.044	.0006	10.0	.021	"
7563	"	30	.043	.0002	9.7	.021	"
7565	60	5	.045	.0001	9.7	.021	13.3
7574	110	"	-	-			8.0
7578	"	"	-	-			13.3
7586	"	30	-	.0001			"
7620	"	"	.053	.00016	9.2	.019	16
7621	"	"	.048	.00019	10.1	.019	"
7624	"	"	.035	.0001	11.2	.019	"
7637	"	"	.031	.0001	10.8	.019	13.3
7640	"	"					"
7647	150	"					16
7649	110	"					"
7653	"	"					"
7660	"	"					"
7664	150	"					"
7667	"	"					"
7674	"	"					"

R. = rubber recessed plate press.
P.F. = #203, wooden plate and flame press.
* = gravity filtration.
** = sent to press without dumping residue from previous batch.

Batch	Press	Cloth	Agitation	Time of Recycling Min.	Filt. Rate Gal/sq.ft/hr.	P.C. Vol. cu.ft.
7549	R.	4/o duck	Sweep	80	2.7	.2
7555	"	"	"	0	3.8	.25
7560	"	"	"	0	7.2	.17
7563	"	"	"	-	14.1	.11
7565	"	"	"	-	5.5	.21
7574	P.F.*	"	"	-	1.4	-
7578	R.	"	Lightning	-	7.8	.13
7586	"	"	"	-	4.1	.04
7620	P.F.	"	Sweep	182	2.9	.21
7621	"	"	"	80	2.3	.21
7624	"	"	"	145	2.7	.15
7637	R	"	"	4	7.0	-
7640	"	"	"	9	7.5	.11
7647	P.F.	XX chain	"	54	6.0	.14
7649	"	"	"	20	7.3	.12
7653	"	"	"	7	9.2	.36
7660	"	"	"	3	16.4	.19
7664	"	"	"	37	9.5)	.31
7667	***	"	"	15	4.7)	
7674	"	"	Lightning	9	9.1	

Yellow Purif.

Rubout versus Standard

Batch #	Batch#	Masstone	Drawdown	Strength	Tint	Lightfastnes
7554	7549	v.s.red & cln.	-	s.str.	s.grn.	better
7556	7555	s.red & cln.	-	s.str.	s.grn.	better
7561	7560	v.s.red, v.v.s.cln.	s.str.	s.str.	s.cln.	s.better
7566	7563	v.s.red, v.v.s.cln.	s.str.	s.str.	s.cln.	s.better
7572	7565	v.s.dark & red v.v.s.dirty	s.red	s.str.	s.red & cln.	better
7579	7574	v.v.s.red & cln.	O.K.	O.K.	v.v.s.red & cln.	better
7584	7578	s.red & dirty	dirty, s.red	s.str.	s.red & cln.	s.better
7588	7586	s.red & dirty	red, s. dirty	v.s.str.	v.s.cln.	s.better
7625	7620	s.red	s.red & dirty	str.	cln.	s.better
7639	7621	v.v.s.red, v.s. dirty	s.red	s.str.	s.cln.	s.better
7638	7624	v.s.red	v.v.s.red	s.str.	s.gr.& cln.	v.s.better
7643	7637	v.s.dirty & flat	s.red	s.str.	s.gr.& cln.	s.better

<u>Yellow</u>	<u>Purif.</u>	<u>Rubout versus Standard</u>				
<u>Batch #</u>	<u>Batch #</u>	<u>Masstone</u>	<u>Drawdown</u>	<u>Strength</u>	<u>Tint</u>	<u>Light- fastness.</u>
7644	7640	s.grn.& dirty	s.red	s.str.	s.grn& cln.	s.better
7654	7647	v.s.grn,s.dirty	s.red	s.str.	s.cln.	better
7655	7649	v.s.grn,s.dirty	s.red	s.str.	s.cln.	better
7659	7653	v.v.s.grn,v.s.dirty	v.s.red	s.str.	s.cln.	s.better

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PIGMENT COLOR RESEARCH REPORT

C.P. CHROME GREENS. G-409-L - 412-D

PRODUCTION

SUBMITTED BY:

J. Kallbach U.S. Berdinger

APPROVED BY:

U. Chalupotki

FILE: CHROME GREEN

DATE: JANUARY 1935.

INTRODUCTION

One batch of each member of this line was made in the plant for stock.

SUMMARY AND CONCLUSIONS

Difficulty was experienced from redness in the greening yellows and the corresponding olive and dirty masstones of the greens. Two of the above greening yellows were improved in a measure by the addition of fifty pounds (actual weight) of acetic acid before greening. It seems probable, therefore, that this trouble can be corrected by an increase in the acid in the strike.

EXPERIMENTAL DETAILS

See notebook #494. pp. 48-55.

Code	Batch No.	Formula	M.T.	D.D.	Str.	Tint
G-409-D	47097	Standard Form. but 50# actual wt., acetic acid added to yellow before greening.	olive.	s. yellow	v.s. weak	v.s. yellow. dirty.
G-410-D	47078	Standard Form.	lt. & olive	yellow		
G-411-D	47079	As in 47097	s. dark and olive; dirty and changeable.	blue		
G-412-D	47098	Standard Form.	dark, s. blue, s. yellow dirty, changeable.	s. yellow	s. weak	s. yellow dirty.

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PIGMENT COLOR RESEARCH REPORT

C.P. CHROME GREENS, G-415-D AND G-416-D

SUBMITTED BY:

APPROVED BY:

J. William A. D. Reidinger
H. Chalupski

FILE: CHROME GREENS

DATE: JANUARY 1936

INTRODUCTION

Three batches of each of these colors were made on the formula (439-89A) recently used in an effort to obtain a strong type G-389-D. This was done to restandardize G-415-D and G-416-D (G-390-D and G-391-D standardized for rubber). This was necessary because the original standards contained copper.

SUMMARY AND CONCLUSIONS

The formula actually used was a modification of the laboratory formula previously reported as SW 7530 and G-389-D, plant batch 46839.

These batches showed a decided tendency to flatness and chalkiness in masstone by rubout against both the rubber and regular G-389- line standards. Against G-390-D standard a mix of the three G-415-D batches brought to equal depth with a little G-416-D, showed about 5% extra strength. A similar G-416-D mix was about 10% strong versus G-391-D standard.

Satisfactory new copper-free standard mixes were made using the six batches made.

See Table 1.

Mixes have been made and submitted for standard in rubber as follows, the mixes containing a small amount of lead sulphate to allow for the extra strength of this production:

G-415-D, 16662 consists of:

G-415-D	47118	1330#
"	47156	1470#
"	47183	1098#
G-344-D	46618	190#
PbSO ₄		285#
		<u>4363#</u>

G-416-D, 16698 consists of:

G-416-D	47119	1680#
"	47155	1835#
"	47205	1780#
G-415-D	47183	371#
"	46682	212#
PbSO ₄		152#
		<u>6037#</u>

TABLE I

EXPERIMENTAL DETAILS

N.B. #494 p. 10 - 21.

BATCH	VS. STD.	MASSTONE	DRAWDOWN	STRENGTH	TINT
G-415-D					
47118	G-415-D G-390-D	light and flat light, s.flat & chalky	s. yellow yellow	s. weak	yellow
47156	G-415-D G-390-D	light and flat vs changeable s.light, s.flat s. chalky	s. yellow yellow & strong	s. strong	yellow & clean
47183	G-415-D G-390-D	light, s.flat s.changeable light, s.flat	s.yellow yellow	O.K.	yellow & clean
mix.	G-390-D	vs.dull, s.blue and chalky	s.yellow & strong	5% strong	yellow & clean
16662 16661	G-415-D G-415-D in rubber	light & flat (O.K.migration)	s.yellow	O.K. O.K.	yellow s.clean
G-416-D					
47119	G-416-D	s.light (muddy) (flat) s.change- able	s.yellow		
	G-391-D	s.light, s.flat s.chalky	yellow	s.weak	yellow

TABLE I (CONTD.)

BATCH	VS. STD.	MASSTONE	DRAWDOWN	STRENGTH	TINT
G-416-D	(Contd.)				
47155	G-416-D	dark, blue vs. dirty s. changeable	blue		
	G-391-D	dark, blue, flat chalky	s. yellow, s. clean - s. strong	strong	clean
47205	G-416-D	s. dark (blue) changeable, s. dirty	blue		
	G-391-D	s. dark, blue, flat, chalky	s. yellow and strong	strong	s. clean
mix	G-391-D	blue and chalky s. dull	yellow	± 10% strong	O.K.
16697 and 16698	G-416-D	light, muddy & flat	s. yellow	vs weak	yellow
	G-416-D in rubber	(O.K. migration)		± 5% strong	vs clean

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B

SUBMITTED BY: *Alfred Segel*
APPROVED BY: *ACE*

FILE: PIGMENT GREEN B
DATE: JANUARY 1936

INTRODUCTION

Work was continued in the laboratory this month on the problem of developing a dry lake of Pigment Green B for dispersion in rubber and for general evaluation.

The investigation on the preparation of dry colors for paper coating continues to be delayed, pending completion of the studies on GL-407-D.

SUMMARY AND CONCLUSIONS

Preparation of a dry lake of Pigment Green B for rubber has been completed. The product K-8172 is a fifty per cent Pigment Green B Lake on alumina hydrate - barium resinate substrata. It disperses well in rubber, 107 parts being equal to 100 parts of both the corresponding later dispersed pulp and Green F D Standard, on an equal pigment content basis. A sample is to be submitted to the Rubber Chemicals Dept. for evaluation.

The work this month was devoted chiefly to studies involving minor modifications of the laking formulation, in order to obtain exactly 50 per cent toner content of the lake and complete precipitation of the water dispersible pigment on the substrata.

The use of barium chloride in place of sodium chloride in breaking the pigment-rosin soap colloid results in complete flocculation of the pigment and slightly softer texture and consequently slightly improved strength as judged by rubber tests. The rosin in the pigment is in the form of barium resinate instead of the free resin acid or soluble sodium resinate.

Altho diglycol stearate gave some improvement in previous products the recently improved lakes are not benefited by diglycol stearate.

Lithographic varnish rubout inks of K-8172 show it to be equal to the best prepared previously and indications are that this one product may be satisfactory for both rubber and general application, excluding paper coating. The problem of development of a Pigment Green B Lake dry powder in the laboratory can be considered complete and this new pigment is now ready for semi-works development, pending acceptance of K-8172 for rubber.

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EXPERIMENTAL DETAILS

Expt. 34, 35 and 36. N.B. 482 p. 106, 110 and 112 respectively.

Substitution of BaCl_2 for NaCl , for precipitation of the dispersible pigment results in slightly softer texture and slightly increased strength, and is preferable, especially since barium resinate instead of free resin acids or sodium resinate is present in the lake.

The use of "Duponol" WA and diglycol stearate have no beneficial effect upon the dry pigment.

Variation in the quantity of precipitant (BaCl_2) influences the flocculation and physical properties of the pigment. Sufficient BaCl_2 to give complete flocculation is essential in obtaining pigment of the desired texture and strength.

Expt. 37 N.B. 482 p. 116

Variation in alumina hydrate base from 30/11 to 30/15 has not appreciable effect upon the pigment. The 30/15 base is preferable.

Expt. 38 N.B. 482 p. 118

Substitution of CaCl_2 for BaCl_2 as the precipitant results in a lower yield and offers no advantage over BaCl_2 .

The quantity of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ was varied from 4 to 12 grams per 20 grams of Pigment Green B dry basis and 6 grams I Wood Resin. Complete flocculation and maximum strength was obtained using 8 grams $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.

Expt. 39 & 40. N.B. 482 p. 120 and 122 respectively.

Variations were made in the quantity of alumina hydrate in order to obtain an exact 50 per cent Pigment Green toner content of the dry product. The composition of the 50 per cent lake, K-8172 as estimated from the amounts of ingredients employed and yield obtained, is probably as follows:

50 parts Pigment Green B 100% toner basis
50 parts Alumina Hydrate
20 parts Barium Resinate

Actual Fe analysis of K-8172 gave 4.29% Fe. equivalent to

$4.29 \times 11.9 (\text{factor for P.G.B.}) = 51.05\% \text{ Pigment Green B}$

PIGMENT COLOR RESEARCH REPORT

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PIGMENT GREEN B PULP, GT-68-P

SUBMITTED BY: E. J. Her
APPROVED BY: V. Chalupski

FILE: PIGMENT GREEN B
DATE: JANUARY 1936

INTRODUCTION

Production was started in the plant on GT-68-P in order to build up the required stock of 1500 pound dry for rubber. A total of three batches were made under Development Section supervision.

SUMMARY AND CONCLUSIONS

The first batch was made to check Lot 41873 except that a new type Turkey Red Oil (Jacques Wolff) was used in preparing the Cadmium Soap. Because of the partial oxidation of the Sodium Bisulfite used in this batch the Nitroso Beta Naphthol was not fully esterified with the result that there is a large excess of Ferric oxalate and of Cadmium Soap in the finished pulp. The resultant color was therefore weak and dirty in both rubber and paper and with a yield of only 70% of estimate. In order to consume this off batch it will be necessary to mix off small portions of it in subsequent production of rubber material. Steps have been taken to prevent a recurrence of production of sub-standard material by arranging to discard all Sodium Bisulfite that has deteriorated (hardened in barrels) and by securing analyses of all material under suspicion. It has also been decided that subsequent orders for Sodium Bisulfite will be placed only as needed for current production in order to keep loss of material due to oxidation to a minimum.

The next two batches were made to check the first except that in the case of the last batch the regular General Aniline Turkey Red Oil was used in preparing the Cadmium Soap in order to have a control batch to use as a cross-check on the use of Jacques Wolff's Turkey Red Oil. Both of these batches were satisfactory in both rubber and paper by Krebs test and checked each other almost exactly by rubber test. The Jacques Wolff Turkey Red Oil is therefore satisfactory for use in GT-68-P on the basis of this test.

Yields were satisfactory averaging 96½% of estimate in the use of the last two batches which checks the yield on previous plant production.

The Dye Works has requested that no more stump barrels or kegs be included in the shipments of GT-68-P. This will of course necessitate the holding of stump batches in stock until enough has been accumulated to make full barrels (± 400 pounds of pulp). Uniform mixing of these stump lots will be rather difficult as the only equipment available at present for pulp mixing is the Semi-Works Straub-mill or Mikro-Pulverizer which are not designed for work of this type. The Dye Works has returned stumps on six batches of GT-68-P for paper. These have been mixed and there are two more mixes in process which will consume all stump material on hand at present.

Shipments over the past two months have consisted of 2473# pulp (656 # dry) shipped for paper. Stump barrels on the two batches shipped were retained as requested.

Production results are tabulated in Table I, mixes in Table II and shipments in Table III.

EXPERIMENTAL DETAILS

3.

N.B. 473, pages 101-103

TABLE I

GT-68-P PRODUCTION

Batch	Formula	Pulp Yield		Est. Yield	Pulp Character	Rubber Test vs Lot 13926	Paper Test vs Lot 41873	Remarks
		Original %	After rework Dry %	Dry				
47276	Check 41873 N-75	J.W. 30.9	265#	-	380# watery	5% wk-bl & dty	dirty	NG rubber & paper. Deteriorated N-78. In SSS to be mixed
47302	Check 41873 J.W. N-75	27	368#	28.0	356# firm, breaks down	O.K. ± O.K.	5% str - O.K.	OK rubber & paper. (Krebs)
47342	Check 41873 G.A. N-75	26.6	378#	26.8	378# firm, breaks down	O.K. ± O.K.	5% str - O.K.	OK rubber & paper (Krebs) 86# pulp (stump) to 16695

TABLE II

GT-68-P MIX

Lot	Composition	Total Wt.		Rubber Test vs Lot 13926	Paper Test vs Lot 41873	Disp.
		Full	bbls.			
16541	44581 83# pulp, 44402 135# pulp, 44371 102# pulp, 44224 137# pulp, 44426 124# pulp, 15342 73# pulp.	666#	at 30% =	vs wk- vs dty	5-10% str - O.K.	OK rubber. 266# pulp (stump to 16721

TABLE III

GT-68-P SHIPMENTS

Batch	Rubber Test vs Lot 13926	Paper Test vs Lot 41873	Disposition	Weight Shipped and Destination
46076	O.K. - O.K.	7% wk - O.K.	O.K. rubber O.K. paper	1215# at 26.5% = 322# dry. Shipped for paper
46148	"	5% wk - vs bl	O.K. rubber O.K. paper	1255# at 26.6% = 334# dry. Shipped for paper.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN LAKE FOR PAPER, GL-407-D

SUBMITTED BY: *Ad Reidinger*
APPROVED BY: *V. Chalupka*

DATE: JANUARY 1936
FILE: PIGMENT GREEN B

INTRODUCTION

Three trial batches of this lake were made on the K-8137 (sodium resinate) formula to test practicability of large scale operation. A plant size Drum Dryer located at the Dyeworks was used.

SUMMARY AND CONCLUSIONS

Although the material made on standard formula was weak on paper test, it is believed that by proper adjustment of the toner content, a satisfactory product can be obtained with use of drum drying. Analysis of the lakes produced showed only a 44.2% toner content on the two batches intended to be 50% toner. This discrepancy is explained by the yields obtained which were higher than on small previous batches and much closer to theoretical. Recalculation of the toner content on basis of theoretical yield gives a figure of 44.5%.

On basis of control tests which indicated the need of a weak shading batch a third batch was made to be 31% toner and was of course too weak for present use. The toner content by analysis was 26.48%. Dispersion in water was reported as somewhat inferior to the other batches.

Operation of the formula appears to be satisfactory. For regular manufacture provision for a vat in which to prepare sodium resinate would be necessary as use of an iron drum as done in other trial runs is cumbersome and unsafe. An agitated iron vat was used to slurry the green toner and resinate. No colloid mill was available and is probably not required.

The actual drying proceeded without difficulty, the material coming off the rolls in rather fine condition with only a small amount of wet lumps in the edgings. Approximately 8% of the product is carried into the ducts and scrubber from which it can be recovered.

The total yield obtained for the series was 549# or 93.5% of theoretical. The latter is calculated from the dry weight of Pigment Green, plus the weight of rosin, the sodium equivalent of the caustic soda used and 2-1/2% moisture.

EXPERIMENTAL DETAILS

N.B. #291, p. 29.

The Drum Dryer used is the one located in the Basic Pink Bldg. (#292) at the Dyeworks. The set up included a 445 gal. iron vat from which the color is fed to the rolls. A dust collecting system consisting of flues and a scrubbing tower is used. A drying rate of

about 100# per hour was obtained. Steam pressure was from 30 to 40#. ~~Drum reversed~~ ^{revolved} at a rate of 4 R.P.M.

The Green Toner Pulp (GT-68-P) used in the series was on lot #45093. This was treated material which was O.K. for rubber and for paper.

Paper tests on final grinds (main portion).

SW-7626	-	10-15%	weak	-	Dispersion	-	good.
SW-7628	-	"	"	-	"	-	"
SW-7031	-	30%	"	-	"	-	"

SW	Dyeworks Charge	CT-68-P #	Pulp Dry				# Rosin	# NaOH	# Steam on Dryer	% Toner Calc'd	Anal.	Weight
			#	#	#	#						
7626	1		344	90	100	13	35	44.5	43.44	160		
7627	"		-	-	-	-	-	-	-	8		
7628	2		344	90	100	13	33	44.5	44.93	184		
7629	"		-	-	-	-	-	-	-	3		
7630	3		191	50	120	16	40	27.3	26.48	164		
7631	"		-	-	-	-	-	-	-	8		
7632	"		-	-	-	-	-	-	-	4		
7633	1-2-3		-	-	-	-	-	-	-	18		
TOTAL			879	230								
			Theoretical Yield									549
												587

PIGMENT COLOR RESEARCH REPORT

SPECIAL ORGANIC PIGMENTS

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SUBMITTED BY: *Alfred Legil.*
APPROVED BY: *Paul*

FILE: MISC. LAKES & TONERS
DATE: JANUARY 1936

INTRODUCTION

Laboratory investigation of new intermediates, dyes, lakes, etc. was confined this month to the preparation and evaluation of dyes and lakes of the anilide of beta-oxynaphthoic acid as coupling component with 2 chlor 4 amino 5 sulfonic acid (Red 2 B diazo component) and 4 chlor aniline 2 sulfonic acid (Scarlet 2 YL diazo component) as diazo components.

Tests were made of 2.5 dichloraniline of Verona Chem. Co. and Lithosol Orange R lot 1 of the Dye Works.

SUMMARY AND CONCLUSIONS

Dyes and corresponding pigments of the alkaline earth metal salts were prepared in the laboratory from

A-diazotized 2 chlor 4 amino toluene 5 sulfonic acid combined with the anilide of 2 naphthol 3 carboxylic acid and

B-diazotized 4 chlor aniline 2 sulfonic acid combined with the anilide of 2 naphthol 3 carboxylic acid

The barium and calcium toners of the "A" combination are reds of medium depth and of yellowish shades, the calcium pigment being darker, and bluer than the barium. Both, however, are much darker and weaker than the Calcium toner of Lithosol Red 2 B K-7853 and both bleed slightly in linseed oil and distinctly in lacquer. This bleed, however, may result from decomposition products formed during coupling or incomplete conversion of the dye to the alkali earth metal salt. Altho light fastness tests have not been completed the results so far indicate that both the calcium and barium pigments are inferior to K-7853 (CaRed 2 B Toner) in light fastness.

The barium and calcium toners of the "B" combination are deep reds of yellowish shade, the barium pigment being darker and bluer than the calcium. Both are much darker than pigments of the "A" dye and very much darker, duller, bluer and less clean than the strontium toner of Scarlet 2 YL, K-7985. The calcium and barium pigments exhibit a slight linseed oil and lacquer bleed. This bleed, however, may result from decomposition products formed during coupling or incomplete conversion of the dye to the alkali earth metal salt. Altho light fastness tests have not been completed, the results so far indicate that both the calcium and barium pigments are inferior to K-7985 in this respect.

The work on the above new dyes and lakes so far has been strictly of an orientation nature and a conclusion at this time as to the merits of any of these products would be premature. Continued investigation is proposed.

A sample of 2:5 dichloraniline of The Verona Chemical Co. appears to be satisfactory for the preparation of Monolite Fast Red 2 R (2:5 dichloraniline -- anilide of beta-oxynaphthoic acid). This color is a very close match to Teluidine Red RT-386-D.

Tests of laboratory prepared dye and duPont "Lithosol" Orange R lot 1 showed indications of the latter being contaminated with black dye. This was subsequently confirmed by The Technical Laboratory.

EXPERIMENTAL DETAILS

Expt. 49 N.B. #487 p. 168

Preparation of

Dye: diazotized 2 chlor 4 amino toluene 5 sulfonic acid → Naphthanil A.S., and Barium and calcium toners. Series A₂ A₃ A₄ A₅ and A₆.

Preparation of

Dye: diazotized 4 chloraniline 2 sulfonic acid → Naphthanil A.S., and Barium and calcium toners. Series B₂ B₃ B₄ B₅ and B₆.

Expt. 47 N.B. #487 p. 164

R.M. test of Verona Chem. Co.'s 2:5 dichloraniline

Expt. 45 N.B. #487 p. 160

R.M. test and investigation concerning off-color of duPont "Lithosol" Orange R-lot-1.

The dye lot 1 appears to be contaminated with black dye. This was confirmed at a later date by the Technical Laboratory of The Dye Works.

PIGMENT COLOR RESEARCH REPORT

FLUSHED INORGANIC PIGMENTS

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SUBMITTED BY: *H E Burdick*
APPROVED BY: *W E E*

FILE: PIGMENTS IN OIL
DATE: JANUARY 1936

INTRODUCTION

Orientation work on the flushing of iron blues and chrome yellows has been continued.

SUMMARY AND CONCLUSIONS

The effect of the flushing process on the tinting strength of iron blue is still in doubt although indications are that flushing causes an increase in tinting strength. Metallic oxide (i.e. ZnO and MgO) flushing assistants decrease the tinting strength probably by reacting with the iron blue and increase the rate of livering of the finished ink.

Long chain alkyl amines and the corresponding quaternary ammonium salts apparently coat the iron blue particles so that they are no longer wet by water causing a thick iron blue pulp to become thin and watery (this same effect is produced by stearic acid, sodium hexametaphosphate and the sodium salts of lignin sulphonie acids (D-2262) but these agents do not cause the iron blue (in pulp form) to transfer into the oil phase easily in such a manner as, for instance, Zinc Oxide does. In some instances, fixanol has caused an actual transfer but consistent results have not been obtained and it is evident that the optimum conditions have not been determined. The results obtained at the Experimental Station with a 1% slurry of iron blue have been confirmed in this laboratory.

Zinc naphthenate (D-2240) is relatively successful while the following additional agents are tentatively unsuccessful as assisting agents for the "flushing" of iron blue in pulp form:

- | | |
|---|----------|
| 1. Active silica adsorbent | (D-2241) |
| 2. Merck's medicinal charcoal | (D-2242) |
| 3. Sulphonated olive oil | (D-2233) |
| 4. Diglycol laurate | (D-2315) |
| 5. Free lignin sulphonie acids | (D-2261) |
| 6. Sodium salts of lignin sulphonie acids | (D-2262) |
| 7. Crude stearyl amine | (D-2286) |
| 8. Sodium hexametaphosphate | |
| 9. Stearic acid | |
| 10. Furoic acid | (D-1267) |
| 11. Cetyl trimethyl ammonium bromide | (D-2274) |
| 12. Aqualoid | (D-2328) |
| 13. Lecivatt | (D-2329) |

Y-316-D does not flush into vegetable oils unless Zinc Oxide is added. B-66-D and Y-316-D will not flush into Nujol even with the addition of Zinc Oxide. Providing sufficient water is present, B-66-D and Y-316-D mixed with Nujol will produce a crude emulsion with water as the continuous phase.

Zinc naphthenate was relatively successful while Zinc Oxide, lead sulphate and soya lecithin (D-1561) were tentatively unsuccessful for flushing Y-202-D into #1 transparent varnish.

REVIEW OF PREVIOUS WORK

The immediate effect of treating water-wet pigments with oil as recorded in the various experimental note books is summarized in Table I.

EXPERIMENTAL DETAILS

N.B. 483.

Expt. 13. Reaction of Kadox with wet and dry B-66-D.

Expt. 14. Effect of White Seal ZnO and MgO on flushed and dry control inks of B-66-D.

Expt. 15. Orientation experiment on flushing B-66-D and Y-316-D.

Expt. 33. Fixanol as assisting agent for B-66-D.

Expt. 34. Affect of fixanol on strength of B-66-D. Experiment to be repeated.

Expt. 35. Affect of various amines and quaternary salts on rate of filtration of B-66-D slurry (1%).

Expt. 42. Flushing assistants for B-66-D.

Expt. 46. Flushing assistants for B-66-D and Y-202-D.

TABLE I

EFFECT OF TREATMENT OF INORGANIC PIGMENTS WITH OIL

Pigment	Code Number	Notebook and experiment	Stirring with oil	Ink milling with oil	Colloid milling with oil
Chinese Blue	B-63-D	359-84, 78, 81, 83 381-170, 172 431-120, 125, 128, 131 451-13, 24 483-6, 7, 8	No transfer Oil is emulsified	Does not flush	Passes mill easily with 100% oil - no transfer
Milori Blue	B-66-D	359-78, 82 451-1, 3, 6, 15, 18, 25 483-8, 10, 11, 13, 14	No transfer Oil is emulsified	Does not flush	Passes mill easily with 100% oil - no transfer
Milori Blue	B-109-D	417-73			
Prussian Blue	B-140-D	451-14	No transfer Oil is emulsified	Does not flush	Passes mill easily with 100% oil - no transfer
Opaque Blue	CS-7469	431-132, 451-2,			
Extended Chrome Green	GX-309-D	359-77 431-126 451-29			Passes mill easily with 43% castor oil - no transfer
Extended Chrome Green	GX-312-D	359-77			
Excelsior Yellow	Y-12-D	451-5			Passes mill easily with 8% Najol - probably no transfer
Excelsior Yellow	Y-309-D	359-78 451-17, 12		Does not flush	Passes mill easily with 33% mineral oil - no transfer
Excelsior Yellow	K-7903	451-27, 28			Passes mill easily with 5% Najol - probably no transfer
M-Lithopone		451-13, 22			

PIGMENT COLOR RESEARCH REPORT

FLUSHED LITHOL RED

36-23 55

SUBMITTED BY: *H E Burdick*
APPROVED BY: *con 6*

FILE: PIGMENTS IN OIL
DATE: JANUARY 1936

INTRODUCTION

A consistency study of the effect of flushing on RT-364-D has been made.

SUMMARY AND CONCLUSIONS

Flushed inks of RT-364-D are consistently slightly weak and blue in tint and very slightly light and dull against corresponding dry control inks. It makes no difference whether the dry control inks are ground from lump dry color or from dry color which has been pulverized on paper by rolling with a bottle. The above relationship of the fresh tints changes on standing. Apparently the masstone, undertone and tint are more closely duplicated for flushed inks than for dry control inks within a single plant batch. Between plant batches, the absolute tinctorial values, of course, vary greatly but the relative tinctorial values of flushed and dry control inks are quite consistent.

A preliminary experiment indicates that RT-364-D can be flushed into RC-203 "Dulux" resin containing thinner on the hot ink mill.

The extraction method of analysis for pigment content of inks does not appreciably affect RT-364-D tinctorially.

Apparently there is less bleed into oil in flushed inks than in dry control inks of RT-364-D since when 483-31 and 483-36 inks (1:1) were thinned down to 2:3 inks and mounted on paper, the oil that soaked into the paper showed less bleed for the flushed inks than for the dry control inks.

REVIEW OF EXPERIMENTAL WORK

A consistency series (483-36) was run in order to determine if the relationship of flushed and dry control inks of RT-364-D would be the same from batch to batch. Apparently this is true since in both series 483-31 and 483-36, the fresh tints of the flushed inks are slightly weak and blue and the masstones slightly light and dull against the dry control inks. Apparently the masstone and tint are more closely duplicated for flushed inks than for dry control inks. The conclusion seems justified that within a single plant batch, the masstones and tints of flushed inks can be duplicated. There is a great variation, of course, from plant batch to batch of RT-364-D. The results of 483-36 series are tabulated in Table I of this report and should be compared with the results of 483-31 series tabulated in Table III of the "Flushed Lithol Red" report for December, 1935.

EXPERIMENTAL DETAILS

N.B. #483.

483-36. Consistency study on RT-364-D flushed and dry control inks.

483-37. Flushing of RT-364-D into BRC-95490 resin.

483-40. Flushing of RT-364-D into #3 regular varnish and into #3 transparent varnish.

483-44. Effect of extraction procedure on RT-364.

TABLE I

EVALUATION OF FLUSHED AND DRY CONTROL LITHOL
RED INKS

Sample (1)	% Yield of ink	Water Content (2)	Pigment Content (2)	Strength (5) (1:100 ext.)	Length (3)	Body (3)	Masstone (3)	Undertone (3)
483-36A ₁	99.0	0.8	50.73					
-36B ₁	100.7	1.0	49.60	1% weak & bl vs. A ₁	vs long vs. A ₁	vs soft vs. A ₁	vs light vs. A ₁	= to A ₁
-36A ₂	99.0	1.8		= to A ₁	vs long vs. A ₁	= to A ₁	vs light vs. A ₁	= to A ₁
-36B ₂	99.1	2.0		1% weak and bl vs. A ₁	vs long vs. A ₁	vs soft vs. A ₁	vs light vs. A ₁	= to A ₁
-36C ₄ (4)	97.2			= to A ₁	vs long vs. A ₁	vs soft vs. A ₁	vs light vs. A ₁	= to A ₁

Notes to Table I:

- "A" samples are laboratory dried control inks. "B" samples are flushed on ink mill inks. "C" is plant ground dry control ink. All samples are 1:1 inks.
- Analyses for water content by toluene distillation method and for pigment content by extraction method carried out by S. W. Rumbel.
- Body, length, masstone and undertone readings were made on 1:2 inks. Thinned inks (1:2) of A₁ and B₁ had no flow and texture readings of 20 and 20+ respectively.
- Rolls of the ink mill were not previously wet with ink.
- These readings were made while the tints were fresh. The relationships changed on standing. 36 series tints were blue and dirty vs. 31 series tints.

PIGMENT COLOR RESEARCH REPORT

36-24

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FLUSHED P.T.A. PIGMENTS

SUBMITTED BY: *H E Burdick*
APPROVED BY: *acc E.*

FILE: PIGMENTS IN OIL
DATE: JANUARY 1936

INTRODUCTION

A cooperative study with F. H. Levey Co. on methods of flushing and evaluation of flushed inks has been initiated.

SUMMARY AND CONCLUSIONS

A large sample (over 3 pounds) of BT-125-D flushed ink and corresponding dry control and pulp control have been prepared so that F.H. Levey Co. can compare their methods with our method of flushing. Also a 3 pound sample of Blue Toner, K-7982, dry color ground in an equal weight of #0 regular varnish has been prepared for Levey to compare in properties against competitive flushed inks.

Neither flushed nor dry control PTA pigment #0 regular varnish inks showed an appreciable amount of livering after standing in cans at room temperature for three months.

Flushed and dry control inks of GT-347-D extended in hydrate ink (1:50 and 1:100) by grinding on the ink mill showed no difference in tinting strength.

EXPERIMENTAL DETAILS

N.B. 483

483-41. Preparation of three pound sample of K-7982: #0 regular varnish (1:1) ink for Levey.

K-7982 is stronger and is better in light fastness vs. BT-150-D.

483-43. Preparation of large samples of BT-125-D (Lot #47391) for Levey.

A. Dry Control. Press Cake dried over night in steam oven 140°F and micro pulverized in semi-works.

B. Flushed Ink. 3400 g. pulp (833 g. dry color) ± 833 g. #0 regular varnish flushed on large ink mill. Four passes over hot mill after removal of water.

C. Pulp control.

483-45. Extension of 483-29 inks (GT-347-D) with hydrate ink.

PIGMENT COLOR RESEARCH REPORT

FLUSHED LAKE RED C

36-25

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SUBMITTED BY: *H E Burdick*
APPROVED BY: *Am E.*

FILE: PIGMENTS IN OIL
DATE: JANUARY 1936

INTRODUCTION

An accurate evaluation of flushed and dry control inks of RT-313-D has been made.

SUMMARY AND CONCLUSIONS

On the ink mill, RT-313-D flushes readily into an equal weight of #0 regular varnish with only a negligible amount of pigment remaining in the flushed water.

Duplicate experiments show that RT-313-D flushed inks have no advantages in tinting strength, flow, masstone and undertone and slight advantages in body, length, texture and light fastness as compared to corresponding dry control inks.

The experimental evidence upon which these conclusions are based is summarized in Table I.

TABLE I

EVALUATION OF FLUSHED AND DRY CONTROL INKS
OF RT-313-D

Sample (1)	% Yield of Ink	% Water (2)	% Pigment (3)	Texture	Tint. Strength	Body	Length	Flow (5)	Masstone (6)	Light Fastness
483-38A ₁	99.7	0.5	52.35	17				3		
-38B ₁	99.2	0.2	53.32	18	= to A ₁	s soft vs. s long A ₁	A ₁	4	vs light vs. A ₁	vs better vs. A ₁
-38A ₂	101.0			17	= to A ₁	vs soft vs. A ₁	= to A ₁	5	= to A ₁	= to A ₁
-38B ₂	100.4			18	= to A ₁	s soft vs. s long A ₁	A ₁	2	vs light vs. A ₁	vs better v A ₁
-38C (4)	96.0			17+	= to A ₁	vs soft vs. A ₁	= to A ₁	2	vs light vs. A ₁	vs better v A ₁

Notes to Table I:

- "A" samples are lump dry control inks ground on the ink mill
"B" " " flushed on the ink mill inks.
"C" " " plant pulverized dry control inks.
- Analyzed by S. W. Rumbel using Toluene distillation method.
- " " " " extraction method.
- The ink rolls were not initially wet with ink.
- Flow measurements were made on 1:2 inks.
- Masstone mounts " " " 1:2 inks and on 1:1 inks. On standing all the masstones became more equal. All the undertones were equal.

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PIGMENT COLOR RESEARCH REPORT

NEW TYPE PRINTING INKS - ROTOGRAVURE INKS

SUBMITTED BY: *A. Stratton*
APPROVED BY: *K. E.*

FILE: PRINTING INK COLORS
DATE: JANUARY 1936

INTRODUCTION

This problem has been pursued actively in the laboratory the past month. A large number of inks have been prepared and evaluated in so far as we are able to evaluate them and conclusions drawn as to the formulation of inks with satisfactory gloss. This phase of the investigation appears to be substantially complete. Some investigation of the merits of various resins remains to be done, as well as the final preparation of sizeable samples of inks to be evaluated by the user by actual printing tests.

SUMMARY AND CONCLUSIONS

The following resume of the previous work is introduced to make this report complete in itself.

The Technical laboratory at the Dyeworks was asked in February 1935 to recommend an ink for use on a new type of rotogravure press by one of their customers. After considerable investigation, they selected RH - 35 - D (the high melting modification of the R & H Department's new resin) as the binder for the ink with Solvent Naphtha (H-504) as the solvent. They formulated an ink using Hansa Yellow as the pigment which pleased the customer very much. The approximate composition was given to us as

Hansa Yellow	6.6%
RH-35-D	40.6%
Solvent Naphtha	52.8%
	<u>100.0%</u>

(Attempts in this laboratory to duplicate the above ink have cast some doubt on the accuracy of these figures).

The ink was prepared by pasting the yellow with part of the solvent and adding the resin dissolved in the remainder of the solvent. No grinding was necessary and the ink prints with a very desirable gloss.

This laboratory took up the problem with the idea of incorporating part or all of the resin with the pigment in the hope that it would greatly facilitate the mixing into an ink and possibly eliminate grinding where it would otherwise be necessary. Attempts were made to incorporate the resin by adding a solution in mineral spirits to the color in process, finishing by the usual development, filtering and drying, the solvent evaporating along with the water.

This treatment was applied to a lithol red by adding the

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resin solution in a colloid mill to the finished pigment and by adding the resin solution to the soda salt suspension in the vat with subsequent treatment as if it were not present. The treatment was also applied to BT-100-D by adding the resin solution just after adding the PTA solution to the dye and before the development at the boil. In each case the resin was incorporated and the dry product was somewhat harsh and brittle to the touch but softened very readily when added to solvent naphtha. There was good evidence that these products did in some cases show better dispersion in the solvent without grinding than did the untreated products. However, there was also evidence that complete dispersion would be very difficult to obtain without some grinding action such as a short ball mill cycle. When such a grinding action was used the effect of the resin in the pigment was no longer apparent.

There was also evidence that such a treatment did in some case result in a marked tinctorial change although in other cases this was not apparent.

It would appear that any resin could probably be incorporated in most pigments in some such manner as here described. It was, however, decided to drop this phase of the work and concentrate on a determination of the real merit of RH-35-D with a representative group of pigments and to formulate with each of them a satisfactory ink.

The first preparations made with a lithol red and with BT-100-D (PTA Blue) lead to the conclusion that each color would require a different formulation to obtain the desired gloss. This work was done in cooperation with the Technical Laboratory but attempts to duplicate it at Newark have lead to a new view of the problem which is more fully set forth in the following discussion of the work undertaken in January.

During this month two preparations of Para Brown have been made and have shown the process to have satisfactory reproducibility. A large sample is available for a K sample and for preparing an ink for examination by the customer who has expressed a desire for such a color.

The remainder of the work during the month has consisted of the preparation and testing of roto inks using RH-35-D resin as the binder and solvent naphtha (H-504) as the solvent. The following pigments have been examined:

- | | |
|---------------|--------------------------------|
| 1. RT-242-D | Duol Red |
| 2. RT-232-D | Toluidine Red |
| 3. RT-329-D | P.T.A. - Rhodamine 6 GDN |
| 4. YT-291-D | Hansa Yellow |
| 5. BT-125-D | P.T.A. - Victoria Pure Blue BO |
| 6. Para Brown | |

A small amount of time has also been given to the examination of other resins in comparison to RH-35-D.

The method of testing the inks is fully described under the Experimental Details. As more fully set forth there, our evaluation of the inks is limited in its accuracy because of the thick film of ink and slow rate of evaporation of the solvent on the cold printing

machine. In actual practise a much higher amount of solvent could undoubtedly be tolerated than the maximum permissible under the conditions which we are using for testing.

A quite exhaustive study has been made of the relations of the three ingredients of the inks using each of the six colors listed above. Contrary to our previous statements that each pigment would require a different formulation, the conclusion now appears unmistakable that a suitable relation between resin and pigment can be found which will apply to all of these pigments without material change. It appears that no gloss can be obtained when the pigment-resin ratio is below 1:4 and that about 6 parts of resin to one part of pigment are required to give the best results. Higher amounts of resin can be used but appear to be very impractical.

With the printing equipment available and using a pigment-resin ratio of 1:6 it is necessary to keep the solvent below 45% of the total and preferably below 40% to give the best detail of print and the highest gloss. As noted above, this factor of solvent content will certainly be influenced very largely by the thin film of ink used in typical roto work as well as by the very rapid evaporation on the heated presses.

As to the effect of different resins, we have some confirmation of the reported merits of the higher melting varieties of RH resins. The natural resins such as East India Gum and Damar, give viscous solutions with resulting good detail of print but are somewhat deficient in gloss. Other synthetics like Amberol and Gumar show fair gloss and give promise of being quite satisfactory when the solvent is properly adjusted. We are now inclined to the belief that RH-35-D (or RH-211) is probably the best of the resins so far tested. However, we further believe that other resins can be so formulated as to give results substantially comparable even if slightly inferior. We have ordered and plan to examine a number of commercially available products and to select certain of the best from this group to prepare inks for examination by our customer along with the contemplated inks from RH-35-D.

We are now proceeding to make up one gallon samples of the various colors noted above (omitting RT-329-D) using RH-35-D and we will use RT-232-D to make up similar samples using the various resins. These inks will consist of

Pigment	8.4
Resin	51.6
Solvent	40.0

we will hope to keep accurate records of the additional solvent, if any, used by the customer.

EXPERIMENTAL DETAILS

N.B.#493, P. 50-95.

Also - evaluation of inks as reported in December.

All evaluations of rotogravure inks as noted in this report have been carried out on a small textile printer with an engraved roll operating against a rubber roll. The engraving is quite deep in respect to the usual rotogravure etching. The ink is

distributed from a blade with a second "doctor" blade to remove the excess. The machine operates at a speed of about 20 feet per minute and there is no heat so that drying is by evaporation in the open air. As a consequence of this situation inks of relatively low solvent content are required to obtain a satisfactory gloss. With higher solvent contents, the solvent penetrates the paper and takes so much of the resin with it that the finish is quite flat. Also inks of low viscosity do not print a sharp, clear design when the engraving is deep. All tests have been carried out on a rotogravure paper obtained from The International Paper Co. Ratings have been based on the clearness or detail of the print, on the gloss and on the penetration of the paper.

493-5 series evaluations p. 25-27. These inks were prepared to duplicate the inks prepared in November at the Technical Laboratory of the Dyeworks and also to study the effect of grinding in a ball mill.

It is obvious that all the inks of this series have more solvent than was used in the earlier work. They are thin, give blurred prints with little gloss and considerable penetration.

Grinding very definitely improves the P.T.A. blues but appears to have very little effect on the Hansa Yellow and Lithol Red inks.

493-6 series evaluation p. 28-29. East India Gum vs. Cumar Resin with Hansa Yellow and Lithol Red.

In each case the East India Gum gives a more viscous ink and a sharp print but poor gloss. The Cumar resin gives a thin ink with a blurred print and poor gloss.

493-12 series evaluation p. 46-47. These inks were prepared from the brown pigments of 493-9 and 493-10 series.

"A" was from 493-9C Basic Brown P.T.A. and was a very poor ink. The pigment was hard and did not grind out. The other inks were from Copper Para Browns. Although all the prints were blurred and showed bad penetration, there was some suggestion of a gloss which gave promise of formulating a satisfactory ink.

493-13 series evaluation p. 48-49. This series consisted of the Cumar resin inks prepared according to Mr. Duhring's formula. They had no gloss and did not dry completely. The formula is certainly not applicable to the work at hand.

493 p. 50-51. An attempt was made to flush BT-100-D press cake in a mixture of RH-35-D in solvent naphtha (H-504) in a vacuum mixer arranged to recirculate the solvent.

Due to the high rate of evaporation of the solvent it came over much more rapidly than the water and recirculation proved to be well-nigh impossible. The work was stopped because of lack of time but the resulting very heavy paste still contained a large amount ($\pm$ 30%)

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of water but only about 12% of the original solvent. It has since been shown that the remaining water and solvent can be removed on a hot roller mill leaving a slightly plastic mass which solidifies on cooling and can be dispersed in the solvent by heating. The whole process appears, however, to be quite impractical and no further work is planned.

493-14. A study with variations in amount of color and amount of solvent using a Lithol Red as the pigment. This series seems to place an upper limit of 43-45% as the maximum solvent possible with the present testing equipment to give a print of satisfactory detail and gloss. The variation in pigment over the range selected seems to have little effect.

493-15. Clear varnishes with variation in solvent content. When printed, 40-45% solvent seems to be the maximum which is possible and still obtain the desired gloss.

493-16. A further study of Hansa Yellow inks on formulas previously used. No gloss was obtained and it is obvious the inks have more solvent than the ink obtained from the technical laboratory, as representative of the sample examined by their customer.

493-17. Formulation of Roto inks from RT-242-D using increasing amounts of pigment with variations in solvent content.

Where the solvent is kept below 45% a fair gloss results with fairly sharp prints over the whole range of the amount of pigment used. However, even these inks give too much penetration.

493-18. A study of the use of Alumina Hydrate to give gloss and of increasing the pigment still further

The Alumina Hydrate does not increase the gloss but rather tends to act as additional pigment which is further shown to give reduced gloss but better detail of print. To a ratio of 1 part pigment to 4 parts resin there is some gloss but at a 1:1 ratio there is none and the products are quite thixotropic and require more solvent to have a workable body.

493-19. A further study of pigment-resin ratios with RT-242-D varying from 1:1 to 1:3.75. Below 1:3 there is almost no gloss but there is good detail of print. With 1:3.75 the ink seems to be fairly satisfactory in all respects.

It is therefore tentatively concluded that the pigment binder ratio is the deciding factor as to gloss with the amount of solvent a function of the manner in which the ink is to be used, these conclusions assuming the use of RH-35-D as the resin.

493-20. Formulation of Roto inks from RT-232-D.

This series varied the pigment-binder ratio through equal steps from 1:1 to 1:8. The resin solution was made up to contain 8 parts of resin to 7 parts of H-504 except that additional solvent was necessary in the first two members of the series to give a workable body to the ink.

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Good detail of print is obtained up to a pigment-binder ratio of 1:6. However, there is no gloss below 1:4 and it requires at least 5 or possibly 6 parts of resin to give the desired gloss and freedom from penetration.

493-21. Formulation of Roto inks from RT-329-D.

The variables were the same as in 20 series and the conclusions are the same except that none of the inks from this series give the desired gloss.

493-22. Formulation of Roto inks from BT-125-D.

The same variables and the same conclusions apply as in 493-20 series.

493-23. Formulation of Roto inks from YT-291-D.

The same variables were applied as in 493-20. However, there was a much greater thickening effect at the lower ratios than in the previous series. Even these inks left something to be desired.

493-24. Solvent variable with YT-291-D using a pigment binder ratio of 1:6. It is conclusively demonstrated that such an amount of resin will give a print which is excellent in every respect if the solvent is sufficiently low (below 40%).

493-25. A study of the use of different resins using RT-232-D with a pigment binder of 1:6 and a resin solution containing 8 parts resin to 7 parts of H-504.

	<u>Resin</u>	<u>Detail of Print</u>	<u>Gloss</u>	<u>Remarks</u>
A	RH-35-D	Good	Good	
B	RH-35	Poor	Poor	Low melting variety
C	RH-211	Good	Excellent	Higher melting than RH-35-D
D	East India Bold	Good	Fair	Required additional solvent to grind
E	Gum Damar	Good	Slight	
F	East India Fossil Gum	Good	Slight	
G	Elemi Gum	Poor	None	
H	Amberol Resin	Good	Very Slight	Required additional solvent to grind
I	Manila Bold	-	-	Insoluble - no tests
J	Cumar $\frac{1}{2}$	Poor	Very Slight	
K	" $\frac{1}{2}$	Poor	Slight	
L	Ester Gum	Poor	Slight	
M	I Rosin	Poor	None	

The high melting RH resins gave the best prints in every respect. The natural resins gave good detail but low gloss. The other synthetics as Amberol and the Cumars were promising and should be studied with a view to adjusting to the desired properties if possible. The Elemi Gum and the rosins were quite unsatisfactory.

493-26. A large sample of Para Brown was prepared on a formula very much like that used in 493-10B. The products check the previous work very closely.

493-27. Solvent variable on RT-242-D

493-28. Solvent variable on RT-232-D

493-29. Solvent variable on RT-329-D

493-30. Solvent variable on BT-125-D

These four series are like that of 493-24 in varying the solvent on a pigment-resin ratio of 1:6. The same conclusions hold in every case, that a sufficiently low solvent content (below 40%) gives an excellent print of good detail and gloss.

493-31. The preparation of Roto inks from 493-26 - Para Browns.

The same variable as used in 493-20 was applied (variation in pigment-resin ratio). The same conclusions also apply. 6 parts of resin to 1 of pigment are necessary for good gloss.

493-32. Solvent variable on Para Brown.

See 493-27 to 30 above for method and conclusions.

493-33. Solvent variable using Cumar W $\frac{1}{2}$ with RT-232-D.

A very respectable print is obtained with solvent below 40% but the best in this series appears to be somewhat less glossy than the best obtained from RH-35-D.

493-34. An additional large sample of Para Brown was prepared as in 493-26 series and four samples are very good checks. This material will be used to prepare a large sample of ink for examination by a customer.

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PIGMENT COLOR RESEARCH REPORT

SPECTROPHOTOMETRIC STUDY - CHROME YELLOWS

SUBMITTED BY: *V. Chalupski & H. Young*

FILE: PHYSICAL STUDIES
DATE: JAN. 22nd, 1936.

INTRODUCTION

Spectrophotometric measurements have been made of a large number of paper mounts ("skindowns") of inks prepared from Krebs Chrome Yellow standards, various mixes, and several competitive Cadmium Yellows. C.I.E. tristimulus specifications were computed from the spectrophotometric curves, and these results, as well as the curves, have been carefully examined.

SUMMARY AND CONCLUSIONS

This study gave no unexpected results as to shapes of curves or the tristimulus color specifications. All of the curves are of the usual form for Chrome Yellows, showing strong absorption (even for the thin ink films examined) in the violet, blue, and green-blue, reflecting less than ten percent of the incident light in this region; in the green-yellow, yellow, orange and red, reflectance is high, about 75-85 percent. Spectrophotometric data agrees with visual observation in a qualitative way.

The influence of quality of the pigment preparation and mount on colorimetric measurements was observed, particularly in the case of mounts of identical inks mounted on different paper. It is probable that "skindown" mounts are not well suited for colorimetric measurements because of difficulty of duplication.

The hue range of chrome yellows runs from 582.5 to 573.0 millimicrons dominant wave length. The sensibility of the eye to hue change at the average excitation purity of chrome yellows (74-78 percent) may be roughly estimated at 0.1 millimicron in dominant wave length. (According to extensive data in the literature, the sensibility of the eye to wave length change is at a maximum in the vicinity of 580 millimicrons. There is another maximum at 490).

Because of the similarity of the curves of all the yellows examined, mixes of various of these are very similar to the intermediate yellows in visual appearance and shape of curve.

The curves of mixes fall between the curves of the individual components of the mixes, in an approximate way. Deviations from this rule are attributed to non-uniformity of ink film thickness in the skindowns.

The three Cadmium Yellows examined are similar to chrome Yellows in their color characteristics but are all higher in purity than corresponding chrome yellows. The spectral curves show more complete absorption in the absorption bands (less than seven percent)

and somewhat steeper slopes than chrome yellows of approximately the same hue.

DETAILS

The Yellows examined fall into three groups:-

I. Chrome Yellow Standards (Krebs)

A. Regular ("nitrate") Yellows. (The following tabulation is arranged in order of hue).

1. Y-359-D (reddest)
2. Y-190-D
3. Y-309-D (high finish)
4. Y-171-D
5. Y-180-D
6. Y-303-D
7. Y-305-D (high finish)
8. Y-315-D (" ") (greenest)

B. Lemon Yellows.

9. Y-318-D
10. Y-202-D

II. Chrome Yellow Mixes.

Mixes of various members of the IA group to match other members of the same group. Mixes were made of standards one step apart to match the intermediate standards; a mix was also made of the extremes to match a standard in the middle of the range. Several mixes were made of standards two or three steps apart.

III. Three competitive Cadmium Yellows.

Visual examination of the Yellows were made before the mounts were measured spectrophotometrically; these results were reported in a letter (V.C. to A.W. Kenney 1/18/35), a copy of which is appended to this report.

It should be kept in mind that all measurements discussed were made on "skindowns"; masstones and tints were not considered in this study. Skindowns were prepared in the usual manner, by scraping a thin film of the yellow inks (made from the pigments ground with litho varnish) over two kinds of paper - bond paper and litho label stock. Mounts of the latter are designated by the subscript "A".

CHROME YELLOW STANDARDS

Skindowns of the ordinary types of Chrome Yellows show an excitation purity between 74.51% and 76.83%, a brightness factor of 43.4% - 48.07%, and vary in dominant wave length between 582.48 (medium) and 573.0 millimicrons (primrose). Y-309-D has an abnormally high purity, 78.39%.

(A) Dominant Wave Length. The classification of chrome yellows according to their dominant wave lengths rather than their "shade" is an unfamiliar one. The visual color differences, as graded by the pigment makers' ordinary methods, are compared below with the dominant wave length differences.

Standards	Dominant Wave Lengths	Wave Length Differences	Visual differences
Y-359-D -Y-190-D	582.5 - 578.1	4.4	Y-190-D is very much greener.
Y-190-D -Y-171-D	578.1 - 577.4	0.7	Y-171-D is slightly greener.
Y-171-D -Y-180-D	577.4 - 576.6	0.8	Y-180-D " " "
Y-180-D -Y-303-D	576.6 - 575.5	1.1	Y-303-D is much greener.
Y-303-D -Y-305-D	575.5 - 573.6	1.9	Y-305-D is very much greener.
Y-305-D -Y-315-D	573.6 - 573.0	0.6	Y-315-D is slightly greener.

Y-309-D (577.6) appears slightly greener than Y-190-D (578.1) and very slightly redder than Y-171-D (577.35); because of its high finish, Y-309-D looks more brilliant or stronger than the others.

From this tabulation, the sensitivity of the eye to hue change in this range is estimated to be in the order of 0.1 millimicron; shipping limits would require standardization to within about twice this limit.

(b) Purity and brightness. The variation in purity may be largely accidental, due to variations in quality of the mounts which were measured. Although every effort was made to make these uniform, noticeable visual differences were often seen in duplicate pulldowns. Accidental variations might be expected in which purity would go up and brightness go down, or vice versa, corresponding to a thicker or thinner ink film. There is a gradual increase in brightness as we go from the redder to the greener yellows; roughly, the brightness increases with increasing lead sulphate (and decreasing lead chromate) content:-

Code	Brightness	percent lead sulphate
Y-359-D	53.52	0
Y-190-D	64.06	28%
Y-171-D	66.00	31
Y-180-D	67.47	42
Y-303-D	68.91	48
Y-305-D	72.97	44
Y-315-D	74.55	52

The spectrophotometric curves (Figure 1)* show this as a widening of the absorption band with increasing percentage of lead chromate.

*Figure 1 follows.

REFLECTANCE

100
80
70
60
50
40
30
20
10
0

400 420 440 460 480 500 520 540 560 580 600 620 640 660 680

WAVELENGTH

Fig. 1

Chrome Yellow Standards

- 1 - Y-359-D
- 2 - Y-190-D
- 3 - Y-309-D
- 4 - Y-171-D
- 5 - Y-180-D
- 6 - Y-303-D
- 7 - Y-305-D
- 8 - Y-315-D

A tabulation of calculated Dominant Wave Length, excitation, purity, and brightness factor for the various yellow standards is given below.

Standard Code	Dom. Wave Length	Excitation	
		Purity	Brightness
Y-359-D	582.48	76.36	53.52
Y-190-D	578.13	74.61	64.06
Y-309-D	577.63	78.39	65.50
Y-171-D	577.35	76.83	66.00
Y-180-D	576.64	76.76	67.47
Y-303-D	575.53	75.80	68.91
Y-305-D	573.55	76.94	72.97
Y-315-D	573.00	75.02	74.55

An examination of the spectrophotometric curves in figure 1. shows nothing unexpected in their general characteristics. The shapes of all the curves are similar, showing a strong absorption band in the violet and blue, and having a fairly sharp cut off between 500 (blue-green) and 540 (yellow-green).

The curves for Y-305-D and Y-315-D show lower reflectance in the blue than any of the other yellows. If this is not caused by a accidental variation in quality of the mounts, it would indicate that there is room for improvement in the other yellows in the blue end of the spectrum. The curves are approximately parallel, except for the red end of the Y-359-D curve; the arrangement and spacings are about what would be expected from visual examination.

Lemon Yellows

A tabulation of the two "lemon yellows", Y-202-D and Y-318-D, compared with two regular yellows of similar hue is shown below.

Standard Code	Dom. Wave Length	Excitation	
		Purity	Brightness
Y-180-D	576.64	76.76	67.47
Y-318-D	575.73	78.47	67.69
Y-202-D	575.33	73.03	65.81
Y-303-D	575.53	75.80	68.91

Lemon Yellows are Chrome Yellows differing from the ordinary type in their working properties when made into printing inks because of their crystal form and particle size. Y-202-D is a "pure" lemon yellow, while Y-318-D is intermediate between a lemon and a regular type yellow.

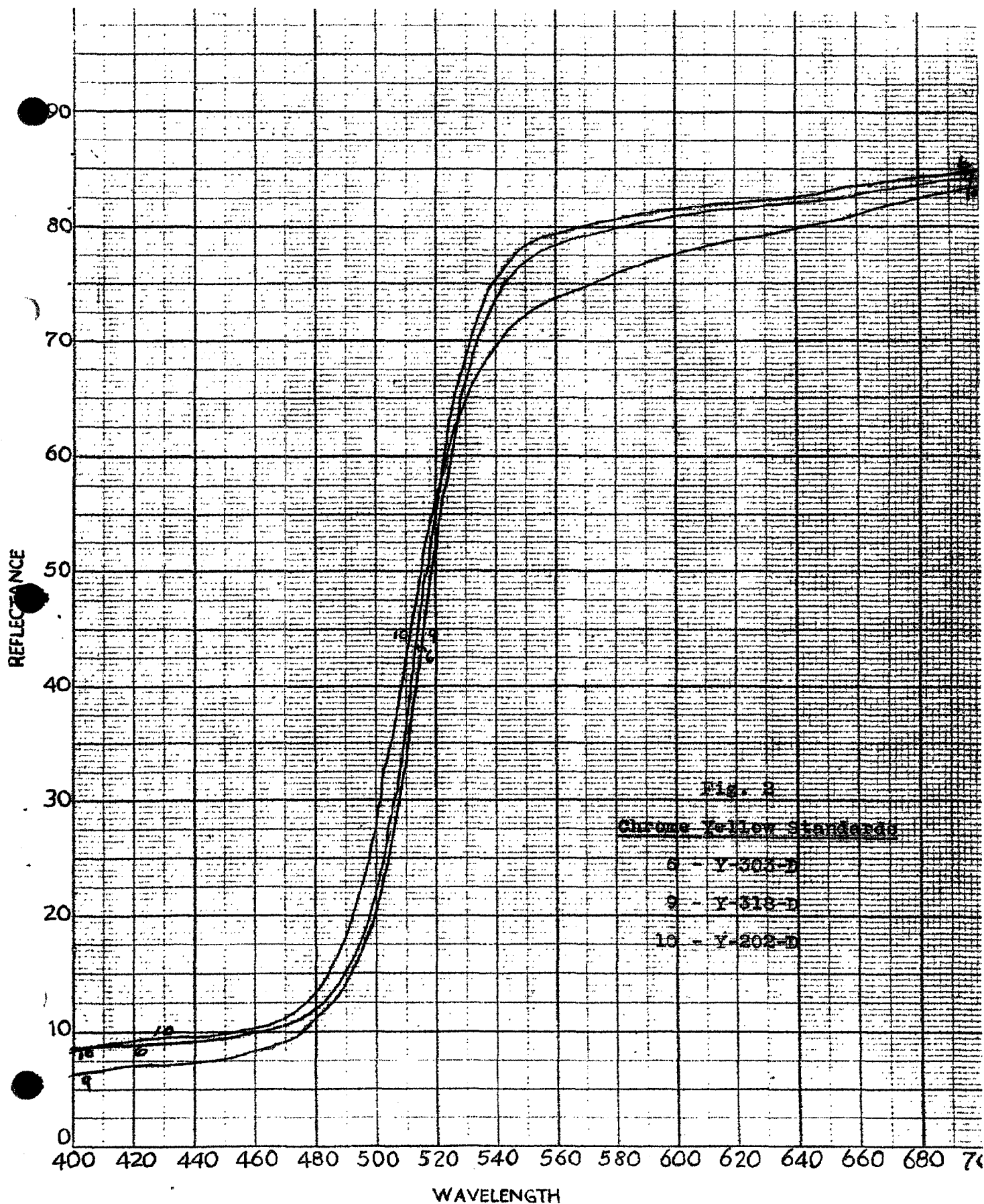
Spectrophotometric and visual examination of these mounts do not show the expected superiority of the lemon yellows; this may be attributed to the method of preparation of the mount.

Examination of inks made on an ink mill and printed on a proofing press would probably show the lemon yellows to better advantage.

Figure 2* shows the curves of the two lemon yellows.

Three Cadmium Yellows (Cadmium Sulphide) -C-31670, 31671, 31672 - were examined. A tabulation of the tristimulus specifications is given below; several chrome yellows of similar hue are included.

*Figure 2 follows.



g

	<u>Dom. Wave Length</u>	<u>Excitation Purity</u>	<u>Brightness</u>
C-31671	577.39	82.91	64.48
C-31672	574.84	81.15	70.51
C-31670	573.80	78.62	72.80
Y-171-D (chr. Yellow)	577.35	76.83	66.00
Y-305-D " "	573.55	76.94	72.97

Figures 2A* and 2B* show the greater steepness in the curves and lower reflectance in the blue and violet for the Cadmium Yellows as compared to Chrome Yellows.

Figure 3* shows the chromaticity of all of the yellows discussed above plotted on the C.I.E. coordinates.

Effect of Quality of Paper

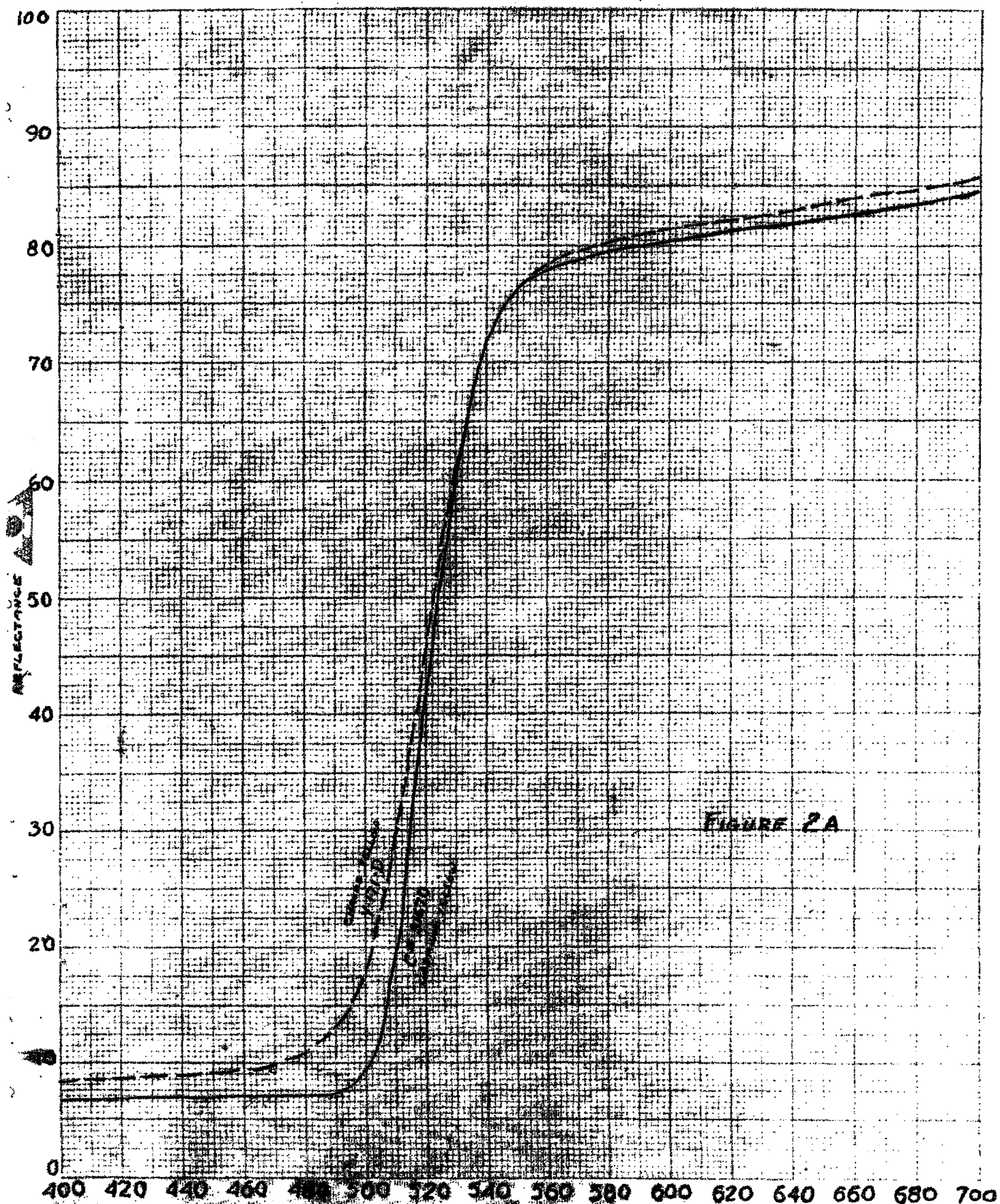
As discussed previously, "skindowns" were made on two kinds of paper. A comparison of these is shown in the following tabulation.

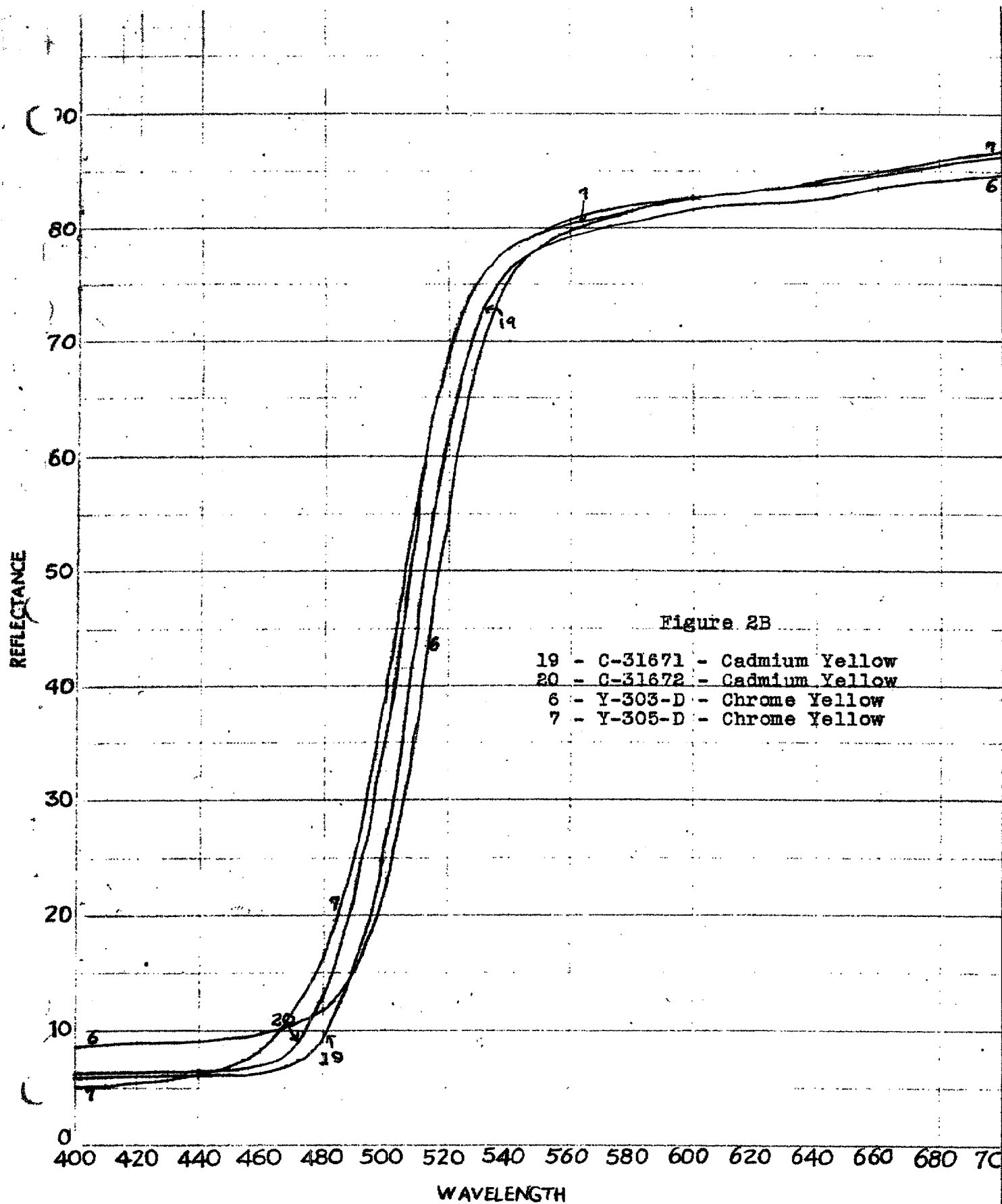
<u>Curves</u>	<u>Standard Code</u>	<u>Paper</u>	<u>Dom. Wave Length</u>	<u>Excitation Purity</u>	<u>Brightness</u>
1	Y-359-D	bond	582.48	76.36	53.52
1A	"	litho label	581.48	80.06	57.32
2	Y-190-D	bond	578.13	74.51	64.06
2A	"	litho label	574.27	69.03	74.13
3	Y-309-D	bond	577.63	78.59	65.50
3A	"	litho label	576.05	80.15	69.49
4	Y-171-D	bond	577.35	76.83	66.00
4A	"	litho label	573.61	77.62	73.18
5	Y-180-D	bond	576.64	76.76	67.47
5A	"	litho label	573.77	73.53	75.27
6	Y-303-D	bond	575.53	75.89	68.91
6A	"	litho label	574.25	77.31	73.32
7	Y-305-D	bond	573.55	76.94	72.97
7A	"	litho label	571.98	72.06	78.46
8	Y-315-D	bond	573.00	75.02	74.55
8A	"	litho label	571.99	73.21	78.30
9	Y-318-D	bond	575.73	78.47	67.69
9A	"	litho label	574.79	80.85	71.64
10	Y-202-D	bond	575.33	73.03	65.81
10A	"	litho label	574.47	71.15	71.17

* Figures 2A and 2B follow

* " 2 follows

* " 3 "





In the above data the difficulty in preparing satisfactory uniform "skindowns" on the smooth surfaced litho label stock must be taken into consideration; the conclusions drawn are therefore tentative. The following table shows the differences found between the bond and litho label mounts.

<u>Differences (litho label vs bond)</u>			
	<u>Dom. Wave. Length</u>	<u>Purity</u>	<u>Brightness</u>
Y-359-D	- 1.00	+ 3.70	+ 3.80
Y-190-D	- 3.86	- 5.48	+10.05
Y-309-D	- 1.58	+ 1.76	+ 3.99
Y-171-D	+ 3.74	+ 0.79	+ 7.18
Y-180-D	- 2.87	- 3.23	+ 7.80
Y-303-D	- 1.28	+ 1.51	+ 4.41
Y-305-D	- 1.57	- 4.88	+ 5.49
Y-315-D	- 1.01	- 1.81	+ 3.75
Y-318-D	- 0.94	+ 2.38	+ 3.95
Y-202-D	- 0.86	- 1.88	+ 5.36

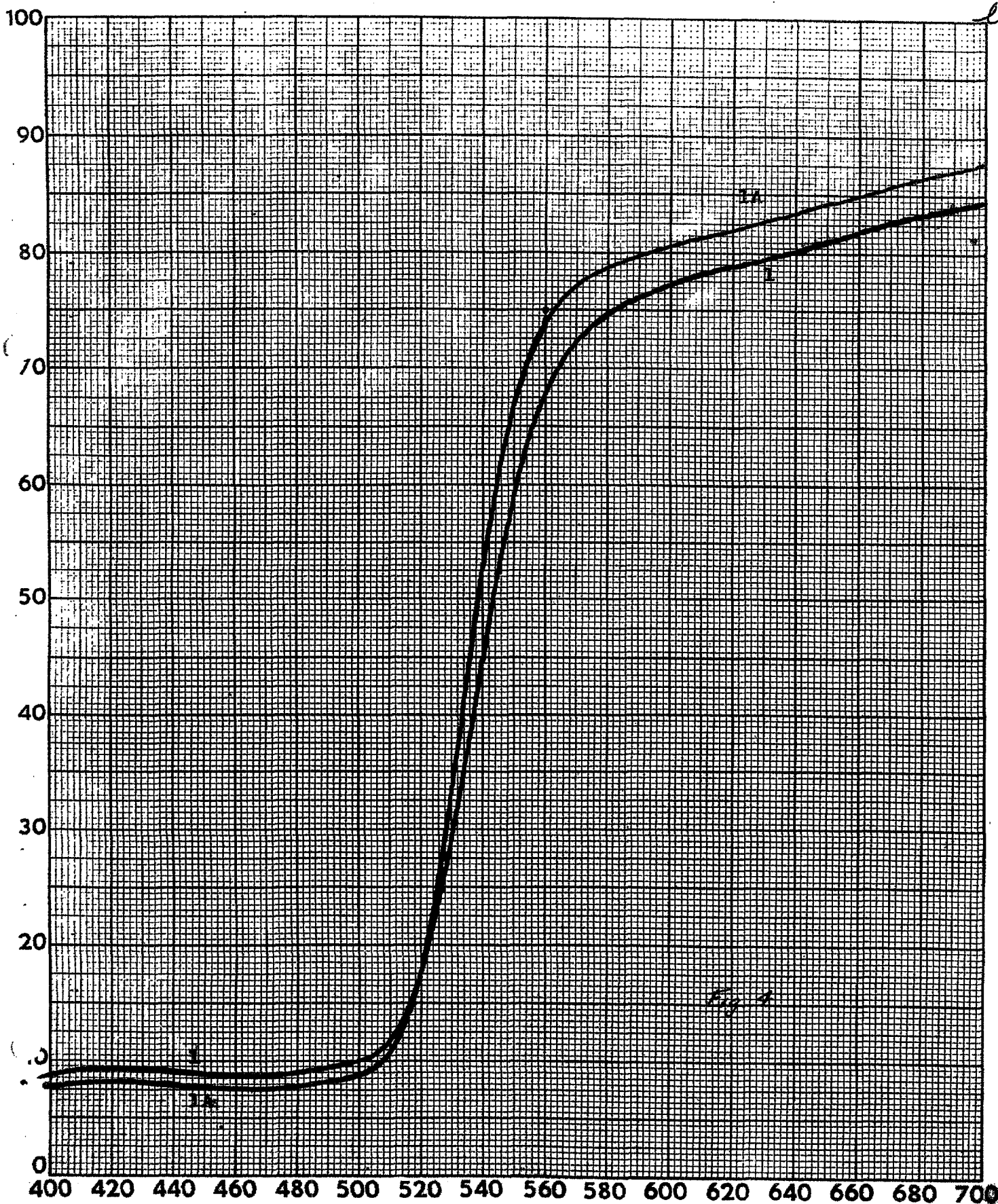
The dominant wave length is shifted to the green side in all cases. Excitation purity differences vary erratically, probably depending on the quality of the mounts (i.e., the effect of more or less of the white paper showing thru the ink film). Brightness is increased in all cases by the use of the litho label stock.

The above data emphasizes the importance of the type of pigment mount on the color of the pigment preparation.

Curves for all these yellows are shown in figures 4 to 13* inclusive; in each case the subscript "A" indicates the litho label skindown.

Fig. 4	Curve #1	Y-359-D
5	2	Y-190-D
6	3	Y-309-D
7	4	Y-171-D
8	5	Y-180-D
9	6	Y-303-D
10	7	Y-305-D
11	8	Y-315-D
12	9	Y-318-D
13	10	Y-202-D

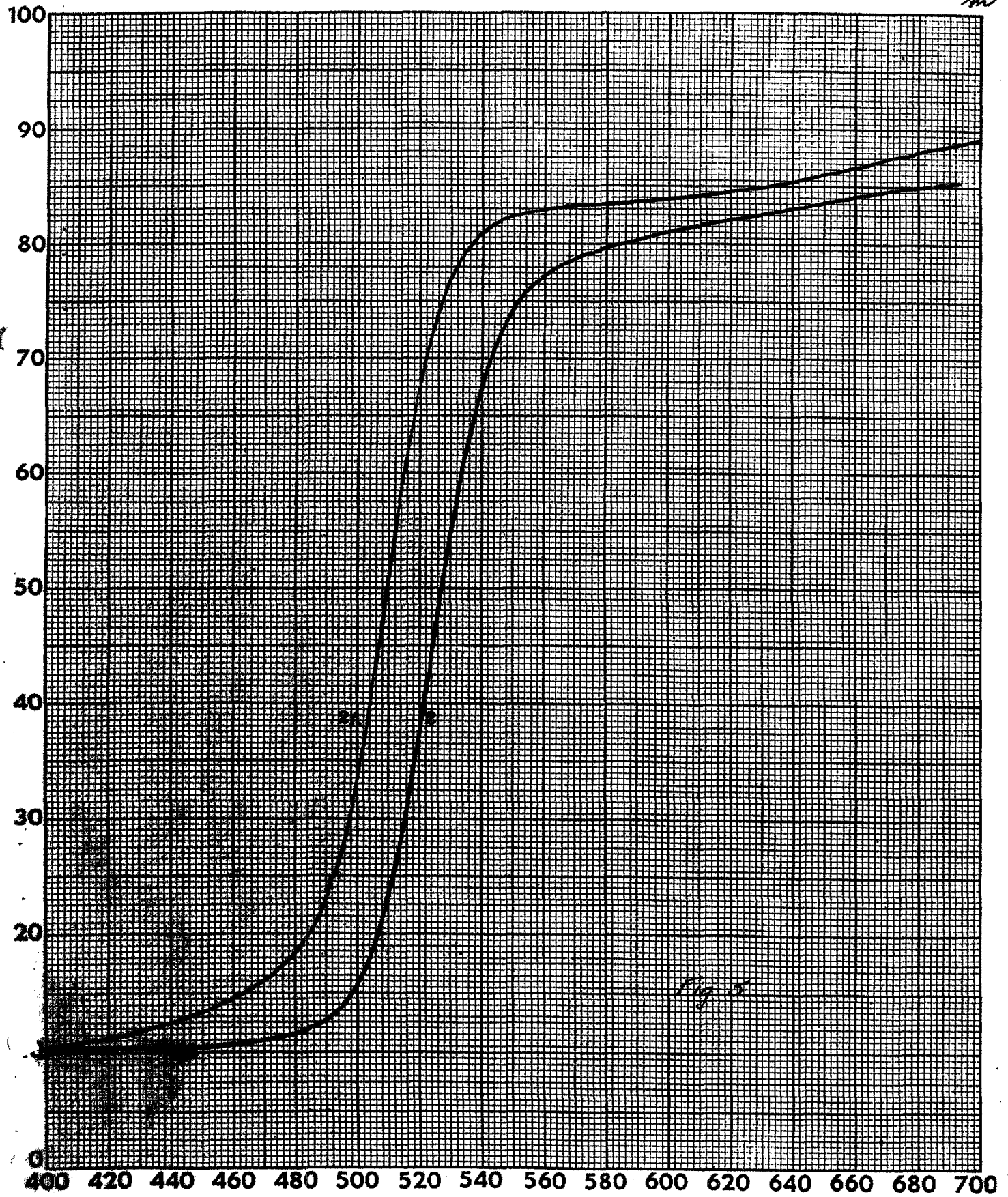
* Figures 4 - 13 Follow.



Date April 6, 1938

WAVELENGTH

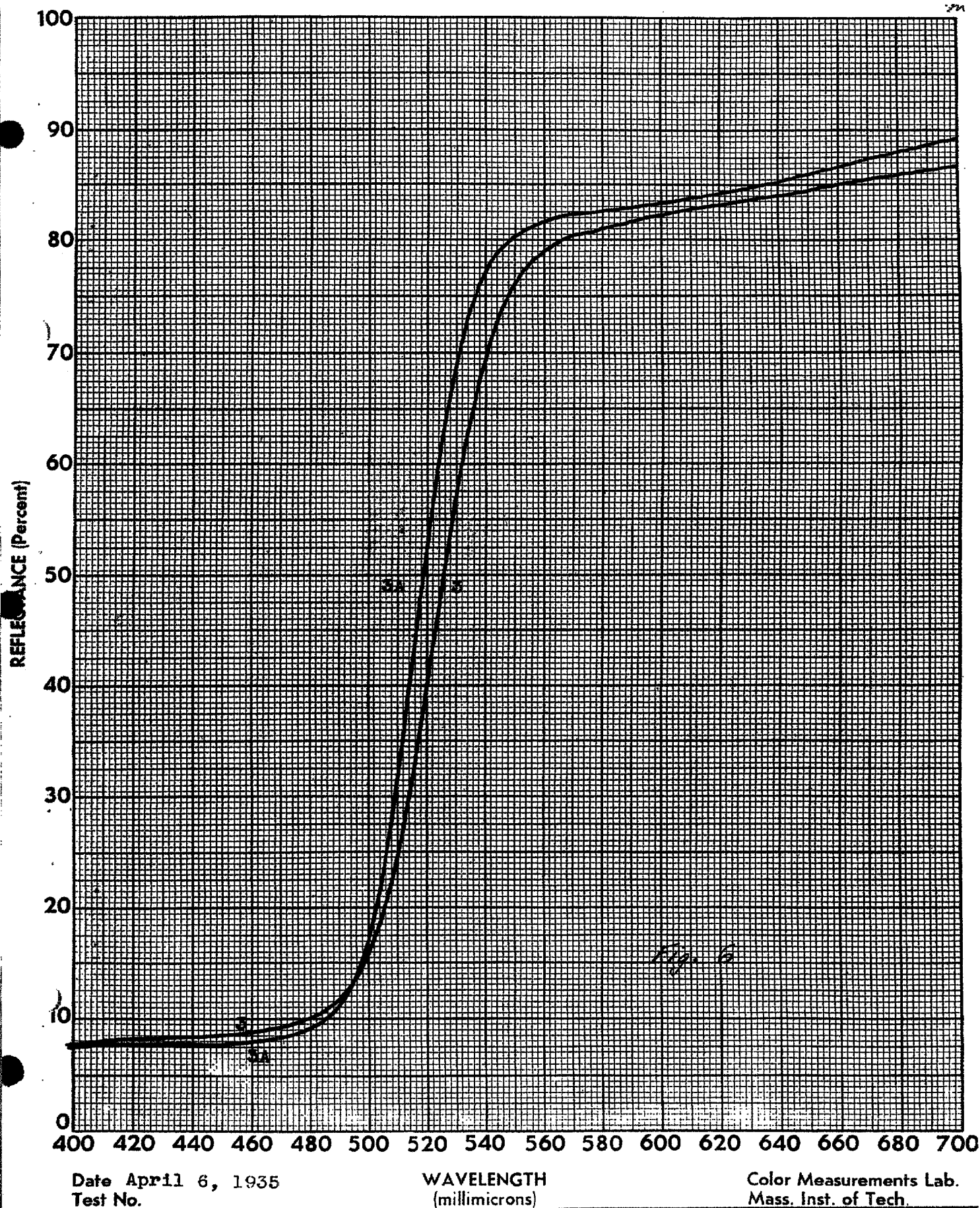
Color Measurements Lab

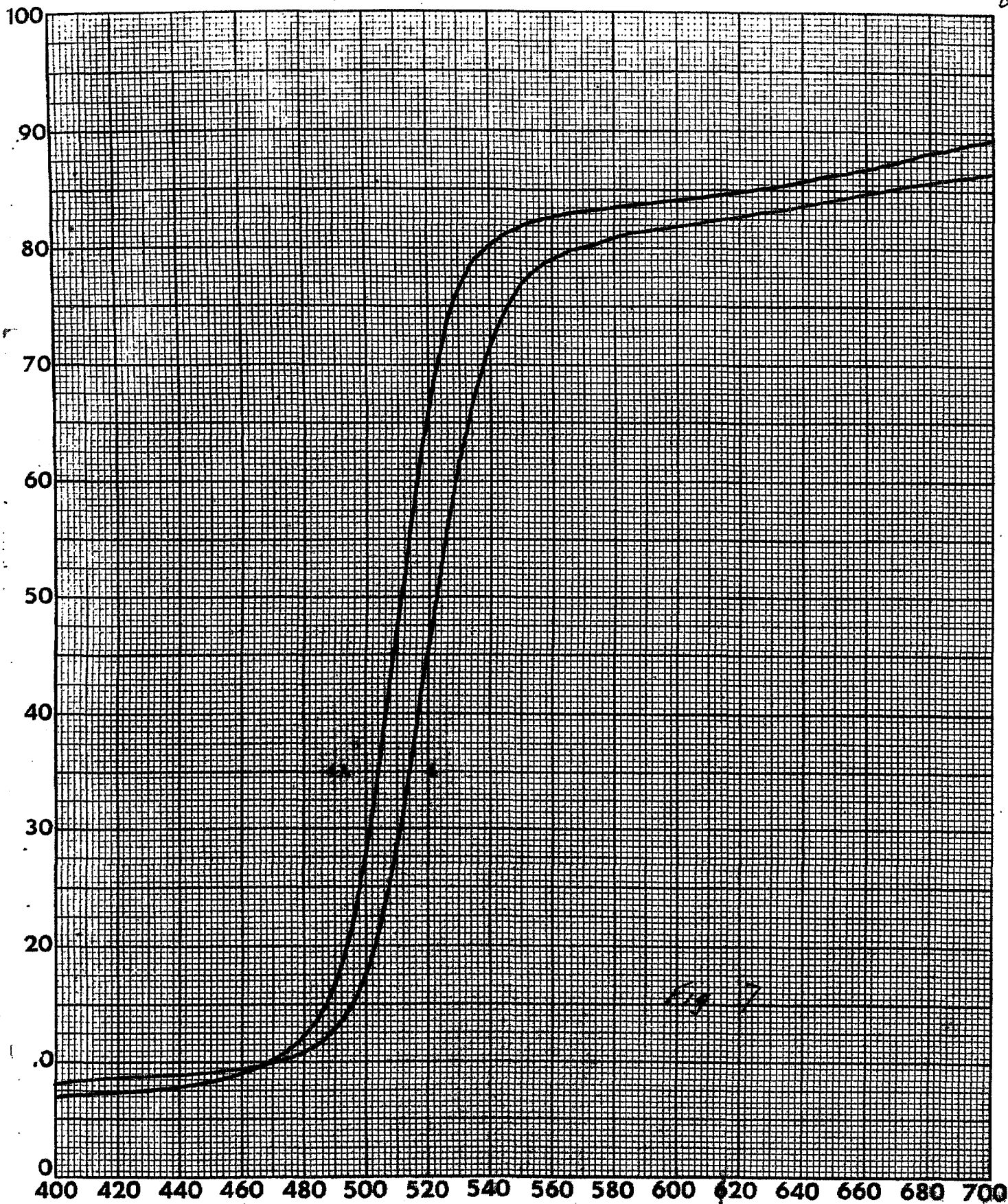


Date April 6, 1935

WAVELENGTH

Color Measurements Lab.

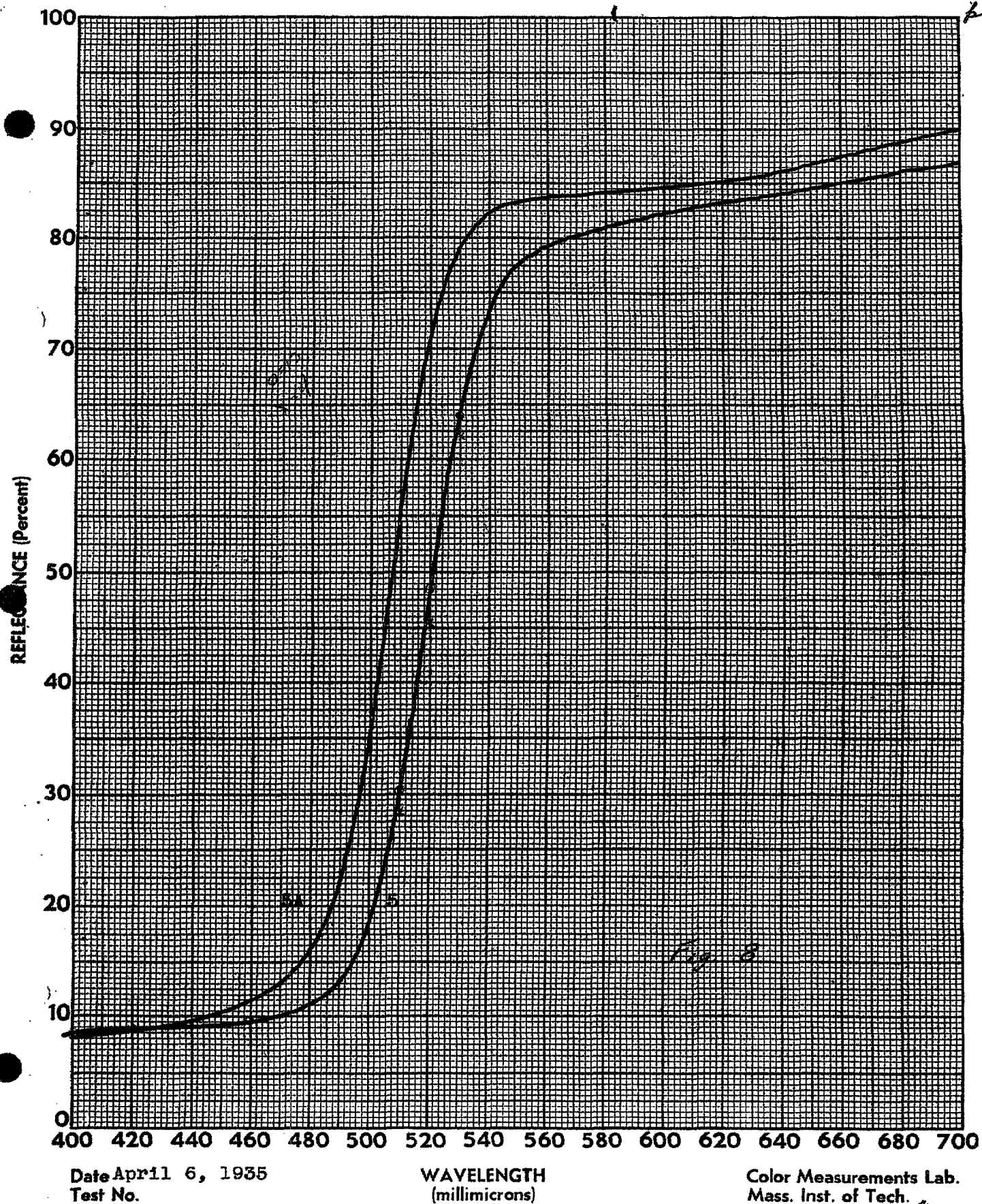


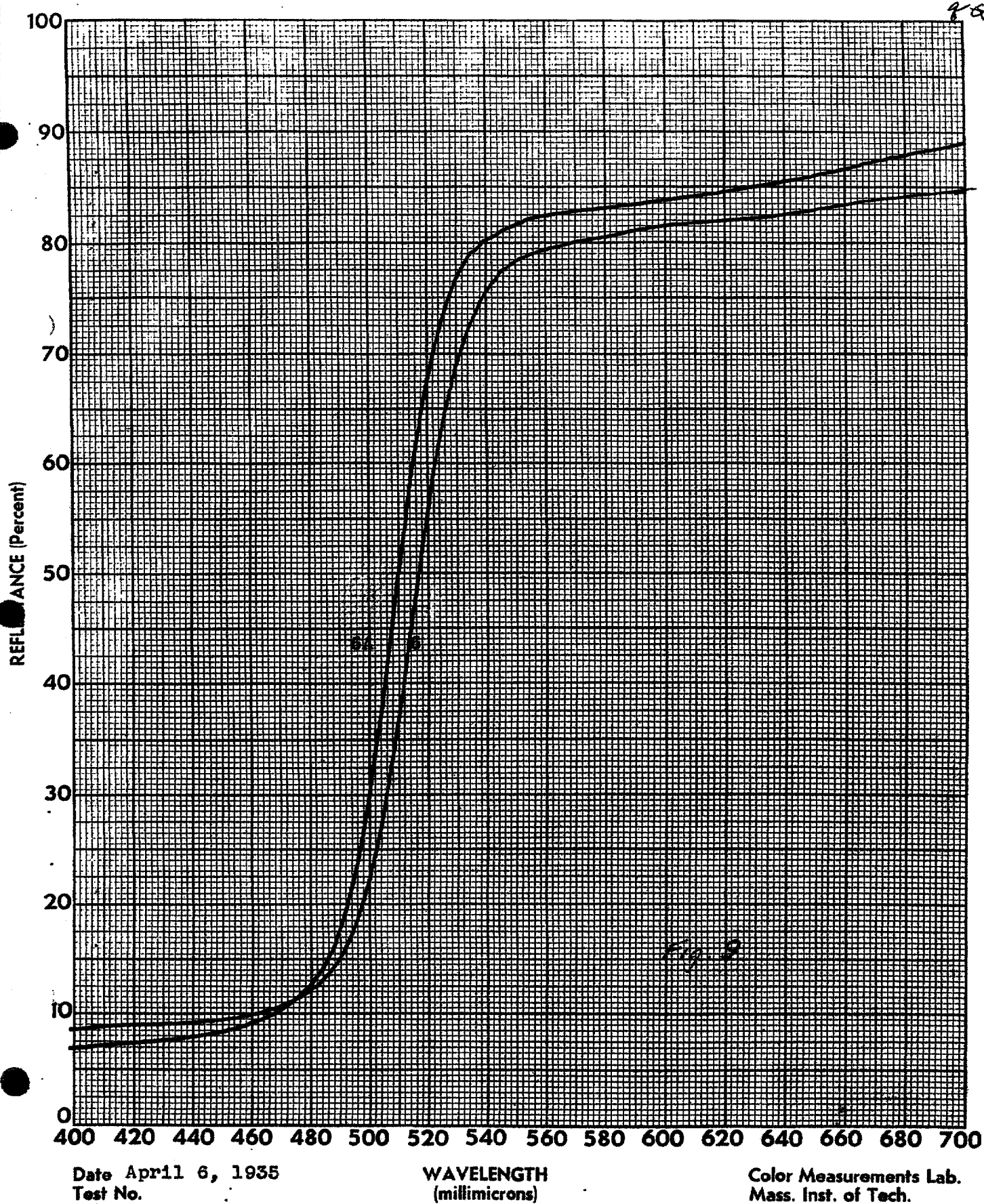


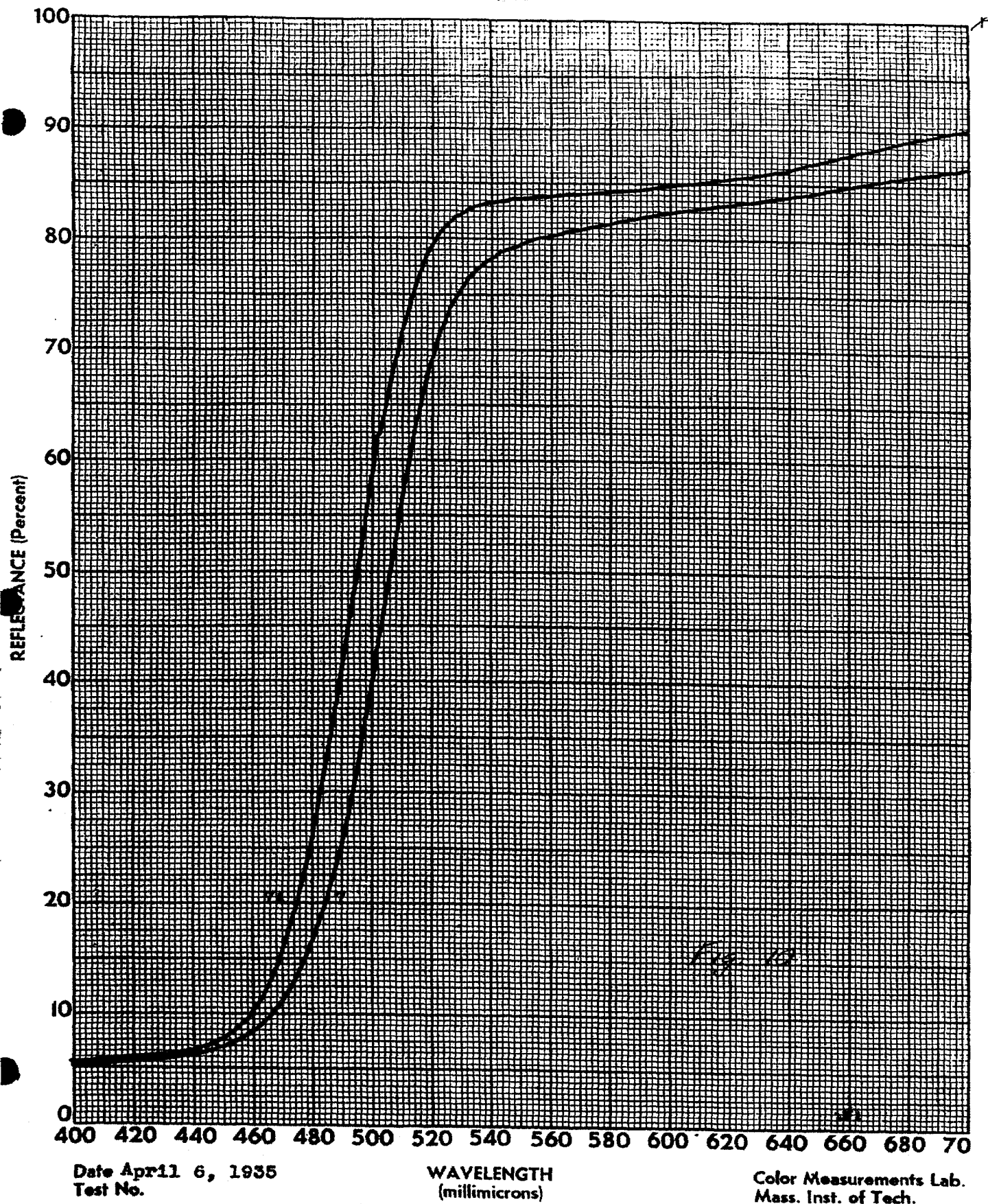
Date April 6, 1935

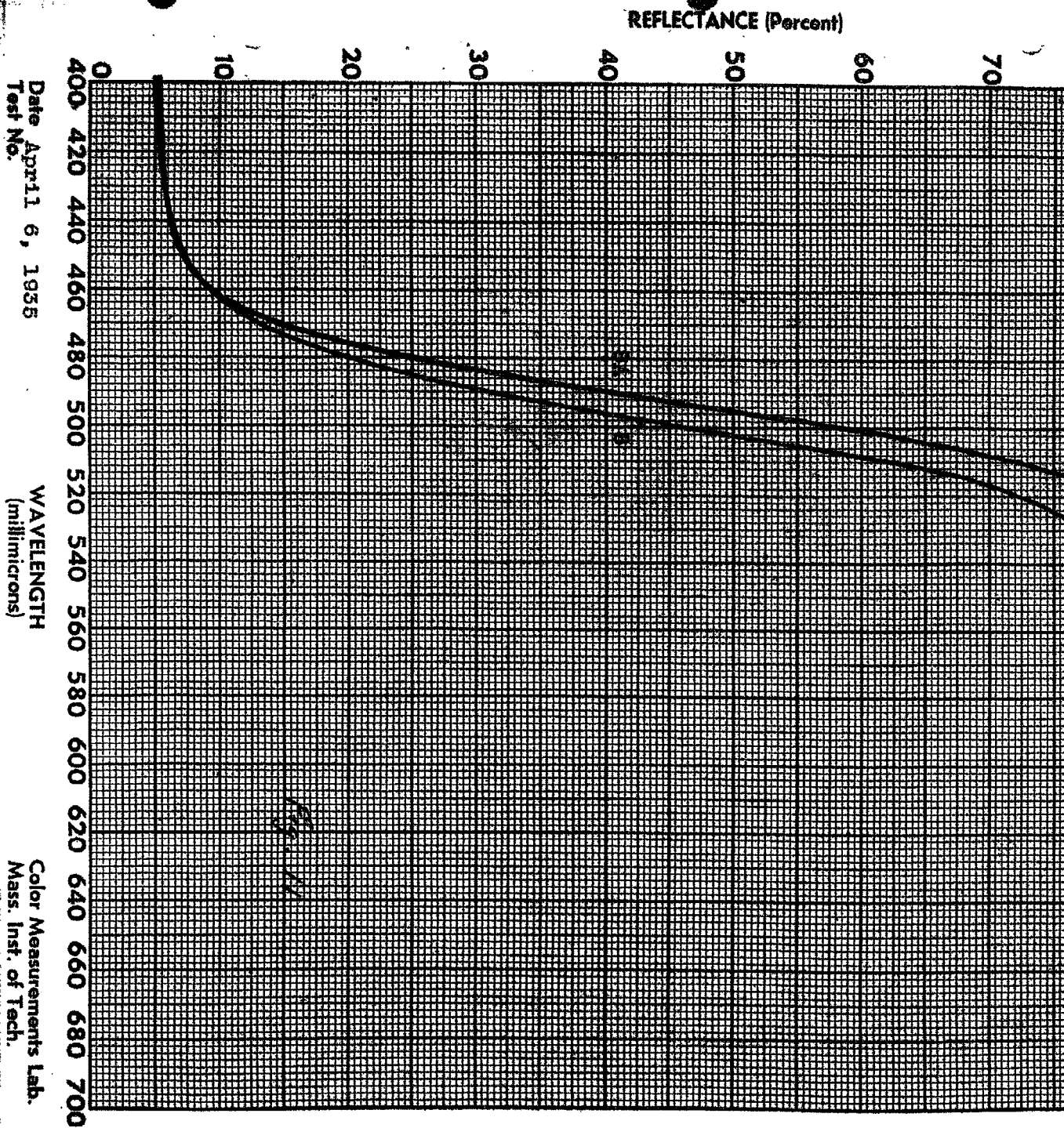
WAVELENGTH

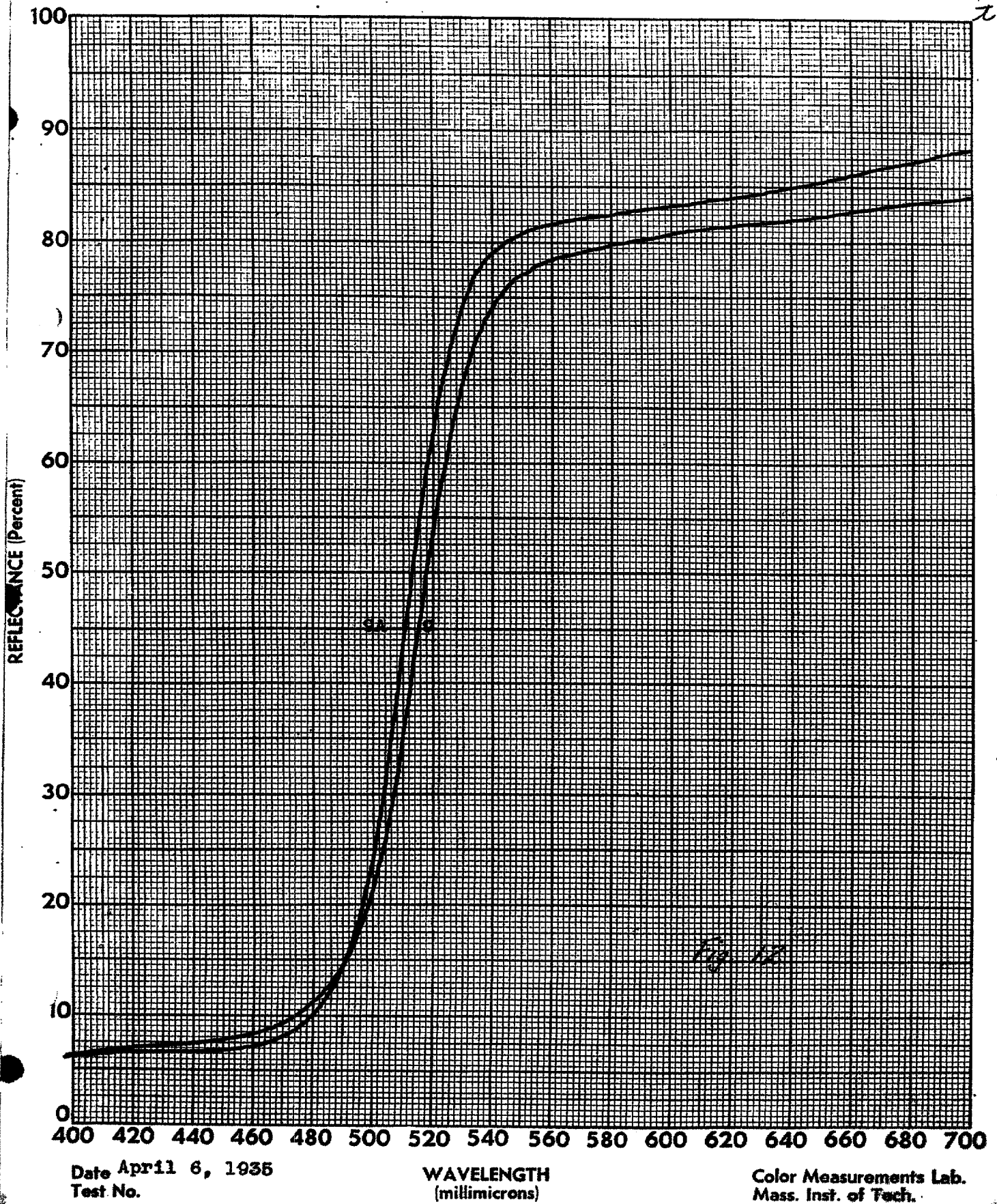
Color Measurements Lab.

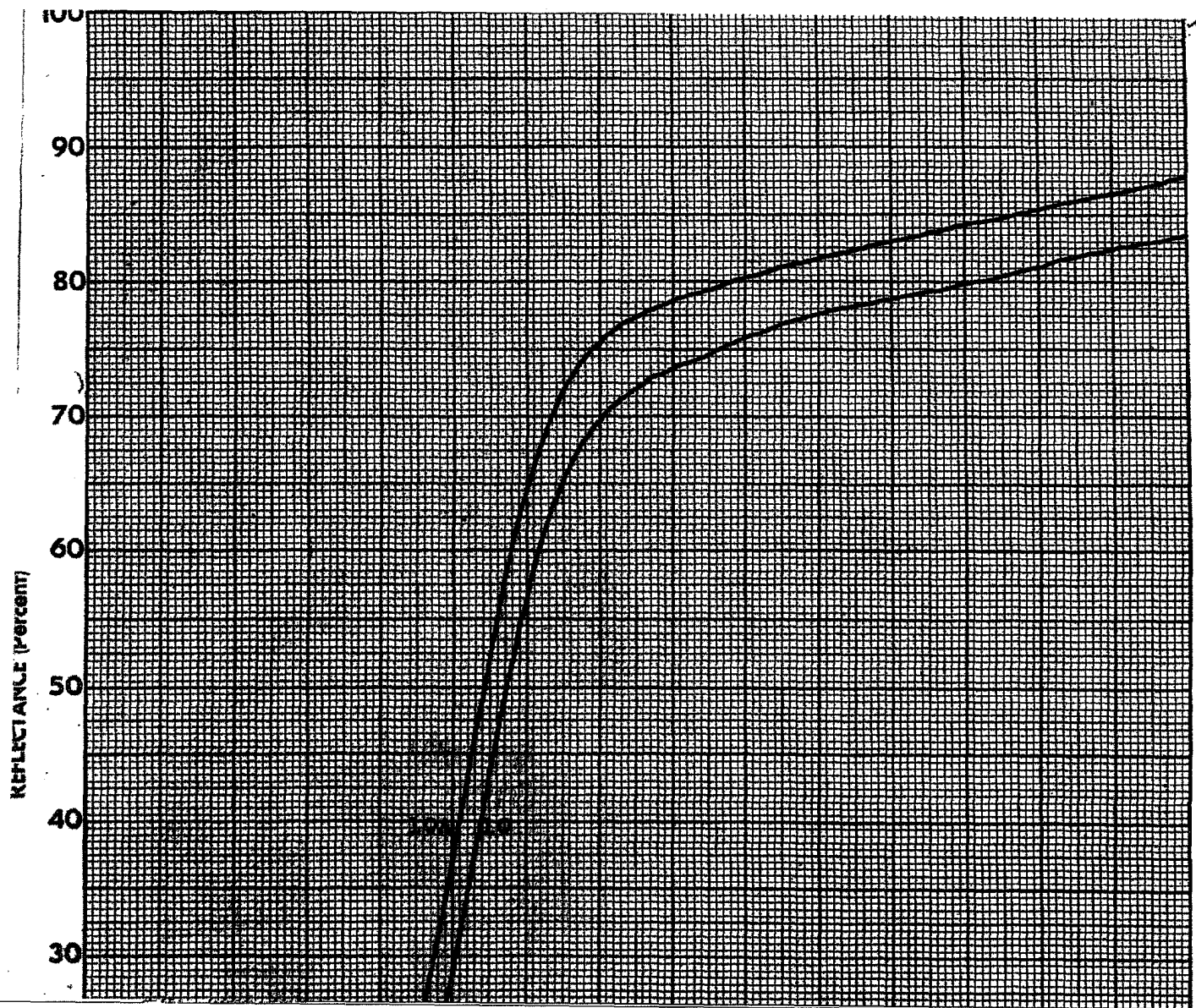






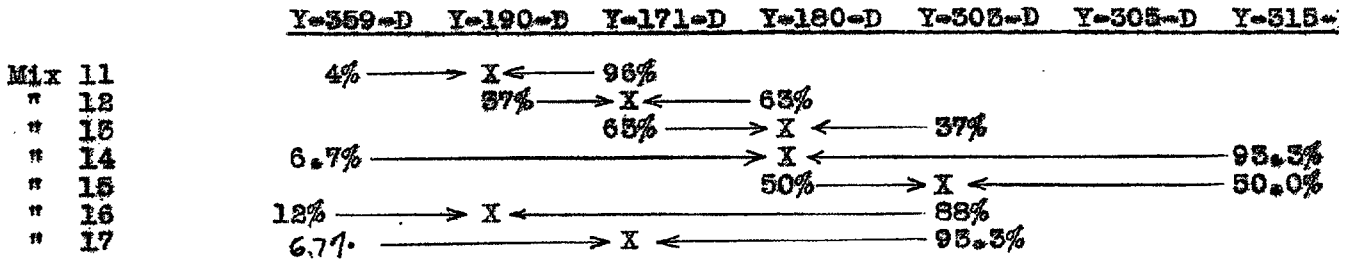






CHROME YELLOW MIXES

Seven mixes of standard yellows were made to match intermediate shades. The composition of these mixes is shown graphically below.



Mix No.	Mixes of	Dominant Wave Length		Purity		Brightness		Mix versus standard Descriptive difference from Spectrophotometric data		Difference - Visual Examination	
		Std.	Mix	Std.	Mix	Std.	Mix	Std.	Mix	Std.	Mix
11	Y-359-D Y-171-D	577.63	578.13	73.91	74.51	64.71	64.06	trace green; trace weak	v.v.s.e strong		
12	Y-190-D Y-180-D	576.96	577.35	75.99	76.83	67.98	66.00	trace green; trace weak	s. weak		
13	Y-171-B Y-303-D	576.68	576.64	76.57	76.76	67.28	67.47	close	v.s. green		
14	Y-359-D Y-315-B	575.89	576.64	76.54	76.76	70.03	67.47	trace green and clean	v.s. green and weak		
15	Y-180-D Y-315-D	575.54	575.53	77.96	75.80	68.93	68.91	trace strong	v.s. strong		
16	Y-359-D Y-303-D	576.00	578.13	78.23	74.51	70.20	64.06	slightly green and stro	v.s. clean		
17	Y-359-D Y-303-D	577.87	577.35	76.29	76.83	65.20	66.00	trace red	s. weak		

In general, it may be concluded:-

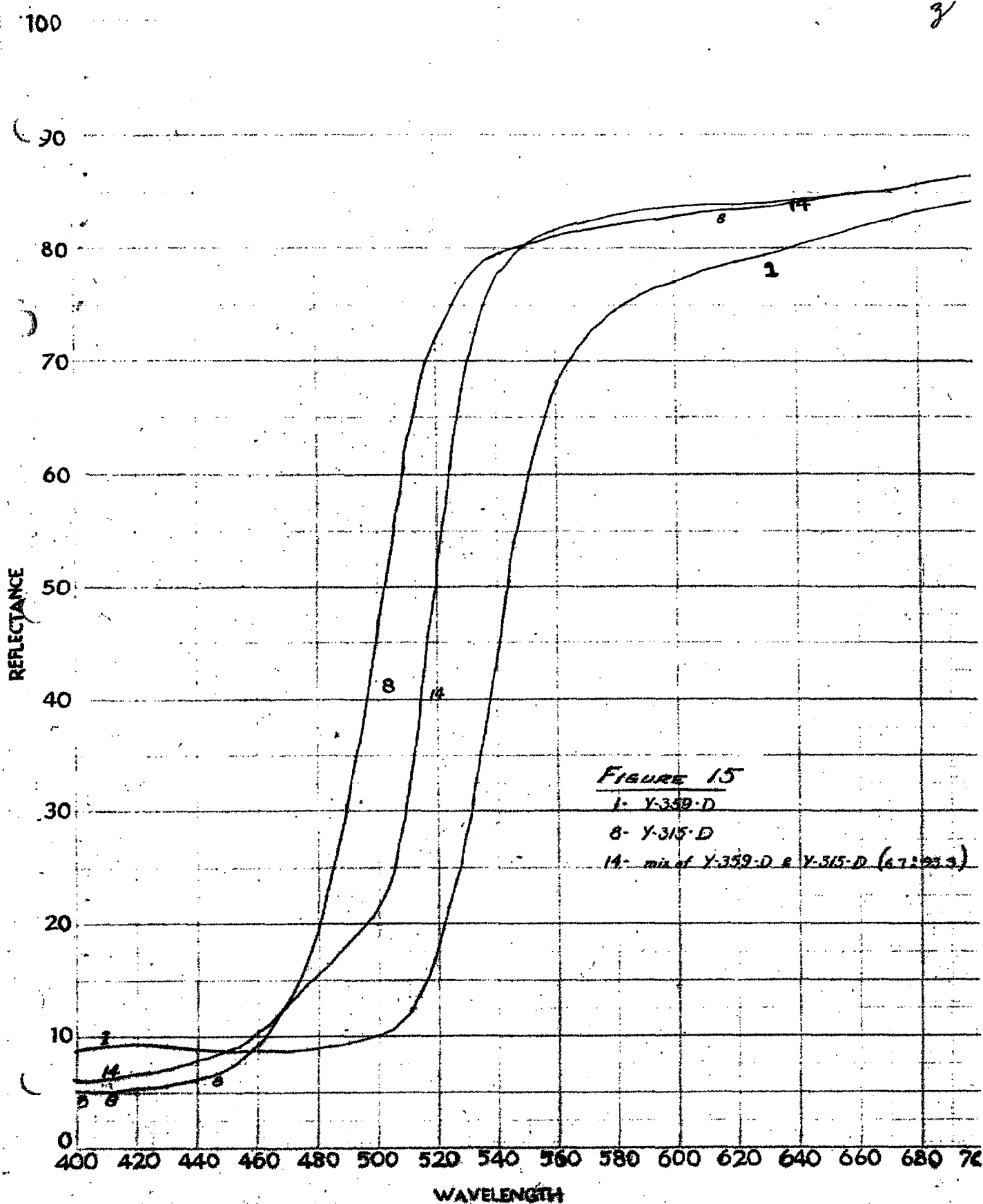
- 1) Spectrophotometric data agree fairly well with visual readings of mixes and standards.
- 2) All of the yellows in the regular line, from primrose to medium shades are "compatible" with each other, giving satisfactory matches for intermediate standards in skindown mounts prepared as specified earlier in this report.

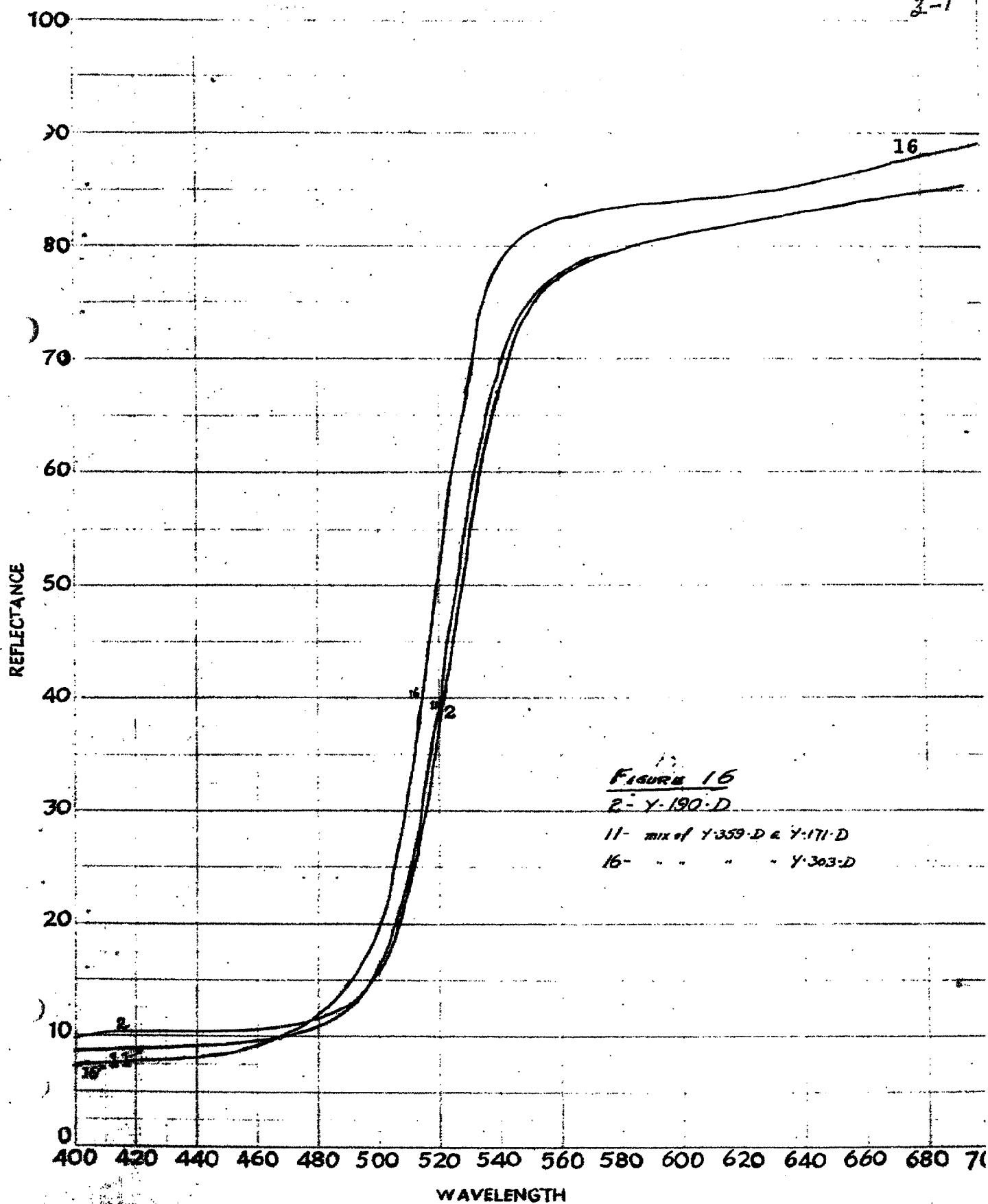
Spectrometric Curves of Mixes and Standards

Figure 14* shows curves for two standard yellows, Y-359-D and Y-171-D, and a curve for the mixtures of these two yellows. As might be expected, the curve for the mix falls between the curves of the two components. A similar set of curves for two other yellows is shown in figure 15*; in this case, the curve for the mix is somewhat above either component from 550 to 660 millimicrons; this is probably due to variation in quality of the pigment mounts.

Figure 16* shows curves of two mixes (11 and 16) designed to match standard Y-190-D (2). Mix 11 is a good match; mix 16 is not as close.

Figures 14, 15, and 16 follow.





Dr. A.W. Kenney,
Experimental Station,
E.I. du Pont de Nemours & Co.,
Wilmington, Del.

CHROME YELLOWS

We are sending you attached herewith forty "pulldowns" which we have prepared for spectrometric examination by Dr. Hardy and your subsequent study of C.I.E. curves. These exhibits have been made from our important standard Chrome Yellows and mixtures of these standards and also include six mounts of competitive cadmium yellows.

There are two mounts of each pigment or pigment mixture, one on a bond paper (Parchmont Bond) and one on a coated paper ("litho label"). The latter mounts are designated by the subscript "a". No chrome oranges are included in this series, because visual examination of tentative mixes indicated that differences in oil absorption, finish, etc. between the oranges and the yellows made significant mixtures of the two types impossible.

The yellows used are tabulated below, with identifying numbers with which the pulldowns are marked;

<u>Pulldown No.</u>	<u>Standard Code</u>
429-1-1 and 1a	Y-359-D
" 2 " 2a	Y-190-D
" 3 " 3a	Y-309-D
" 4 " 4a	Y-171-D
" 5 " 5a	Y-180-D
" 6 " 6a	Y-303-D
" 7 " 7a	Y-305-D
" 8 " 8a	Y-315-D
" 9 " 9a	Y-318-D
" 10 " 10a	Y-208-D
" 11 " 11a	mix to match Y-190-D (4% Y-359-D, 96% Y-171-D)
" 12 " 12a	mix to match Y-171-D (37% Y-190-D, 63% Y-180-D)
" 13 " 13a	mix to match Y-180-D (63% Y-171-D, 37% Y-303-D)
" 14 " 14a	mix to match Y-180-D (6.7% Y-359-D, 93.3% Y-315-D)
" 15 " 15a	mix to match Y-303-D (50% Y-180-D, 50% Y-315-D)
" 16 " 16a	mix to match Y-190-D (12% Y-359-D, 88% Y-303-D)
" 17 " 17a	mix to match Y-171-D (6.7% Y-359-D, 93.3% Y-303-D)
" 18 " 18a	C-31671 Cadmium Yellow
" 19 " 19a	C-31672 Cadmium Yellow
" 20 " 20a	C-31670 Cadmium Yellow

Mixes 11 to 17 inclusive were made to be fairly close visual matches for masstone and undertone on bond paper, mounted in the usual manner. In many cases, differences in finish, masstone; undertone relation-ship, strength, etc. made close matches impossible. The following list is a tabulation of readings of the mixes versus the standards they are designed to match. The readings are in the orthodox manner used at Newark;

3-3

the abbreviations s., v.s., and v.v.s., mean "slightly", "very slightly", and "very very slightly". Strength is the apparent tinting power when the pigment is extended 1 part to 15 (by weight) with zinc oxide. Tint is the chromaticity of such an extension. (These tints were made for general information and are not being sent to you.

Readings - mix versus standard

Mix	Standard	Masstone	Undertone	Strength	Tint
11	2 (Y-190-D)	vvs green	vs strong	vs strong	s green & clear
12	4 (Y-171-D)	vs red & clean	s.weak	vs weak	s red
13	5 (Y-180-D)	vs green	vs green	equal	vvs green
14	5 (Y-180-D)	s red	vs weak & green	weak	equal
15	6 (Y-303-D)	vs red & clean	vs strong	s weak	s red
16	2 (Y-190-D)	vs lt.cl.&grn.	vs clean	s weak	vs green & dirty
17	4 (Y-171-D)	vs red	s weak	weak	vs red

The above readings are the writer's, of mounts on bond paper.

In order to give you an idea of other than color differences between these yellows, the following tabulation indicates the composition and special characteristics of the various pigments,

Std.Code	Pulldown No.	%PbCrO4	"Shade"	Type	Remarks
Y-359-D	1	100%	Medium	regular	regular medium type
Y-190-D	2	72%	dk.excelsior	"	regular excelsior type; somewhat wk.in tint.
Y-171-D	4	69%	lt. "	"	regular excelsior type
Y-180-D	5	58%	canary	"	regular canary type
Y-303-D	6	52%	lt.canary	"	light, weak canary
Y-309-D	3	70%	excelsior	high fin.	high fin. good str.
Y-305-D	7	56%	primrose	"	high fin. primrose
Y-315-D	8	48%	lt.primrose	"	high fin. light, weak primrose.
Y-318-D	9	85%	lem.(canary lemon shade)	lemon	high fin., good printing tone and oil retention.
Y-202-D	10	79%	lemon	lemon	good str., poor light stability.

In most of these yellows, the additional component besides lead chromate is chiefly lead sulphate; in the high finish type, there are small amounts of alumina hydrate; in the lemon yellows there is also lead phosphate.

Pulldowns were made instead of prints because the former are much easier to prepare, and approximate the results obtained in prints. Large pulldowns were made so that they could be cut up, if necessary, for several runs in the spectrophotometer. I am enclosing several sheets of the

3-T

papers used. In running curves on these mounts, I believe they should be backed by several sheets of paper, to give a non-selective opaque backing.

Visually, Y-359-D is much redder than Y-190-D; Y-309-D, Y-171-D, and Y-180-D are greener in approximately equal small steps; Y-303-D is distinctly greener than Y-180-D; Y-305-D is greener than Y-303-D the difference being large; Y-315-D is very slightly greener than Y-305-D. Y-318-D is somewhat greener and more saturated than Y-180-D; Y-202-D is much greener, and more saturated than Y-180-D.

V. CHALUPSKI.

36-27 69

PIGMENT COLOR RESEARCH REPORT

COLORIMETRIC STUDY OF MILORI BLUES

SUBMITTED BY: *H. Young + V. Chalupski*
APPROVED BY: *V. Chalupski*

DATE: JANUARY 1936
FILE: PHYSICAL STUDIES

INTRODUCTION

Spectrophotometric curves have been obtained on a number of blue ink preparations, with the object of studying their inherent color properties, and to determine the extent to which the ordinary Milori Blue fall short of the "ideal" minus red. From the data obtained, certain conclusions can be drawn as to possibilities of improvement.

SUMMARY AND CONCLUSIONS

Two standard Milori Blues (B-57-D and B-66-D) were examined, let down to approximately printing strength with alumina hydrate ink and printed on a proof press. A curve was also run on Peacock Blue Lake, BL-72-D, for comparison. In the case of B-66-D, curves were run for three different extensions (1:3, 1:9, 1:15), and also on a thin film of ink mounted between glass plates.

The following is a tabulation of the results calculated to the C.I.E. tristimulus specifications (Illuminant C).

	<u>Dominant Wave Length</u>	<u>Excitation Purity</u>	<u>Brightness Factor</u>
BL-72-D print	482.70	62.43	17.47
B-57-D (1:2.3 Al. hydrate) print	478.11	58.95	12.95
B-66-D (1:3 " " " "	478.46	57.15	14.16
" (1:9 " " " "	481.76	40.03	29.43
" (1:15 " " " "	482.56	31.70	36.05
" transmission-film between glass	478.95	73.07	17.86
" reflection (of above film, over white paper)	473.98	74.09	7.05
idea/minus red	490.85	30.15	81.82

The color difference between the B-57-D and B-66-D prints is quite small, as might be expected.

In the B-66-D series, successive reductions shift the dominant wave length toward the green. Purity is reduced, and brightness is increased, as expected. The transmission curve of the ink film of B-66-D between glass slides represents the inherent light absorption of the blue in varnish; examination of the curve and the above tristimulus specifications shows a high transmission in the violet and blue, rapidly decreasing transmission thru the green, a very small transmission in the yellow, and practically zero transmission in the red (less than 0.25 percent). The reflection curve, made by placing this ink-between-

glass mount on a sheet of white paper and treating this as a reflection mount, shows no peculiar results; the data from the transmission curve calculated to such a reflection curve (taking into consideration the reflection characteristics of the paper) agrees very closely with the actual curve, as shown in the following tabulation. This indicates that there are no complications introduced by bronze or surface color phenomena. The absence of any pigment-air interface would of course prevent any very marked surface selective reflection.

		Cal. Reflection		Actual Reflection
Wave Length	(a) Transmission of Mount	(b) Refl. of paper	$[(a)^2 \times b] + K^*$	from curve
400	43.2%	73%	15.9%	15.8%
420	53.2	76	23.8	23.6
440	60.5	77.6	30.7	30.6
460	62.8	79	33.5	33.5
480	58.3	80	29.5	29.1
500	48.2	80.2	21.0	21.8
520	35.0	80.1	12.1	11.3
540	21.7	79.8	6.1	5.7
560	11.0	80.0	3.3	3.1

*K = constant of reflection from glass-air interfaces, taken as 2.3%, based on reflectance in the red and of reflection curve.

Figure 1 shows the curves for the three extensions of B-66-D, and the curve for Peacock Blue Lake, BL-72-D. The reflection curve for the paper used is included.

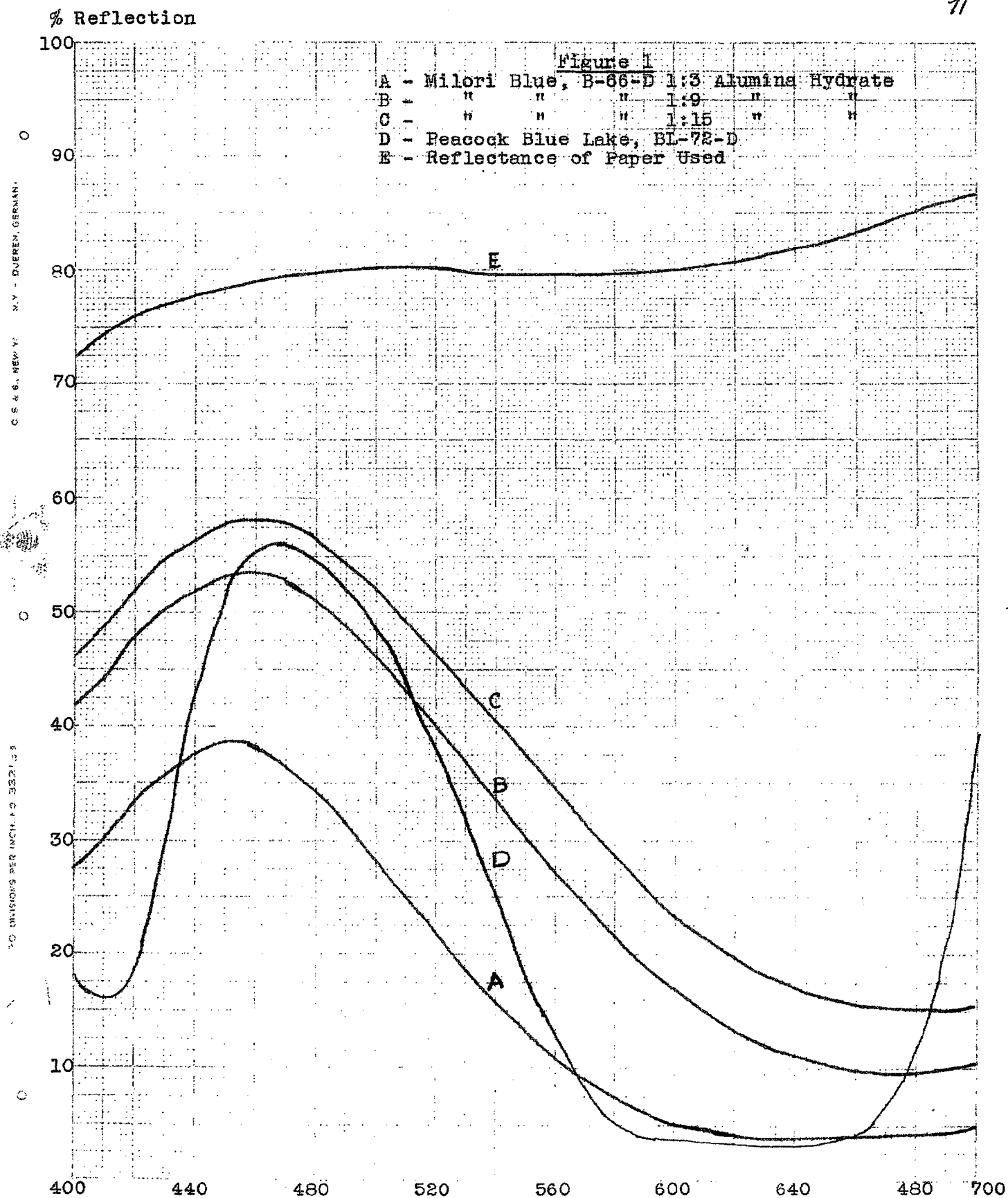
Figure 2 shows the comparison between B-66-D and B-57-D. B-66-D is extended somewhat more than the B-57-D in these prints.

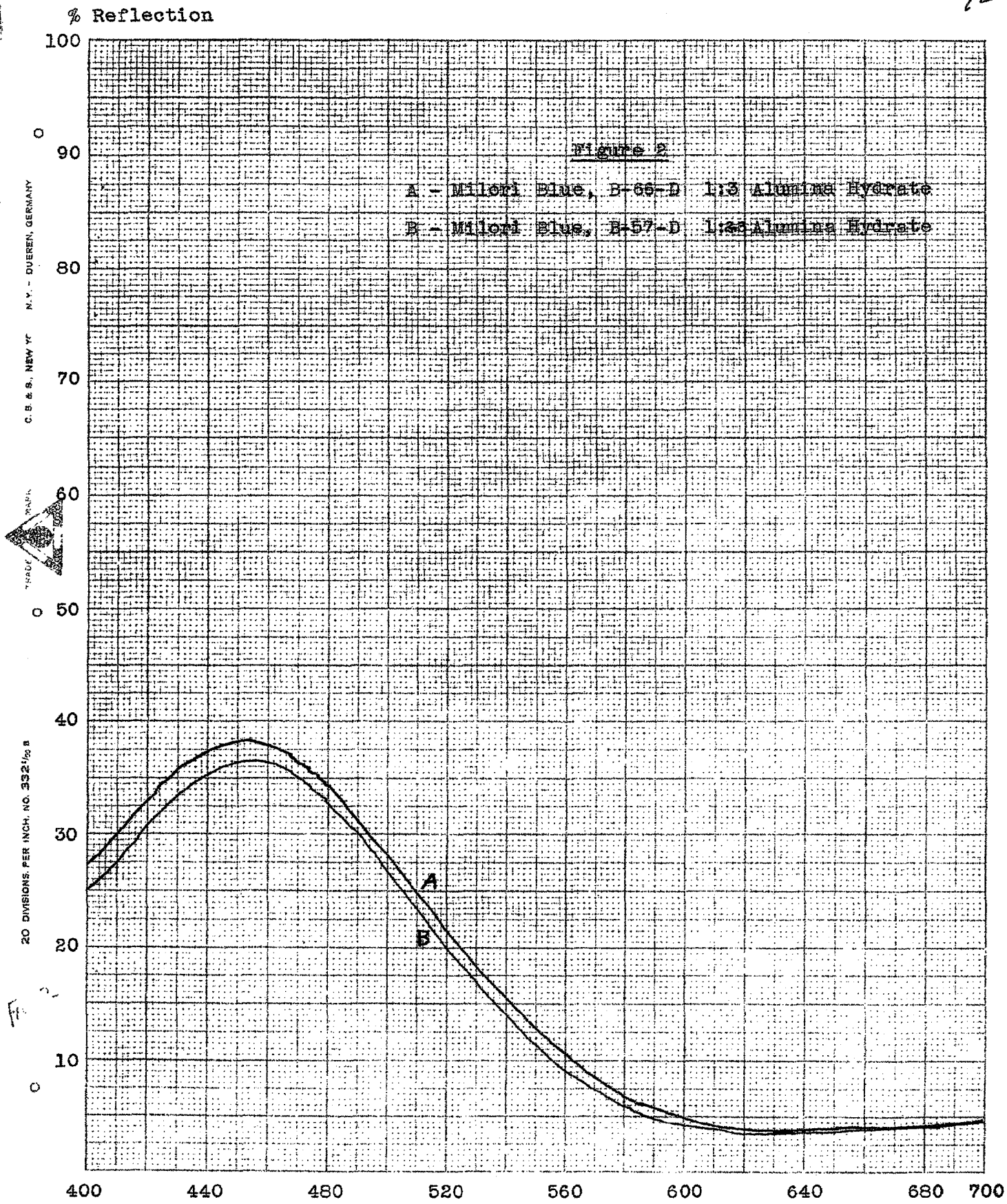
Figure 3 shows the transmission of the B-66-D ink diluted between glass slides. The curve for reflection of this film over paper, and the curve of a print (same as in Figure 1) is also included. It may be noted that curves B and C are both steeper than A.

Figure 4 shows a portion of the C.I.E. triangle, showing the location of these blues on this plot.

All of the blues examined fall far short of the ideal minus red; the spectrophotometric reflectance curves indicate this perhaps more than the C.I.E. graph. None of the blues reflect nearly enough green; all are very low in the violet, and their maxima are all quite low. None of the blues has a very steep curve. A comparison of curves for BL-72-D and B-66-D with a hypothetical blue equal in "chromatic efficiency" to an ordinary chrome yellow (made by reversing a chrome yellow curve right to left) is shown in figure 5. The inferiority of the two blue pigments is obvious.

PG.





% Reflection

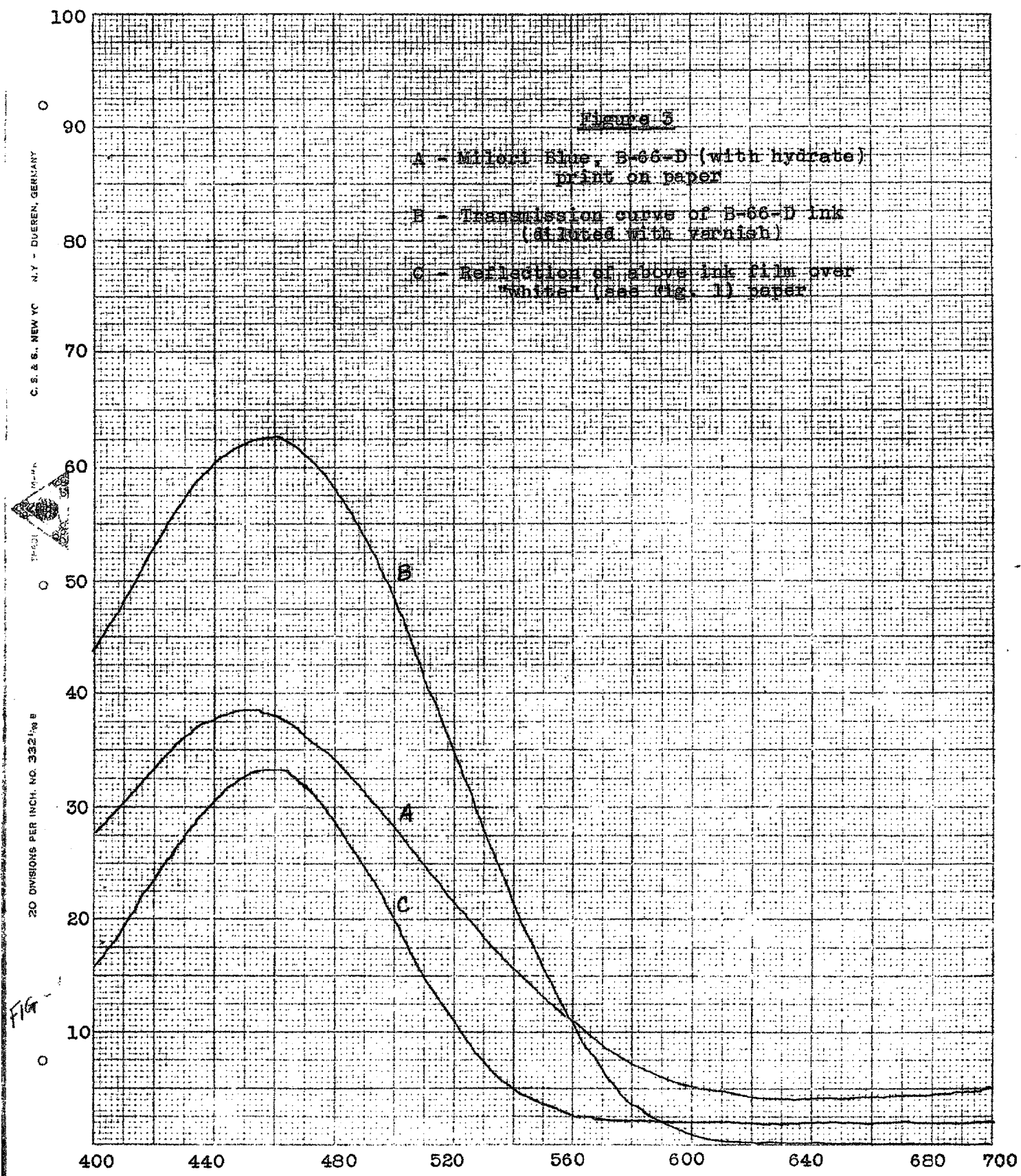


Figure 3

- A - Mileori Blue, B-66-D (with hydrate) print on paper
- B - Transmission curve of B-66-D ink (diluted with varnish)
- C - Reflection of above ink film over "white" (see fig. 1) paper

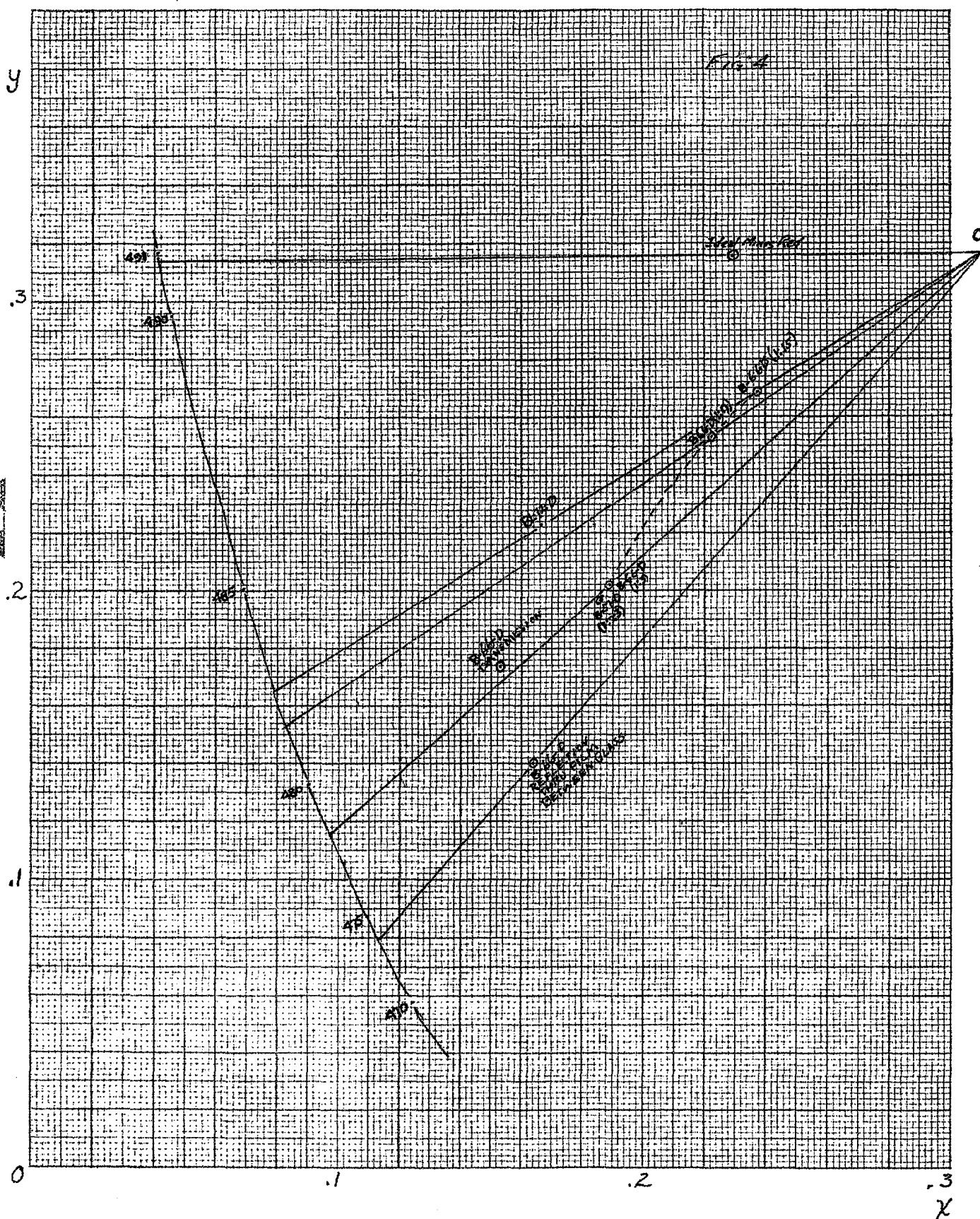


20 DIVISIONS PER INCH. NO. 3321-10 B

Fig.



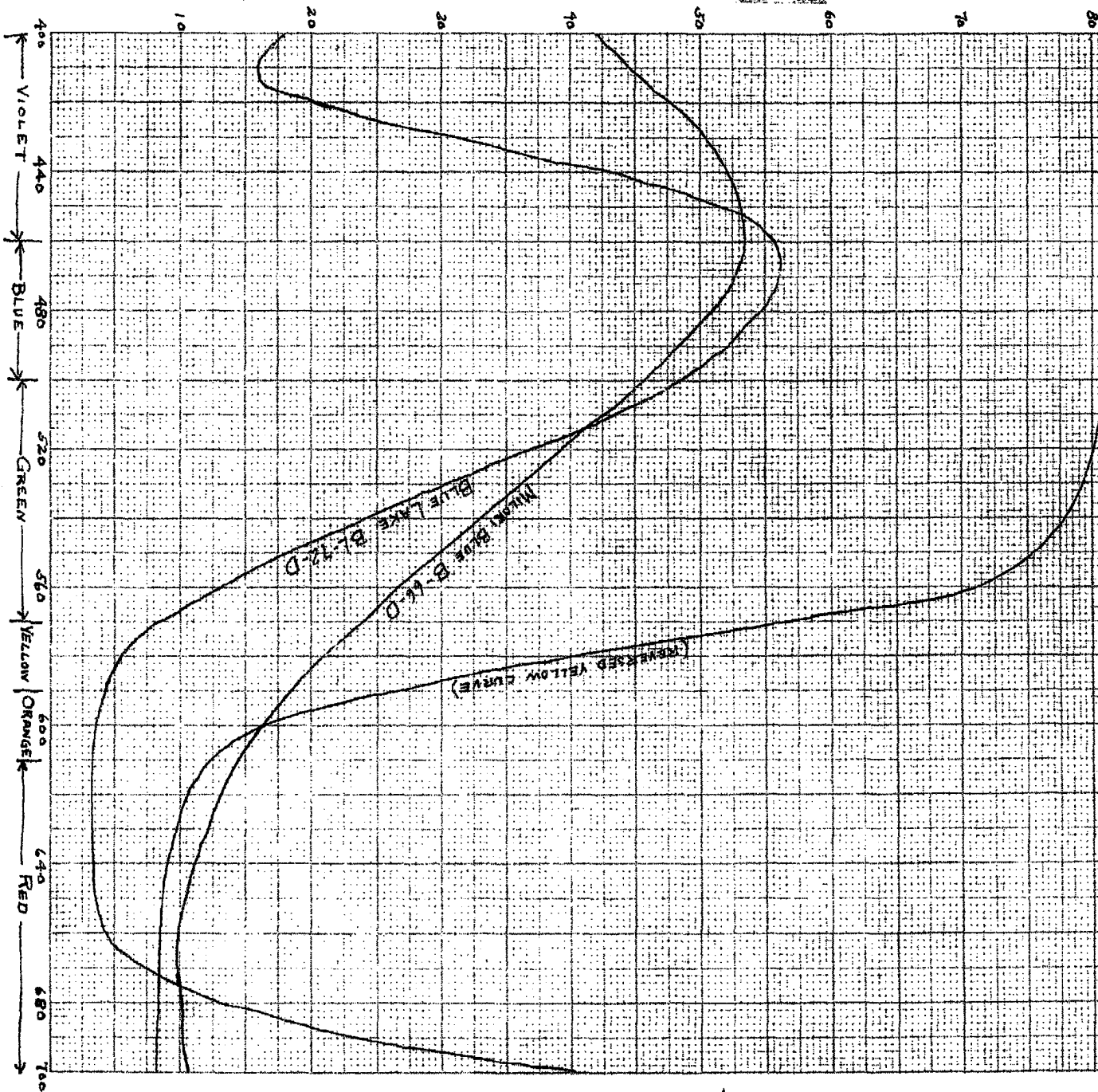
20 DIVISIONS PER INCH. NO. 332180 B



20 DIVISIONS PER INCH. NO. 3321/20 B



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PIGMENT COLOR RESEARCH REPORT

PHYSICAL LABORATORY - PROGRESS REPORT

SUBMITTED BY: *H. H. Young*
APPROVED BY: *V. Chalupka*

FILE: PHYSICAL STUDIES
DATE: JANUARY 1936.

This report covers the work done in the Physical Laboratory for the past month. Inasmuch as most of the work now being done is of a continuous or semi-continuous nature, no attempt will be made to draw conclusions in this report. The work divides itself naturally into three types, namely, microscopic studies, color analysis, and consistency measurements as a part of the general problem of fundamental physical measurements of the physical properties of pigmented vehicle preparations. This latter type was inactive during the month.

Microscopic studies - Considerable work has been done in assisting Dr. J.V. Scudi in his investigation of Para Reds. A number of samples have been examined by transmitted light, reflected light, polarized light, and dark field illumination in an attempt to discover the physical nature of the pigment and differences between samples. For a complete discussion of the work done, reference is made to Dr. Scudi's report on Para Red - Blue Shade for January 1936. In this examination, it was found that the use of sodium silicate as an immersion medium was of considerable value inasmuch as Para Reds, in common with many other organic pigments, are somewhat soluble in the ordinary immersion mediums.

In addition to the above, microscopic examination has been made of flushed inks as compared to regular inks and of a copper phthalocyanine ink, for dispersion. Results in both cases were inconclusive due to difficulties in dispersing a pigmented vehicle and in securing uniform mounts.

Under color analysis, three types of work have been in progress this month. First, mounts have been and are being prepared for Dr. A.W. Kenney of the Experimental Station for use in his study of the possibility of defining numerical shipping limits. Mounts showing differences within shipping limits have been prepared on masstones, extensions, and brushouts and mounts on lacquer tests and linoleum tests are now being prepared. Our part of this study, that is preparation and visual reading of the mounts, should be completed within the next two weeks.

Second, continuation of the work on securing physical data on our more important standards is proceeding. Spectrophotometric curves have been run on ten red pigments and completion of work on this particular group should occur within the next month. At such a time a report will be written drawing such tentative conclusions as may be indicated by the data obtained thus far. It may be mentioned that five curves are being run on each standard, i.e. masstone, skindown, print, 1:30 extension, and 1:100 extension. Calculation of dominant wavelength, excitation purity, and brightness factor from these curves is being delayed by the lack of a calculating machine.

Third, work has been done in the study of blues, especially of the copper phthalocyanine pigments. Prints have been made and spectrophotometric curves obtained for several such blues in various degrees of extension with alumina hydrate. A special report on a number of blue pigments is now in the process of preparation.

A report on the spectrophotometric study of chrome yellows (drawdowns) has been prepared, and is filed under "Physical Studies."

In the preparation of mounts on RT-22-D standard for spectrophotometric analysis, average thickness of printing ink film was determined by the area-volume method as .00239 millimeters or .000094 inches. This value should, of course, be regarded as an approximation, but one which indicates the order of thickness of a printing ink film as applied to litho label paper by the plate press in the Sales Service Laboratory.

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PIGMENT COLOR RESEARCH REPORT

PULVERIZATION STUDIES

SUBMITTED BY: *J. Kalbach*
APPROVED BY: *V. Chalupsky*

FILE: MFG. STUDIES
DATE: JANUARY 1936.

INTRODUCTION

A series of grinds was made in the semi-works to match the performance of the usual settling of the semi-works #1 12-inch pulverizer for the grinding of iron blues with that of a settling of the 8-inch pulverizers.

SUMMARY AND CONCLUSIONS

A batch of B-66-D lump from lot 4429 was ground in the semi-works #1 12-inch pulverizer at 6800 R.P.M. through the .035 inch HB screen and in the 8-inch pulverizer with various settings. The smaller pulverizer best matched the performance of the larger one when running at 5812 R.P.M. with a 3/64 inch screen.

EXPERIMENTAL DETAILS

See notebook #467, p.110-111.

Speed R.P.M.	Screen	Residue on screen in %			
		80	150	200	325
12-inch pulverizer					
6800	.035 HB			17.14	11.48
8-inch pulverizer					
5566	.039 in.			46.26	12.72
"	3/64			45.78	11.96
"	1/16			63.34	11.60
"	1/4			79.30	9.92
4980	.039			21.12	14.56
"	3/64			27.84	16.00
"	1/16			46.60	11.64
"	1/4			53.20	13.26
5812	.039			15.64	17.02
"	3/64			19.84	15.00
"	1/16			31.96	14.28
"	1/4			46.68	17.02
12-inch pulverizer					
6800	.035 HB	1.04	5.48	10.62	11.48
8-inch pulverizer					
4980	.039	1.08	8.00	12.04	14.56
5812	.039	0.76	6.04	8.84	17.02
5812	3/64	0.92	5.98	12.94	15.00
8030	3/64	0.32	1.32	4.02	10.80
8030	1/16	0.86	3.52	7.00	13.04
8030	1/4	4.24	12.50	12.54	14.22

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PIGMENT COLOR RESEARCH REPORT

PARA RED - BLUE SHADE

SUBMITTED BY:
APPROVED BY:

John V. Scuderi
Alfred Siegel

FILE: PARA RED
DATE: JANUARY 1936

INTRODUCTION

Work was continued along the following lines:

- A-Physical studies
- B-Variations in the relative rates of formation of Para Red and Para F
- C-Variations in the concentration and kind of dispersing agent
- D-Barium salt formation of the incorporated Para F
- E- Some semi-works observations

SUMMARY AND CONCLUSIONS

(A) In the course of this work it was believed necessary to obtain certain physical data on the microscopic nature of Para Red-Blue Shade. The Physical Department has made these measurements.

Products prepared in the absence of Para Soap are composed of hard glossy aggregates which may be factured giving irregular particles. The major portion of these particles is optically inactive, that is, amorphous, probably as a result of solid solution. In silicate immersion at 2200 magnifications, anisotropic pin-point spots and occasional needles were observed. Comparison with C-38319 showed the latter to contain a much smaller amount of optically active spots and practically no needles. RT-70-D contains as few if not less than the C number. From these data it is inferred that the latter two are probably in more complete solid solution than the former.

When dusted on a microscope slide, RT-70-D aggregates appear larger than those of the C number. Attempts to break down the agglomerates caused smearing. These smears are due to the Para Soap present in the pigment since products prepared with out Para Soap do not smear. Oblique illumination of these smears in a dark field showed no scattering. Hence these red smears are true solutions containing molecularly dispersed Para Red, Para F or both, as well as possible impurities. The Para Soap (c. 2% by wt.) may merely impart fluidity to the solid solution, acting as a third component. This seems more feasible than a surface coating of the indefinite lumps of solid solution.

The extent of smearing may be considered an approximate measure of the Para Soap content. RT-70-D smears more than C-38319, and both smeared more than a baked sample of 490-20 B which was prepared initially in the presence of twice the amount of soap used in the preparation of R¹-70-D.

It is not impossible that plant processing changes may be concerned with the loss of Para Soap. Foreexample, higher temperatures as in baking, or over-drying, or the heat and shear of grinding may produce a slow evaporation of the Para Soap.

There is no apparent way to determine the nature of these optically active points and needles which appear in the final pigment. As previously indicated Para F and Para Red are probably isomorphous. The angles of extinction of the two substances in polarized light are approximately equal, and equal to the extinction angles observed for the anisotropic points and needles observed in the pigments. Consequently, these may be crystalline Para Red, Para F or mixed crystals. Considering the relative proportions of the Para Red and Para F, it appears improbable that these active spots are the result of mixed crystals. On the basis of proportions, it would seem more probable that these crystals are Para Red which crystallized by way of the Para Soap, and this aging would be accelerated by heating. The higher the temperature and the longer the time of heating, the greater the perfection and the larger the crystals should become. Cementation might even occur in baking treatments and this would be in agreement with the tendency of the products to go weak and blue on baking. This may possibly account for plant processing changes, but would not explain the occurrence of the anisotropic pin points and needles in laboratory products, particularly since these action points are observed in pigments prepared in the absence of Para Soap. It seems most probable that these crystals are Para F incorporated by nature of the mode of coupling. In the methods used, Para F is formed prior to the Para Red. During the course of coupling there is an increasing formation of NaCl. This may salt out crystalline particles which are mechanically included in the micelle (On standing, the bleeds yield definitely crystalline Para F.)

Such a picture is equivalent to a very fine dry mix of the Para F with a solid solution. Since dry mixing does not in general attain the maximum brightness of either component, it would appear desirable to seek a uniform molecular dispersion of the Para F in the Para Red.

Such a molecular dispersion will obviously depend largely on the relative rates of coupling of P.N.A. to B.N. and mono acid F. The ideal case would probably require that something of the order 50 Para Red molecules be formed per single Para F molecule. Another major factor involved appears to be the Para Soap. Consequently, experimentation was carried out along these two lines. (see parts B and C).

(B) Coupling has been carried out in alkaline media; e.g. in NaOH - Na₂CO₃ as per RT-70-D, or in NaOH - NaHCO₃, and these couplings have ended on the alkaline side. Coupling in a medium of NaOH - NaC₂H₃O₂, ending acid to Congo, but not to Clayton Yellow has given certain interesting results. The products were bright, but light in masstone and the loss in Para F was considered wasteful since equal masstones could be obtained in an NaOH - NaHCO₃ medium with less mono acid F.

By inverse coupling (adding BN component to the diazo) it is possible to begin on the acid side and end at any desired pH. Thus we have an additional empirical method of governing the relative reaction rates.

Ending slightly on the alkaline side gave hard, brown weak products even though T.R.O. was used. These products were not examined microscopically, but from their appearance and yields, it was apparent that the Para F was not satisfactorily dispersed. Ending at a high pH gave brown products which were improved by acidification, but were not as strong as those obtained heretofore. There was some blueness of undertone, although this may merely be a result of weakness.

When coupling ended strongly acid to Congo products were obtained in improved yields. They filtered very rapidly, were soft in texture and had much more blueness in undertone than has been obtained heretofore. These products were tinctorially unlike those obtained by the usual diazo to B.N. coupling method in NaOH- $\text{NaC}_2\text{H}_3\text{O}_2$ only in having more blueness in undertone. They were light and weak vs. C-36670 but the undertone approached that of the C number somewhat, and appeared to justify further investigation. Increasing the amount of Para F present gave hard brown products typical of mechanical mixtures of the Para F and Para Red unless the products were developed. When developed and filtered hot, the strength increased to about that normally obtained (490-20B) but the masstone remained very light vs. 490-20B. The products have about the same depth as RT-70-D. They are light, and very bright in masstone and s. blue in undertone versus 490-20B. These products (490-37C² and D²) are close to a match for C-36670 in masstone and brightness. In tint and undertone they are strong and yellow. These colors may be expected to develop further blueness on processing. However, they may become lighter in masstone. If they do not this may be a method of matching the C number.

Series were run in which variations in final acidity were studied. In general, increasing the final acidity yields lighter masstones and the strength increases to a maximum.

From an approximate examination of the bleeds it seems that less Para F is retained by these reverse couplings, but some is definitely retained by the pigment under the experimental conditions. More accurate data will be accumulated. The Para F is present as the free acid and is probably incorporated as such rather than as the Na salt which is probably the usual case in alkaline couplings. This difference may readily account for the tinctorial differences noted.

The products obtained by this inverse coupling method appear to be almost entirely crystalline. They smear when rubbed in a slide due to the T.R.O. present, but the smears contain a large number of crystals. Since some Para F is apparently retained, it appears that instead of solid solutions, mixed crystals were obtained. This uniformity may account for the corresponding increase in brightness. These same phenomena were also observed for product prepared in the presence of a large excess of Para F. The average particle size is roughly between 0.5 - 1.0 μ . It is interesting to note that 37A, made under the same conditions in the absence of mono acid F did not possess blueness of undertone, and yet the particles were of the same order of magnitude in size. From this it would appear that the "adsorbed dye" restraining action is not very great compared to the dispersing power of the T.R.O.

and further that the blue shade in the Para Red depends on the incorporation of Para F.

It is interesting to note that the products are yellow in the reaction slurry. If this slurry is smeared on glass and allowed to air dry the product remains yellow, and possesses no apparent blueness. Yet this blueness develops when the product is finished up as usual. It may be that the dissolved Para F exerts an inhibition toward crystal perfection (Kolthoff and Rosenblum: J.A.C.S. 57, 607 etc.) but after filtration and washing, the dye on the internal surfaces is included in the perfection of the crystals on drying at 60°C.

Thus, it appears that in the inverse method of coupling mixed crystals are obtained whereas in alkaline couplings the products (C number and RT-70-D types) are probably solid solutions. These phenomena may possibly be related to differences in the lattice parameters of the free acid and the soda salt of Para F. In the crystalline products, the masstones are light and bright with blue undertones while the products isolated from alkaline media are dark in masstone and yellow in undertone and tint. The strength, which appears to be concerned with hot development and may therefore be an aging process, is approximately equal in both types of product. It does not seem highly probable that these two products will have a common meeting ground in which the advantages of both may be combined, unless it is accomplished by a dry mix.

It is interesting to note that these crystalline products and their aging may be studied quite rigorously, not only by their optical properties, but also by ionic exchange and Para F extraction. These methods would furnish insight into the kind and amount of surface present. Surface is obviously a measure of particle size, but the kind of surface appears to be equally significant since from the present evidence it appears that the nature of the Para F incorporated may be quite important tinctorially. It seems not impossible that incorporation of the free acid of Para F may be responsible for the blue undertone. The influence of this free sulfonic acid group in the pigment may have some affect on the vehicle in which the pigment is used.

(C) Series were run in which a study was made of the kind and the amount of soap used. In alkaline couplings of the NaOH - NaHCO_3 type it was observed that diminution of Para Soap caused the product to become light and flat whereas increasing the Para Soap caused bronzing. This bronzing may be the result of the solvent action of excess Para Soap on the Para Red as discussed in part A. It is interesting to note in this connection that bronzing is practically eliminated in the semi works during grinding. For a medium of NaOH - Na_2CO_3 , a large increase in Para Soap caused bronzing, in the masstone the product became weak and flat.

Diglycal stearate gave promising results when used in amounts about equal to those of Para Soap normally used. The products were slightly lighter but brighter. Sodium abietyl sulfonate gave stronger products which were lighter and brighter than those produced by Para Soap.

(D) As indicated in earlier reports the Uhlich sample C-36670 was shown to contain barium, and certain experimentation was conducted along these lines. The Ba salt of Para F is best prepared in acid media. This requires removal of the excess nitrite in alkaline couplings. This may be accomplished by urea treatment of the diazo, use of insufficient nitrite or removal of excess NO_2^- from the slurry by washing. All three methods were compared. In general, the yields are increased, but the product is no longer ashless. On the other hand, C-36670 contains 3.38% residue. The product obtained herein contained 3-3.5% residue. In the absence of Ba salt formation the ash content is 0.8%.

As indicated in the December report, the rate of filtration of these products was increased by precipitation of the colloidal Para F as the Ba salt.

In the series in which the excess nitrite was removed by washing, the product acidified and struck with Ba, the products were darker than those not treated with Ba. This may be due to increased retention of Para F since the Ba salt is less soluble than the Na salt, although there might have been a trace of nitrite present to form nitroso - BN. Studying the temperature of development, it was observed that there is little tinctorial difference between colors struck at 22°C and those at 65°C . However when developed to 95°C the colors become light. Ba salt formation cuts down the Para F bleed considerably. Compared to the Na salt the ratios are:

$22^\circ\text{C} = 1:10$; $65^\circ = 5:2$; at $95^\circ\text{C} = 5:3$

In other words at equal temperatures there is a greater loss of the Para F when present as the Na salt. Thus the additional depth in color may be due to increased retention of Para F. This same observation obtains in comparing the Ba containing product at the three temperatures studied. The higher the temperature of development the lighter the masstone. These colors were all filtered hot.

Colors prepared by coupling urea treated diazo to BN in the usual way ($\text{NaOH} - \text{NaHCO}_3$ media and development to 65°C) when compared with a similar product which was acidified and struck with Ba, were dark and brown. The Ba containing product filtered faster and was obtained in higher yields.

By restricting the amount of nitrite used rather than the urea treatment, striking cold in the usual medium $\text{pH} = 7 - 7.3$ and developing to 65°C the brightest product was obtained. (acidification caused brownness, due undoubtedly to a slight excess of nitrite. It appears that small amounts of nitroso - BN produce marked tinctorial deterioration.) There was a slight increase in yield and a good increase in the rate of filtration. The product was softer in texture than 490-20B and appears to be as bright and possibly a trace darker. On baking, this product went light in masstone, weak and blue in tint. The Uhlich blueness was not reached.

Barium was incorporated in the diazo in the inverse coupling method and the products were filtered cold. The product was light in masstone, and strong in undertone. However, the product was weak versus products obtained by development in alkaline couplings.

No comparison can be made at the present time since these products were not developed. It is planned to study this point further since the products obtained were dark and bright against RT-70-D and bright against 490-20B, although light and weak.

(E) Two batches were made in the semi-works, and the details will be reported elsewhere, although certain observations may be discussed here.

The outlet surface of the press cakes after washing showed the greenish fluorescence of Para F. This type of diffusion phenomenon may be related to bronzing, and as has been previously indicated the tedious filtration is related to the Para F present in colloidal suspension in the reaction vehicle. Ba salt formation would probably eliminate both these objectionable features.

It is well known that Para Red - Blue shade is sensitive to development at 95° c. On the other hand, the pigment may be held at 65°c for many hours at pH 7 - 7.3 without any great change tintorially. It is interesting, however, that there is a physical change, viz., an increase in yield. This anomalous increase was discussed in December report, and a possible explanation was given.

EXPERIMENTAL DETAILS

N.B. #490

Series 490 - 30. A non-bleeding Para Red for paper coating to replace RA-26-D. Barium salt formation, its influence on bleed in water, and the rate of filtration. Residue determinations.

Series 490 - 31. Continuation of 490 - 31. Urea treated diazos and those prepared with reduced nitrite concentration. Extension of products with Gloss White.

Page 140 - Notes on semi-works trials.

Page 144 - Microscopic examinations.

Series 490 - 32. The incorporation of diglycol stearate and sodium abietyl sulfonate: Barium salt formations and concentration variations.

Series 490 - 33. Inverse couplings: decreasing final pH values.

Series 490 - 34. Variations in Para Soap: coupling medium of NaOH - NaHCO₃.

Series 490 - 35. Variations in Para Soap: coupling medium of NaOH - Na₂CO₃.

Series 490 - 36. Inverse couplings: final pH values low, intermediate, and high.

Series 490 - 37. Inverse couplings: increasing concentrations of mono acid F; the influence of developing at 22°c, 65°c and 95°c.

Page 172 - Microscopic examinations.

Series 490 - 38. Alkaline couplings with reduce mono acid F concentrations.

Inverse couplings with Ba salt formation of the Para F.

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PIGMENT COLOR RESEARCH REPORT

PARA RED - YELLOW SHADE

SUBMITTED BY:
APPROVED BY:

John V. Lueder
Alfred Siegel

FILE: PARA RED
DATE: JANUARY 1936

INTRODUCTION

In the course of the investigation of Para Red - Blue shade, it became necessary to conduct certain experimentation to obtain some insight into that nature of Para Red - Yellow Shade. Since, at the present time, certain difficulties are being encountered in production, the following data are presented. No intensive research was carried out and the results are those of several isolated series.

SUMMARY AND CONCLUSIONS

The method of diazotization of P.N.A. used through^{out} was #276-44. Whiting was omitted. Consequently, the yields were diminished by approximately 10%. For that reason, products called "strong" herein are approximated only on the strength beyond the initial 10%.

In alkaline coupling media (NaOH - Na₂CO₃ or NaOH - NaHCO₃) the presence of Para Soap appears to be contra-indicated. The products are strengthened considerably but the masstones are dark and brown.

A coupling medium #276-60C (NaOH-NaC₂H₃O₂) which is initially alkaline but finishes acid to Congo, but not to Clayton Yellow gave promising results. The products were light and bright in masstone and quite strong versus RT-22-D. This formula seems reasonably stable since it was checked in a second run.

Running the diazo into a suspension of B.N. precipitated by HCl appears to be entirely unsatisfactory. The product is very bright but very weak and the time of coupling unduly long.

It was found possible to use Turkey Red Oil without the usually accompanying brownness of masstone by an inverse method of coupling #490-37A. The strength was materially increased with the decreasing particle size. The crystalline particles appear to be of the order of magnitude of 0.5 - 1.0 μ .

EXPERIMENTAL DETAILS

See notebooks #276 and #490.

Series 276-44: Method of diazotisation.
Series 276-57: Comparison of various alkaline media for the coupling of RT-22-D.
Series 276-60: Acid couplings.
Series 276-61: Notably "A" a check for 276-60°C.
Series 490-37: Inverse couplings using Turkey Red Oil.

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PIGMENT COLOR RESEARCH REPORT

PARA RED - RA-26-D

SUBMITTED BY:
APPROVED BY:

John V. Scudi
Alfred Siegel

FILE: PARA RED
DATE: JANUARY 1935.

INTRODUCTION

The preparation of an RA-26-D for paper coating which is non-bleeding in water.

SUMMARY AND CONCLUSIONS

On the assumption that the water bleed of dark Para Reds is due solely to the soluble Para F contained, it is possible to prevent this bleed by Barium Salt formation, since the Ba salt of Para F is more insoluble than the Na salt or the free acid in water and acids. However, it appears to be completely hydrolysed in alkaline media.

The procedure for the preparation of RA-26-D involves a slightly alkaline coupling medium. This cannot be directly acidified due to the excess of nitrite present, but it is possible to restrict the amount of nitrite used or to remove this excess by washing of the product prior to acidification, or by urea treatment of the diazo solution. These three methods were used without detriment to the color and the slurries were struck with BaCl₂ in slightly acid media. The products were studied at this point for a more vigorous comparison of water-bleed, rather than after the extension with the gloss white.

It was shown colorimetrically that the bleed at 22°C. of the RA-26-D type is approximately ten times that of the product struck in slightly acid medium. At 65°C the ratio fell to 5 to 2 and at 95°C it became 5 to 3. Thus increasing temperatures cause a more marked increase in solubility of the Ba salt than for the Na salt, although the Ba salt is always the less soluble of the two salts.

When extended with gloss white, the products were tested for water bleed according to TF-7-2, and colorimetrically. The products struck on the acid side did not bleed (the color test used is sensitive to 1 part in 1,000,000), whereas those struck on the alkaline side did bleed and the color ratio of the two bleeds was found to be 1 to 10, in close agreement with the values formed for the unextended colors.

EXPERIMENTAL DETAILS

See notebook 490.

Series 490-30: pages 126 - 133.

Study of Ba salt formation, its influence on the rate of filtration, extent of bleed and tinctorial properties. Colorimetric method of comparison of Para F bleeds, and measurement data - for unextended products.

Series 490-31: pages 134 - 139

Methods used for reduction of nitrite, their influence

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tinctorially in the toners and lakes. Bleed comparisons and baking influence in the toner.

PIGMENT COLOR RESEARCH REPORT

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PARA RED DARK IMPROVED RT-70-D

SUBMITTED BY: *E. J. Hess*
APPROVED BY: *V. Chalupski*

PARA RED
FILE: ~~MISC. TONERS~~
DATE: JANUARY 1938

INTRODUCTION

Two trial runs were made in the Semi-Works on an experimental Para Red B formula (490-20B). The products were quite bright in masstone (by rubout), but did not have the blue undertone and tint required to match the competitive product. Further work in the Semi-Works has been postponed because of pressure of more important problems, but will probably be resumed during the next month.

SUMMARY AND CONCLUSIONS

Both batches were made on the same basic formulation. (One-fourth mol batches were made in each case). The formula differs in some details from K-7347, Semi-Works development for ashless para B which has been inactive since 1933. The chief differences are in (a) method of preparation of the diazo (a modified cold method is used) (b) a very slight increase in Mono Acid F (c) an increase in amount of bicarbonate in the coupling.

One semi-works batch was washed with cold water, and the other washed with warm (150°F) water. The latter batch, as expected, was lighter, stronger, and yellower than the former. Washing time was of course decreased (cold water, 212 gallons in 13 hours; warm water 106 gallons in 3½ hours).

Results on these two batches were satisfactory versus RT-70-D both being darker, vs lighter, stronger and yellower than this standard. Hot water washing gave a lighter, vs brighter, stronger and a bluer results than the cold water washed material. Against a competitive sample C-38319 (Uhlich) this hot water washed material was vs bright, and strong, but yellow in tint. Work is in progress in the laboratory in an effort to match the very blue tint of the competitive sample.

Results are tabulated in Table I.

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EXPERIMENTAL DETAILS

2.

N.B. 455, pages 50-51

TABLE I

IMPROVED RT-70-D DEVELOPMENT

Batch SW	PMA N-40 Lot	Formula	Coupling Tests	Vol.	Washing Time	Rate	Masstone	Drawdown	Strength	Tint	Std.	Yield Est.
7604	1/4 x 490- 20B cold water wash	G-408 10	No diazo, xs BN, pH 7.3	212 gal.	12-3/4 hrs.	.277 gal/min	dk, brt. s dk vs dk & brt	vs str yel & str str, vs bl	str " "	yel " vvs dty	RT-70 G38319 K7347	72 69
7607	1/4 x 490- 20B hot water wash	G-408 10	No diazo, xs BN, pH 7.0	106 gal.	3-1/2 hrs.	.505 gal/min	dk & brt. vs brt s lt & brt s lt & brt s cl	vs str. yel & str str s cl	str " "	yel " vs bl & cl s bl	RT-70 G38319 K7347 SW7604	72 72

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL TONERS, RT-379-D AND RT-300-D

SUBMITTED BY: *E. J. Heas*
APPROVED BY: *V. Chalapati*

FILE: LITHOLS
DATE: JANUARY 1936

INTRODUCTION

Plant production was high on Calcium Lithol Toner, RT-379-D, during the month a total of ten batches being reported. Bright masstone material was the principal type product required although complaints have been made by the Control Laboratory concerning the high strength of present plant production which necessitates the use of Blanc Fixe as a shading material. Extension of the toner with Blanc Fixe is undesirable because of the difficulty of securing uniform material.

Two batches of RT-300-D were also made with material satisfactory for rubber being required.

SUMMARY AND CONCLUSIONS

The first four batches of RT-379-D are outstanding from the latter part of last month. Control on these batches was satisfactory the desired light and strong material being secured in the case of the first batch and three batches of required dark, bright and strong material then being secured. The usual variables (temperature of development, amount of TOBW used and amount of calcium chloride used) were used to secure the desired results.

The next four batches, made the first part of January, were also made to secure dark, bright and strong material with fairly consistent results. The first three batches were satisfactory but the fourth, made with a new lot of "Lithosol" Acid TOB (lot 160) was slightly muddy in masstone.

A complaint was then received from the Control Laboratory that present plant production on this Lithol Toner was too strong, which made it necessary to use Blanc Fixe in shading the color and that there was a possibility of complaint by customers because of the presence of this white pigment in the Red Toner.

The next two batches were made using variables that tend to give weaker material. The first batch was made using 75% TOB - 25% TOBW and striking in the low coupling volume. This variation heretofore had the tendency to give weaker material but with the disadvantage of giving muddy masstone. The resultant product was bright but remained slightly stronger than the standard. The next batch was made to check this batch except that the caustic soda in the coupling was cut 4%. The combination of the two weakening variables gave material that was only vvs stronger than the standard RT-379-D but the undesirable slightly dirty masstone was also secured.

Recent shipments of "Lithosol" Acid TOB (lots 158, 159 and 160) have shown a tendency to give blue tint RT-379-D a type which we have not been able to make satisfactorily for some time. These results were not consistently obtained with these lots, however, as yellow tint material was also obtained with practically no variation in the formula used.

Three satisfactory mixes have been made totalling 3767 pounds which consumes a major portion of present plant production. There are at present 1846 pounds of RT-379-D in sub-standard stock.

Production is tabulated in Table I and the mixes in Table II.

Two batches of RT-300-D were made during the month using the standard K-7038 formulation. Satisfactory rubber material was the type required. Control on this product has not been very satisfactory the material made with recent shipments of "Lithosol" Acid TOB (lot 156, 160) giving dirty results in rubber versus Red SX standard. A batch made with lot 159, however, gave satisfactory results. As production is low on this color there is no opportunity to study and check results secured using variations in raw material.

A satisfactory 685 pound mix has been secured consuming most of present production. RT-300-D sub-standard stock is low totalling 277 pounds.

Production is tabulated in Table III and the mix in Table IV.

3.

EXPERIMENTAL DETAILS

TABLE I

RT-379-D PRODUCTION

Batch	Lot Amt.	TOB Lot Amt.	TOBW Lot Amt.	N-7 in BN	Coupling pH	Strike Vol. Amt.	Develop. Temp.	Massstone	Results vs RT-379-D Std.	Drawdown	Strength	Tint	Yield Est. Lump
46825	158	80%	33	20%	61-1/2#	10.6	160°F	s lt, fl & cl	vs str, s	s str	yel & cl	305	306
46863	158	65%	33	35%	61-1/2#	11.5	140°F	s dk, vs brt	s str & cl	str	vs cl & bl	305	296
46966	159	65%	33	35%	61-1/2#	12.6	140°F	dk, brt	vs str, s	s str	s yel & cl	305	292
46979	159	65%	33	35%	61-1/2#	12.6	140°F	s dk	vs yel, cl	s str	vvs bl, vs cl	305	262
47034	159	65%	33	35%	61-1/2#	-	140°F	dk	s yel	s str	cl, vs yel	305	302
47050	159	65%	33	35%	61-1/2#	12.3	140°F	dk	s yel & cl	vs str	vs yel, s	305	304
47066	158	65%	33	35%	61-1/2#	12.5	140°F	dk	vs bl	s str	s bl	305	310
47122	160	65%	33	35%	61-1/2#	11.9	140°F	dk, s muddy	s bl, vs dty	s str	s bl	305	286
47195	160	75%	33	25%	61-1/2#	11.9	140°F	dk, vs brt	s yel, vs str	s str	cl, vs yel	305	304
47213	160	75%	33	25%	59#	10.3	140°F	dk, s dty	s yel, & cl	vvs str	vs yel, & cl	305	304

TABLE II

RT-379-D MIXES

Lot	Composition	Total wt.	Results vs RT-379-D Std.			
			Masstone	Drawdown	Strength	Tint Disp.
16403	46447 282#, 3703 12#, 46320 300#, Blanc Fixe 45#, (RT-7594) 40509 48#	687#	vvs lt, vs muddy	vs yel & cl	vvs wk	vs yel & cl O.K.
16464	46863 290#, 46966 290#, 45016 8#, 46825 305#, N-65 59#, 14549 1#, 15490 13#, 15873 40#, 16403 246#	1254	vvs lt, vs brown	vs yel	vvs str	vs yel & cl O.K.
16546	47066 305#, 47050 300#, 47034 300#, 46979 260#, 46593 105#, 46778 300#, 46071 233#, N-65 23#	1826	vvs lt, vs flat	vs yel & cl	O.K.	vs yel & cl O.K.

TABLE III

RT-300-D PRODUCTION

N.B. 433 pages 127-129

Batch	Formula	TOB Lot	Rubout Results vs RT-300 Std.			Yield	
			Masstone	Drawdown	Strength	Tint	Rubber Test vs Red SX Est. Lump Disp.
46942	Check std. K-7038	159	s dk	vs yel & cl	s str	cl, vs yel	O.K. - s yel 305 286 O.K. rubber
47130	" " "	160	s dk & muddy	s bl & dty	O.K.	s bl (dty)	8% str-s bl 305 296 SSS & dty

TABLE IV

RT-300-D MIX

Lot	Composition	Total Wt.	Masstone	Rubout Results vs RT-300-D Std.			Rubber Test	
				Drawdown	Strength	Tint	vs Red SX 1st test	Disp.
16629	47130 290#, 46565 290#, 4154 30#, 3638 75#	688#	muddy	s bl	s wk	s bl (dty)	10% wk-s dull 2nd test	N G 5% wk-vs yel O.K.

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL TONER RT-8108-D

SUBMITTED BY: *E. J. Kern*
APPROVED BY: *A. Chalupski*

FILE: LITHOLS
DATE: JANUARY 1936

INTRODUCTION

Development work was started in the Semi-Works on a new Calcium Lithol Toner to replace RT-379-D. A 0.2 molar batch size was used in all cases with K-8108 being used as the basic formulation. A total of five batches were made and studied during the month.

SUMMARY AND CONCLUSIONS

The K-8108 formula differs mainly from that of RT-379-D in that Mono Acid F is used in the Beta Naphthol solution to secure the dark and blue effects desired rather than the use of TOBW mixed with the TOB in the diazo (which gives the same effect in RT-379-D). The use of ammonium chloride as a buffer in the coupling is another variation that may be noted.

The first batch was made to check the standard K-8108 formula using TOB lot 159. Some difficulty was encountered in making the diazo, the TOB precipitating before adding the sodium nitrite when using the formulated volume of 390 gal/mol. An increase to 480 gal/mol was found necessary to prevent precipitation. This last volume was used in all subsequent batches. Because of this trouble or for some other unknown reason this first batch was off, being considerably darker, bluer and weaker than either RT-379-D standard or K-8108.

The next batch was made to exactly check the first including the use of TOB lot 159 and the higher diazo volume. Results were satisfactory, the product being darker, stronger and vs bluer than RT-379-D standard and vs light and bright, vvs strong, and s yellow versus K-8108.

The next two batches were made to exactly check this second batch except that TOB lots 158 and 160 were used respectively. Fairly good checks to the second batch were secured even when using TOB lot 158 which had given abnormal results when used in making Barium Lithol Toner (see RT-364-D report for January 1936).

TOB lot 160 was selected as satisfactory for use in making a variable study and the fifth batch was made to check the above except that the Mono Acid F was omitted and the Beta Naphthol was increased in molecular proportion. A 2% excess of Beta Naphthol was used in this batch and the Mono Acid F is used as 72% material. This formula, of course, resembles the RT-300-D standard more closely than RT-379-D. The resultant product was, as expected, considerably lighter and yellower than either RT-379-D or K-8108. Rubout

and rubber tests were then made versus RT-300-D with the following results: by rubout the material was s dark and considerably brighter, s strong and s yellow versus RT-300-D and by rubber test was vs strong, very yellow and clean in tint versus Red SX (the RT-300-D rubber standard). A rubout was then made versus a competitive sample of Calcium Lithol Toner C-38413 (Sherwin Williams) which is browner in masstone, vs blue in tint but 10% stronger than RT-379-D standard. Our new product was vvs light but bright, vvs weak and yellow versus this C#. It is believed that use of a small amount of Mono Acid V in this type formula will enable us to match the competitive product. A variable study will be made in the Semi-Works before this formula is released for plant production.

Results are tabulated in Table I.

TABLE I

RT-8108-D DEVELOPMENT

Batch SW	Formula	TOB Lot	Mono Acid F Lot	Amt.	BN Amt.	Coupling Tests	Std.	Rubout Results	Strength	Yield
								Drawdown	Int.	Est.
7590	.2 x K-8108	159	10	2.16	28.4	xs diazo & BN pH = 9.7	RT-379 K8108	dk dk	bl bl,cl, wk	83 83 82
7598	.2 x K-8108	159	10	2.16	28.4	No diazo, xs pH 10.3	BN RT-379 K-8108	dk vs lt & vs cl brt.	str vvs str s yel	83 83 85
7612	.2 x K-8108	158	10	2.16	28.4	No diazo, xs BN pH 10.7	RT-379 K-8108	dk & brt vs lt s yel	vs bl vvs str s yel	83 83 84
7614	.2 x K-8108	160	10	2.16	28.4	No diazo, xs BN pH 10.3	RT-379 K-8108	vs lt, vs brt	yes vvs str yel	83 83 84
7623	.2 x K-8108 except omit N-40. Increase N-3 in mol propor- tion.	160	10	-	29.4	No diazo, xs BN pH 10.5	RT-379 K-8108 RT-300	lt lt s dk, brt	yel yel yel, vs str, cl	83 83 84
Rubber Test										
Red Sx O.K. - yel & clean										

Tests versus Competitive Sample

SW-7623 vs C-38413: vvs lt, brt - vs wk - vvs wk - yel.

PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL LAKE RL-8109-D

SUBMITTED BY: *E. J. Hess*
 APPROVED BY: *A. Chalupski*

FILE: LITHOLS
 DATE: JANUARY 1936

INTRODUCTION

In conjunction with the Semi-Works development of Calcium Lithol Toner, K-8108, a study was made on a Calcium Lithol Lake, K-8109, to replace RL-408-D (extended RT-379-D for rubber). Two batches were made during the month.

SUMMARY AND CONCLUSIONS

The K-8109 Toner formulation is an exact check to K-8108 except that the Mono Acid F is omitted with the Beta Naphthol increased in molecular proportion. The coupled soda salt is extended with 162#/mol of Blanc Fixe and the slurry struck and developed as in K-8108. The finished RL-8109 contains 72% toner as compared with 30% toner in RL-408-D standard.

70%
 Two checks to the standard K-8109 formulation were made, TOB lot 159 being used in each case. Check rubout results were secured both batches being dark, strong and vs blue versus K-8109 and light, strong and yellow versus RL-408-D. In rubber both batches were practically identical to K-8109 and about 12% stronger and s cleaner than RL-408-D.

Work will be continued in the Semi-Works to secure a complete variable study before finally releasing the formula.

Results are tabulated in Table I.

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Batch	Formula	TOB Lot	Coupling Tests	After Devel-oment	Std.	Mass- Draw- down	Rubout Results	Str.	Tint	Rubber Test Std.	Yield Est.	Lump
SW-7594	Check K-8109 (.2 size)	159	xs BN, no diazo pH 10.3	no pH=10.7	K-8109	dk	vs str s str	vs bl & cl	vs bl & cl	K-8109 RL-408	OK tr yel 12% str -s clean	115# 113#
SW-7601	Check K-8109 (.2 size)	159	xs BN, no diazo pH 10.3	pH 9.9	K-8109	dk	vs str str	vs bl & cl	vs bl & cl	K-8109 RL-408	OK-OK 12% str -s clean	115# 116#

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL TONER, RT-364-D

SUBMITTED BY: E. J. Heas
APPROVED BY: V. Chalupski

FILE: LITHOLS
DATE: JANUARY 1936

INTRODUCTION

Production in the plant on this color continued under Development Section supervision. A total of five batches are reported this month including three batches outstanding since December, 1935. Light and strong material was required for shading purposes.

SUMMARY AND CONCLUSIONS

Control on this color remains unsatisfactory the principal difficulty being the production of material no more than very slightly dark in masstone, satisfactory in strength and O.K. or blue in tint versus RT-364-D standard.

The first batch reported was made checking the present plant standard formula (check to K-7290 except using 45#/mol of Para soap instead of 60#/mol) except that the Beta Naphthol used was dissolved at a temperature of 750 - 800°F instead of 160°F. Solution is secured by adding the Beta Naphthol to the cold water with agitation, the caustic soda, in solution, is added to the Beta Naphthol in suspension and the mixture stirred to solution. The mixture may be heated slightly, if necessary, to secure complete solution. This variable has given lighter results in a Barium Lithol Lake (RL-402-D). The resultant color was dark, blue and vs strong, an abnormal result, and was due to the use of "Lithosol" acid TCB, lot 158. This result was checked in a subsequent batch omitting the Beta Naphthol solution variable and cutting the Para Soap which is a variable used to give lighter material. The abnormal dark result was again secured using TCB lot 158.

The remaining three batches were made to secure light material with satisfactory strength. To reserve this result the cut in Para Soap (24#/mol and 18#/mol instead of 60#/mol) was used together with TCB lot 156, and 160. Results were normal the resultant products being light, O.K. in strength and yellow versus the standard.

A satisfactory 1635 pound mix has been made consuming part of recent production. Sub-standard stock on this color remains high being 2255 pounds.

Production is tabulated in Table I and the mix in Table II.

EXPERIMENTAL DETAILS

2.

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TABLE I

RT-364-D PRODUCTION

Batch	Formula	TOB Lot	Amt. N-39	Coupling pH	Devel. pH	Massstone	Results vs RT-364-D Std. Drawdown	Strength	Tint	Yield Est. Lump	Notes
46793	Check K 7290 except 32#/mol N-39 used	158	24#	7.9	7.9	dk	bl	vs str	bl	360 350	BN dissolved at 75°F
46852	Check K-7290 except 24#/mol N-39 used	156	18#	7.0	6.4	s lt & flat	vs wk, vs yel	O.K.	yel & cl	360 343	
46886	Check K-7290 except 24#/mol N-39 used	158	18#	7.3	6.5	dk	s str & bl	s str	s yel	360 334	
47102	Check K-7290 except 18#/mol N-39 used	160	13-1/2#	7.1	6.4	s lt (flat)	vs yel & cl	O.K.	yel & cl	360 356	
47236	Check K-7290 except 18#/mol N-39 used	160	13-1/2#	7.1	6.3	s lt, flat	vs yel & dty	s str	yel & cl	360 348	

TABLE II

RT-364-D MIX

Lot	Composition	Total wt.	Results vs RT-364-D Std.			
			Massstone	Drawdowns	Strength	Dispersion
16612	46852 340#, 43913 49#, 45612 470#, 45463 350#, 47102 356#, 46793 70#	1635#	vs dk & muddy	vs str & yel	vs str	vs yel O.K.

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PIGMENT COLOR RESEARCH REPORT

MEDIUM CALCIUM DUOL TONER, RT-265-D

SUBMITTED BY: E. J. Hess
APPROVED BY: V. Chalupski

FILE: DUOLS
DATE: JANUARY 1936

INTRODUCTION

Production on RT-265-D was started in the plant to manufacture dark and vs blue shading material to mix off approximately 800 pounds of sub-standard stock averaging light and vs yellow versus standard RT-265-D. Two batches were made under Development Section supervision.

SUMMARY AND CONCLUSIONS

Considerable difficulty has been encountered in recent months in securing blue tint RT-265-D. The one variable that consistently gives blue results, that of substituting "Lithosol" acid TOBW for part of the "Lithosol" Acid TOB used in the coupling, also gives weak and dirty tint and is undesirable for this reason.

The variables used in these batches were those that had given blue results in RT-265-D production during 1934. These variables included use of a mixture of M Gum Rosin and B Wood Rosin, decrease in alkalinity of the coupling by cutting the caustic soda used in the Beta Naphthol solution, and cuts in the volume of soda salt development and strike volume. The first batch was made omitting the cut in volume of soda salt but the second batch was made using all these variables to give blueness. Results were unsatisfactory both batches being dark and bright, strong, but yellow in tint versus RT-265-D. The only reason that can be given for these consistently yellow results as compared with previous production is changes in the quality of the rosin or Lithosol Acid TOB.

A satisfactory mix totalling 1257 pounds has been made, however, consuming all but 85 pounds of outstanding sub-standard stock together with one batch of this month's production. A small amount (less than 1%) of Mapico Red light was used to secure the blue tint required to secure a satisfactory mix. Sub-standard stock is low totalling 529 pounds which includes one of the dark, bright yellow batches made this month (weight 444 pounds).

Production is tabulated in Table I and the mix in Table II.

EXPERIMENTAL DETAILS

2.

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TABLE I

RT-265-D PRODUCTION

Batch	TOB Lot	N-7 in BN	Coupling pH	Resin Amt. B	M	After Devel. pH	Mass. Drawdown	Results vs. RT-265-D Std. Str.	Tint	Yield Est. Lump	Notes
47275	158	80#	10.2	40	100	10.9	dk, s brt.	dk, s brt.	s yel cl	440 445	
47339	158	77	10.3	40	100	-	dk	s str, yel str	yel	440 444	as 47275 except variations in develop. vol.

TABLE II

RT-265-D MIXES

Lot	Composition	Total Wt.	Results vs RT-265-D Std. Mastone Drawdown	Strength	Tint	Disp.
16650	44849 243#, 45061 80#, 45110 454#, 47275 440#, N-341 10#, N-65 25#	1257#	vvs dk, s bl vs bluish	vs str	vs bl, vvs dirty	O.K.

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PIGMENT COLOR RESEARCH REPORT

HANSA YELLOW LAKE, YP-324-D

SUBMITTED BY: *E. J. Hess*
APPROVED BY: *V. Chalupski*

FILE: HANSA YELLOW
DATE: JANUARY 1936

INTRODUCTION

Production was started in the Semi-Works on YP-324-D to build up standard stock. There is no sub-standard stock on this color so that satisfactory material was desired. A total of eight batches were made during the month.

SUMMARY AND CONCLUSIONS

All eight batches were made on the same formulation checking SW-7208 which had given satisfactory material in recent production. All eight batches were satisfactory averaging vs green and dirty in toptone, O.K. in strength, and vs green and clean in tint.

A variation from the standard method of dissolving the aceto acetanilid, that of adding the dry material directly to the dilute alkaline solution without pulping with water beforehand, (as in the standard formula), was tried in two of these batches with practically no effect on the quality of the solution or on the final product. In the case of one batch in which the diazo was contaminated with lead nitrate it was necessary to clarify the diazo in order to separate the precipitated lead compound (lead chloride?). This batch was also satisfactory.

Yields were low on these eight batches averaging 96½% of estimate. This is a 3% decrease in yield under that secured in the last Semi-Works series. The use of a weak lot of MNPT (lot 456) may be partially responsible for this low yield.

A satisfactory board mix has been made consuming all of these eight batches and the mix is now in process.

Results are tabulated in Table I.

TABLE I

YP-324-D PRODUCTION

Batch	Formula	Lot MNPT	Lot AAA	Coupling Vol.	pH	Results vs YP-324 Std.		Strength	Tint	Opacity	Yield	
						Masstone	Drawdown				Est.	Lump
SW 7636	Check SW-7208	456	16	450 gal.	4.6	vs dk & dty	vs grn	O.K.	s green	s more transp.	118	113
SW 7641	Check SW-7208	456	16	"	4.5	vvs lt, grn, s dty	"	Tr str	"	vvs more transp.	118	113
SW 7646	Check-7208 except did not pulp N-222. 1/11 BARS. lost	456	16	"	4.5	vvs lt, s grn	vs grn & cl	vvs str	"	vvs more transp.	118	113
SW 7650	Check 7208	456	16	"	4.5	lt & grn	s grn & cl	vvs str	green vs cl	vvs less transp.	118	111
SW 7656	Check 7208. Diazo con- taminated with Pb	456	16	"	4.5	vs lt & gr s dty	vs grn & cl	O.K.	s green	vs more transp.	118	113
SW 7661	Check 7208	456 458	16	"	4.5	lt & grn	vs grn & cl	vvs str	green vs cl	vvs less transp.	118	116
SW 7662	Check 7208, Did not pulp N-22, N-28 in N-222 sol cut to 2# 100%	458	16	"	4.7	s dk, vs gr & dty	s grn & cl	O.K.	s green	s more transp.	118	116
SW 7669	Check 7208. Did not pulp N-222	458	16	490 gal.	4.5	s lt & grn	s grn & cl	vvs str	green	equal transp.	118	116

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PIGMENT COLOR RESEARCH REPORT

RED TONER K-7699

SUBMITTED BY: *Fr. L. Truffer*
APPROVED BY: *Alfred Siegel*

FILE: MISC. TONERS
DATE: JANUARY 1936

INTRODUCTION

Work on K-7699 (MNPA-BN) was concluded during this month. Material very close to the original K-7699 was obtained and a large sample has been prepared for Philadelphia.

SUMMARY AND CONCLUSIONS

The main difficulties in matching K-7699 were due to low purity of the Meta-nitro para anisidine (D-2234). Increases in depth were obtained by increase in MNPA and decrease in the amount of second component. This combined with a slight increase in Hydrochloric and acetic acid resulted in material of satisfactory masstone, brighter, cleaner undertone and an increase in strength of over 10% compared with K-7699. A composite mix was made of four 0.5 mol. laboratory batches which checked each other closely. This mixture K-7699 (487-53) tested in lacquer is slightly light and bright versus K-7699. A pound sample was shipped to Philadelphia for evaluation.

EXPERIMENTAL DETAILS

487-46. Series. Increase in Meta Nitro Para Anisidine by 5, 10 and 15%.

A 5% increase resulted in an increase in brightness, whereas a 10% and 15% increase resulted in increased depth accompanied by a loss in brightness.

487-48. Lowering the amount of second component to balance the Meta Nitro Para Anisidine D-2234, which is of low purity, and increase in acetic and Hydrochloric acid each by 5% were studied. Hydrochloric acid was added to the diazo. Distinct improvements in depth were obtained with the increase in acidity and the depth of K-7699 was reached. Material of a darker masstone but possessing a faint brownish cast was obtained with the increase in Acetic Acid.

487-50. Two 0.5 mol. batches were made on the formulation 48D (increased Hydrochloric acid). Both batches were decidedly lighter than material obtained on the mol. couplings. During coupling the development of carbon dioxide caused severe foaming. No development of CO₂ was observed in the 0.1 mol. couplings.

487-51. Two 0.5 mol. couplings similar to Expt. 50, but with increase in Acetic Acid. The Sodium bicarbonate was added to the PN solution just prior to addition of the diazo. The Sodium bicarbonate is added undissolved and shall dissolve progressively during the coupling

process so balancing the alkalinity of coupling. No carbonate development takes place when the sodium bicarbonate is added just before coupling, whereas a strong development occurs when the sodium bicarbonate is completely in solution. At the start of coupling this causes trouble by foaming and results in undesirable shade.

487-52. Check runs on 51. Material close to 51 was obtained.

487-53. Composite mix of 51 and 52.

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PIGMENT COLOR RESEARCH REPORT

M.N.P.A. - N.B.S. MAROON

SUBMITTED BY: *John V. Scuderi*

APPROVED BY: *Robert E. Siegel*

FILE: MISC. TONERS

DATE: JANUARY 1936.

INTRODUCTION

Preliminary couplings of M.N.P.A. to N.B.S. were made with the object of obtaining dark maroons for "Dulux".

SUMMARY AND CONCLUSIONS

The diazonium of M.N.P.A. was coupled to N.B.S. in an alkaline ($\text{Na}_2\text{CO}_3 - \text{NaOH}$) medium. The N.B.S. was taken into caustic solution by preliminary wetting with dioxan.

In the presence or absence of Turkey Red Oil, heat development of the product in alkaline media gives improved darkness and brightness. If the medium is acidified the reverse is true. The products tend to become light and flat in development. The use of Turkey Red Oil appears to be advantageous. The products are not only softer in texture, but darker in masstone. The best product was obtained using T.R.O. and development in alkaline medium.

The products show some promise. They have good hiding power. In this respect they are better than RT-297-D and probably better than RT-354-D. The products are darker than either standard. The increase in depth comparing product 1C² with RT-354-D is greater than the increase in depth observed in comparing RT-354-D with RT-297-D.

EXPERIMENTAL DETAILS.

Notebook #499, page 8. The influence of development in alkaline coupled products.

The influence of acidification, and development of the acidified product.

The influence of T.R.O. Development and acidification of T.R.O. containing products.

PIGMENT COLOR RESEARCH REPORT

ALIZARINE TONER RT-332-D

SUBMITTED BY: *H. Rotherham*
 APPROVED BY: *V. Chalupski*

FILE: MISC.TONERS
 DATE: JANUARY 1936

INTRODUCTION

Three laboratory experiments were run in order to produce brighter plant material. Plant production has been lacking in depth and particularly in brightness.

SUMMARY AND CONCLUSIONS

This series included a study of lower development temperature, shorter time of development and the adjustment to a pH of 6.0 before development. A pigment very slightly dark and slightly bright versus standard was produced by adjusting a sample of plant slurry (Lot 47305) to a pH 6.0 and developing in the laboratory.

EXPERIMENTAL DETAILS

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Observe the effect of holding at 165°C for three hours, then bringing to a boil

A - Plant slurry (Lot 47274) filtered and washed
 B - " " " " held at 165°F for 1 hr.
 C - " " " " " " 165°F " 2 hr.
 D - " " " " " " 165°F for 3 hr.
 E - D brought to a boil and boil 2 minutes

Results

A vs Std. dk. muddy, blue; O.K., blue - Light fastness - equal
 B vs " " " " " " " " " " " "
 C " " " " " " " " " " " "
 D " " " " " " " " " " " "
 E " " " " " " " " " " " "
 B vs A lt, muddy, O.K; str. vs clean - Light fastness - equal
 C vs A " " vs yellow; str, " - " " - vvs better
 D vs " " " " " " " " - " " - vs better
 E " " " " " " equal sl yellow - Light fastness vs better

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Study the effect of adjusting plant slurry to pH 6.0 before development

A - Control (Lot 47305)-usual development
 B - Adjusted to pH 6.0

Results

A vs Std. dk. O.K., vvs. str. -vvs str, s blue - Light fastness - vs better
 B vs " vs dk, s bright, vs str. - vs str, vs blue - " " - "

B vs A sl. lt, vs bright, O.K. - O.K, vvs yellow - Light fastness - equal

P. 60

Rapid development of plant slurry

A - Control (Lot 47305) usual development

B - B - brought to boil as fast as possible and
boiled 2 hours

Results

A vs Std. dk, vs brown, vs str; O.K, sl blue - Light fastness - vs better

B vs " " " " " " " " " " " " " " " "

B vs A, vs lt., O.K., O.K; O.K., vs dirty - Light fastness - equal

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PIGMENT COLOR RESEARCH REPORT

LACQUER MAROON TONER, RT-354-D

SUBMITTED BY: *A. S. Readings*
APPROVED BY: *1 Chalupski*

FILE: BORDO MAROONS
DATE: JANUARY 1936.

INTRODUCTION

Work on this problem was continued in the semi-works. Attention was given to materials used and chiefly to selection of a suitable lot of T.O.B.W. Batch size was reduced to 0.1 mol to prevent increase in substandard stock. In the plant one batch of RT-130-P was made for Parlin.

SUMMARY AND CONCLUSIONS

With Lot #34 T.O.B.W., which had been used in most of the work to date, the product obtained on the smaller charge was satisfactory in depth and brightness.

However, Lot 33 could not be used although several variations were tried. The resulting products were too light.

Lot #35 gave a dark product with satisfactory brightness on both 0.1 and 0.2 mol charges although it was necessary to keep the alkalinity of coupling very low. 2000# of this lot were ordered for use on RT-354-D. Material obtained with this lot was used to make up 2500# of standard material in which was included 1800# of a slightly light "off" mix carried in substandard stock. Substandard is now 2193# and consists almost entirely of light material.

The shift to the smaller coupling equipment used for the reduced charges had the effect of giving a slightly lighter and brighter product in lacquer. It is thought that this tendency was due to the vigor with which the small charge was boiled in the large size development vat and in latter batches more moderate boiling was employed.

The red appearance of the diazo from T.O.B.W. lot 33 led to the erroneous conclusion that this lot contained too much Bronner's Acid. On this basis, mixture with various amounts of T.O.B. Regular were tried without improving the product. It was learned from the Dyeworks that Lot 33 was actually low in percent of Bronner's, containing only 5% as compared to 7.4% in Lot #34 which was satisfactory. Adding Bronner's Acid to Lot #33 to make up this difference was not successful.

The mixtures of T.O.B. Regular and 6 or 7% of Brönner's Acid which had previously given sufficient depth resulted in light products. However, the poor results of this series can be fairly definitely ascribed to the type of B.O.N. (beta oxynaphthoic acid) used. This assumption is supported by the fact that B.O.N. lot #145 which was used, also gave the only light products obtained with the satisfactory T.O.B.W. lot #35 while B.O.N. lot #154 gave dark products. Further comparison of these two lots will be made in the laboratory. The first thought that the difference may be due to moisture content was found to be incorrect.

It is believed that with intermediates of the type indicated above no further difficulty will be experienced in the semi-works, at least during cold weather, in obtaining depth and brightness. Some doubt is felt about maintaining brightness during summer months due to possible instability of the soda salt. Use of ice on or after coupling may correct this apparent difficulty.

The batch of RT-130-P, made in the plant, was similar to that made last month in formula and in lacquer test on slurry control. A sample of pulp dried in a 1 inch cake was slightly light and brown in lacquer. However, the material appears to have been satisfactory for Parlin's needs.

EXPERIMENTAL DETAILS

N.B. #480 - p. 151 to 170.

O.1 mol charges were made this month with exception of the last two batches. The small coupling equipment (Mats 15 and 33) was used for both sizes.

SW.	Variations	T.O.B.W. Lot	B.O.N. Lot	Coupling Test	Development (pH)	Lacquer Test vs. RT-354-D
7557	1/2 of SW-7546 using T.O.B.W. (O.1 mol charge)	Lot # 34	153	Positive BY Slight R.L.	9.55	s.dark & brt.
7559	check SW-7557 using T.O.B.W. Lot #33		"	Strong B.Y. Positive R.L.	9.4	light
7564	check SW-7559 except use T.O.B.W. 94%; T.O.B.Reg. 6%	"	"	positive B.Y. slight R.L.	9.55	light, v.brt.
7567	check SW-7559 except use T.O.B.W. 85%; T.O.B.Reg. 15%	"	153) 145)	slight B.Y. V.sl.R.L.	9.5	light
7569	check SW-7559 except use T.O.B.W. 75%- T.O.B.Reg. 25%	"	145	Postige B.Y. Slight R.L.	9.4	light, v.s.brt.
7602	Check SW-7559 except use T.O.B.W. 97.5%; Bronner's Acid 2.5%	"	154	v.sl.B.Y. sl.R.L.	9.5	light
7573	check SW-7557 except use T.O.B.Reg. lot 155, 94%; Bronner's Acid 6%	"	145	positive B.Y. slight R.L.	9.5	very light
7575	check SW-7573 except use T.O.B.Reg. lot 158.	-	"	"	9.6	light, v.s.brt.
7577	check SW-7573 except use T.O.B.Reg. lot 159.	-	"	slight B.Y. v.sl.R+L.	9.5	light
7615	Check 7577 except use T.O.B.Reg. lot 159, 93%; Bronner's Acid 7%	-	"	"	-	light
7583	check 7557 except use T.O.B.W. lot 35		"	slight B.Y. slight R.L.	9.7	light, s.brt.

S.W.	Variations	T.O.B.W. Lot	B.O.N. Lot	Coupling Test	Development (pH)	Lacquer Test vs RT-354-D
7587	Check 7583	35	154	v.slight B.Y. sl.R.Lo	9.4	dark
7593	check 7587 except couple in 15 mins. instead of 1/2 hour.	"	"	slight B.Y. v.sl.R.Lo	9.5	dark
7605	check 7593 except use full (std.) amt. of NaOH in final development.	"	"	v.sl.B.Y. V.sl.R.Lo	9.4	s.dark & brt.
7606	check 7587 except use full (std.) amt. of NaOH in final development.	"	"	"	9.6	s.dark
7611	check 7611 except use B.O.N. lot #145	"	145	v.v.slight B.Y.	9.7	light, s.brt.
7619	2 x 7606 (0.2 mol charge)	"	154	v.sl.B.Y. " R. h.	9.4	dark, v.s.brt.
7622	check 7619	"	"	"	9.4	dark

Lot 47168 - RT-130-P. Same as 46384 Slurry Control - Vat 105 dark Vat 106 - s.dark
Dried Pulp - slightly light.

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PIGMENT COLOR RESEARCH REPORT

LACQUER MAROON RT-354-D

SUBMITTED BY: *Fr. A. B. B.*
APPROVED BY: *Alfred Sigel*

FILE: *BORD* MAROONS
DATE: *1* JANUARY 1936

INTRODUCTION

Laboratory work was started this month on RT-354-D with the object of improving the stability of the formula K-7507. Offshade material is occasionally obtained due to difficulties encountered in control of the present formulation.

A further object of this investigation is to improve the depth and brightness of RT-354-D over its present standard, in order to match Harmon's Deep Infrex Maroon.

SUMMARY AND CONCLUSIONS

The difficulties in controlling the process for RT-354-D appear to be in the coupling which does not go to completion. The coupling reaction ends with a slight excess of diazo and a faint excess of B.O.N. The excess diazo remains through the various steps of the process and disappears only when the development temperature reaches approximately 70°C. It has not been determined whether the decomposition products of the diazo have a beneficial or harmful effect upon the pigment properties but undoubtedly reactions of this kind are difficult to control. Work was directed to eliminate the excess diazo. An increase in alkalinity forced the coupling to completion, but resulted, however, in a lighter shade owing to insufficient excess B.O.N. which is necessary for the formation of the iron salt of Nitroso-B.O.N., upon which RT-354-D is dependent for its depth. However, the depth of RT-354-D was obtained by increasing the quantity of B.O.N.

The T.O.B.W. is another factor which causes inconsistencies in shade: first, through the tendency of the quick crystal growth of the T.O.B.W. diazo which influences the rate of coupling and causes variations in the excess of diazo, and secondly, through variation in the amount of Broenner's acid in the T.O.B.W.

Experiments in which the quantity of Broenner's Acid was varied in the T.O.B. regular in the laboratory indicate that the maximum depth and brightness is obtained with 7-8% Broenner's Acid. This is in agreement with recent semi-works studies, in which 7.5% Broenner's acid was found to give maximum depth. Therefore, 7.5 percent added Broenner's Acid is recommended in the future preparation of RT-354-D. Efforts were made to eliminate the T.O.B.W. by T.O.B. reg. Neither slurry mixes of T.O.B. reg.-B.O.N. couplings with Pigment Green B, nor couplings with T.O.B. reg.-B.O.N. + Mena acid F showed any promise.

No definite improvements in depth and brightness over standard RT-354-D were obtained. However, some promising leads, which warrant further investigation, have been uncovered.

Semi-works trial of the higher alkaline complete coupling method, using 7.5 per cent Broenner's and an increase in B.O.N. excess, is recommended.

EXPERIMENTAL DETAILS

424-27. Substitution of T.C.B. reg. for T.O.B.W. results in very light and flat material versus control on RT-354-D formulation.

Addition of Pigment Green B and Green Toner GL-407-D gave only a very slight increase in depth and caused poor dispersion in lacquer.

424-28. Decrease in volume by 50% had no marked effect. T.O.B. reg. - B.O.N. Mono Acid F coupling resulted in light and dull material; however, it was darker and brighter than the straight T.O.B. reg - B.O.N. coupling.

Applying modification of alkaline buffered coupling and long digestion [adopting working procedure of K-8108 (calcium-Lithol)] material was obtained which showed a distinct improvement over its control. This series, however, was made without the formation of the Nitroso B.O.N. Iron compound and therefore very light versus RT-354-D.

424-29. Increase in alkalinity of coupling by 5, 10 and 20% in NaOH gave complete coupling (absence of excess Diazo) with 10% increase in NaOH resulting in lighter but slightly brighter material. A 20% increase in NaOH showed a tendency toward brownness.

424-30. Modifications of the improved Calcium Lithol formulation (K-8108) were applied to RT-354-D. The variation consisted of an increase in Sodium acetate, slight increase in alkalinity, use of Ammonium chloride and longer digestion time. These variations resulted in light material.

424-31. Increase in copperas. A 10% and 20% increase in copperas, and an increase of 10% excess B.O.N. in the higher alkaline coupling 29C procedure resulted in no improvement in depth.

424-32. Neutralization of the Sodium salt of the T.O.B.W.-B.O.N. dye with HCl immediately after coupling (lowering pH from 10.2 to 7.) resulted in slightly darker material versus the control but lighter versus standard RT-354-D.

Lowering the pH to 6.8 resulted in decreased depth. Addition of the excess B.O.N. as nitroso compound (formed separately) resulted in decreased depth.

424-33. Study made on 29C formulation. Addition of excess B.O.N. as nitroso compound resulted in slightly lighter material, checks results of 32. 10% increase in copperas resulted in a slightly lighter material. A 10% substitution of B.O.N. by Mono Acid F resulted in a lighter shade. The dye of T.O.B.W. - Mono Acid F is lighter than the dye of T.O.B.W. - B.O.N.

424-34,39. Adding Broenner's acid to T.O.B. reg. in various amounts.

T.O.B. reg.	98%	96%	94%	93%	92%	91%
Broenner's Acid	2%	4%	6%	7%	8%	9%

Increasing amounts of Broenner's acid in T.O.B.W. resulted in an increase in depth, maximum depth being obtained with a Broenner's acid content of 7 to 8%.

424-35. Separate diazotisation of T.O.B. reg. and Broenner's acid, mixed together before coupling, and separate coupling of Broenner's acid diazo followed by T.O.B. reg. diazo, resulted in no improvements, in the laboratory, which would justify a change from our present standard procedure. However, these changes in procedure are worthy of some consideration and should be checked in the semi-works. This was however studied on the formulation with high alkalinity of coupling which seems to be less susceptible to the effect of crystal growth of the T.O.B.W.diazo.

424-36. Development of the Sodium salt of the T.O.B.W.-B.O.N. dye at the boil results in an increase of crystal size of the dye. The fine irregular particles of the sodium salt obtained are distinctly crystalline. Preparation of RT-354 from heat developed dye resulted in very light material. Filtration and reslurrying of the boiled sodium salt results in a much brighter and darker product than obtained with the unfiltered dye but which is still much lighter than RT-354-D.

424-37. Increase in Hydrochloric acid in order to balance the high alkinity of the completely coupled 29C dye, and a similar variation on a dye prepared by substituting Nitroso B.O.N. for B.O.N. as coupling component, resulted in darker and slightly brighter pigment than the control. The nitroso-B.O.N. coupling is of interest since all excess B.O.N. is already in the nitroso form, which is necessary for composing the Iron complex. The material is dark, slightly brown versus control but still light versus RT-354-D.

424-38. Readjustment of pH to 9.6 on the 37 formulation. This study was made with the sodium salts of 37 series. No conclusions can be drawn as the sodium salt was detrimentally influenced by long standing after diluting.

424-40. Addition of the excess B.O.N. as Nitroso B.O.N. dissolved in caustic resulted in a slight increase in depth but gave a distinct brownish cast. No increase in depth was obtained by this procedure on the formulation with high alkalinity of coupling (29-C).

424-41. Reducing the rate of coupling to 1/3 and eliminating the digestion on 29-C formulation showed no considerable change. An increase in depth was obtained with an increase in Hydrochloric acid by 10%, this checks previous results (37).

424-42. Pouring the sodium salt of the T.O.B.W.-B.O.N. dye into the water containing Hydrochloric acid and sodium nitrite should assist the formation of nitroso B.O.N; however, no appreciable improvement in shade was obtained in this experiment.

424-43. Increase in excess of B.O.N. by 10%, 20% and 40%. A 10% increase gave a slight and a 20% increase a very distinct improvement in depth. The depth of RT-354-D was reached in the laboratory with the 20% increase in excess B.O.N. in the formulation employing completely coupled dye.

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PIGMENT COLOR RESEARCH REPORT

GREEN TONER, GT-337-D

SUBMITTED BY: E. J. Hess
APPROVED BY: V. Chalupski

FILE: P.T.A. COLORS
DATE: JANUARY 1936

INTRODUCTION

Semi-Works production was started the latter part of last month on the manufacture of clean GT-337-D for shading sub-standard stock. A total of five batches were made with a total estimated weight of 175 pounds.

SUMMARY AND CONCLUSIONS

The five batches were made using Brilliant Green B as a shading dyestuff to produce the clean results desired.

Rubout results were satisfactory average results being dark, vvs strong and vs blue and clean versus GT-337-D.

Yields were satisfactory, the lump yield averaging 103% of estimate.

A satisfactory 306 pound mix has been made consuming the first three batches made in the Semi-Works (total wt. 106#) together with 200 pounds of dirty tint sub-standard material. Sub-standard stock now consists of 531 pounds of dirty tint, O.K. strength material and 75 pounds of clean material representing the last two batches of Semi-Works production.

Production is tabulated in Table I and the mix in Table II.

EXPERIMENTAL DETAILS

TABLE I

N.B. 405

GT-337-D PRODUCTION

Batch	Dye Type	Lot	Amt.	PTA PH	Results vs GT-337-D Std. Masstone Drawdown	Strength	Tint	Yield Est. Lump	Notes
SW7543	N-109 N-338	6864 6875	12.96# 1.44#	1.6	dk	vs str, bl	str bl clean	34 35	Lot 4435
SW7548	N-109 N-338	6864 6875	11.5# 2.9#	1.6	dk	vs str & bl	str clean	34 36	Lot 4436
SW7553	N-109 N-338	6864 6875	10.1# 4.3#	1.6	dk	vvs str, vs bl	" vs bl clean	34 35	" 4437
SW7558	N-109 N-338	6864 6875	10.1# 4.3#	1.5	dk	vvs str, cl	" vs bl clean	34 35	" 4438
SW7562	N-109 N-338	6864 6875	10.1# 4.3#	1.6	dk	clean vvs wk	clean clean	39 40	" 4439 5# N-65 added to struck color before pressing

TABLE II

GT-337-D MIX

Lot	Composition	Total wt.	Masstone	Results vs GT-337-D Std. Drawdown	Strength	Tint	Disp.
16582	3897 35#, 3844 110#, 4435 35#, 4436 36#, 4437 35#, 28693 30#, 28781 25#	306#	s lt	vs wk	vs wk (floods vs str)	s clean	O.K.

PIGMENT COLOR RESEARCH REPORTORANGE LAKE, K-8098 and 8099

SUBMITTED BY: *G. L. Reiding*
APPROVED BY: *V. Chalupka*

FILE: MISC. LAKES
DATE: JANUARY 1936.

INTRODUCTION

Three batches of Orange II lake were made in the semi-works in a trial of the laboratory formula K-8098. This was developed to match Ansbacher-Siegle's product, C-58038, by dry mixing with Y-299-D and blanc fixe.

SUMMARY AND CONCLUSIONS

Semi-works products were duller in masstone and yellower in undertone than the laboratory standard. This is due to rate of strike used. In the semi-works as in the plant, 2 minutes is as fast as the barium chloride can be added thru a line while in the laboratory the precipitating agent was dumped in. Increasing the time of strike from 2 to 6 minutes gave a lighter yellower product with greater strength. Striking at 150 instead of 130°F had a similar effect although the contrary was expected from laboratory results.

Drying at a constant humidity of 30% gave a very slightly weaker and slightly bluer product.

All the material made was mixed as K-8099 and will be submitted to the customer, Binney and Smith. The mix which employed less Y-299-D and more blanc fixe showed some of the dullness and yellowness of the straight orange lake.

EXPERIMENTAL DETAILS

N.B. #430, p. 57.

All charges were 1/20th of K-8098 using 20# Orange II dye lot 17 which was also used in Laboratory work.
SW-7581 - BaCl₂ added in 2 minutes at 130°F vs K-8098 - v.s.lt. muddy; yellow, s.strong; yellow.

SW-7591 - BaCl₂ added in 6 minutes at 130°F vs K-8098 - light and flat; yellow; strong; yellow.

SW-7596 - BaCl₂ added in 2 minutes at 150°F vs K-8098 - light and flat, yellow, s.strong, yellow.

The following are portions of the above batches dried in the P & S Dryer at 140°F 30% Humidity. Gradings are against the portion dried in the Freas Dryer at 140°F.

SW-7582 vs 7581 - s.dark; v.s.bl. & wk., s.wk, s.blue.
SW-7592 vs 7591 - v.s.dark, v.s.blue, w.s. wk., v.s.blue.
SW-7597 vs 7596 - v.s.bright, v.s.blue, v.s.wk, s.blue.

Lot 4458 - YL-8099-D Mix of above (113#) plus Y-299-D (14#) and
blanc fixe (32#) -
vs K-8099 - v.s. dark & dirty; yellow; v.s. weak; s yellow
vs C-38038 - v.s. dark & dirty; yellow; s. str.; yellow, s. clean

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PIGMENT COLOR RESEARCH REPORT

PEACOCK BLUE LAKE BL-164-D

SUBMITTED BY: *E. J. Hess*
APPROVED BY: *V. Chalupski*

FILE: MISCELLANEOUS LAKES
DATE: JANUARY 1936

INTRODUCTION

Production on this high finish Peacock Blue Lake has been continued at intervals over the past two months in both plant and Semi-Works. High finish material was needed to shade existing low finish sub-standard stock.

SUMMARY AND CONCLUSIONS

Two check batches of BL-164-D were made in the Semi-Works using the following variations from standard K-7986: the soda ash in the base was increased 11% to secure improved finish and the dye was decreased 7% in order to secure a product a trifle closer to standard in strength than has been secured in recent production. The reason for this increased strength may be the use of a new lot of Lithosol Brilliant Blue E (lot 31 instead of lot 28) or the difficulties involved in controlling drying during the winter months. The use of controlled humidity drying should reduce this last possible variation to a minimum, however. Both batches were dried in the same manner at 140°F - 40% Humidity and the resultant color in each case was very close to standard in finish but was strong and s red and dirty in tint.

An attempt was made to make a satisfactory mix with this material together with some high and low finish material from sub-standard stock but with no success. The material available is, on the average, too high in strength and too low in finish to blend satisfactory.

A plant batch was then made to check the standard K-7986 formulation except increasing the soda ash in the base 20% in order to secure improved finish. The product showed the desired increased finish but was strong and s red and dirty in tint versus BL-164-D standard. Because of the high strength this material was also unsatisfactory for shading present sub-standard stock.

A second plant batch is in process checking the Semi-Works batches reported this month except that the dye was cut 39% below the standard in order to make a weak, high finish batch for shading material. Results on this batch will be reported next month.

Semi-Works production is tabulated in Table I and Plant production in Table II.

EXPERIMENTAL DETAILS

2.

N.B. 430, p. 167

TABLE I

BL-164-D SEMI WORKS PRODUCTION

Batch	Formula	N-311 Dye		Base				Results vs BL-164-D Std.				Yield	
		Lot	Amt.	N-15	N-3	N-6	pH	Mass.	Draw.	Strength	Tint	Finish	Est. Lmp
SW 7523	Check K-7986 except in-crease N-3 11%, decrease N-311 7%	31	13.7#	199.8# 100%	75# 100%	61.5# 100%	7.1	-	str	s str	s red s dty	vvs low	90 87
SW 7536	Check SW-7523	31	13.7#	199.8# 100%	75# 100%	61.5# 100%	7.1	*	str	str	s red s dty	vvs high	90 76

TABLE II

BL-164-D PLANT PRODUCTION

Batch	Formula	N-311 Dye Lot Amt.	Base			pH	Results vs BL-164-D Std.				Yield	
			N-15	N-3	N-6		Mass.	Draw.	Str.	Tint	Finish	Est. Lump
46982	Check K-7986 except increase N-3 20%	31 49#	666# 100%	270# 100%	205# 100%	6.9	dk	str & red	str	red, vs dty	high	280# 262# 140° F - 30%

N.B. 457, p. 51

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PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: *Al Pearson*

APPROVED BY: *V. Chalupski*

FILE: RAW MATERIALS

DATE: JANUARY 1936.

The two items outstanding from the November report remains to be tested.

This month 92 shipments of raw materials were received and have the following status: 28 tested satisfactorily in the laboratory; 4 used without testing; 6 satisfactory when previously tested; 5 remaining to be tested; 15 awaiting completion of analysis; one shipment returned, wrong material; and 32 reported without test.

A shipment of M.N.P.T. from the duPont Dye works produced a flat masstone when tested on the RT-386-D formula. This material was included in the investigation mentioned in the December report.

Raw Material Samples

Thirteen samples were received this month.

- RMS-1484 P.C.O.N.A. from Verona Chem.Co.
Satisfactory.
- RMS-1485 - Sod. Molybdate, du Pont.
- RMS-1486 - M.N.P.T.) Calco.
- RMS-1487 - ") Unsatisfactory.
- RMS-1488 - M.N.P.T. from Sherwin-Williams.
Satisfactory;
- RMS-1489 - Bleached Barytes from
F.E. Shundler. Unsatisfactory.
- RMS-1490 - Naphthol AS-BS from General Dyestuffs Corp. Unsatisfactory.
- RMS-1491 - P.C.O.N.A. - Dry - from du Pont.
This material is the dried and ground P.C.O.N.A. Crude Press
cake, RMS-1472. To be tried on production scale.
- RMS-1492 - Tri-sulphoil Soap from Scholler Bros.
Satisfactory/
- RMS-1493 - Lithosol Scarlet 3BI from du Pont.
Satisfactory/
- RMS-1494 - M.N.P.T. A) from Calco
- RMS-1495 - " B) Unsatisfactory
- RMS-1496 - Naphthol AS-BS from General Dyestuffs Corp.
Unsatisfactory.

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PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: *Harry Arnold*
APPROVED BY: *V. Chakrapani*

FILE: CR-200
DATE: JANUARY 1936

INTRODUCTION

86 batches of 13 different colors, and 13 lead nitrate purification were made during January; these included 11 batches of RT-354-D, 5 batches of RT-8108-D, 13 batches of Y-359-D, 17 batches of YO-8074-D, 8 batches of YP-324-D and 6 batches of GL-407-D.

SUMMARY

RT-354-D, Maroon Toner - Eleven batches were made in a process study on this color with the production of bright material for shading as the principal object. Effect of varying lots of BON was also tested in this series.

RT-8108-D, Calcium Lithol Toner - Five batches were made in a process study of the new calcium Lithol Toner to replace RT-379-D.

RT-70-D, Para Toner, Dark - Two batches were made in a process study of a new formula for ashless Para Red Dark.

RT-280-D, Para Toner, Light - Six batches were made in an effort to make bright material to fill an order.

RT-8109-D, Calcium Lithol Lake - Two batches were made in a process study of a new calcium Lithol lake formula to replace RL-408-D.

Y-359-D, Medium Yellow - Thirteen batches were made in connection with the study of lead nitrate purification by the YPS method.

Y-8021-D, Excelsior Yellow - Three batches were made in a process study of this recently standardized color.

Y-8124-D, Primrose Yellow - Two batches were made in a yield study.

YO-8074-D, Molybdate Chrome Orange - Seventeen batches were made. Thirteen of them in a process study of the color, and the remainder in a development study of a stronger type.

YL-8098-D, Yellow Lake - Six batches were made to use as shading material in manufacture of YL-8099-D.

YP-324-D, Hansa Yellow - Eight batches were made for production of stock.

GL-407-D, Treated Pigment Green - 6 batches were made at the dye-works in order to try drum drying.

BE-114-D, Extended PTA Toner - One batch was made for stock.

Lead Nitrate Purification - Thirteen purifications were carried out in a study of the YPS method for the removal of copper by precipitation.

One grind of dry color, 14 grinds of pulp color and 13 mixes were made in the Research Semi-Works during January 1936.

<u>Dry Grinds</u>	<u>Charge</u>	<u>Batches</u>	<u>Lbs.</u>
C.P. Yellows	739-S	1	100

<u>Pulp Grinds</u>	<u>Charge</u>	<u>Batches</u>	<u>Bbls.</u>	<u>Lbs.</u>	
Red Toners	737-S	4	14	1044	100%
Green "	"	5	15	1320	"
C.P. Yellows	"	6	24	7024	"
Total		15	53	9388	"

<u>Mixes</u>	<u>Charge</u>	<u>Batches</u>	<u>Lbs.</u>
C.P. Greens	755-S	2	859
C.P. Yellows	753-S	1	703
C.P. Blues	756-S	2	382
C.P. Oranges	754-S	1	965
Ext. Yellow	752-S	1	132
Blue PTA Toner	751-S	3	423
Green " "	"	1	306
Red " "	"	1	339
Red Toner	"	1	255
Total		13	4364

#10 Bldg. Production Report for January 1936

Batch	Code	Lbs. Struck	Lbs. Pack	% Yield	Batch	Code	Lbs. Struck	Lbs. Pack	% Yield	Batch	Code	Lbs. Struck	Lbs. Pack	% Yield
7576	YO-8074-D	30	31	103.3	7611	RT-354-D	55	58	105.3	7646	YP-324-D	120	113	94.2
77	RT-354-D	55	58	105.4	12	RT-8108-D	83	82	98.8	47	Lead Nitrate Purification			-
78	Lead Nitrate Purification			-	13	YO-8074-D	30	31	103.3	48	BE-114-D	592	584	98.7
79	Y-359-D	31	35	112.8	14	RT-8108-D	83	83	100.0	49	Lead Nitrate Purification			-
80	YO-8074-D	30	31	103.3	15	RT-354-D	55	59	107.3	50	YP-324-D	120	111	92.5
7581	YL-8098-D	20	18	90.0	7616	Y-8124-D	155	152	98.1	7651	RT-280-D	23	23	100.0
82	"	20	21	105.0	17	YO-8074-D	30	31	103.3	52	YO-8074-D	30	31	103.3
83	RT-354-D	55	54	98.2	18	"	30	31	103.3	53	Lead Nitrate Purification			-
84	Y-359-D	31	31	100.0	19	RT-354-D	109	119	109.1	54	Y-359-D	31	31	100.0
85	YO-8074-D	30	31	103.3	20	Lead Nitrate Purification			-	55	"	31	31	100.0
7586	Lead Nitrate Purification			-	7621	"			-	7656	YP-324-D	120	113	94.2
87	RT-354-D	55	61	110.9	22	RT-354-D	109	114	104.6	57	RT-280-D	24	23	95.9
88	Y-359-D	31	30	96.8	23	RT-8108-D	83	84	101.2	58	"	24	24	100.0
89	Y-8021-D	150	150	100.0	24	Lead Nitrate Purification			-	59	Y-359-D	32	32	100.0
90	RT-8108-D	83	81	97.6	25	Y-359-D	32	32	100.0	60	Lead Nitrate Purification			-
7591	YL-8098-D	20	21	105.0	7626	GL-407-D	160	160	100.0	7661	YP-324-D	120	116	96.7
92	"	20	19	95.0	27	"	8	8	100.0	62	"	120	116	96.7
93	RT-354-D	55	57	103.6	28	"	184	184	100.0	63	Y-359-D	32	31	96.8
94	RL-8109-D	115	111	96.5	29	"	"	"	-	64	Lead Nitrate Purification			-
95	YO-8074-D	30	30	100.0	30	"	164	164	100.0	65	RT-280-D	24	23	104.3
7596	YL-8098-D	20	20	100.0	7631	"	8	8	100.0	7666	YO-8074-D	30	31	103.3
97	"	20	20	100.0	32	"	-	-	-	67	Lead Nitrate Purification			-
98	RT-8108-D	83	81	97.6	33	"	18	18	100.0	68	Y-359-D	31	30	96.8
99	Y-8021-D	150	155	103.3	34	YO-8074-D	30	32	106.7	69	YP-324-D	120	116	96.7
7600	YO-8074-D	30	30	100.0	35	"	30	31	103.3	70	RT-280-D	23	23	100.0
01	RL-8109-D	115	115	100.0	7636	YP-324-D	120	113	94.2	7671	YO-8074-D	30	32	106.6
02	RT-354-D	55	56	101.8	37	Lead Nitrate Purification			-	72	"	30	30	100.0
03	Y-8021-D	150	163	108.6	38	Y-359-D	32	32	100.0	73	RT-280-D	39	42	107.7
04	RT-70-D	72	69	95.9	39	"	32	32	100.0	74	Lead Nitrate Purification			-
05	RT-354-D	55	59	107.3	40	Lead Nitrate Purification			-	Totals		5553	5551	101.0%
7606	"	55	60	109.1	7641	YP-324-D	120	113	94.2					
07	RT-70-D	72	72	100.0	42	YO-8074-D	30	30	100.0					
08	YO-8074-D	30	31	103.3	43	Y-359-D	32	31	96.8					
09	"	-	-	-	44	"	32	31	96.8					
10	Y-8124-D	155	150	96.8	45	YO-8074-D	30	30	100.0					

SUMMARY OF #10 BLDG & PRODUCTION REPORT FOR JANUARY 1936

Code	Lbs. Struck	Lbs. Packed	% Yield
RT-354-D	713	755	105.8
RT-8108-D	415	411	99.1
RT-70-D	144	141	97.8
RT-280-D	157	159	101.3
RL-8109-D	230	226	97.0
Y-359-D	410	409	99.7
Y-8021-D	450	468	104.0
Y-8124-D	310	302	97.4
YP-324-D	960	911	94.9
YO-8074-D	510	524	102.7
YL-8098-D	120	119	99.2
GL-407-D	542	542	100.0
BE-114-D	<u>592</u>	<u>584</u>	<u>98.7</u>
	5553	5551	99.8%

MATERIAL TRANSFERRED DURING JANUARY 1936 FROM RESEARCH SEMI-WORKS

<u>Lot</u>	<u>CR-200</u>	<u>Color Code</u>	<u>lbs.</u>	<u>Group</u>	<u>Cost Unit</u>	<u>Total Cost</u>
4441	1	RT-354-D	58	2	110.07	
42	1	"	62	2		
43	1	"	60	2		
44	1	"	58	2		
49	1	"	63	2		
50	1	"	58	2		
59	1	"	57	2		
60	1	"	61	2		
61	1	"	61	2		
62	1	"	59	2		
63	1	"	59	2		
64	1	"	119	2		
65	1	"	115	2		
4467	1	RT-70-D	140	2	45.40	
4445	3	Y-359-D	210	2	8.80	
46	3	Y-8021-D	158	2	8.80	
47	3	"	156	2		
48	3	Y-180-D	368	2	8.11	
68	3	Y-8024-D	150	2	8.70	
69	3	"	153	2		
70	3	Y-8021-D	149	2	8.80	
71	3	"	156	2		
72	3	"	164	2		
4413	8	YO-34-D	45	1	7.29	
4466	8	"	106	2		
4440	8	YO-38-D	305	1	7.34	
4457	8	"	288	1	7.34	
4434	6	BE-149-D	269	2	14.54	
4411	11	BL-72-D	29	1	52.82	
4451	11	BL-164-D	96	2	44.22	
4458	11	YL-8099-D	157	1	20.72	
4410	25	BT-122-D	29	1	150.75	
4412	25	RT-338-D	31	1	299.41	
4435	25	GT-337-D	35	2	176.94	
36	25	"	37	2		
37	25	"	35	2		
38	25	"	37	2		
39	25	"	41	2		
4452	115	RT-7853-D	40	2	133.56	
4453	125	RL-408-D	48	1	49.25	
4455	151	YO-8074-D		2	13.98	
56	151	"		2		

RECAPITULATION

Colors transferred from Semi-Works to Plant during
January 1936.

Toners	1070#
Yellows	1664#
"RE" Lakes	157
Oranges	1503
"RL" Lakes	173
PTA Toners	<u>244</u>
Total	4811#

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