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KW 46-22

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

MEDIUM CHROME YELLOW Y-547-D: DEVELOPMENT OF CONTINUOUS MANUFACTURING PROCESS - #6 BLDG. PILOT UNIT.

Period Covered: October 5, 1945 - June 17, 1946.

FILE:

DATE: 7/5/46

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

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Period Covered: October 5, 1945 - June 17, 1946.

SUBMITTED BY: E. F. Klemke, Jr. DATE SUBMITTED: 6/18/46.

APPROVED BY: J. O. Morrison DATE ISSUED: 7/5/46

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INDEX

INTRODUCTION

The development work involved, in making a product equal to the Semi-Works improved medium yellow (K-2217, lot 8314) in the 6 Bldg. continuous manufacture pilot unit, is reported below. The final accepted process was coded K-2352; this formula, together with detailed operating instructions, is also included.

SUMMARY AND CONCLUSIONS

During this period the K-2352 formula was developed for making material, substantially equal tinctorially to the Semi-Works K-2217 (lot 8314), in the 6 Bldg. continuous manufacture pilot unit. K-2217, the standard set up from Semi-Works continuously manufactured improved Medium Yellow, is, in turn, only slightly inferior to material prepared by the laboratory batchwise K-1958 formula.

Since the proposed 12 Bldg. continuous manufacture unit is to be geometrically the same as and only twice the size of the 6 Bldg. unit, it is believed that the development information accumulated in the pilot unit will be directly applicable in the full scale plant.

Development work in the 6 Bldg. pilot unit was based on the Semi-Works formula for K-2217 (see KM-46-8). By applying variables evaluated during the Semi-Works development work, and some developed during the plant study, a formula was arrived at which gave satisfactory material; this is coded Y-547-D (K-2352).

Rub out evaluations of the previously mentioned standards, in H-108 and alkyd vehicles, gave the cross comparisons tabulated below:

RUB OUT EVALUATIONS

			H-108			
			Masstone	Strength	Tint	Alkyd Masstone
K-2352 (lot 33601)	vs	K-1958 (lot 7697)	vvs-vs red, vvs dk	OK	OK	vvs red
K-2352	vs	K-2217 (lot 8314)	s lt, s grn & clean	str	vs grn	vs-s lt, vs green
K-2352	vs	Y-547-D (lot 8340)	vs-s red, vs dk	vvs wk	OK	vs red, vs dk.
K-2217 (lot 8314)	vs	K-1958 (lot 7697)	s dk, s red	wk	s red	s red, s dk
K-2217	vs	Y-547-D (lot 8340)	vs red	wk	vs red	s red, vs dk
Y-547-D (lot 8340)	vs	K-1958 (lot 7697)	vs dk, vs dull	vvs str	OK	vvs-vs dk

Preliminary printing ink evaluations of K-2352 indicate a tendency toward reactivity; further study of this property is desirable in connection with plant manufacture of Y-547-D. Compared with a tentative plant standard of similar material (lot 8342), approved by Sales for shipment to Areo, K-2352 was vs 1%, and vs clean in masstone, OK in strength and tint. (by H-108 rub out)

The plant produced material was considered to be a close enough tinctorial check to K-2217 to warrant discontinuing development work. However, in view of the existing demand for an improved medium yellow of the Y-547-D (lot 8340) type, to meet customer commitments, the pilot unit K-2352 final formula is being turned over to the Plant for production use.

Additional information concerning continuously manufactured Medium Yellow can be found in the following reports: (see page 28)

DISCUSSIONS

The successive basic steps involved in making improved medium Chrome Yellow, continuously, may be very briefly summarized as follows:-

1. Introduction of Lead Nitrate and Sodium Chromate solutions into a precipitation zone with intense agitation to precipitate Lead Chromate (slight chrome excess present).
2. Two stage heat development of the Lead Chromate crystals at a selected pH.
3. Digestion, or ageing, of the crystals at the final development temperature.
4. Pipe line introduction of alum ($Al_2(SO_4)_3.18H_2O$) and Titanium Oxide to coat the crystals with hydrous oxides and prevent further crystal growth and development.
5. Pipe line introduction of Lead Nitrate to precipitate Lead Sulfate and Lead Chromate.
6. Addition of Soda Ash for final pH control.

Although the 6 Bldg. pilot unit and the Semi-Works unit are similar in principle, they differ geometrically and, in some cases, physically. Since this difference proved to be important, a diagram of each unit is included (diagrams I and II). Also included is a copy of the operating sheet used for operating the 6 Bldg. Pilot Unit, together with an explanation of the operation.

Because of its location and its integration with the operation of the Plant Yellow Units, the pilot unit was initially operated and supervised by Plant personnel. The Research department supplied the formulas, followed each run, collected data and samples, and initiated the necessary changes.

For simplicity of operation it was decided to make the first few runs using pre-diluted Lead and Chrome solutions instead of continuously diluting them in the lines, therefore, the formula was adjusted to suit the rotameter limits. To do this some of the vat retention times had to be altered, therefore the first Plant Unit formula used did not exactly duplicate all the conditions of the Semi Works Unit.

Table I tabulates the unit conditions for each subsequent change in formula involving a change in rate, and table II contains the changes involved in the development of the final formula.

Briefly summarizing these tabulations the major formula and processing changes made were:-

1. Increased concentration of Lead and Chrome solution (±53% total increase), producing lighter masstone.

2. Increased heat development temperatures, giving redder masstone.

3. Immediate de watering of slurry from final treatment vat (by pumping directly to the centrifuge) to give lighter, cleaner masstone.

4. Continuous Petronate treatment, to increase centrifuge rate.

5. Increase development time (±10%) to give closer masstone match.

TABLE I

	VAT RETENTION TIMES - MINUTES					
RATE	STRIKE	1st HEAT DEV.	2nd HEAT DEV.	AGEING	FINAL TREATMENT	LOT NOS.
630 #/hr.	0.90	0.95	11.7	11.7	6.8	26035-26157
800#/hr.*	0.96	1.1	13.6	13.6	7.8	27354-27420
1100 #/hr.	0.70	0.80	10.8	10.8	5.6	27557 -

*Lead and chrome solution concentration increased.

TABLE II

Lot No.	Variables	FMC Results (Vs K-2217) Masstone	Strength	Tint
26035	First plant batch; 630 #/hr. rate	dk, dull	OK	OK
26157	Increased Lead and Chrome sol'n. conc. (±33%) to lighter MT	vvs lt.	OK	OK
27354	Check 26157, but increase rate to 800 #/hr.	dk, dull	str	OK
27420	Check 27354, but increase conc. of Lead and Chrome ±15%	s lt, grn	vvs str	OK
27557	Check 27420 but use concentrated lead and chrome sol'n. and dilute them in line to strike unit.	dk, dull	str	s grn.
27592	Check conditions of 27557 but increase rate to 1100 #/hr.	vvs grn.	vs str.	OK
27992	Check 27592 but increase development temps. to give redder MT.	vs lt.	vvs str.	OK
28289	Check 27992 but dilute 2nd lead in line	s dk, grn	stre.	OK
29840	Check 28289 but continuously Petronate treat	dk, grn	str	OK
30823	Check 28289 but centrifuge slurry as rapidly as it is made	s dk, grn	str	OK
31451	Check 20823 but continuously Petronate treat	vvs dk	vs-s str	OK

An important step in the program of this development work was the transfer of the operation and supervision responsibilities, of the plant pilot unit and processing equipment to the Research department. This change was made in recognition of the fact that very close control of all factors involved in making and processing the continuous unit products was absolutely essential to the success of the development work. The results justified the change; thirteen out of twenty subsequent batches being satisfactory checks. It was interesting to note that of the seven unsatisfactory batches, five were made during the period when Zinc Yellows were being centrifuged immediately before each batch of experimental medium yellow.

Semi-Works experience with K-2217 indicated that, with Lead and Chrome solutions of accurately pre-adjusted pH, the pH after strike could be used to indicate the Lead to Chrome flow rate ratio more correctly and more accurately than could be done with rotameters alone. Therefore, once the desired strike ration of Lead to Chrome was determined, it merely became necessary to measure the strike pH and use it as a control point for subsequent check runs, provided sufficient care was exercised in preparing the component solutions.

During the final stage of the Plant development work the Lead and Chrome solutions were analysed and the flow rates were adjusted solely on the basis of analysis. Subsequently a frequent strike pH variance (5.5 to 6.5) was noted from batch to batch, and each time this difference occurred, the quality of the resulting pigment was inferior. The method of control was changed, therefore, and the chrome solution rate of flow was adjusted each time the strike pH was beyond allowable limits. Using this technique (and continuing to analyse the solutions for checking purposes) a series of successful check batches was run. Analysing this information and the attending results, it was decided that the solutions (Lead and Chrome) could not be sampled and/or analysed accurately enough to warrant operating by calculated flows only. It is therefore established that, for K-2352, solution analyses should be used to check the accuracy of solution make-up and provide a calculated flow rate for the Lead solution. The chrome solution flow rate, however, should be regulated by the resulting strike pH. With good sampling and accurate analyses the rotameter adjustment should not normally exceed ±0.5%.

In the early plant development work, the color would frequently thicken up in the first and second heat development vats, so badly at times that it caused the vats to overflow. The cause of this was found to be a lead excess after striking, instead of a very slight chrome excess. This was attended in every case by a low strike pH. It is therfore recommended that the heat not be introduced to the development vats until the strike pH has been checked and stabilized. The normal chrome excess after strike should be ±0.5 #/min. (calculated for K-2352)

As indicated in the summary of major formula and processing changes, the final processing conditions had an appreciable affect on the quality of the pigment produced. Closely allied with the indicated need for immediate de watering of the slurry, was the need for increasing the solids content of the finished pulp from ±40% to ±45% (wet basis), to meet the specifications for the continuous dryer guarantee. To accomplish this, a centrifugation study was made, coincidental with the main study; this is reported in KN-46-27. This study indicated that the second-pass centrifugation rate for K-2352 is increased when the quantity of re pulp water (equipalent to wash water) is reduced from 1.5 gallons to approximately 1 gallon per pound (dry color). This causes an increase in calculated water soluble salts in the finished pigment. Due to the increased centrifugation rate and the reduced quantity of re pulp water, the temperature of the re pulped material entering the centrifuge for the second pass is higher - resulting in the desired higher solids content pulp for drier pan loading. There are no data at the present time to indicate the effect of this treatment, if any, on the texture

of the finished pigment. One run was made using a ±1.5 gallon per pound (dry basis) re pulp rate, and adjusting the temperature of the entire quantity of re pulped material to 90° before the second centrifugation; preliminary evaluations indicated that the tinctorial properties of the pigment were not effected. Since the solids content of the final pulp was ±47% (wet basis) using this procedure, confirmation runs are planned.

In conclusion, there are some necessary precautions to be taken regarding equipment checking and maintenance, as noted during the experimental operation of the 6 Bldg. pilot unit. Table III contains a list of the items requiring attention. The most important factor involved, in successfully bringing the unit to equilibrium, is making certain the operator keeps all the rotameters accurately adjusted at all times. There is a tendency for new operators to believe a rotameter can be set once and need no more attention - a very incorrect assumption.

YIELD CALCULATIONS

The yield for continuously manufactured products can be calculated most easily by using a one minute time basis and the respective flow rates of the ingredients. The yield for K-2352 is calculated in this manner, as follows:

Using the following flow rates (see formula)

$Pb(NO_3)_2$	17.000 lbs./min.
Na_2CrO_4 (as $Na_2Cr_2O_7 \cdot 2H_2O$)	8.220 "
$Al_2(SO_4)_3 \cdot 18H_2O$	1.623 "
"L" Liquor (TiO_2 equiv.)	0.351 "
2nd Lead - $Pb(NO_3)_2$	1.785
Petronate (as is)	0.187

1. $PbCrO_4$ precipitated at strike - $(17.00)\left(\frac{323.22}{331.23}\right) = 16.589$

   Chrome excess will be - $(8.22) - \left[\left(\frac{149.0}{334.23}\right)(17.04)\right] = 0.587$

2. Hydrate - from Alum treatment - $(1/3)\ (1.623) = 0.541$

3. Hydrate - "L" Liquor " - $0.351 = 0.351$

4. $PbCrO_4$ precipitated by 2nd lead - $\frac{(0.587)(323.22)}{(149.0)} = 1.274$

5. $PbSO_4$ precipitated by 2nd Lead - $\left[1.785 - (0.587)\left(\frac{331.23}{149.0}\right)\right]\left(\frac{303.27}{331.22}\right) = 0.440$

6. Petronate (as is) - $0.187 = 0.187$

Calculated Yield per minute → 19.382 #/min.

Using the figures calculated for production per minute, the % composition can be calculated as follows:

$$PbCrO_4 = \left(\frac{16.589 + 1.274}{19.382}\right)(100) = 92.16\%$$

$$PbSO_4 = \left(\frac{0.449}{19.382}\right)(100) = 2.27\%$$

$$\text{Hydrates} = \left(\frac{0.541 + 0.351}{19.382}\right)(100) = 4.60\%$$

$$\text{Organic Agent} = \left(\frac{0.187}{19.382}\right)(100) = 0.96\%$$

$$\text{TOTAL} = 99.99\%$$

The yields for continuously manufactured K-2352 are calculated as follows:

Total Yield

Divide the total quantity of 100% Lead Nitrate made up, by the combined quantities of First and Second Lead additions (per minute); this will determine the total "run time."

Multiply the total "run time", by the calculated yield per minute (see "Calculations") to determine the total yield for the run.

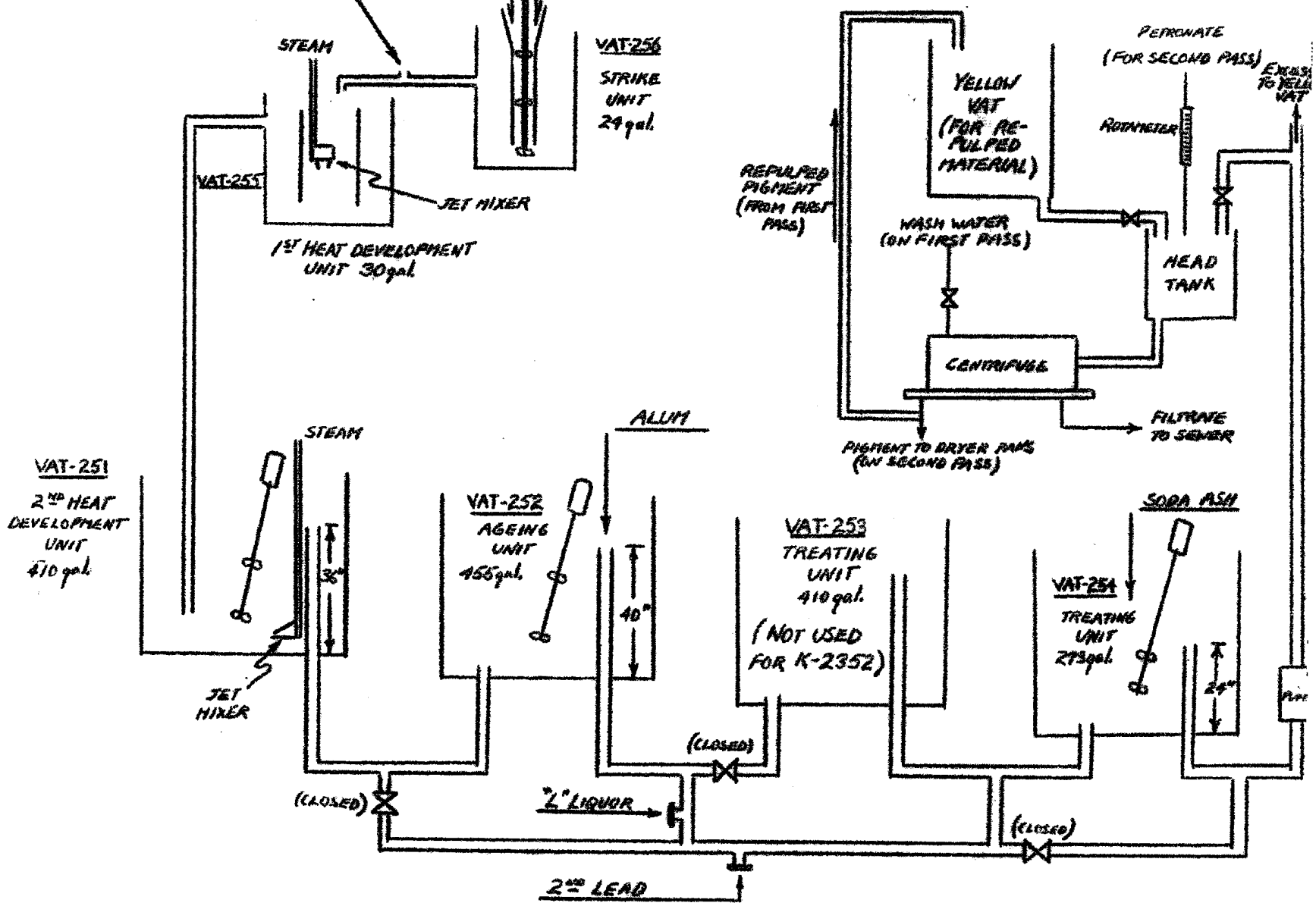
Yield of Centrifuged Material

Assume equilibrium material is being discharged from the final treating unit at the end of 40 minutes. Subtract the 40 minutes from the total run time (see above) to determine the centrifugation time. Multiply the predetermined average centrifugation rate (see EM-46-27) of 10.5 lbs/minute for the first pass, by the centrifugation time to determine the estimated yield for centrifuged material.

From these calculations it can be seen that, if the unit is not brought to equilibrium quickly and maintained there, the yield for centrifuged material will be low.

DIAGRAM II

6 BLOC PILOT UNIT



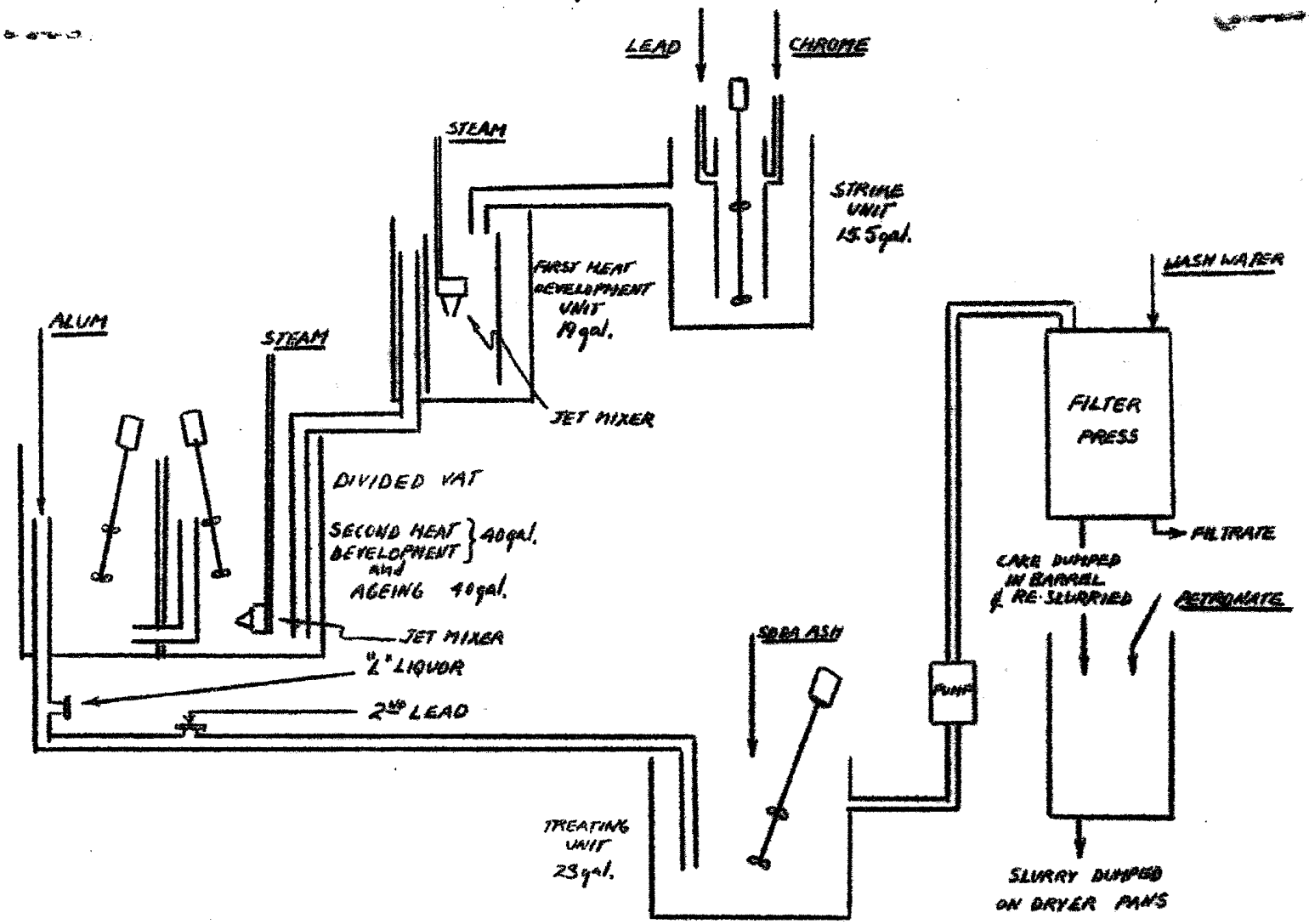


DIAGRAM I
SEMI-WORKS UNIT

K-13398

FORMULA SHEET

DUPONT

COLOR CODE K-547-D DATE 6/17/46 NAME Chrome Yellow 2352 (Pit)

OBJECT Same as K-2340 but increase batch size.

BASED ON Plant Production, batch 33601 etc.

BATCH SIZE-MOLS 28.25 BATCH No. OF "K" SAMPLE

LBS. OR GMS.	MATERIAL	100% WT.	GAL.	C. C.	MANIPULATION	N/
					Dissolve	
441	Alum ($Al_2(SO_4)_3.18H_2O$)	441	685		in (15 1/2" Vat 240) at 90°-100°F. Pump to Vat 344 - Do Not Wash In.	15
					Wash out Vat 240, then dissolve in it	
826	Soda Ash (Na_2CO_3)	826	752		in (17" Vat 240) at 90°-100°F. Pump ±1/2 to Vat 238 - Do Not Wash In	3
					Weigh into Vat 240	
6040	Lead Nitrate $Pb(NO_3)_2$	6040	3930		Adjust volume to (79" Vat 241) at 79°-81°F. Adjust pH to 3.15-3.25 with	41
±2.0	Nitric Acid (±60% HNO_3 sol'n.)	±2.0			Record pH. Have sol'n. analysed for #/gal. $Pb(NO_3)_2$ Pump sol'n. to Vat 372 (see notes)	13
					Weigh into Vat 356	
2700	Sodium Chromate (±31% sol'n., Calc. as $Na_2Cr_2O_7.2H_2O$)	2700	1720		and adjust volume to (61" Vat 356) at 79°-81°F. Adjust pH to 7.35-7.45 with	10
±50	Nitric Acid (±60% HNO_3 sol'n.)	±50			Record pH. Have sol'n. analysed for #/gal.-expressed as $Na_2Cr_2O_7.2H_2O$	13
					Weigh into Vat 352	
99	"L" Liquor (±7% sol'n.)	99 "as is"	241		Dilute with water to (14" Vat 352) - stir well.	330
					Weigh into Petronate Sol'n. vat	
30	Petronate (as is)	30	70		Dissolve in water at 140°-160°F. and make to Stir well.	40c

FORMULA SHEET

DUPONT

COLOR CODE DATE NAME K-

OBJECT

BASED ON

BATCH SIZE-MOLS. BATCH No. OF "K" SAMPLE

LBS. OR GMS.	MATERIAL	~~XXXXX~~ Lbs/Min	~~XX~~ Lbs/Gal	~~XXX~~ Gal/Min	MANIPULATION	N#
					Equipment and lines must be properly connected and checked, as directed in KN-46-22. Then proceed as follows:-	
					Start agitation in Strike Vat 256.	
					Adjust Lead sol'n. diluting water rotameter flow to	
	Water	-	-	10.45		
					Adjust Chrome sol'n. diluting water rotameter flow to	
	Water	-	-	8.1		
					Adjust Chrome sol'n. rotameter flow to	
	$Na_2Cr_2O_7.2H_2O$	8.22	±1.57	±5.2	(actual gal/min. calculated from analysis- see notes).	
					Simultaneously adjust Lead Nitrate sol'n. rotameter flow to	
	$Pb(NO_3)_2$	17.00	±1.537	±11.0	(actual gal/min. calculated from analysis see notes).	
					Adjust pH after strike to 5.9-6.0 by adjusting Chrome sol'n. rate of flow.	
					After strike pH has been adjusted, turn on steam in "First Heat Development" Vat 255 and set temperature controller at 165°F.	
					Turn on agitator and steam in "Second Heat Development" Vat 251, and set temperature controller at 205°F.	
					Start agitator in "AGeing Vat 252 when slurry reaches sufficient depth.	
					When slurry begins to flow from Vat 252, adjust alum soln. rotameter flow to	
	$Al_2(SO_4)_3.18H_2O$	1.62	0.692	2.35		
					Adjust "L" Liquor sol'n. rotameter flow to	
	"L" Liquor	0.351	0.41	0.86		
					Adjust 2nd Lead sol'n. rotameter flow to	
	$Pb(NO_3)_2$	1.785	±1.537	±1.16	(actual gal/min calculated from analyses - see notes)	
					Adjust 2nd Lead sol'n. diluting water rotameter flow to	
	Water	-	-	1.1		

FORMULA SHEET

DUPONT

COLOR CODE | DATE | NAME | K-

OBJECT

BASED ON

BATCH SIZE-MOLS. | BATCH No. OF "K" SAMPLE

LBS. OR GMS.	MATERIAL	100% WT. XXXXX	GAL. XXX	C.C. XXX	MANIPULATION	NI
			Lbs/Min	Lbs/Gal	Gal/Min	
	Na_2CO_3	2.84	1.21	2.35	Start agitator in "Final Treating" Vat 254 and adjust Soda Ash sol'n. rotameter flow to Adjust pH in Vat 254 to 6.0 - 6.5 by regulating Soda Ash sol'n. flow. When slurry in Vat 254 reaches the level of the stand pipe - start pumping it to the centrifuge, keeping a ±4 inch slurry depth in the centrifuge head tank. Re-pulp the centrifuged material (by adding #/gal. water per lb. color - dry basis) using 1 1/2 opening turns of the re-pulp water valve. Pump re-pulped material into a yellow vat and (after it is all collected) re centrifuge, adding continuously to the centrifuge head tank
	Petronate	0.187	0.40	0.47	through the rotameter. (see notes) Centrifuge the final material onto dryer pans.

CALCULATED COMPOSITION

$PbCrO_4$	=	92.16%
$PbSO_4$	=	2.27%
Hydrates	=	4.60%
Organic Agent	=	0.96%
		99.99%

ANALYSED COMPOSITION

$PbCrO_4$	=
$PbSO_4$	=

PACKAGE | LBS. PER | COST GROUP | SIGNATURE E. F. KLENKE, JR.

GOAL YIELD 6240# as is, 6140# anhydrous, 1.5% moisture (see KN-46-22 for calc.)

K-13399

COLOR CODE [illegible] K [illegible] NAME Green Yellow DATE 6/28/46 GOAL YLD. 6140

MAKING

VAT USE	PREFERRED VAT NO.	PREFERRED GAL./IN.	ALTERNATE VATS VAT NO.	ALTERNATE VATS GAL./IN.	Required Mixing Vat Volumes
Lead Nitrate	242	49.8	Strike	256	1.59	256 - 24 gal.
Sod. Chromate	356	20.2	1st Heat Envelop.	255	1.83	255 - 30 gal.
Alum	240	44.2	2nd " "	251	11.4	252 - 410 gal.
" (storage)	244	43.4	Ageing	252	11.4	252 - 455 gal.
Soda Ash	352	17.16	Treating	254	11.4	254 - 273 gal.
Soda Ash	240	44.2	Re pulp	243 - 247	89.8	243-247

PRESSING

	PREFERRED
PRESS NUMBER	
TYPE PUMP	
CUBIC FEET PER STANDARD BATCH	
FRAMES PER STANDARD BATCH	Centrifuge
TYPE CLOTH	
NUMBER OF CLOTHS PER PLATE	to be
TIME OF FILLING	
TIME OF WASHING	used
WASH WATER—TEMPERATURE	
WASH WATER—PRESSURE	
END POINT	
DRY CONTENT OF CAKE	44.0% +
PRESS WITH WITHOUT AGITATION	

SPECIAL CONDITIONS

DRYING

	PREFERRED
TYPE DRYER	High velocity P A F
NUMBER OF CARS	3
PANS PER CAR	20
TIME OF DRYING	10 hrs. till dry
TEMPERATURE OF DRYING (DRY BULB)	160° F.
WET BULB TEMPERATURE	"
HUMIDITY OF DRYING	"
TEST FOR DRYNESS	Feel

SPECIAL CONDITIONS

GRINDING

	PREFERRED
NUMBER OF GRINDER	#2 20"
MILL SPEED	1750
HAMMER TYPE	Fairfax
SCREEN	3/8 round perforated
FEED SPEED	"
MINUTES PER BARREL	"
FINENESS SPEC.	"
DRY ICE	"
POUNDS PER PACKAGE	
PACKAGE TYPE	
LINER TYPE	

SPECIAL CONDITIONS

REMARKS:

SIGNATURE E. F. Evans Jr.

DUP050150525

Y-647-D, K-2352 Notes

K-2352 describes the process for making improved medium yellow in the 6 Bldg. continuous manufacture pilot unit; development work was based on the Semi-works K-2217 (lot 8314) formula.

It is imperative, for good quantone quality, that the effluent slurry from 254 Vat be de-watered immediately. Because of the inadequate capacity of the centrifuge, a portion (±8 lbs./ min) of the ±19 lbs./minute production must be diverted to a yellow vat and vat washed, treated with Retronate and pressed. The portion so treated will be of inferior quality compared to the balance of the material, however it is consumeable as, or mixed with, Y-469-D and/or Y-424-D.

The ±11 lbs./ min. rate of centrifugation was based solely on average centrifuge performance and must not be used directly to determine or predict batch yields. Several pilot unit yields for centrifuged material (per 2750 lb. total run yield) averaged ±900 lbs. This low yield was due principally to solution concentrations being lower than required, resulting in the exhaustion of supply of the respective solution in less time than normal. Since the time to reach equilibrium (before starting centrifugations) is fixed, this time reduction affects the total centrifugation time - thereby reducing the yield of centrifuged material. When solution concentration were what they should be, the centrifuged material yield averaged ±1100 lbs. (per 2750 lb. total yield).

The following notes represent pertinent information gathered from Semi-works as well as 6 Bldg. pilot unit experience. The processing notes pertain only to the centrifuged portion of the batch, except where otherwise specified.

1. It is necessary that the Lead Nitrate solution and the Sodium Chromate solution be analysed for #/gal. $Pb(NO_3)_2$ and $Na_2Cr_2O_7.2H_2O$ respectively, and the specific gravity of each determined. This information is necessary to enable calculation of the respective rates of flow.

2. After the necessary solutions have been made up, and the Lead Nitrate and Sodium Chromate solution analyses have been received, the Lead and Chrome flow rates are calculated. Since the rotameters were calibrated with solutions of different specific gravity from those to be used, calculations are also necessary to correct for this. A typical set of calculations, necessary to fill in the information needed on the operating sheets (see second page of sample sheet), would be as follows:-

Solution	Rot. Reading	#/gal.	Formulated #/min.	gal/min.	Specific gravity
N-41		1.537*	17.0	11.05	1.162*

*From analyses

Using the desired 17.0 #/min figure, and the #/gal. analysis, the necessary gal/min flow is calculated. Then, using the equation shown*, the specific gravity compensation factor is calculated as follows:

$$\frac{Q_1}{Q_2} = \frac{\left(\frac{Pf - P_2}{P_2}\right) 1/2}{\left(\frac{Pf - P_1}{P_1}\right) 1/2}$$

Q_1 = rate of flow of liquid 1 (used for calibration)
Q_2 = " " " " " 2 (to be used for run)
Pf = density of rotameter float (7.92 for 6 Bldg. rotameters)
P_1 = density of liquid 1 (used for calibration) { = 1.048 rotameter calibration for N-41 / = 1.046 for N-10
P_2 = density of liquid 2 (from anal.)

*"Performance of the Rotameter" by Schoenborn & Colburn
Trans. A I Ch. E. - 35 , 359 (1939)

Using 1.048 for P_1 and 1.162 for P_2, this equation resolves to:-

$$\frac{Q_1}{Q_2} = \frac{\left(\frac{7.920 - 1.162}{1.162}\right) 1/2}{\left(\frac{7.920 - 1.048}{1.048}\right) 1/2} = 0.942$$

The calculated flow (11.05 gal/min) divided by this factor (0.942) will give the corrected flow rate (11.7# not shown in above table). Using this corrected flow rate figure, and the calibration curve for the rotameter involved, the rotameter reading is determined.

3. Do not turn steam on in development units until strike pH has been adjusted. If the strike pH is low (indicating a Lead excess) the slurry will thicken in the development units and the vats will overflow.

4. Provided the strike and final pH have been adjusted, and no rotameter changes have been made in the interval, equilibrium material will begin to be discharged from the unit 20 minutes after the desired development temperatures have been reached and stabilized. The minimum total time required, to begin to discharge equilibrium material from the final treating vat, averaged 40 minutes from the time the chrome and Lead flows were started.

5. An increase in development temperature and/or time will cause a loss in strength and a redder masstone and tint. There is little or no accompanying effect on light stability.

6. An increase in concentration of the Lead and chrome solutions will lighten the masstone and conversely decreased concentration causes a darker masstone.

7. An increased Alum addition increases the tendency for the pigment to liver in printing ink vehicle, however it tends to improve the lightfastness properties.

8. An increased quantity of Petronate would tend to cause a darker, dirtier masstone, and decreased light stability, but decreases the livering tendencies in printing ink vehicles.

9. A high final pH (close to 6.5) causes a decreased centrifugation rate.

10. Immediate de-watering of the slurry, after leaving 254 Vat, is essential to good masstone quality. To facilitate this, the level of slurry in the centrifuge head tank is limited to 4 inches. If the slurry is not de-watered immediately, the masstone will become dark, green and dirty.

11. A repulp rate of 1 gal. of wash water per lb. of pigment (dry basis) will give the best centrifuge rate. See report KN-46-27.

12. The sequence and position of addition of the ingredients is very important and should not be varied in the 6 Bldg. unit, except under Research supervision.

13. Instructions in "Operating Details" and "Inspection and Maintenance of Equipment", given in Research report KN-46-22, must be strictly observed.

14. The proper Petronate solution flow rate is determined by making an original rotameter setting (±40) and adjusting this, during the second centrifugation period, so that the rate of decrease in volume of the Petronate sol'n. is proportional to the decrease in volume of the repulped slurry.

APPROVED
JAC: 6-12-46
JOM: 6-12-46

CHECKED BY
CODE Y-547-D K #
GOAL 6140 SIZE +3000
VATS SST

ACCT. LBS.	N-CODE	USE LBS	INSTRUCTIONS
			MEDIUM YELLOW
6040	41		@ % M- Fe-
			MAKE TO 79" AT 80°F VAT 241
			ADJUST PH TO 3.15 TO 3.25 WITH
	33		RECORD PH ____.
	7		RECORD Be ____. RECORD INCHES ____.
2700	10		@ % M T-
			MAKE TO 61" AT 80°F VAT 356.
			ADJUST PH TO 7.35 TO 7.45 WITH
	33		RECORD PH ____. RECORD INCHES ____.
	7		RECORD Be ____.
			DISSOLVE
441	15	441	IN 15 1/2" AT 90-100°F IN VAT 240.
			PUMP TO VAT 344.
			DO NOT WASH IN.
99	390		@ % M- DILUTE TO 14" VAT 352.
			STIR WELL.
			DISSOLVE
825	3	826	IN 12" AT 90-100°F IN VAT 240.
			PUMP TO VAT 238.
			PUMP FINISHED SLURRY TO CENTRIFUGE.
			EXCESS SLURRY IS PUMPED UNCENTRIFUGED
			TO VAT ____ AND FILED FOR Y-469-0124.
			REPULP (± 1 GAL/LB.) TO VAT ____.
			RE-CENTRIFUGE, AND TREAT CONTINUOUSLY
			IN CENTRIFUGE HEAD TANK, WITH
30	490	30	DISSOLVED IN 45-50 GALLONS AT
			140-160°F IN 55 GALLON DRUMS.
			CENTRIFUGE ONTO PANS, WITH AGITATION
			IN VAT

TIME OF START _____ DATE _____
TIME COMPLETED _____

ROTAMETER	ROT. READING	#/GAL.	#/MIN.	GALLONS PER MIN.	SPECIFIC GRAVITY
#1 N-41		1.537	17.0		
#2 WATER	10.45	X	X	10.45	
#3 N-10		1.57	8.22		
#4 WATER	8.1	X	X	8.1	

ADJUST PH AT STRIKE TO 5.9-6.0 BY N-10 FLOW RATE.
TIME
N-IC
PH

WATER TEMP 80°F, 1ST HEAT DEV 65°F 2ND HEAT DEV 205°F
USE "0" DEPTH IN 252 VAT

#5 N-15	2.32	0.692	1.623	2.35	
#6 N-390	0.84	0.41	0.351	0.856	
#7 N-41		1.537	1.785		
#8 WATER	1.1	X	X	1.094	
#9 N-3	23.0	1.21	2.84	2.35	

ADJUST FINAL PH TO 6.0 6.5 BY N-3 ADDITION
TIME
N-3
PH

TIME STARTED _____ TIME FINISHED _____ OPERATOR _____

" " _____ " " _____ " _____

" " _____ " " _____ " _____

APPROVED HA: 6-13-46
JAC: 6-14-46
EK: 6-14-46

CHECKED BY
CODE Y-469-D424 3140
COOL SIZE
VAT 3

ACCT. LBS.	N-CODE	USE LBS.	INSTRUCTIONS
			MEDIUM YELLOW FROM Y-547-D
			COLLECT EXCESS SLURRY FROM UNCENTRIFUGED Y-547-D IN VAT ____. MEASURE ____" AT END AND FLOOD TO 153".
			DRAW 3 WATERS AT 1 1/2, 2, AND 4 HOUR INTERVALS.
			AFTER DRAWING LAST WATER, TREAT WITH
31	490	31	DISSOLVED IN 50-55 GAL. AT 140-160°F.
			STIR ±10 MINUTES AND PRESS

OPERATING INSTRUCTION

The steps to be followed in starting the unit are listed below in the order of their sequence: (assuming the checks and precautions of Table III have been done)

1. Start agitator in strike unit.
2. Start flow of diluting water for Lead and Chrome, and adjust.
3. Start Lead and Chrome flows, and adjust.
4. Check strike pH, after allowing ±5 minutes for unit to come to equilibrium.
5. Make chrome flow rate adjustment, if necessary.
6. When strike pH is within limits, start steam in first heat development unit.
7. Start agitator in second heat development unit.
8. " steam " " " " "
9. " agitator in ageing unit (when slurry level is high enough).
10. Start alum flow when material in ageing unit begins to enter standpipe.
11. Start "L" liquor flow immediately after the alum.
12. Start the 2nd lead, and its diluting water, flow immediately after the "L" Liquor.
13. Start the soda ash flow immediately after the second Lead.
14. Start the agitator in the final treating unit, when the slurry level is high enough.
15. Start the pump when the material in the final treating unit covers the standpipe.
16. Check the pH in the final treating vat.
17. Make soda ash adjustment, if necessary.
18. If both pH's are within limits; if all the rotameters are at their correct settings; and if the material in the development units has reached the proper temperatures - the unit may be considered to be at equilibrium conditions. Provided no changes of any kind are made or noticed, 20 minutes from this time the unit will be filled with and discharging material representative of the conditions of operating card.

Due to equipment limitation the material from the final treatment vat was disposed of in a complex manner. For clarity the subsequent steps will be preceeded by an explanation.

As indicated in Table II, immediate de-watering of the finished pigment is essential for good quality. To do this, the material from the final treating unit was pumped to a "Bird" continuous centrifuge.

The maximum capacity of the centrifuge (at maximum allowable filtrate bleed) is limited to ±10 lbs. (dry basis) of K-2352 per minute. The rate of output from the unit, however, is ± 20 lbs. (dry basis) of K-2352 per minute. To make immediate de-watering possible, under these circumstances, a line was connected to the existing line from the unit pump to the centrifuge, and extended to a yellow vat. It was then possible, by regulation of the inlet valve to the centrifuge head tank, to divert, to a yellow vat[2], the material the centrifuge could not handle (±10 lbs/min, dry basis).

[2](see diagram II)

The centrifuged material was re-pulped into a yellow vat for collection and, when the run was finished, re-centrifugation (with continuous Petronate treatment); the final centrifuge pulp discharge going into dryer pans.

The diverted material, collected in the other yellow vat, was vat washed, Petronate treated, and pressed.

Continuing with the sequential steps of operation (for the centrifuged portion only):[3]

19. Start the centrifuge.
20. Open slurry inlet valve to centrifuge head tank (provided conditions of step 18 have been fully satisfied), and regulate depth in head tank to ±4 inches.
21. Open centrifuge feed valve and regulate to give acceptable filtrate bleed.
22. Open re-pulp water valve to desired position (± 1 gal/lb.) - 1 1/2 turns (or slightly more)
23. Start re-pulped slurry pump.
24. When all the material has been centrifuged, and re-pulped into a yellow vat, measure, and start agitator in the yellow vat and open the valves to the centrifuge head tank.
25. Start the agitator in the head tank.
26. Start the Petronate flow into the head tank and regulate (1% treatment)
27. Open feed valve to centrifuge and regulate to give acceptable filtrate bleed.
28. Load the discharged pulp onto dryer pans.

If the proper care is taken when adjusting the centrifuge feed valves, and in making certain equilibrium has been reached at the selected valve position, there should be no difficulty in obtaining satisfactorily processed material.

TABLE III.

The following tables contain a list of the equipment items that need attention and the respective time cycles for inspection and checking etc. Included also, is a list of some operating checks and precautions.

EQUIPMENT CHECK LIST

Item	Attention Required	Cycle Frequency
1. Rotameters	Clean Out - (see equipment instructions)	Monthly
2. Screens in Rot.Feed Lines	Clean Out	After Each Run
3. Temperature Controllers	Mechanical Check	Monthly
4. Air Operated Steam Valves	Mechanical Check & Lubrication	Monthly
5. pH Meter	Clean Electrode (send to Research Lab)	Weekly
6. Agitators	Motor Check-Up	Routine
7. Pumps	Packing, Shaft Alignment, Etc.	Routine
8. Stand Pipes & Joining Lines	Clean Out Scale & Pigment Build Up	Monthly
9. Screens in Head Tanks	Clean Out	After Each Run
10. Thermocouples	Check With Hand Thermometer	Weekly
11.		

OPERATING & PRE-OPERATING CHECKS

Item	Attention Required
1. Rotameters	See if floats are free, and meter is not plugged.
2. Temperature Controllers	Turn on air and move pointer to see if controller responds.
3. pH Meter	Calibrate and re-check.
4. Pumps	Check Operate.
5. Valves	Check positions before run.
6. Tanks	Make certain all are available before starting run.
7. Centrifuge	" " it is " " " "
8. Lines	Make certain lines to and from centrifuge and unit are properly hooked up.
9.	
10.	

The following references include reports, to date, concerning the general aspects of continuous manufacture as well as specific information pertaining to medium yellows.

KN-40-155 - Cont. Strike Sheet -precipitation studies (Lab.)

KN-40-215 - Comparisons of Simultaneous strike vs Batch strike.

KN-41-246 Plant Scale Simultaneous Strike.

KN-44-77 - Cont. Mfgr. of Pigment Colors.

KN-44-78 - Cont. Mfgr. of Y-469-D in Lab unit.

KN-44-80 - Cont. Mfgr. of med. yellow in #6 Bldg. Pilot Unit.

KN-45-5 - Summary of Lab. Work on Cont. Mfgr. of Chrome Yellow.

KN-45-10 - S. W. develop. of Cont. process for Excelsior Yellow.

KN-46-3 - Cont. Mfgr. of med. yellow in #6 Bldg. Pilot Unit.

KN-46-8 - Cont. Mfgr. of Improved Med. Yellow, K-2217. Sam-Works Study.