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PIGMENT COLOR RESEARCH REPORT

Final Report

"OK STRAIGHT" STUDY - DRY COLORS

MARCH 1941 - MAY 1942

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

"OK STRAIGHT" STUDY - DRY COLORS

MARCH 1941 - MAY 1942

SUBMITTED BY: L. Stewart *[Signature]* DATE SUBMITTED: 7/23/46
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"OK STRAIGHT" STUDY - DRY COLORS

This report has been prepared to summarize and consolidate the information developed in the course of a research program which was carried out in 1941 and 1942. It has been designated the "OK Straight Study" since the object was to improve plant performance in respect to reproducibility from batch to batch. The study as such was discontinued in May, 1942 but has never been formally reported.

The following was compiled from a partially completed draft of a progress report prepared by Dr. Stewart at the time, together with copies of memoranda summarizing the status of the work from month to month. Other pertinent information has been included when readily available.

Attention was focussed for the most part on chrome oranges; however, some consideration was given to chrome yellows and to zinc yellow.

R.R.D.

INTRODUCTION:

Dry color which is not satisfactory for shipment on completion of the normal precessing procedure must be brought to specification by dry blending with other material. Standardization by dry blending is both time consuming and expensive.

Because of the increasing demand by the trade for more rigid standardization, the amount of dry color finishing equal to standard without dry blending, known as "OK Straight", decreased steadily from 39% of production in 1930 to an all time low of 14% in 1935. At that time the seriousness of the situation was recognized and steps were taken to improve the performance. Improvement was shown, until, for the last quarter of 1940, the "OK Straight" performance was 36.9%. Cost calculations made at that time indicated a savings of approximately \$40,000 per year could be obtained by increasing this to 85.0%. In view of the potential savings involved the Research Department was requested to assign a chemist to full time study of this problem. During 1941 and the first half of 1942 full time was given to the problem. However, during the last half of 1942 little attention, other than that of a general supervisory nature, was given the problem because of the pressure of other work.

GENERAL SUMMARY

As a result of the combined efforts of the Production and Research Departments, the "OK Straight" performance on colors was increased from an average of 36.9% for the last quarter of 1940 (the period immediately preceding the study) to an average of 78.4% for the last quarter of 1942 when the study was discontinued. This improvement in performance, 41.5%, was due primarily to two factors: -

- (1) An improvement in quality control.
- (2) An increase in zinc yellow production which usually runs 95-100% "OK Straight".

However, the increase in zinc yellow production at that time was relatively small and calculations in which correction is made for the effect of the increase in zinc yellow production show that approximately 75% of the improvement was due to improved quality control. Further confirmation of this fact is obtained by an examination of the performance of the individual color groups. A decided improvement is to be found on some groups as well as a general improvement on all groups.

As is to be expected, the above improvement has resulted in increased efficiency and reduced costs, especially in the grind shop. The amount of color processed in the grind shop increased from 300 lbs. per man-hour to 540 lbs. per man-hour and the labor cost per barrel of color standardized was reduced, roughly, from \$1.20 to \$0.65. Rough cost calculations show that these savings amount to approximately \$30,000 per year.

The individual studies leading to the above described improvement in "OK Straight" were: -

1. A general educational program based on a survey of color shop operations.
2. Revision and standardization of the tri-basic lead acetate process (See KN-41-159 and SF-25 formula, K-1482)
3. Revision and standardization of the lead nitrate process (See KN-44-46 and SF-10 formula, K-1720)
4. A general revision of formulas with emphasis being placed on control points, tolerances, etc.
5. A standard practice manual was written describing in detail the proper method of performing the various plant operations.

6. The chrome orange process was studied in detail and control refinements introduced into the formulas.
7. A general study of the chrome yellow processes was started.
8. A definite system of classification of ground color was established. This system was based on the criterion that color which grinds out "OK Straight" is the most desirable type from an economical standpoint.

Items 1 and 8 are discussed in the following section:

DISCUSSION OF DETAILS

Educational Program based on Survey of Color Shop

It was known in the beginning that the properties of a pigment depend primarily on its chemical composition, and the size and shape of its particles. The problem of improving quality control, therefore, resolves itself into establishing the necessary control over all factors affecting chemical composition and particle size and shape. In general, the principles governing particle formation were known. For example, it was known that the amounts of raw material, their purity, their acidity, etc., determine the composition of the pigment, while the size and shape of the particles are affected by the rate and temperature of strike, amount of agitation, etc. In other words, in order to produce "OK Straight" material standardized raw materials must be processed under standardized conditions.

Bearing this in mind, a program was set up to check the following:

- (a) Formulas - for duplicability, material balances, control points, equipment limitations, etc.
- (b) Raw Materials - for variation, purity, methods of analysis, methods of sampling, etc.
- (c) Equipment - for suitability, mechanical condition, accuracy (scales, thermometers, etc.), etc.
- (d) Operating Technique - for accuracy, methods, etc.
- (e) Evaluation of Product - for accuracy of method, choice of standard, practicability of shipping limits, etc.
- (f) Performance - by individual code so that the "bad actors" could be spotted. Normal practice had been to show the performance by group.

Classification of Ground Color

All of the lump color ground in the plant falls into one of the following classes:

1. "OK Straight" - Material satisfactory for the purpose for which it was made and shippable without further work being done on it. Material of this type leads to lower labor costs, decreased investment charges, and good customer service.

2. "Shippable Straight" - this includes material unsatisfactory for the purpose for which it was made but satisfactory, without additional labor being expended on it, for another purpose. Material of this type also leads to lower labor costs, but the amount that can be carried in stock is usually limited and its manufacture is accompanied by poor customer service.

3. "Sub-standard" - this includes material which is not satisfactory for the purpose for which it was made and must be blended with other material before it can be shipped. Because of the additional work involved in blending, the manufacture of this type material is relatively costly. The object of this study is to eliminate, as far as possible, the manufacture of this type material.

4. "Component" - in some cases it is more convenient to make a desired product by blending two or more standard colors. Material falling in this category is classed as a "component" since it obviously cannot be "OK Straight" as struck.

5. "Development" - all experimental material falls in this category. It does not affect "OK straight" performance.

6. "Shading Material" - this represents material deliberately made "off" standard for shading existing sub-standard and comprises 3 sub-groups -

- a) OK as shading material. Class as (4)
- b) Unsuitable for shading but shippable straight.
Class as (2)
- c) Unsuitable for either (a) or (b). Class as (3).

The "OK Straight" performance for any given period is obtained by dividing the pounds of color falling in class (1) by the sum of the pounds in (1), (2), and (3).

MONTHLY PROGRESS REPORTS ON "OK STRAIGHT" PROBLEM (Stewart)

MARCH, 1941

I - Chrome Oranges

A. YO-27-D

Observation in the plant has shown the reverse strike to be unsatisfactory from the standpoint of being able to control conditions of temperature, time, volumes, etc. In the laboratory good duplicability has been obtained using the direct strike which can be controlled.

Plant trial of the direct strike shows that conditions can be controlled satisfactorily but lighter material is obtained. Variables are being applied to this type of formula to give standard material.

B. YO-38-D

This is a chronic "pick list" color and the OK Straight performance has always been poor. Observation in both plant and laboratory indicates that the trouble is probably due to the method of making the "white base". In order to make a 6000 lb. batch in the plant it is necessary from volume considerations to add dry sodium bicarbonate to the lead liquor when precipitating the base. This method gives a lumpy non-uniform base and its conversion is therefore erratic. Laboratory study shows that check results can be obtained if the bicarbonate is added as a solution instead of dry. This would involve cutting the batch size from 6000 lbs. to 5000 lbs. Other means of overcoming this difficulty were tried but without success.

Plant work is now being directed towards determining the performance to be expected with all conditions controlled as closely as possible except for the use of dry bicarbonate. Following this, three batches will be made at a smaller batch size using controlled conditions and the bicarbonate in solution. A comparison of the performance by both methods will then be made to determine whether the decrease in batch size can be justified.

C. Light Granges

Plant performance on YO-263-D and YO-260-D has always been very poor. A laboratory study is now in progress to develop more information and better control of these formulae. Some of the variables being studied are acidity, chrome concentration, and development temperature. No results are available.

II - Miscellaneous

A. Although the plant OK Straight performance is reported monthly for each group, the performance for each individual code is not calculated at present. This has been done for January and February and will be done monthly in the future.

APRIL, 1941

I - Chrome Oranges

The new recording thermometer which was purchased for the chrome orange strikevat has been installed and was placed in service on April 28th. It was checked against a standard thermometer before use. Its use has confirmed our belief that temperature control on chrome oranges, especially Y0-38-D, was very poor. With the elimination of this important variable and the use of controlled formulae, the one important variable remaining to be controlled is concentration and acidity of lead liquor. Work will be started on this during the coming week.

A. Y0-27-D

The study of variables to give standard material was continued. Two batches were followed in the plant. Both were just outside shipping limits, one being too dark and the other too light. The study of control variables will be continued.

B. Y0-38-D

Five batches were followed in the plant. Two of these were made with variable changes to adjust the formula to give standard material. One of the remaining three batches, which were made on the same formula, was OK straight. It has been found, however, since the new thermometer was installed, that the temperature control on Y0-38-D was poor. Evaluation of the Y0-38-D formula using dry bicarbonate is being continued.

In connection with the laboratory study of Y0-260 -D it was noticed that several of the batches made were quite dark in masstone. Rubouts showed one of these to be quite close to Y0-38-D. This is of interest as it may be a means of making our Dark Oranges on a formula that can be controlled, that is, eliminate the dry bicarbonate method. This lead will be investigated further in the near future.

C. Light Oranges

The laboratory study of the Y0-260-D and Y0-263-D formulae has been completed and plant batches are now being followed. No results are available on the batch of Y0-260-D which was followed.

Investigation has shown that practically all Y0-260-D and Y0-263-D are sold as "specials". These four or five "specials" are made by shading with small amounts of chrome yellow or other orange. Work should be directed, therefore, towards striking these "specials" OK Straight rather than Y0-260-D and Y0-263-D. The Production Department has been asked to supply a list of such "specials" for consideration.

II - Zinc Yellow

Our present knowledge of the zinc yellow formulae indicates that the controlling variable, provided standard formula is followed in other respects, is the amount of impurities in the zinc liquor. The amount of impurities required to give standard material is not known. Variation in quality in the plant is due, no doubt, to the fact that a fixed amount of impurity is added to the zinc liquor without any consideration being given to the amount of impurities already in the zinc liquor. A preliminary survey shows that the impurities in the zinc liquor vary considerably. Work is being directed towards establishing a relationship between the amount and kind of impurities in the zinc liquor and tinctorial properties.

A. Y-232-D

Four batches were followed on standard formula in the plant and a close check on impurities made. All four batches were OK Straight. This work is being continued.

B. Y-467-D

Two batches were followed very closely for the purpose of improving the OK Straight performance and also to develop texture equal to our "HY-CAP" standard. Samples of these batches are to be submitted to the Ink Laboratory for evaluation. No further work is planned until results are obtained on these batches.

III - Miscellaneous

A. Plant OK Straight performance has been calculated for April for each individual code.

MAY, 1941

I - Chrome Oranges

A. YO-27-D

Three batches were made in the plant using the direct type of strike and controlled conditions. None of these were OK Straight. These results indicate that yellower material is to be expected from the direct strike than from the reverse strike. The reverse type of strike is to be avoided, however, as processing conditions are not readily duplicated due to equipment limitations. Control variables will be applied to the direct type of strike in an attempt to obtain more "in-line" material.

B. YO-34-D

The initial trial of YO-34-D in the plant using controlled conditions resulted in OK straight material. This batch will be checked when production is resumed.

C. YO-38-D

Study of the YO-38-D process in the plant was continued. The first two batches, check batches, made during the month were OK Straight, but, when production was resumed about a week later, using the same formula, very dark material was obtained. Apparently the plant had encountered one of its chromic "off" periods which necessitates a readjustment of the formula. The trouble has been traced to a shortage of lead in the strike and an average of bicarbonate of soda, both of which tend to give darker material. In order to prevent the reoccurrence of this type of trouble the plant has been asked to control the lead liquor within definite limits and the bicarbonate is being weighed instead of being used at the net weight specified on the bag. The formula has been adjusted to give standard material on this basis and a performance series is being run.

The YO-260-D type formula, which was developed in the laboratory last month for the manufacture of the Dark Oranges, was tried in the semi-works during the month. This formula, since it eliminates the solid phase or "white base" step in the regular process should be much more easily controlled and, therefore, give a higher OK Straight performance. The results of the semi-works batch are quite promising and another batch is to be scheduled as soon as equipment is available.

D. YO-260-D

Study of the YO-260-D process was started in the plant. Two batches were made using the direct type of strike and controlled conditions. Both batches, which were made for shading purposes, gave satisfactory mixes. This study will be continued when production is resumed.

E. YO-263-D

Study of this formula was also started in the plant. A batch, which was made for use in YO-422-D, a dry mix, was satisfactory for this purpose.

II - Zinc Yellow

Study of the zinc yellow processes was interrupted early in the month while the Development Section at the Operating Department's request, determines the maximum batch size for the present equipment. Before this work was discontinued, however, three batches of Y-467-D with "HY-CAP" texture were made. The development of "HY-CAP" texture on Y-467-D is being carried along as a part of the OK Straight study. This work will be resumed when the study of batch size is completed.

III - Chrome Yellows

With the discontinuance of the OK Straight work on zinc yellows, attention was turned to Chrome Yellows. Calculations, based on the first four months of 1941, show that the three yellows, Y-403-D, Y-424-D, and Y-490-D, which are dry mixes, comprise 43.5% of the color sold as dry mixes. If these three yellows were to be struck OK Straight, and the problem does not appear too difficult, a savings of approximately \$8000 per year could be realized. With this in mind a letter is being written requesting the Research Department to take up the study of this problem in the laboratory. An added reason for the study of Y-490-D, which is a special for G.P.I. and probably the largest single yellow consumed in the country, is that we only get a small part of this business. If the yellows were struck direct and OK Straight, it should lead to a much more uniform and satisfactory product.

IV - Miscellaneous

A. Plant OK Straight performance for April has been calculated for each individual code.

JUNE, 1941

Control of chrome oranges depends on the control of chemical composition and particle size. To control the chemical composition, it is necessary to control closely the amounts of raw materials used, especially those that effect the alkalinity of the system. To control particle size, physical conditions, such as temperature, volume, etc., must be controlled.

It is felt that the physical conditions and some of the raw materials are now controlled satisfactorily; however, the lead liquor has varied greatly and has been far from satisfactory for use on chrome oranges. Little progress has been made by the plant in meeting the specifications set up in a letter of May 21, 1941. During the early part of the month approximately 50% of the lead liquors used contained precipitated material while during the latter part of the month the liquors have been too acid.

Rapid methods for the determination of alkalinity and lead content have been developed in the laboratory. It has been suggested that the plant use these methods for control purposes and arrangements have been made to check all finished liquors in the laboratory by these methods.

Attached is a table giving the details on batches of orange followed in the plant and semi-works during the month.

I - Chrome Oranges

A. YQ-27-D

All the Light Oranges made in the plant to date by the direct strike have been muddy and yellow. Comparison of the reverse and direct type of strike, both in the laboratory and the plant, shows that the reverse strike gives cleaner, brighter and redder material than the direct strike. Although the direct strike is more desirable from a control and ease of operation standpoint, rather than embark on a long and costly study to eliminate the muddiness, it has been decided to improve the reverse strike as much as possible and determine the "OK Straight" performance to be expected.

B. YQ-34-D

The three batches made were within shipping limits of each other but two were too dark to be "OK Straight". Lighter material will be made when production is resumed.

C. YO-38-D

In general, batches made on the same formula were within shipping limits of each other provided the bicarbonate of soda was weighed rather than used at bag weight. Three batches, however, made with lead liquor which was found to be "off" were quite dark and muddy and did not check among themselves.

Trial of the YO-260-D type formula in the semi-works gave material which, although muddy, was relatively close to YO-38-D in shade. As the direct strike was used, it is believed that the reverse strike will eliminate the muddiness. A batch will be made by the reverse strike method when equipment is available.

D. YO-260-D

No production in plant.

E. YO-263-D

The reverse strike gave cleaner and brighter material here too. Variables will be applied to the reverse strike to give standard material and a performance study run.

II - Zinc Yellows

The "OK Straight" and development of "HY-CAP" texture studies have been inactive pending the completion of the study of batch size in the plant.

III - Chrome Yellows

The Research Laboratory is actively engaged in developing a formula for Y-490-D as a struck color. Y-490-D at present is made as a dry mix. Satisfactory progress is being made on this problem.

IV Miscellaneous

A. Plant "OK Straight" performance for May has been calculated for each individual code.

CHROME ORANGES - JUNE 1941

<u>Code</u>	<u>Batch</u>	<u>Results</u>	<u>Disposition</u>	<u>Remarks</u>
Y0-27-D	85981	lt; yel, vs wk -	SSS	Trial of reverse strike and controlled conditions.
"	86169	dk, red, s brt; s red, vs wk -	"	Same as 85981, but darker -
Y0-34-D	85422	vs dk, s red & brt; vs red -	OK	Controlled conditions for std. material
	85775	dk, red; red, vs wk -	SSS	Same as 85422 - too dk for std., but within shipping limits of 85422
	86302	dk, red, vs brt; vs red -	"	Same as 85775
Y0-38-D	85537	lt; yel -	"	Controlled conditions - Study of bicarbonate
	85538	"	"	Check on 85537 - Checks within shipping limits.
	85539	vs lt, s red, flat, chromy, vs yel - OK	OK	Controlled conditions - Study of bicarbonate
	85679	vs lt, s red & flat; s yel -	OK	Check 85539
	85680	dk, red, s chromy; red, vs wk	SSS	Controlled conditions - Study of bicarbonate
	85776	s dk, red, s chromy; vs wk -	OK for shading	Check 85680 - Requested s dk shading mtl.
	86088	v dk, red, muddy & chromy -	SSS	" " - TBL off
	86389	dk, red, muddy, chromy -	"	" 85539 - " "
	86390	dk, red, s chromy -	"	" 85539 - " "
	SW-4720	lt, muddy; yel -	-	Trial of Y0-260-D type formula - direct strike
Y0-263-D	86167	s lt (muddy, flat); yel, vs str - SSS	SSS	Direct strike and controlled conditions - requested vs lt material for Y0-422-D mix.
	86168	lt; s yel -	OK for shading	Check 86167, but reverse strike - requested vs lt material for shading

JULY, 1941

The "OK Straight" work was inactive during the period July 20th - August 3, 1941 while the staff was on vacation.

The control of alkalinity and lead content of lead liquors during the month was satisfactory. The improvement was obtained by adjusting the liquors, as soon as available, using the rapid methods developed in the laboratory for the determination of alkalinity and lead content. Examination of retained samples indicate that all of the lead liquors used during the month were stable, that is, had no tendency to precipitate on standing.

Preliminary results indicate that further improvement in the control of chrome orange manufacture has been obtained thru using pH as a measure of alkalinity rather than total pounds of acetic acid. This change has necessitated the readjustment of some of the control variables but it appears to be giving more uniform conditions.

Attached is a table giving the details on batches followed in the plant and semi-works during the month.

I - Chrome Oranges

A. YO-27-D

The two batches made were too light to be "OK Straight" and further variable changes must be made to get darker material.

B. YO-34-D

The single batch made was too dark. A further decrease in alkalinity will be made to obtain lighter material.

C. YO-38-D

Five batches were made in the plant. The last three were "OK Straight" and one of the others was OK in depth but was too red. This redness is common to both Plant and Development controlled material and has been a chronic complaint on this orange for several years. It probably will be necessary to make a study of this sometime in the near future.

Another batch of orange was made in the semi-works on the YO-260-D type formula using the reverse strike. The material was light and muddy against YO-38-D. Although it is felt that the product could undoubtedly be improved by changes in formulation, no further work is planned for the present since the regular YO-38-D formula is showing an improved performance.

D. YO-260-D

The single batch made finished too light in masstone. Control variables will be applied on the next manufacture to give darker material.

E. YO-263-D

The single batch made was light in masstone as requested but was too dirty in tint for use in YO-422-D, the purpose for which it was made. It was shaded without difficulty as YO-263-D.

II - Zinc Yellows

Inactive. Still awaiting completion of batch size study.

III - Chrome Yellows

The Research Laboratory has been able to develop a formula for making Y-490-D as a struck color. This is being turned over to the Development Section for further study. It is estimated that eliminating dry mixing on Y-490-D will result in a savings of approximately \$1500 per year.

IV - Miscellaneous

A. Plant "OK Straight" performance for June has been calculated for each individual code.

CHROME ORANGES - JULY 1941

<u>Code</u>	<u>Batch</u>	<u>Results</u>	<u>Disposition</u>	<u>Remarks</u>
Y0-27-D	86735	lt; s yel - 3% wk; s yel, cl	SSS	Reverse strike; acidity of TBL controlled by pH; study of control variable.
	86960	s lt (red & cl); s yel - OK; yel "	"	Same as 86735, except alkalinity increased for darker material.
Y0-34-D	87059	dk, red; s red - 3% wk; red	SSS	Acidity TBL controlled by pH; decreased alkalinity for lighter material.
Y0-38-D	86736	s dk (red & flat); s red -	SSS	Acidity TBL controlled by pH; material too red and flat to be OK Straight.
	86821	dk (red & chromy); s red -	SSS	Same as 86736; material just outside OK Straight range in depth.
	86822	OK (red, flat & chromy); vs yel - OK		same as 86736.
	86823	OK (red, flat & chromy); vs yel - OK		same as 86736.
	87060	vs dk, s red; vvs strg. -	OK	same as 86736.
	SW-4818	lt, muddy & flat; yellow	-	Trial of Y0-260-D type formula - reverse strike.
Y0-260-D	86553	lt, flat, s muddy; yel, vs str -	SSS	Trial of reverse strike and controlled conditions; muddiness not apparent when shaded to standard depth.
Y0-263-D	86391	lt, flat, s muddy; yel -	-	Reverse strike; variable study; requested light material for Y0-422-D - too dirty in tint. Satisfactory in Y0-263-D mix.

AUGUST, 1941

Supervision and control of the manufacture of lead liquor was continued during the month. Control of concentration and alkalinity was satisfactory. A detailed formula for the manufacture and control of the lead liquor process is being written preparatory to returning the supervision of this process to the Plant staff.

Attached is a table summarizing the batches followed in the plant during the month. A detailed discussion of these batches is given below.

I - Chrome Oranges

A. YO-27-D

The adjustment of control variables to give OK Straight material was continued. The first two batches, made on the same formula, checked each other quite closely but one was a little too dark to be OK Straight. The next two batches were made to be lighter but while they checked each other quite closely, one was a little too light to be OK Straight. The fifth batch which was made to be a little darker than the two previous batches was still a little too light to be OK Straight. Further changes will be made until OK Straight material is obtained. A performance study will then be run.

B. YO-34-D

No production.

C. YO-38-D

A performance study was run during the month using the formula developed during July. The results indicate that several difficulties must be overcome before OK Straight material can be made consistently. They are: -

1. Bicarbonate of soda which has been stored for two-three weeks becomes hard. This gives a lumpy "white base" and probably erratic results. This is the first time this trouble has been encountered and further investigation is necessary.

2. The chronic complaint of red and chrome masstones was encountered. Several recent batches were satisfactory in depth of masstone but were too red and chromy. This problem has been studied in the laboratory several times during the past few years but little progress has been made in its solution. Since this problem is now on the plant "sick list" and since it must be overcome before OK Straight YO-38-D can be made, it has been decided to study it in the OK Straight laboratory. Its successful solution will probably require a long and tedious study of the fundamentals involved in the manufacture of YO-38-D.

3. Two "off" batches were made during the performance study. Analyses are now being made on all the retained samples of raw materials, slurries, and backwaters to determine the cause. Preliminary results indicate that the balance between the lead oxide and bicarbonate is close enough to the upset occasionally by a combination of variations in raw material analyses, weights, etc. It is known that an unstable product will result if there is no soluble lead present at the "white base" stage. It has been decided to return this formula to the laboratory to determine definitely the causes of "off" batches and to develop a more stable formula. Plant work on YO-38-D will be discontinued until this work is complete.

D. YO-260-D

No production.

E. YO-263-D

The adjustment of control variables was continued from last month. The single batch made was OK Straight. A performance study will now be run using this formula.

II. Zinc Yellows

Inactive. Plant OK Straight performance is very good. Development of "HY-CAP" properties is awaiting completion of batch size study.

III. Chrome Yellows

The Research Department is now actively engaged in the development of a formula for making Y-424-D, the duPont medium yellow, as a struck color.

IV. Miscellaneous

A. The Plant OK Straight performance by individual code for the first six months of 1941 is being issued as a matter of general information.

CHROME ORANGES - AUGUST 1941

<u>Code</u>	<u>Batch</u>	<u>Rebout</u>	<u>Disposition</u>	<u>Remarks</u>
YO-27-D	87710	dk, red, vs brt; red, vs wk -	SSS	Study of control variable. Within shipping limits of 87711 but too dark to be OK. Check on 87710
	87711	vs dk, red, s brt; vs wk - 8% wk; red	OK	
	87880	s lt & red & brt; s yel, vs wk - 2% wk; yel	OK	Check 87710 except control variable used to give lighter material
	87881	lt, s flat; vs wk - 3% wk; yel	SSS	Check on 87880. Quite close to 87880 but too light to be OK
	88200	lt, s flat; s yel - vvs wk; yel	SSS	Control variable to give material between 87710 and 87880
YO-38-D	87609	s dk (red, muddy, chromy); s red, vs wk -	SSS	Check on 86823. Depth of massstone OK but too red and chromy.
	87775	lt, flat, s yel, chromy; yel, vs str -	SSS	Check on 86823. Bicarbonate very hard and lumpy from long storage.
	87776	vs dk (red, muddy, chromy); vs red - 2% wk; vs yel	OK	Check on 86823. New bicarbonate used.
	87879	dk, red, muddy, chromy; s wk -	OK	
	87976	lt, yel, s flat; yel, s str -	SSS	Requested dark shading material - OK for this purpose.
	88074	vs dk & muddy (red & chromy); vs red - vvs wk; vvs yel.	OK	" " "
	88075	s dk & muddy (red); vs wk - 5% wk; vs dirty	OK	" " "
	88076	lt, muddy, flat; yel -	SSS	" " "
	88202	v dk & red & muddy, chromy; wk -	SSS	Requested dark shading material.
	88296	dk, v muddy & chromy (red); wk -	SSS	Check on 86823
YO-263-D	88201	s red & brt; vvs yel - vvs str; yel & s clean	OK	Study of control variables.

SEPTEMBER, 1941

I - Lead Liquor

Study of the lead liquor process has been completed, a process report written, and control of the process returned to the plant staff. The important changes introduced were: -

1. Use of standard batch cards giving detailed operating instructions.
2. Use of more efficient operating conditions.
For example, it has been shown that one 1-hour run over the feathered lead gives satisfactory liquor, whereas two runs of 2-3 hours each were formerly used.
3. Conditions under which lead liquor is stable, does not precipitate on standing, have been determined and all liquors are being adjusted to these conditions.
4. Rapid control tests have been developed and are being used for the control of the lead liquor process.

II - Chrome Oranges

A. YO-38-D

Work on YO-38-D was confined to the laboratory. Two problems were studied during the month. They are: -

1. Causes of flopping - Two definite causes of flopping have been found to date. Flopped batches in the plant are probably due to either or both of these conditions. The present formula will be revised, based on this knowledge, to give a more stable formula.
2. Causes of red-chromy masstones - The problem is being attacked from several angles.
 - a. Laboratory study of strike variables - No definite leads have been uncovered to date.
 - b. Particle size, shape and composition - Samples of satisfactory material and red-chromy material have been sent to the Experimental Station for comparison under the electron microscope and a spectrochemical analysis.
 - c. Testing Method - All complaints to date have been based on rubout results. Paint grinds, etc., are being made to determine whether the redness and chrominess is evident under actual conditions of usage. Preliminary results indicate that there is no correlation between rubout results and those obtained in other systems.

B. YO-260-D

Two batches were made in the plant. They were very close checks, one being "OK Straight" and the other a little too dark. A performance run will be made using this formula when production is resumed.

III - Zinc Yellows

The plant "OK Straight" performance continues satisfactory and no work is planned for the present.

IV - Chrome Yellows

Work on the development of a formula for making Y-424-D, the duPont medium yellow, as a struck color, was continued in the Research Laboratory. The results, to date, have been rather erratic, probably because of the difficulty of controlling the composition by precipitation methods.

V - Miscellaneous

- A. A process report for the control and manufacture of lead liquor has been written.
- B. The plant "OK Straight" performance by individual code for the first six months of 1941 has been issued for the guidance of those interested in the improvement of "OK Straight".

CHROME ORANGES - SEPTEMBER 1941

<u>Code</u>	<u>Batch</u>	<u>Rubout</u>	<u>Disposition</u>	<u>Remarks</u>
Y0-260-D	88399	vvs dk, s red; vs yel - 2% str; vs red	OK	Study of control variable to give standard material.
	88400	vs dk (red) floods s dk; vvs wk - vvs wk; s red	SSS	Study of control variable to give standard material.

OCTOBER, 1941

The other "OK Straight" work was interrupted early in the month in order to give full time to checking the lead nitrate operation. Chrome Yellows made from lead nitrate have been running too dark and dirty in masstone and it was felt that this was due for the most part to the lead nitrate.

I - Lead Nitrate

As a matter of record and in order to avoid their reoccurrence, a detailed discussion of conditions which were responsible for the poor grade of lead nitrate being made are listed below. Recommendations are also included for further improving the lead nitrate operation.

1. A brown sludge which has been shown to give dark and dirty yellows was found in all the lead nitrate storage tanks. The tanks have been cleaned and are to be cleaned on schedule in the future.
2. The electric eye which is the sole guard against the brown sludge leaking into the storage tanks was in such poor mechanical condition that it was useless as a protective device. A new lamp is to be installed and the system rearranged. The electric eye should be scheduled for inspection at least once a month in the future.
3. The pump switch which is normally actuated by the electric eye was too rusty for automatic operation. This has been remedied.
4. Assuming the electric eye to have been in good condition, it would not have been possible to utilize it for control purposes because of the following conditions: -
 - a. One of the glasses in the trough was broken. It is being replaced.
 - b. Too much wood fibre suspended in the solution. The press is in very poor condition. New plates are being made. These should be painted with a protective material to increase their life.
 - c. Too much entrained air in the solution. This was due to the pump needing packing and the poor condition of the press and accessories. The pump has been packed; the press is being reconditioned, and small changes have been made in the pressing procedure. A screen has also been installed in the trough to break up the air bubbles and eliminate the wood fibre.
 - d. Too many lead nitrate crystals entrained in the solution. Conditions under which lead nitrate solutions are stable are being determined and all lead nitrate will be adjusted to these conditions.

5. The test for iron under the present conditions was found to be worthless due to the relatively large amount of nitrite present in the lead nitrate. A new test is being developed.

6. In general, the operation was sloppy and important differences existed in the methods used by the three operators. The operators are being instructed and trained as the work progresses.

The following suggestions are offered as a means of further improving the lead nitrate operation: -

1. Increase the lead nitrate concentration to an average of 22%. It has been running 18-19% for some time. This is now being studied.

2. Improve the agitation in the purification tank. The poor agitation increases the time of purification and adjustment, and leads to lower lead yields and non-uniformity of reaction.

3. The present press pump has too much capacity for the present system, resulting in leaks in the press, blowing air, and higher maintenance and power costs. A slower speed motor on the pump would lead to more efficient operation.

4. Lack of storage capacity results in hurried operation during peak production periods. This has been exaggerated by the recent increase in batch size on lead nitrate colors. Hurried operation is resulting in sloppy operation and poorer quality lead nitrate. Consideration should be given to installing another storage tank in the rear of #18 Building and larger scale tanks in #6 Building. It should be possible to use wooden tanks for this purpose.

5. As a check on the lead nitrate operation a continuous sampler should be installed on the line from the clarification press.

6. The present method of adding nitric acid for adjustment purposes should be eliminated due to the hazard involved and higher maintenance costs.

II - Lead Liquor

The quality and control of the lead liquor operation continues satisfactory.

III - Chrome Oranges

A. YO-27-D

Two batches were made for the purpose of studying control variables. Both were a little too dark to be "OK Straight" but were readily shadeable.

B. YO-38-D

A single batch was made to try a new and more stable formula. It was too light and it will be necessary to apply known variables to give darker material.

CHROME ORANGES - OCTOBER 1941

<u>Code</u>	<u>Batch</u>	<u>Rubout</u>	<u>Disposition</u>	<u>Remarks</u>
Y0-27-D	89254 89145	dk red, s brt; red, s wk - s dk & brt (red); s wk -	SSS) SSS)	Study of control variables to give standard material
Y0-38-D	89444	lt, yel; yel, vs str -	SSS	Trial of new and more stable formula

NOVEMBER, 1941

Full time was given to improving the lead nitrate operation and process. Except for occasional batches, the lead nitrate was satisfactory throughout the month. Elimination of the poor batches and a further general improvement depends primarily on the completion of pending equipment changes. The improvement in lead nitrate quality to date has resulted in much less dark - dirty masstone chrome yellow being made.

A brief discussion of the status of the lead nitrate work is given below:

1. The storage tanks were cleaned during the month. The storage tanks are being cleaned once per month and the scale tanks once per week.

2. The electric eye has been mounted on a permanent base so that it is not affected by vibrations, spillage, and condensation. The old lamp should be replaced and the cover to the lamp and eye locked to prevent tampering.

3. The rheostat which controls the eye has been locked to prevent tampering. The present ammeter is to be replaced as it is in need of repairs. When this replacement is made it will be possible to calibrate the system so that its efficiency can be checked at any future date.

4. There are ten bad plates in the press. An order has been issued for replacements.

5. The majority of the spigots and hose on the press are worn out. Replacements are on order.

6. The agitation on the purification tank is unsatisfactory. A project has been written to install a larger agitator.

7. The press pump is too large for the present system. The Mechanical Department is trying to locate a slower speed motor for this work.

8. The Industrial Engineers have installed a temperature recorder on the make tank. This will provide a much needed means of control for this operation.

9. A project is being prepared to install a small pump for use in adjusting the lead nitrate.

10. The lights in #19 Building have been moved so that the spigots and trough can be observed during the night shift. A study is being made for the purpose of providing sufficient light for operation at night.

11. Consideration is being given to installing a continuous sampler on lead nitrate. This would provide a much needed check on the lead nitrate operation.

12. Operating instructions covering the lead nitrate process have been written and posted in the leadshop and #19 Building for the guidance of the operators.

13. A new test for iron has been developed and is now on trial in the plant.

14. Corrosion tests are being run to determine the effect of lead nitrate on iron pipe. The new test for iron is also being used to determine the iron pick-up in the present system.

15. For the present, all lead nitrate is being adjusted to approximately pH 4.0. This appears to prevent the formation of crystals and yet is not acid enough to attack the iron piping.

DECEMBER, 1941

The lead nitrate operation was checked throughout the month. Although there were several periods of poor quality, in general, the quality of lead nitrate showed an improvement over last month. These "off" periods are due to our inability to control the pH of the lead nitrate with the present equipment and methods. The possibility of adapting conductivity equipment from the lithopone plant to the measurement of pH is being investigated.

The new iron test which has been on trial during the past month appears to be satisfactory. The test is also proving valuable in the study of corrosion and iron pick-up in various parts of the system. Preliminary results indicate that the pH in the storage tank must be kept above 3.5 to avoid contamination with iron while pumping to the scale tanks in #6 Building.

A new lamp and micro-ammeter have recently been installed in the electric eye circuit. These are now being calibrated so that the efficiency of the system can be checked in the future. After a trial period an inspection schedule will be set up. Several tests have shown that the eye is working satisfactorily and that no solids are passing into the storage tanks.

Chrome Yellows have been made in the laboratory from plant lead nitrate containing iron to determine the maximum amount of iron which can be tolerated under present conditions. Preliminary results indicate that the plant is now operating below the maximum amount of iron which can be tolerated, whereas several months ago they frequently were above the maximum.

Several batches of lead nitrate which were made on standard formula were followed. When the Industrial Engineers complete their study on nitric acid yields, the making operation will be studied further with the object of shortening and improving the control of this operation.

JANUARY, 1942

The lead nitrate operation was checked throughout the month. Daily samples of the finished liquor indicated that, except for one day, the lead nitrate was satisfactory. The one exception was due to the mechanical failure of an acid valve. Very little dark and dirty masstone chrome yellow was made as a result of the improved performance on lead nitrate.

All of the recommended equipment repairs and improvements, except the installation of a pH electrometer and continuous sampler, have been made. The equipment is now in excellent condition and little trouble is to be expected in this respect in the future. Consideration is still being given to the purchase of a pH electrometer and the installation of a continuous sampler.

A detailed study of the lead nitrate purification process has been completed and, except for pH control, the methods are now satisfactory. The only dependable method found for the control of pH is the electrometer.

The electric eye has been calibrated and a method set up for checking its operating efficiency. It is recommended that it be checked at 30 day intervals in the future.

At the Plant's request samples of finished lead nitrate were taken throughout the month for the purpose of determining the average nitrite content. A KCR method for the determination of nitrite has been developed and will be used in determining the nitrite content of these samples.

A trial run was made at the Plant's request to determine whether it is possible to eliminate the purification step by consuming the lead sludges at rates low enough to keep the impurities within specifications. Preliminary results indicate that satisfactory lead nitrate can be made in this manner if close supervision is provided. The process has certain weaknesses and it is felt that there may be more desirable means of accomplishing the same purpose. These have been fully discussed under separate cover.

As of January 12, 1942 the plant has been using, without research supervision, the Chrome Orange formulas developed during the "OK Straight" study. These formulas specify volumes, times, temperatures, pH, etc., which normally are not controlled. At this time, the results are too few to justify any conclusions. It is planned to give a few days to checking the operation on these formulas during the coming month.

FEBRUARY, 1942

Lead Nitrate

Daily tests made on the finished lead nitrate liquor indicate that it was satisfactory for use in the manufacture of colors. The quality of Chrome Yellows made during the month confirm these tests.

A study of the lead nitrate making operation was carried out during the month. A detailed operating procedure has been written, based on this study, and is now on trial in the plant.

A trial run has been in progress for the past three weeks to determine whether it is feasible to manufacture satisfactory lead nitrate without purifying and without clarifying in the filter press. During this period pig lead has been the sole source of lead and the liquor has been clarified by settling. The results to date indicate that satisfactory lead nitrate can be made in this manner provided the following conditions are met: -

1. Pig lead continues to meet specifications.
2. Each batch of lead nitrate is completely finished (pH 4.4) before pumping from make tank.
3. The pH is adjusted within the range 3.8 - 4.2 before settling.
4. The settling period is held to a minimum of 30 minutes.
5. The tank cleaning schedule, recommendations to be made later, is adhered to rigidly.
6. Close supervision is provided - much more than at present.

Chrome Oranges

The "OK Straight" study on Chrome Oranges was resumed on January 28, 1942. This study was interrupted several months ago because of the variable character of the lead liquor. This is the first work on Chrome Oranges since the difficulties on lead liquor were eliminated. During the period January 12 - 28th, the "OK Straight" Chrome Orange formulas were used in the plant without supervision by the Development Section. Attached is a table showing the results on all the batches made on these formulas. Batches marked with an asterisk were not followed by the Development Section. A brief discussion of the performance of each Orange is given below.

YO-27-D

This formula appears, except possibly for a few minor adjustments, to be satisfactory. All the batches made under the Development Section's supervision were "OK Straight" and six of the last eight batches were "OK". The cause of the poor performance at the beginning was located and has now been eliminated.

YO-34-D

This formula now appears to be satisfactory - four of the last five batches were "OK Straight".

YO-38-D

This formula is still unsatisfactory and has been returned to the laboratory for further study. The plant is also having trouble controlling the regular formula at this time. Apparently there is still some unknown variable in this formula which must be located before consistent results are obtained. Occasional batches will be made in the plant as a check on the laboratory work.

YO-260-D

There was only one batch made. Additional experience will be needed before drawing any conclusions concerning this formula.

YO-263-D

Although two of the four batches made appear to be "OK Straight" versus the regular standard, the results are too few to justify any conclusions at this time.

<u>Code</u>	<u>Batch</u>	<u>Disposition</u>	<u>Results</u>	<u>Remarks</u>
Y0-27-D	91806*	SSS	lt, s flat; yel - vvs wk; yel	Possibly "OK Straight" but needed for mix at the time.
	91807*	OK	vs lt, s red; vs yel - 5% wk; vs red & dirty	
	91873*	SSS	s lt & brt; vs yel - OK; vs yel	
	91947*	SSS	lt, vs cln; s yel - vvs str; yel, vs cln	
	92090*	OK	vvs lt (brt) s red; s wk - 2% wk; vs yel	
	92091*	OK	vs lt (red) s brt; vs wk - 6% wk; s red	
	92194*	SSS	lt; yel -	
	92279	OK	vs dk (red) s brt; vs wk - 5% wk; red	
	92362*	OK	vs lt (red) s brt; vs wk - 7% wk; s red	
	92529	OK	vvs lt (red & brt); vs wk - 6% wk; s red	
Y0-34-D	92530*	SSS	s dk (red) vs brt; s wk - 8% wk; red	Formula changed to give lighter material.
	92694	OK	vs dk (red) vs brt; vs wk - 7% wk; red	
	91872*	SSS	dk, red, vs brt; s red -	
	92092*	OK	vs dk, s red & brt; vvs yel - vvs wk; vs red	
	92280	SSS	dk, red, brt; s red, vvs wk -	
	92426	SSS	dk, red, s brt; red, vs wk -	
	92428	SSS	dk, red, s brt; red, vs wk -	
	92529	OK	vvs dk, s red & brt; vs red - OK; vs yel	
	92573	SSS	s dk (red & brt); s red -	
	92575	OK	vvs dk, s brt; vs yel - OK; vs yel	
Y0-38-D	92693	OK	vs dk, s red & brt; vvs cln - OK; vvs red	Formula changed to give lighter material
	92763	OK	vs yel & brt; vs yel - vvs str; s yel	
	91871*	SSS	s lt & red (flat & chromy); yel, s str -	
	92019*	"	dk, v red (muddy & chromy); wk -	
	92020*	"	dk, v red (chromy); wk -	
	92360	"	lt, flat, chromy; s yel -	
	92361	"	lt, flat; yel, vs str -	
	92427	"	lt, s flat; s yel -	
	92574	"	lt, s flat & chromy; yel & str -	
	92192	"	dk, red, vs brt; red & vs wk -	
Y0-260-D	92192	"	dk, red, vs brt; red & vs wk -	TEL did not meet specifications.

CHROME ORANGES - FEBRUARY 1942 Cont'd.

<u>Code</u>	<u>Batch</u>	<u>Disposition</u>	<u>Results</u>	<u>Remarks</u>
YO-263-D	91835*	(SSS)	red & brt; s red, vs wk - vvs str; s cln	Made for YO-422-D "OK Straight" vs regular Std.
	91955*	SSS	s lt (v muddy); yel, vvs str -	
	92153*	SSS	lt & s flat; s yel - 5% str; yel	
	92281	(SSS)	vs lt, s red, brt; - OK; s red	vs ABW std; "OK Straight" vs regular std.

* - denotes made on "OK Straight" formula but without Development Section supervision; all others were made on "OK Straight" formulas under Development Section supervision.

MARCH/APRIL, 1942 - No reports)

MAY, 1942

Chrome Oranges

There was no production on Chrome Oranges during the month. When manufacture is resumed the "OK Straight" formulas will be used on all Oranges.

Chrome Yellows

A general survey of the Chrome Yellow equipment and operation has been made. This was followed by a more detailed study of raw materials and the various operations involved in the delivery of the proper amount of raw material to the strike vat. A brief discussion of the raw materials investigated and the equipment used in handling them is given below: -

Sodium Chromate

analyses - satisfactory
uniformity - samples taken from hose to strike vat check
those taken from storage tank.
alkalinity - varies considerably but majority of formulas
now require adjustment before use.

Sodium Dichromate

analyses - satisfactory
uniformity - "
acidity "

Lead Nitrate

analyses - in general good, but several found outside limits.
uniformity - satisfactory
acidity - at times varies considerably
impurities - contained too much iron three of the eleven days
checked.

Sodium Molybdate

analyses - satisfactory
uniformity - satisfactory
impurities - suggest that brown sludge in storage
tanks be removed.

Sodium Bicarbonate and Soda Ash

uniformity - contains too many large lumps
weights - bag weights are incorrect - should always
be weighed.

Scales and Weighing

Scale Vat #359 - used in weighing sodium molybdate solution.
Found to be in good condition.

Scale Vat #351 - used to weigh chromate, acids, alum, etc.
Found to be in good condition.

Scale Vat #356 - an alternate for #351.
Not satisfactory for use on Molybdate
Orange, possibly satisfactory for Yellows.
Location of beam relative to solution
valves contributes to inaccuracies.

Scale Vat #354 - used for weighing lead nitrate solution.
Inherently a good scale but now sluggish
and tends to stick, requiring considerable
care in its use. Beam not visible from
control value.

In weighing it should be made a general practice to balance and read the scale just before drawing solutions. Several instances were found where the weighing was done some time previous to withdrawing solution and examination before withdrawal showed that the weight had changed. Leaking valves usually are the cause of such changes.

During the course of the general survey several conditions were encountered which should be corrected. They are: -

1. Several of the agitator turbines are so badly worn that little or no agitation is supplied in the upper portion of the vat.
2. The practice of re-using paper sample cups should be discontinued. A sample of lead liquor taken in one of these cups showed 7.2 pH whereas checks in glass ran 9.15 pH which is normal for lead liquor.
3. On several occasions it was found that either the late maker or the nightman had not washed out equipment and hose sufficiently. As an example, a sample of water taken from the hose to Vat #351 contained a white sediment and its pH was 10.7. This solution would normally go into Molybdate Orange which is quite sensitive.

Work to date has been confined to the making operation. The treating (nightman), pressing, drying and grinding operations are still to be studied.

REFERENCES:

Experimental work is recorded in Laboratory Notebooks 884 and 932, and DSN 46-26.