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KW-45-33

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Progress Report

Title: LIGHT CHROME YELLOWS: STUDIES ON REACTIVITY, COLOR STABILITY, ETC.

Period Covered: JUNE 1948 - JUNE 1949

Charge: 1100-11-3 (A-IC-3)
1100-12-3

FILE: 211.1

DATE: July 15, 1949

211.1

N.26279

N42214

Serial No. KN-49-33

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NEWARK PLANT

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SUBMITTED BY: R. R. DENSLOW DATE SUBMITTED: June 27, 1949

APPROVED BY: B. H. PERKINS 7/15/49 DATE ISSUED: July 15, 1949

INTRODUCTION:

This report covers work carried out during the period June 1948 - June 1949. The purpose was to improve our Light Yellows, Y-404-D/Y-405-D in respect to certain properties in order to meet competition, particularly the corresponding Calco products 40-3355 and 40-3615. Laboratory work has been active throughout the period and a number of experimental runs have been made in the 12 Building and 10 Building continuous units. This latter work is not covered in detail here since it is planned to review it in a separate report.

For the most part, attention has been focussed on "color stability" (behavior of masstone exhibits on drying), ink consistency and reactivity. Some work was also carried out on bake resistance (linoleum print paint) a property probably related to color stability. Texture, drying (in ink) and certain other properties are touched on in an incidental way.

SUMMARY AND CONCLUSIONS:

Products superior to standard Y-405-D in all important properties and apparently at least equal to the best competitive material have been made in the laboratory by batch processes. Products of good color, strength, lightfastness, bake resistance and color stability have been made batchwise in the plant. However, they were somewhat deficient in rheological properties against the established goals and too reactive to be satisfactory. Products have been made continuously (in 12 Building) which were superior to standard but inferior to the best batch material and competition particularly in respect to color stability.

An important cause of the high consistency and reactivity is the hydrous alumina precipitated on the yellow to give improved color and lightfastness; reducing the amount of alum gives lower consistency but at the expense of the other properties. The reactivity can also be reduced by adding sodium phosphate (preferably pyrophosphate) to the partly neutralized slurry. The low reactivity in this case is frequently accompanied by slower ink drying which can in turn be partly remedied by the addition of a second portion of lead nitrate. A relatively low final pH is also desirable.

Color stability is influenced by various factors of which possibly the most important is the presence of undeveloped pigment in the product. The problem is more serious with continuous manufacture than with batch manufacture. The relatively poor texture of continuously manufactured yellows is probably due in part at least to the same factor.

Further details are presented in the body of this report.

PATENT SITUATION:

The yellows discussed in this report are treated according to the procedure specified in our U.S.Patent 2,212,917 (S.C.Horning).

In our opinion, none of the information developed in the present study is of a patentable nature.

FUTURE PROGRAM:

Since the preparation of this report, emphasis has been shifted to the development of batch products equal to the best competition but with special attention to the properties discussed below. The immediate program comprises additional laboratory work along with batch runs in the plant (Y-519-D type) using variables which appear likely to give the desired results. A limited amount of work will be carried out in the semi-works continuous unit, chiefly to be of assistance to the Plant Quality and Control Group in their concurrent study of the same general problem in 12 Building.

Following the work on the Y-405-D shade, it is planned to continue with the lighter product, Y-404-D. It is hoped that the work on Y-405-D may make this latter a relatively minor problem.

DETAILED REPORT:

Historical - Origin of the Problem and General Objectives:

In 1948, our current standard (as of that date, batch material) on Y-405-D was tested in H-108 and alkyd against four samples of Calco's 40-3615. Similar tests were run on a temporary shipping standard of Y-404-D against two samples of Calco's 40-3355. Included also were samples of improved products made in the semi-works by batch procedures, namely, K-1957 (Y-404-D type) and K-2024 (Y-405-D type). The following results were obtained:

> "40-3355 is slightly superior to Y-404-D in color by H-108 test. The superiority is less marked in alkyd. Lightfastness is better in both vehicles. Compared to K-1957 it is approximately equal on fresh H-108 rubout but it darkens on drying. It is definitely dark in alkyd, lightfastness is worse in this vehicle.
>
> "40-3615 in H-108 rubout varies in masstone color from dark to vvs light against Y-405-D; two of four samples darkened on drying. Lightfastness was slightly to definitely better. Color in alkyd was quite close to Y-405-D. Lightfastness was vs to s. better. In H-108, the Calco samples varied from dark to vvs light initially against K-2024, but showed a tendency to darken on drying. They were dark in alkyd. Lightfastness (compared with K-2024) was slightly worse to worse in H-108 and definitely worse in alkyd."

It was concluded that while the Calco products showed some superiority to the existing standards, they were inferior to material which we had made successfully in the semi-works.

Our present (12 Building) standards on Y-404-D (lot 10115) and Y-405-D, (lot 10116) were established early in 1948, at which time they were evaluated against not only the older batch

standards but also against new samples of 40-3355 and 40-3615. Y-404-D, lot 10118, was close in color to the old standard but vs stronger (95/100) and better in lightfastness. Against 40-3355 it was red in masstone, vs stronger (90/100) and vs better in lightfastness. Y-405-D, lot 10116, was also close in color to the old standard, equal in strength and slightly superior in lightfastness. Its relationship to 40-3615 was substantially the same as to the old standard.

In rheological properties, Y-404-D, 10118, was inferior to both the old standard and 40-3355. The latter two were quite similar, altho the Calco yellow gave a very slightly softer ink. In the case of Y-405-D, the differences were much less marked altho here also the new standard appeared very slightly heavier than the old one. The new standards were worse in texture than the old ones but no worse than competition. (Reference - Letter H.A.M. - F.E.B.- 2-5-48).

In connection with a sales problem (Armstrong Cork) a limited amount of work was carried out about a year ago with the object of improving the bake resistance of Y-405-D (continuous process material) in linoleum print paint. This resulted in setting up a special standard, Y-577-D, based on modifications of the standard continuous process. Y-577-D was considered to be a satisfactory counter-offering altho inferior in bake resistance to certain batch products.

The problem of color stability arose originally as the result of a complaint by IPI, Chicago (CD-270, April 1948) that exhibits of Y-405-D darkened on drying to a greater extent in their testing vehicle (Cobalt Drier) than similar exhibits made from Calco's 40-3615. Subsequently, it was found that Y-404-D also darkened more than its counterpart 40-3355. In both cases, the difference is one of degree; darkening occurs with the Calco yellows but is less marked than with our products.

Preliminary work on this problem indicated Y-577-D (see above) to be superior to Y-405-D in color stability; however, it was not as good as 40-3615 and like Y-405-D was considered to be too reactive to be generally acceptable. Our so-called Y-519-D type (lots 57028, 57003) was apparently satisfactory in stability but was even more reactive; moreover, it involved a batch operation and has, therefore, not been given serious consideration as a permanent solution to the problem.

The work reviewed above served to set up fairly definite objectives, namely, to improve our continuous process light yellows in color stability, ink consistency, and reactivity. Obviously, these improvements should be obtained without any significant sacrifice of other properties such as masstone color, tinting strength or lightfastness.

Light Chrome Yellows - General Considerations:

While Light Yellow formulas are all superficially similar to each other, they differ in certain points some of which are indicated below for products of the Y-405-D type. Similar products of the Y-404-D type are made by changing the chromate/sulfate ratio.

	Y-405-D	Y-577-D	Y-819 (1)	AY-578 (2)	BY-578 (3)
Type Process	Continuous	Continuous	Batch	Batch	Continuous
Chromate/Mole	108	104	109	109	104
Development	140/180°	140/200°	Boil	120°	125/140°
Wash before treatment	No	No	Yes	No	No
Alum/L Liquor per mole	17/8	17/8	32/8	32/8	17/8
Second Lead Nitrate	No	No	No	Yes	No
Final pH	6.0/6.1	5.2/5.4	5.2/5.5	6.8	5.2/5.4
Final Treatment	Petronate	Petronate	TRO	None	Petronate

(1) Modified K-1852. For related formulas, see K-1132, K-2085, K-2024.

(2) K-2152.

(3) AY-578-D and BY-578-D which are included in the tabulation are of interest because of their high tinting strength. The former was simply a laboratory product; the latter was made in 12 Building but was never completely standardized. Both were developed as offerings against Imperial's X-1899.

Analytical data on Y-404-D, Y-405-D and certain competitive light yellows are presented in the appendix together with selected electron microscope photographs.

Consistency, Reactivity and Texture:

While few, if any, serious complaints regarding consistency or reactivity have been received, our Light Yellows (current 12 Building material) are, as indicated above, somewhat on the defensive compared to competitive products of this type. In the case of batch products showing the best color, strength and lightfastness (Y-819-D type), the deficiencies are even more serious. Considerable work has, therefore, been carried out directed towards developing products more satisfactory in rheological properties and in establishing variables by which they can be controlled.

Reducing the amount of alum used gives lower reactivity but there is a corresponding deterioration in color properties and lightfastness. While some reduction may be possible, it is questionable if the amount can be reduced below that now used in Y-405-D.

The introduction of phosphate into the formulation likewise gives an improvement but again with decreased lightfastness and usually with an increase in ink drying time. It has been found that the level of lightfastness varies widely with the point at which the phosphate is added, the best procedure being to add the major portion of soda ash (precipitation of hydrous oxides and pH adjustment) prior to adding the phosphate. When done in this way, the deterioration in lightfastness is held at a minimum. Attempts to add phosphate with the alum or immediately after the alum and "L" Liquor have given poor results. Apparently satisfactory results were obtained in the continuous unit by adding the soda ash at the inlet of the last vat of the cascade and introducing the phosphate on the opposite side of the same vat. The phosphate was added in a pre-determined amount and the soda ash flow adjusted to give the desired pH. Pyrophosphate appears less harmful to lightfastness than orthophosphate and has been used in most of the plant studies. However, products of good quality have been made in the laboratory with orthophosphate.

Previous work on Light Yellows (Reference KN-44-29) has shown certain advantages in the "second lead" type of formula, i.e. adding lead nitrate after the alum/L Liquor before final precipitation and adjustment with soda ash. Apparently, this has little or no effect on reactivity but when phosphate is also present, it appears at least partially to remedy the slow ink drying resulting from the presence of the latter.

Consistency is affected by the pH to which the slurry is finally adjusted. A low pH gives lower initial consistency but there are indications that products so made tend to body more rapidly in reactive vehicle systems than those which are made at a higher pH. The low pH products are also slower in ink drying.

A limited amount of work has been done with miscellaneous materials added to the slurry or dry blended in the pulveriser in the hope that better ink consistency or lower reactivity might be attained. No important leads were developed. (Reference NB 1145).

It is known that our present Y-405-D is inferior to the previous standard in texture. Apparently, the deficiency is not connected with drying conditions since plant (12 Building) grind samples were substantially equal to vat controls dried at 140° in the laboratory. Tests on laboratory samples containing various amounts of incompletely developed material indicated the latter to be an important possible source of wedge grit.

Color Stability and Its Evaluation:

In view of our limited background on "color stability", considerable time was spent in a study of the phenomenon and in attempts to develop a satisfactory procedure for evaluating it. The term refers to resistance to "after-darkening" in certain vehicles, that is a change in color of masstone exhibits during drying.

Lithographic varnish inks pigmented with Light Chrome Yellow apparently darken to some degree even if no added drier is present and differences between pigments may show up in simple systems of this type. In general, however, they are rather small and possibly insignificant. When driers are added, the ink shows an immediate change in color and the change normally becomes more marked as the exhibit dries. The character and degree of change vary with the type of drier as shown by the following readings of Y-405-D inks containing drier against a control ink without drier.

Type of Drier	Wet	Dry
Lead	vs grn	vvs dark, vs red
Manganese	vvs red	vs dark, s red
Cobalt	vs/s dark, vs grn	dark

With lead, the after darkening was only slight; with manganese and especially with cobalt it is quite evident. The change, which incidentally takes place throughout the film, is roughly proportional to the amount of drier present. The darkening on drying may, of course, be affected not only by the drier but also by the pigment used, the varnish or other components of the system. For example, an ink formulated to give slow drying will ordinarily darken less than one which dries rapidly. The relation between any two pigments may be quite different in different vehicles and the exact comparison will depend on the age of the exhibit.

The color change can be accelerated by a mild baking, e.g. overnight at 140°F. The change in color of enamels, floor coverings, etc. on baking is probably basically the same phenomenon as the after darkening of inks.

The mechanism of the change is not known but the dull greenish color suggests that it involves a reduction of hexavalent chromium to trivalent chromium. Cobalt is known to be a "surface drier" and its catalytic effect is, therefore, presumably on oxidation of the vehicle by air or lead chromate rather than on polymerization. According to this hypothesis, the tendency to darken should be reduced by variables giving slow drying or by coating the pigment surface (e.g. hydrous oxide treatment) so that the area available for reaction with the vehicle is less. Products of relatively large particle size (decreased area in contact with the vehicle) should be more stable than those of extremely small particle size. This seems generally true. Of our various standards

which have been examined, Y-202-D (very small particle size) was the worst, Y-488-D and Y-518-D (because of effective surface coating?) showed only slight darkening. Y-469-DS and Y-500-D were worse than Y-469-D. Experimental samples containing undeveloped material have invariably shown bad darkening. The color change occurs in the dark as well as in the light; in the latter case, the total color change is of course greater.

The phenomenon is difficult to evaluate quantitatively. The most satisfactory procedure comprises rubbing out in a suitable vehicle, drawing down on metal and "baking" a portion of the exhibit at ca. 140°F overnight. A comparison of wet and dry readings furnishes a measure of the darkening. Various vehicles have been used; the most satisfactory was a testing varnish containing cobalt drier, UV-1-A obtained from outside the Company. The change also shows up clearly on the masked portions of exposed Fadeometer exhibits, presumably as a result of the mild baking (110°F) during exposure.

Attempts to measure the change spectrophotometrically gave poor reproducibility due to a combination of several factors, especially the rapid change in color which occurred shortly after preparing the exhibit.

Miscellaneous Variable Studies:

The laboratory study of variables was based for the most part on a batch process similar to the current (continuous) Y-405-D process. It involved a dump strike of a chromate/sulfate/salt solution into lead nitrate, the two solutions being adjusted to give a slurry pH of about 4.7. Development was then effected by heating in 15 minutes to 180° and holding for 10 minutes at this temperature. The hydrous oxide treatment employed the quantities of alum and L Liquor (17 and 8 lbs./mole, respectively) used in the standard formula followed by enough soda ash to give a pH of 5.7. In a number of series pyrophosphate or ortho phosphate was used in order to obtain better rheological properties (See section above on Consistency, etc.).

The material given by the above procedure is usually lighter (sl.lt./lt.) in masstone, better in lightfastness and considerably better in color stability than standard Y-405-D when evaluated in UV-1-A vehicle. A typical sample tested against Calco's 40-3615 (C-61301) was vs/s.red, s.int. in initial color, light, sl.red after baking at 140°F and sl.better in lightfastness.

The superiority of the laboratory material compared with plant production is apparently chiefly the result of more complete development. In fact, it was possible to approximate the color, lightfastness and stability of standard Y-405-D fairly closely by removing ca. 25% of the slurry after the strike, holding it at room temperature and adding it to the main portion after completion of the heat development. The importance of proper development in respect to color stability has been indicated in other experiments, variables known to favor development giving better stability.

The results of miscellaneous experiments are presented below. They are concerned with laboratory studies for the most part but plant work is included when relevant. In much of the early work, the formula outlined above, involving batch (and, therefore, presumably complete) development, was used and as a result the effect of certain variables on stability may have been masked. In the more recent experiments, the modified technique was used, that is, holding out a portion of slurry during development. The 12 Building experimental runs were based for the most part on procedures involving the use of phosphate.

1. The effect of certain anionic and cationic impurities in the raw materials was investigated.

In general, crystal lead nitrate was used. This usually gives slightly better quality than plant liquor although some samples of the latter have been satisfactory. Metallic impurities (copper, iron, bismuth) in small amounts ($> 0.005\%$) as contaminants of the lead nitrate have an adverse effect on color but little effect on color stability. Spectrographic tests showed no apparently significant difference between our standards, current manufacture or the Calco yellows except that the last mentioned contained magnesium in somewhat more than trace amounts. (See Appendix).

Nitrite in amounts greater than ca. 1.0% ($Pb(NO_2)_2$ on $Pb(NO_3)_2$) has a decidedly adverse effect on the color (chiefly initial color) of laboratory batches. It can be eliminated readily by adding sulfamic acid to the alum solution. Urea was less effective in a single laboratory trial. In the semi-works and plant (one experiment each, continuous procedure), sulfamic acid gave no improvement.

A single laboratory series indicated vanadium (a possible contaminant of chromate liquor) to have little effect on color properties.

2. The current practice of striking at a relatively low temperature (80°F) gives better results (color, lightfastness) than striking at an elevated temperature even though the total development time and final development temperature are the same. It is possible that an even lower strike temperature might be desirable; the available evidence has not given a definite answer to this question.

3. The temperature of development has a marked effect. Products struck at 80°F and simply stirred at that temperature or below 150°F are extremely non-uniform and contain a considerable amount of small particle size material, frequently needle-like in character. (See electron microscope photographs in appendix). Dispersed in a vehicle they are dark and green, poor in color stability, poor in lightfastness and poor in texture. As the development temperature is increased (strike temperature constant) redder and more intense products (i.e. Y-405-D type) of better stability and lightfastness are obtained. The improvements in color are accompanied by some loss in strength. The greatest change occurs

between 80°F and ca. 150°F; above this temperature the effect, while present, is less marked. In the laboratory, 180°F has given products of good quality; developing to 200°F gave little or no further improvement. Time of development appears to have the expected effect but it has not been studied in detail. As would be expected, products developed to a low temperature are similar in color and lightfastness to those in which a portion of the slurry has been held out while the remainder is developed to the standard temperature. As indicated above, this latter technique has been used in the laboratory to approximate 12 Building quality more closely than is possible by simply developing to 180°F, the standard plant temperature. (See electron microscope photographs in appendix.)

4. The standard strike procedure involves reacting a relatively acid lead solution with an alkaline chromate/sulfate/salt solution, the resulting slurry having a pH of ca. 4.7. In the laboratory, this was accomplished by adjusting the lead solution to pH 4.1-4.2, if necessary, and the chrome to pH 9.5 and then adding a pre-determined weight of caustic based on experience. This gave good reproducibility (in strike pH) and is believed a more satisfactory procedure than attempting to make any pH adjustment above 9.5. Any desired strike pH can be obtained by varying the quantity of caustic. It is, of course, possible to adjust the lead to a lower pH than 4.1 and compensate by using a more alkaline chrome solution. The pH of the lead can also be increased slightly but care is necessary during adjustment to avoid precipitation of lead hydroxide. Neither of these latter modifications in technique appear to offer any advantage.

5. The slurry pH after the strike should not be less than about 4.5. At pH 4, slightly darker and slightly greener material is obtained. At pH 3.6 the effect is even more marked and the slurry shows definite evidence of flocculation (thickening and high bulk). Electron microscope photographs (see appendix) show the final pigment in this case to comprise material which is more acicular than that obtained at a higher pH. Differences in strike pH between 4.5 and 5.5 have relatively little effect; pigments made at the latter appear very slightly redder. These variations are chiefly in initial color rather than in color stability.

The importance of strike pH apparently lies in its influence on the subsequent development step. A batch struck at low pH finished with normal color if the pH was adjusted to the proper value before heating. Similarly, a batch struck at normal pH showed the characteristic dark masstone of low pH material, if acid was added before development. In the case of batch yellows which are vat washed before treatment (Y-519-D), there are indications that better settling is obtained if the solutions are adjusted to give the pH required for development directly rather than to strike at a low pH and then add soda ash as specified in the current formulas.

6. Only a limited amount of work has been done on variations in chromate/sulfate ratio or in (chromate/sulfate)/lead ratio. In the range investigated, the differenses were rather small. A higher chromate/sulfate ratio gave redder masstone material as did also a reduction in the lead excess. In the latter case, there appeared to be a tendency towards lightness also.

7. A number of experiments were carried out in which sodium fluoride was added to the chrome solution. In yellows struck at a low pH (3.6), the fluoride gave a marked improvement in initial color and lightfastness and some improvement in color stability. At a more normal strike pH (4.6) the effect is much less marked. Electron microscope photographs indicate that the particle shape is altered slightly (wider crystals) and it was, therefore, hoped that sodium fluoride would be effective in accelerating development, especially in 12 Building. Some improvement was obtained but it was rather small.

The action of fluoride appears similar to that of chloride except that a lesser amount is required. As might be predicted, it is more effective when present during the strike than if added later. Sodium bifluoride has been used in a few experiments but offered no obvious advantage in respect to quality. Omitting sodium chloride results in dark dull products of high strength, poor lightfastness and heavy ink consistency. Increases above the amounts normally used are relatively ineffective.

8. The precipitation of hydrous alumina in the slurry has been used for many years as a means for controlling color, light stability and certain working properties of chrome yellows. Subsequently, it was found (U.S. Patent 2,212,917) that a somewhat similar effect could be obtained with hydrous titanium oxide. This had the added advantage of giving less reactive products than those obtained with alum. In the effort to obtain the optimum in lightfastness, combinations of the two treatments were developed involving the use of large amounts of alum and titanyl sulfate (L Liquor). The Y-519-D formula for example employs 32 lbs. of alum and 8 lbs. of L Liquor per mole. In developing our present Y-404-D and Y-405-D formulas (continuous manufacture), it was found necessary to reduce the amount of alum to 17 lbs.

Omitting the alum gave better ink consistency and lower reactivity but lightfastness and stability were worse. Omitting the L Liquor had a similar effect; initial color was not impaired quite so much but stability appeared even worse. Increasing the alum over the standard amount improves color, color stability and lightfastness but results in higher consistency and greater reactivity.

Adding the alum before the L Liquor (standard procedure) appeared to give somewhat better results than the reverse procedure but the results were not clear-cut. Adding a mixed solution of the two ingredients gave as satisfactory results as the standard procedure in a single experiment.

9. Analysis of certain competitive light yellows (see appendix) has indicated them to contain small amounts of phosphate. For this reason and because of the favorable effect on working properties (especially reactivity) which usually results, considerable attention has been given to formulations containing pyrophosphate or orthophosphate. This is discussed in an earlier section of the report.

10. Mention has been made above of the use of a second portion of lead nitrate. In addition to its apparent effect on drying, this also gives some improvement in color stability, the magnitude of the effect depending on the type of formula used and probably on the vehicle system. Interesting results have also been obtained by replacing the lead nitrate with zinc sulfate which, like the lead, is precipitated as phosphate and/or carbonate. This has given an improvement not only in stability but also in lightfastness; reactivity was somewhat greater with zinc sulfate than when lead nitrate was used. Further study of both procedures is recommended.

In view of the magnesium found present in Calco's 40-3615, the use of magnesium sulfate instead of lead or zinc salts was investigated in a preliminary way. In the limited amount of work which was done no obvious difference from the controls was noted.

11. While the effect of alum/L Liquor on lightfastness and other properties is believed to be due largely to a coating effect by the hydrous oxides formed when soda ash is added, the pigment may also be affected by the low pH which prevails prior to the neutralizing step as a result of their use. That this was so was indicated by experiments in which the alum and/or L Liquor was partly neutralized before addition to the yellow slurry, so that the pH during treatment remained above the normal value. The material obtained by this technique was redder and worse in color stability and lightfastness than a control made in the usual way.

Interesting results have been obtained by reversing the above procedure, that is adding sulfuric acid to the alum solution. Since it was believed that the deficiency of Y-405-D in stability was due to incomplete or non-uniform development, part of the slurry was held at room temperature during the heating of the main portion. When acidified alum (15# H_2SO_4/mole Pb) was used, the products were better in color, color stability and lightfastness than a control made with unacidified alum. This has been checked more than once in the laboratory. 12 Building results were inconclusive; it is still under study.

As mentioned above, too low a pH before development is unsatisfactory, large particle, needle-like crystals being formed which are poor in color. However, if the bulk of the slurry has undergone normal development, the acid added with the alum may simply serve to convert the remaining portion to the desired form. Whether it would effect any improvement in an orthodox batch process, when development is substantially complete after the heating step, has not been determined.

12. The importance of the final pH to which the slurry is adjusted before filtration has been mentioned elsewhere in connection with ink properties. It likewise has a marked effect on color, lightfastness and stability. The desirability of a low pH (ca.5) is especially evident in continuous manufacture where there are also indications that the adverse effect of low pH on ink drying is less serious. In the laboratory there is an improvement in drying in going from 5.0 to 5.7 without significant changes in other properties.

13. Rather large differences have been observed in certain 12 Building runs between samples taken at different points, viz. 59 Vat (final pH adjustment, sample washed in laboratory), 71 Vat (final repulp, Petronate treated) and grind. The reason for these differences has not been established definitely. In the laboratory (completely developed material), the presence or absence of Petronate has very little effect on color. Raising the drying temperature from 140° to 180° gave a vs darker masstone; this difference is less than the differences referred to above which were obtained in the plant.

14. A limited amount of work on strike concentration has been carried out in the laboratory, semi-works and plant but the results have been inconclusive. Some further attention to this variable is probably justified.

REFERENCES:

Notebook 1145 - This covers the latter part of the work on AY-578, K-2132 (reported in KN-45-29) and the early stages of work on the present problem. The specific items include:

Work on bake resistance.
Evaluation of competitive products.
Livering - chiefly Y-519-D type.
General work on color stability and its evaluation.
Development of laboratory formula - Y-405-D.
Variable studies on above.

Notebook 1330 - The entire contents of this notebook is concerned with work on this problem. It includes:

Variable studies on laboratory formula.
Effect of raw material quality.
Effect of undeveloped pigment on texture.
Evaluation of 12 Bldg. and 10 Bldg. runs.

Notebook 1339 - Expts. 1 to 8. This is a continuation of NB 1330. The most important new item covered is the effect of acid in the alum solution discussed in the present report.

Relevant research reports include,-

KN-45-10 Semi-works Development of a Continuous process for the manufacture of Excelsior Chrome Yellow.

KN-45-29 Light Chrome Yellow K-2132 Development of a Strong Canary Yellow - Laboratory Work.

KN-47-3 Chrome Yellows: Rheological properties of continuously manufactured products and development of testing methods.

KN-47-37 - Laboratory study of the Application of Continuous Flow Tubular Reactor to the Manufacture of Medium Yellow.

ANALYTICAL DATA:

Competitive Light Yellows. Report of 5-15-49.

Current Standards. Information from Plant Analytical Laboratory, not yet reported.

Spectrographic Analysis Letter: Dunlop - Denslow 1-20-49.

ANALYTICAL DATA

	Y-404-D 10115	40-3355 Calco (1)	Y-405-D 10116	40-3615 Calco (2)	X-1899 Imperial(3)
PbO	66.0	67.5	67.3	65.9	66.2
CrO_3	18.9	19.7	20.4	21.5	21.5
SO_3	7.3	7.6	5.4	5.5	5.3
CO_2	0.3	Nil	0.8	Nil	- (5)
TiO_2	2.4(4)	1.1	2.8(4)	1.7	0.5
Al_2O_3	1.5(4)	1.3	1.5(4)	1.0	1.0
P_2O_5	Nil	0.8	Nil	0.9	2.2
Water Soluble	0.6	- (5)	0.5	- (5)	- (5)
Moisture	0.8	- (5)	0.7	- (5)	2.6
	97.8	98.0(6)	99.4	96.5(6)	99.3

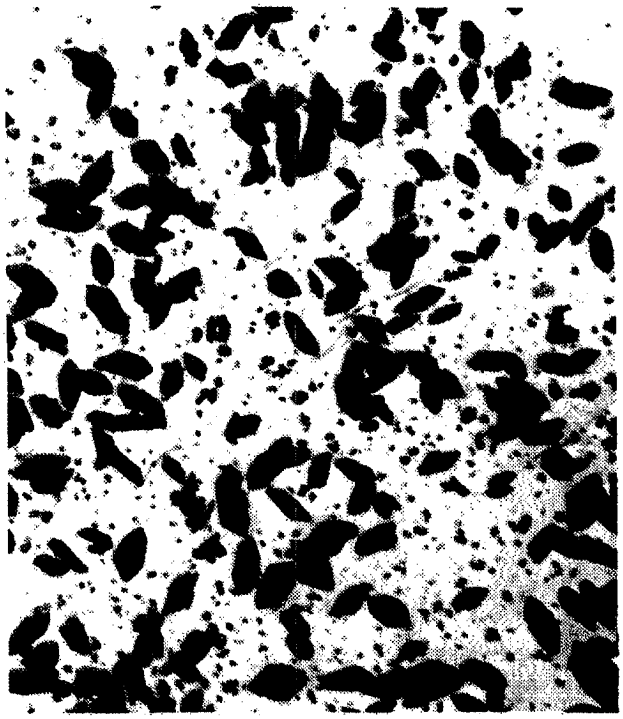
(1) C#51391
(2) C#51301
(3) C#47600 (from Report KN-45-29)
(4) Calculated value
(5) Not determined
(6) C#51391 was found to contain 0.2% Magnesium as MgO. C#51301 was not analyzed for this metal.

APPENDIX

Spectrographic Data

	Major	Minor	Trace
Y-404-D (10115)	Pb Cr	Ti Al	Si Na Mg K Ca Cu
Y-405-D (10116)	Pb Cr	Ti Al	Si Mg Na Ca Cu
Y-577-D (11691)	Pb Cr	Ti Al	Si Mg Na Ca Cu Ag
40-3615 (C-51301)	Pb Cr	Ti Al	Mg Si Ca Na Cu

(Other samples run at the same time comprising plant and laboratory material showed substantially the same qualitative composition. See Letter:Dunlop - Denslow, 1-20-49).



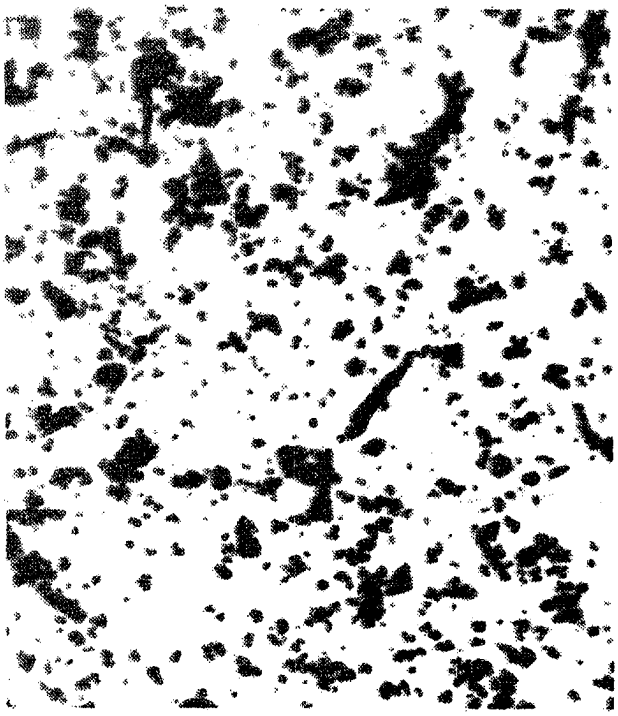
Std. Y-405-D (Plate 781)
Lot 10116

Y-519-D (Plate 1004)
Lot 57928

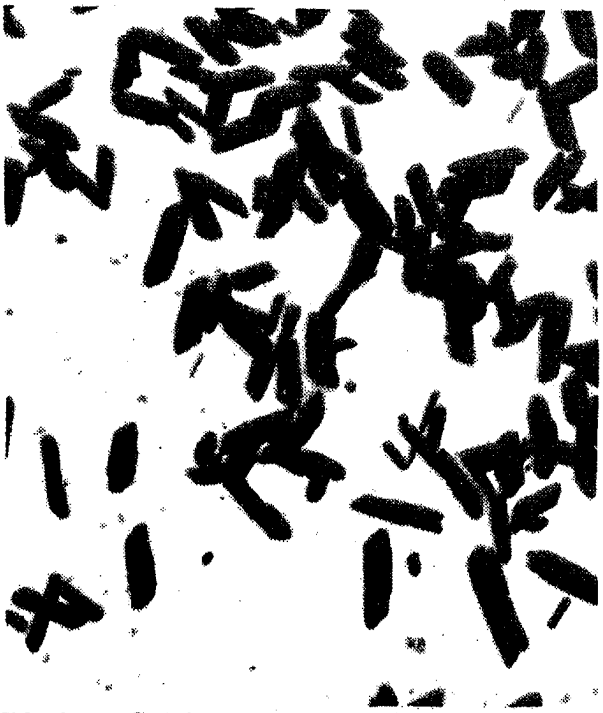
Calco 40-3615 (Plate 782)
C#51301

Imperial X-1899 (Plate 500)
C#47450

FIG. 1:- Miscellaneous Light Chrome Yellows.
Magnification approx. 16000 x

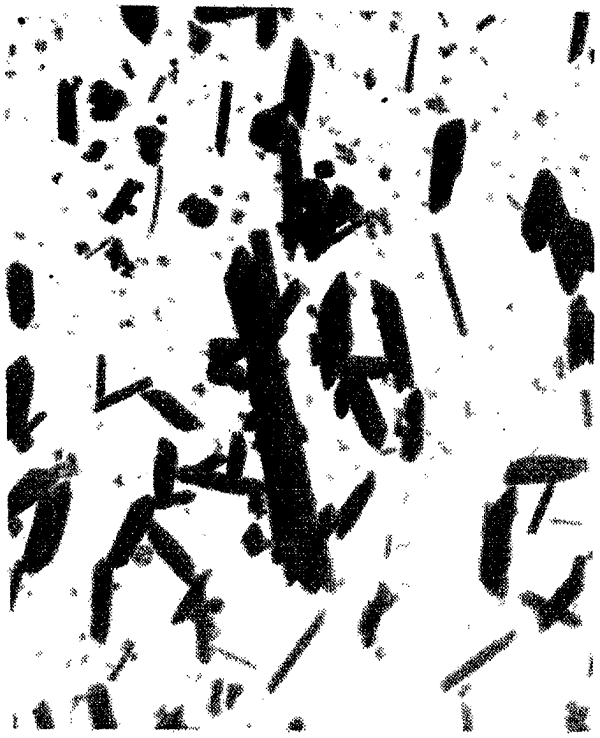


Immediately after strike.
No heat development. (Plate 923)



Finished Pigment.
Standard (180°F) precipitation and development conditions.
(Plate 791)

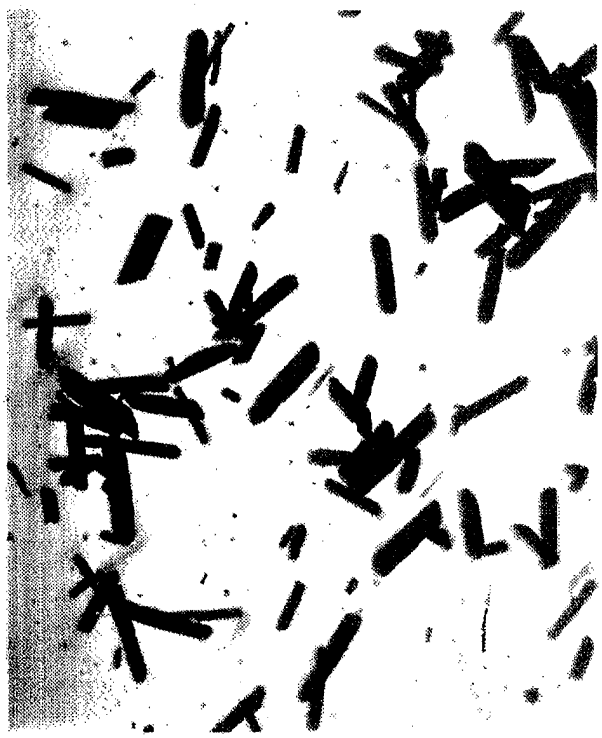
Low temperature (140°F) Development.
(Plate 1011)



25% of slurry held out at room temperature until after 180°F. development of main portion was completed. (Plate 1012)

FIG. 2:- Lab. Light Yellows (Y-405-D type).
Effect of heat development. Magnification approx. 16000 x

Effect of striking and developing at low (3.5) pH. Compare with Plate 791 (Fig. 2).
(Plate 821)



Effect of sodium fluoride on material struck and developed at low (3.5) pH.
(Plate 820)

FIG. 3:- Laboratory Light Yellows (Y-405-D type).
Magnification approx. 16000 x