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E. I. DU PONT DE NEMOURS & COMPANY
PIGMENTS DEPARTMENT
256 VANDERPOOL STREET
NEWARK, NEW JERSEY

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# NEWARK PLANT
## CHEMICAL DIVISION - COLORS

## NON REACTIVE MEDIUM YELLOW Y-662-D FOR USE IN I.P.I'S "K6" AND "VAPOSET" VEHICLES

Period Covered May 1, 1952 - September 15, 1952

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

FINAL REPORT

TITLE: NON REACTIVE MEDIUM YELLOW Y-662-D FOR USE IN I.P.I.'s "K6" and "Vaposet" Vehicles

PERIOD COVERED: May 1, 1952 - September 15, 1952

CHARGE: 1101-11, 12 -3

SUBMITTED BY: Julius Jackson JULIUS JACKSON DATE SUBMITTED: 12/1/52

APPROVED BY: B. H. PERKINS 12/29/52

SUMMARY AND CONCLUSIONS:

1 - Modification of Y-469-D "F" process has resulted in the production of a non-reactive type medium yellow similar to Imperial's X-1810 or Imperial's S-3268.

2 - These modifications involved the following changes expressed in terms of lbs/mol of lead nitrate struck.
(a) Reduction in use of Aluminum sulfate (N15) from 48 lbs. to 16 lbs.
(b) Reduction in use of titanyl sulfate (N390) from 6.8 lbs. to 3.4 lbs.
(c) Reduction in use of "2nd" lead nitrate from 34.73 lbs. to 30.5 lbs.
(d) Ommission of all petronate (N390)
(e) Use of tetra sodium pyrophosphate (N479) at the rate of 6 lbs./mol lead nitrate struck.

3 - A degradation in lightfastness versus Y-469-D has resulted because of these changes. This is not serious since all competitives of the non-reactive type are markedly inferior to Y-469-D in lightfastness.

4 - The product produced and standardized as Y-662-D lot 765 is superior in lightfastness to Y-661-D or the competitive pigments.

5 - Investigations have revealed that zirconia treatment of Y-469-D processes will result in products of improved lightfastness.

6-- The use of recirculation of 68 centrifuge fluid discharge to 66 and 67 centrifuge feed lines offers definite possibilities for improved washing of #12 Building products.

PATENT STATUS:

Nothing of a patentable nature was developed during these studies.

REFERENCES:

N.B. 1358 - 1437
DSN 52-8, 52-6
KN 52-18
KN 52-20

I. DISCUSSION AND BACKGROUND:

The introduction of new vehicle systems in the paint and printing ink fields imposes frequent adjustments in the chemical and

and physical characteristics of chrome yellow pigments. This is not a new situation in the pigment field but is a chronic condition that must be faced as progress is made in the consuming industries. Accordingly, constant revisions and adjustments in our pigment formulation must be made in order to manufacture products suitable for use in these fields.

During the years 1949-1950 it became apparent that our medium yellows Y-469-D or Y-661-D were inadequate to meet the competition of Imperial's X-1810 and S-3268 in rheological characteristics in various vehicles.

Complaints were received from the Socony Paint Products Company concerning Y-469-D reactivity in a phenolic modified alkyd system. The International Printing Ink Division (IPI) of the Interchemical Corporation stated that Y-469-D, Y-434-D and Y-661-D were unsatisfactory in reactivity for use in their "K6" lithographic varnish and "Vaposet" vehicle systems. Minnesota Mining and Manufactoring Co. reported that Y-469-D and Y-661-D were excessively reactive in their traffic paint systems.

In May of 1952, the problem of developing a medium yellow satisfactory for use in the IPI "Vaposet" systems was assigned to the writer. (For a review of this subject see American Ink Maker 27 No. 4 33 April 1949). It is obvious that an economic solution for this problem must involve an evaluation of the working properties of any experimental pigments in all of the fields involved, since continuous manufacture of many different specials would not be a practical solution for the reactivity problem. The goal was set up of a pigment satisfactory in all vehicles mentioned but with emphasis placed on IPI's requirements.

## 2. EXPERIMENTAL:

### A. Evaluation of Standard and Competitive Samples

Investigation revealed that IPI was using a special medium yellow coded S-3268 for "Vaposet" use. Our examination of this product in the customers YV-1304-H vehicle see TF (2403-2) gave the following results versus our standards and various competitive products, when tested according to TF-2403-2. See table I-Page 3. This test has been modified in accordance to Dr. Larsen's instruction to be ground with a roll setting of 120°F in order to minimize effects of moisture variation in the atmosphere. Since the vehicle is sensitive to water, it is imperative to control that factor as rigidly as possible. Considerable variations in values for "good" pigments result in duplicate tests. High viscosity pigments always give no flow on the 45° inclined plane. The test has been simplified in this work to consist of a flow reading alone, instead of the usual body, length and flow measurements. No evidence of "reactivity" in the usual sense, i.e., increase in viscosity with time, has been noted. Usually viscosity drops (flow values increase) with time.

TABLE I

COMPARISONS OF VAPOSET FLOW OF EXISTING STANDARDS

| | | Vaposet Modified TF 2403-2 | | K-6 TF 2405-1 | | Socony Paint Products | |
|---|---|---|---|---|---|---|---|
| Code | C# | Initial Flow | After 7 days at 120°F | Initial KU | After 7 days at 158°F | Initial | 4 weeks |
| Y-469-D | 658 | 0 | 0 | 58 | Gel | 68 | 98-gelled |
| Y-661 | 95500 | 0 | 0 | 48-57 | 69-73 | 73 | 89 |
| Imp. 3268 | 54709 | 3-12 | 17-21 | 30-43 | 44-50 | 70 | 73-77 |
| Imp.X-1810 | 54336 | 0-1 | 0-1 | 32-43 | 59-66 | 72 | 82 |
| Y-528 | 561 | 0 | 0 | --- | --- | -- | -- |
| Calco 45-4500 | 54238 | 3 | 2 | --- | --- | -- | -- |

Readings shown are typical of repeat tests.

Inspection of the above data and other tests revealed that both Y-469-D and Y-661-D standard, as well as typical products, were unsatisfactory in I.P.I. Vaposet and K-6 tests. In addition, the customer stated that Y-661-D was excessively sensitive to darkening on drying. The latter is known to be associated with the use of cobalt drier in the customer's vehicle.

B. Laboratory Studies

a - Factors Found to Increase Vaposet Flow

1. Decrease in Alum usage 48 mol/16 mol to NB 1358,55,56, 57
   a) effect on K6 Reactivity - beneficial - lower initial consistency also.
   b) effect on Socony Paint Products Reactivity - beneficial.

2. Decrease in titanyl sulfate usage NB 1358,57,60 1437,1, 4
   a) effect on K6 Reactivity - ineffective on reactivity but lowered initial consistency.
   b) effect K6 Socony Paint Products - ineffective on reactivity.

3. Ommission of Petronate NB 1358-55,56
   a) effect on K6 reactivity - harmful in high alum processes
   b) effect on Socony Paint Products Reactivity- probably slightly worse

4. Use of tetra sodium pyrophosphate NB 1358-55,56 1437-1,3,7,11
   a) effect on K6 reactivity - beneficial
   b) " " Socony Paint Products Reactivity- beneficial
   c) effect on Minnesota Mining & Manufacturing Reactivity - beneficial

5. Use of Lustrex 820 NB 1358-62 1437-1,8
   a) effect small-inadequate to justify cost
   b) products do not disperse in Varnish Drier.

6. Sequence of Treatments NB 1437-3,8,12
   a) best sequence alum, "L" liquor, sodium pyrophosphate, 2nd PB
   b) effect on K6 reactivity - nil

C. Factors Found to Decrease Vaposet Flow

1. Treatment with fatty acids 4Gms/mol NB 1358-55
   a) effect on K6 reactivity - worsened

2. Use of Zirconium hydrate NB 1358-57,60,63 1437-1,7
   a) effect on K6 reactivity - beneficial(when

alum replaced with zirconia) similar in behavior to $TiO_2$ treatment. The simultaneous use of zirconium and pyrophosphate was not satisfactory for Vaposet usage. Products could be made exhibiting excellent lightfastness and a low level of reactivity in K-6 using 3.4 gr. $TiO_2$ and 6 gr. $ZrO_2$ per mol in place of the usual $Al_2O_3$,$TiO_2$ treatment. Future study of this treatment, in conjunction with variation in alum (N15), "L" liquor (N390) ratios should be undertaken if the problem of improved lightfastness is reopened.

3. Use of Wetting Agents - Aerosol OT, Dupanol WA, Potassium Daxad NB 1358-62
   a) very little or negative effect.

4. Use of High 2nd lead NB 1437-3, 12
   2) effect on K6 reactivity - sl worse
   higher quantities second lead give products of better lightfastness.

5. Increased Final pH NB 1437-6,8
   As pH increased Flow decreased
   pH 6.0 better than higher pH's

6. Underdevelopment of Yellow NB 1437-9
   As percent underdevelopment increases - viscosity rises

7. Casein Wash of Finished Press cake NB 1437-1,2
   (studied in connection with thixotropy)

8. Nopco 2125 Treatment NB 1437-13,14
   (studied in connection with thixotropy)

9. Effect of Washing on Vaposet Flow DSN-52-8
   Complete absence of washing of #12 Building material results in very small Vaposet flow figures. The standard repulp water flow rate is 20 gallons per minute. Lesser quantities have not been extensively studied in #12 Building.

In addition to the previously outlined studies, the effect of redrying was studied. This was found to be ineffective.

Data illustrating these points are given in Tables on Pages 5A,5B.

#12 BUILDING STUDIES:

1. The first attempt (see 1437-2 DSN 52-8) was to make Y-469-D in #12 Building, testing the alum/$Na_4P_2O_7$ variable. These products coded Y-662-D (6758-6761 inclusive) were made on standard formula except-

| | | Y-662-D |
|---|---|---|
| Condition (1) | Alum used at 8 lbs/mol<br>N-479 used 4 lbs/mol<br>no petronate | lots 6758, 59, 60 |

## TABLE 2 - EFFECT OF VARIABLES ON REACTIVITY AND FLOW



ALUM VARIATIONS

| N.B.# | Alum | Per mol Pb "L" as $TiO_2$ | Per mol Pb $Na_4P_2O_7$ | Petronate | IPI Vaposet Flow - Goal* Imp. 5 to S-3268 12 | K-6 Reactivity** Initial | K-6 Reactivity** Final | K-6 Reactivity** Imperial λ-1810 | Comments |
|---|---|---|---|---|---|---|---|---|---|
| 1358-55A | 48 | 6.8 | 0 | 1% | 0 | 39 | 73 | 43→66 | Note wide variation in duplicate tests |
| 42A | 48 | 6.8 | 0 | 1% | | 47 | 300+ | 34→53 | |
| 51A | 48 | 6.8 | 0 | 0 | 3 | | | | |
| 1437-1-I | 48 | 6.8 | 2½ | 0 | 15, 23 | | | | |
| 1358-55C | 48 | 6.8 | 4 | 1% | 0 | 44 | 77 | 43→66 | |
| 1358-55D | 32 | 6.8 | 0 | 1% | 1 | 41 | 61 | 43→66 | |
| 1358-56A | 32 | 6.8 | 4 | 1% | | 34 | 64 | 32→59 | |
| | | | | | | 29 | 190 | 34→53 | |
| 1358-56G | 32 | 6.8 | 4 | 0 | | 23 | 62 | 32→59 | |
| 1358-55E | 32 | 6.8 | 4 | 1% | 0 | 41 | 70 | 43→66 | |
| 1358-52A | 24 | 6.8 | 0 | 0 | 18 | Abnormally high? | | | |
| 1358-55K | 16 | 6.8 | 0 | 1% | 3 | 37 | 50 | 43→66 | |
| 1437-1-C | 16 | 6.8 | 8 | 0 | 17 | | | | |
| 1437-1-D | 16 | 6.8 | 4 | 0 | 6-1/2 | | | | |
| 1437-1-E | 16 | 6.8 | 0 | 0 | 2 | | | | |
| 1358-62B | 16 | 6.8 | 0 | 1% | 0 | | | | |
| 1358-62A | 16 | 6.8 | 0 | 0 | 2 | | | | |
| 1437-1-B | 12 | 6.8 | 6 | 0 | 10- | | | | |
| 1358-56D | 8 | 6.8 | 4 | 1% | 26 | 27 | 40 | 32→59 | |
| 1358-56H | 8 | 6.8 | 4 | 0 | 50, 19 | 20 | 27 | 32→59 | |
| 1437-1-A | 8 | 6.8 | 4 | 0 | 15, 23 | | | | |
| 1437-1-F | 8 | 6.8 | 0 | 0 | 4 | | | | |
| 1358-57F | 0 | 6.8 | 0 | 1% | 1 | 38 | 52 | 32→75 | |

*Larger numbers better
**Smaller numbers better

TABLE 3 - EFFECT OF VARIABLES ON VAPOSET FLOW

| N.B.# | Alum | Per mol Pb "L" as $TiO_2$ | Per mol Pb $Na_4P_2O_7$ | Petronate | IPI Vaposet Flow - Goal* Imp. S-3268 5 - 12 Units | |
|---|---|---|---|---|---|---|
| **LUSTREX TREATMENTS** | | | | | | |
| 1358-62-A | 32 | 6.8 | 0 | None | 2 | |
| " "-F | 32 | 6.8 | 0 | None | 18 | 3% Lustrex 820 Treatment |
| 1437-1-E | 16 | 6.8 | 0 | None | 2 | |
| " "J | 16 | 6.8 | 0 | None | 4 | 3.3% Lustrex 820 |
| **VARIATION IN "L" LIQUOR (Slurry ppt'd. in 12 Bldg. - Treated in Lab.)** | | | | | | |
| 1437-2-D | 8 | 6.8 | 4 | None | 2 | |
| 1437-2-E | 8 | 0 | 4 | None | 17-1/2 | |
| | | | | $TiO_2$ Studies | Initial Flow | Lightfastness vs. X-1810 |
| 1437-4-H | 24 | -- | 4 | None | 28 | |
| " "-A | 16 | 6.8 | 8 | None | 61 | better |
| " "-B | 16 | 3.4 | 8 | None | 77 | m better-best of series |
| -C | 16 | -- | 8 | None | 80 | sl better |
| -G | 16 | -- | 4 | None | 75 | ± |
| -D | 8 | 6.8 | 4 | None | 52 | sl better |
| -E | 8 | 3.4 | 4 | None | 72 | better-next best |
| -F | 8 | -- | 4 | None | 85 | ± |
| Y-661-D | | | | | 0 | |
| **CONCLUSION: Increased Flow with decrease in "L" Liquor** | | | | | | |
| | **Position of addition of N-479** | | | | | Effect of higher 2nd Pb $Na_3PO_4$- ltftness vs X-1810 |
| 1437-3-A | Alum | "L" | 2Pb | N-479 | 65 | sl better |
| -B | N-479 | Alum | "L" | 2Pb | 43 | - |
| -C | Alum | "L" | N-479 | 2Pb | 76 | v sl better |
| -D | Alum | N-479 | "L" | 2Pb | 30 | v sl better |
| -E | Alum | "L" | High 2nd Pb | N-479 | 65 | m better |
| -F | Alum | "L" | " " | $Na_3PO_4$ | 61 | vv sl better |

Condition) Alum used at 16 lbs./mol Lots 6761
2. ) N-479 8 lbs./mol
no petronate

Tinctorial comparisons of these lots follow:

| | | | | |
|---|---|---|---|---|
| Y-662-D, 6758 vs Y-469-D-658 | s.lt-s.grn.s.dull | 98 s grn. | much worse |
| 59 | s.dk- " " | = vvs red | " " |
| 60 | s.dk- " " | = vvs red | " " |
| 61 | vs.dk- " " | = vvs red | " " |
| 6758 vs Y-661-D | s.lt-s.grn | = vvs red | vs-s. better |
| 59 95500 | s.lt-s.grn | 97 s.grn. | s.better |
| 60 | vs.lt-s.grn.s.dull | 97 s.gmn. | s.better |
| 61 | s.lt-vs.grn. | 97 s.grn. | s. worse |
| 6758 vs Imperial | grn.-s.dull | 98 vs.grn | vs.better |
| 59 X-1810 | vs.dk-grn.s.dull | 98 vs.grn | s. better |
| 60 54336 | sl.dk-s.grn.s.dull | 98 vs.grn. | s.better |
| 61 | vs.dk-s.grn.vs " | 98 vs.grn. | vvs worse |

REACTIVITY TESTS

| | K-6 | | IPI VAPOSET |
|---|---|---|---|
| 6758 | 34 | 40 | Initial Flow 0 |
| 59 | 35 | 45 | 1 |
| 60 | 34 | 54 | 1 |
| 61 | 31 | 42 | 1 |
| Y-661-D 95500 | 42 | 106 | 1 |
| Imperial S-3268 | 26 | 39 | 5 |

Final Vat Control cond.(1) 8 alum 4 N479
Lab. washed and dried 7

FVC cond.(2) 16 alum 8 N479 21
Lab. washed and dried Y-661-D 16 alum 8 N479 0

These studies show the beneficial effects of the alum/pyrophosphate variable in K6 reactivity. Vaposet flow was inadequate to meet competition except in the case of the final vat control from cond "2" which was excellent.

Analysis of Lot 6761 showed 1% $PO_4^{\equiv}$ content while final vat control from cond "2" showed 1.62% $PO_4^{\equiv}$. This probably accounts for the difference in behavior of the plant product and the final vat control, and indicated the necessity for longer experimental runs to permit attainment of equilibrium conditions in the plant.

It is suggested that the Quality Control Group might undertake a further trial of this type process which was made without recirculation of centrifuge effluents and which did not show excessive bleed. The attainment of good Vaposet reactivity, which seems possible judging from results of the evaluation of the final vat control from condition 2, might be a desirable simplification of the processes ultimately used for standardization.

2. The second plant run, Y-662-D, A-6988, 89, 92, was based on a modification of the previous procedure in corporating the effects of reduced "L" liquor and 2nd lead studies and differed from that of Y-469-D in the following respects:

| | Standard | Experimental |
|---|---|---|
| N-15 (alum) | 48# | 16# (per mol lead nitrate struck) |
| N-390 ($TiO_2$) | 6.8 | 3.4 " " " " " |
| N-490 (Petronate) | 1% | none " " " " " |
| N-479 ($Na_4P_2O_7$) | none | 8# " " " " " |
| "Second Lead" | 33 | 26 " " " " " |
| Tank 58 used | no | yes (for better aging) |

Some difficulty was experienced during the run due to peptization of the color during washing and resultant centrifuge bleed. Raising repulp temperatures to ±140°F and reducing the amount of N-479 to ±6.3 lbs./mol eliminated this problem.

Comparisons with Y-661-D and Imperial's S-3268 are as follows:

(a) Rubout in Varnish Drier

| | Wet MT | Dry MT | L/F |
|---|---|---|---|
| Y-662-D-6988 vs Y-661-D-95500 | lt-grn | s lt-s grn | vs worse |
| 6989 " | lt-grn | sl lt-s grn | ± equal |
| 6992 " | s lt-grn | vs dk-s grn s dull | vs worse |

| | Wet MT | Dry MT | Str. | Tint | L/F |
|---|---|---|---|---|---|
| 6988 vs Imp. S-3268 | lt-grn | s grn-s dull | 97 | s grn | ±equal |
| 6989 " | s lt-s gr | " " | | | tr better |
| 6992 " | vs grn-s dull | " " | 95 | " " | tr ws |

Rubouts show that color undergoes slightly greater change in drying or baking <u>in this vehicle</u> than does Y-661-D or S-3268.

(b) Rubout in CPV-8677 (I.P.I. Robout Vehicle)

| | Wet MT | Dry MT |
|---|---|---|
| 6988 vs Y-661-D-95500 | v lt-grn-int | v lt-red-v int |
| 6989 " | v lt-grn-int | v lt-red-v int |
| 6992 " | v lt-grn-int | v lt-red-v int |

(b) Rubout in CPV-8677 (cont.)

| | Wet MT | Dry MT |
|---|---|---|
| Y-662-D-6988 vs Imp. S-3268 | lt-grn-int | s lt-red-int |
| 6989 " | lt-grn-int | s lt-red-int |
| 6992 " | lt-grn-int | s lt-red-int |

These rubouts in this vehicle show that the experimental lots are light, red and intense after drying in the customer's vehicle and appear to be more color stable on baking at 100°F.

Reactivity - K-6 - MacMichael Readings

| | Initial | 3 days 158°F | Increase | |
|---|---|---|---|---|
| Y-661-D-95500 | 48 | 69 | 21 | |
| Y-661-D A-7007 (current prod.) | 33 | 98 | 65 | more |
| Y-662-D-6988 | 30 | 65 | 35 | made with / |
| 6989 | 32 | 54 | 32 | N-479 than |
| 6992 | 32 | 78 | 46 | (6992 |
| Imp.S-3268 C#54709 | 30 | 44 | 14 | |
| " X-1810 C#54336 | 35 | 61 | 26 | |

The products are approximately equal in reactivity to Imperial's X-1810 but inferior in this test to Imperial's S-3268, the special for Vaposet.

Reactivity - Socony Paint Products Vehicle

| | Initial KU 7/15 | KU after 3 days 7/18 | KU after 14 days 7/29 | KU after 14 days 4 weeks |
|---|---|---|---|---|
| Y-661-D-95500 | 71 | 79 | 91 | 99 |
| Y-662-D-6988 | 68 | 75 | 86 | 94 |
| Y-662-D-6989 | 69 | 75 | 85 | 94 |
| Y-662-D-6992 | 69 | 78 | 101 | 140+ |
| Imp.X-1810 C#54336 | 70 | 75 | 81 | 87 |
| Y-469-D-658 | 71 | 87 | 121 | 140 |

Vaposet I.P.I. - YV-1304-H

| | Initial Body | Length | Flow (initial) | Flow (7 days/120°) |
|---|---|---|---|---|
| Y-661-D-95500 | s heavy | s short | 0 | 0 |
| Imp.S-3268 C#54707 | * | * | 8 | 18 |
| Y-662-6988 | vs heavy | " | 6 | 15 |
| Y-662-D-6989 | vvs heavy | " | 5 | 15 |
| Y-662-D-6992 | " | " | 11 | 13 |

These results indicated that material of the type of lot 6992 was satisfactory for use in IPI vehicles, but unsatisfactory in the Socony Paint Products Phenolic-Alkyd. A sample was sent to

IPI for test.

3. The next run Y-662-D-7386 was designed to overcome the Socony Paint Products reactivity exhibited by Y-662-6992. Sister lots from the (Y-662-D, 6988, 89) but made with higher N-479 content than the Y-662-D-6992 were satisfactory in this vehicle, but less desirable in the Vaposet vehicle. An attempt was made in this #12 Building run, to obtain material satisfactory in all three uses. Considerable bleed occurred during portion of the run which used an alum/$Na_4P_2O_7$ ratio of 16/6.3 per mol lead struck. The bleeding does not occur from the dewatering step, but only from the centrifuge used after repulping of the dewatered color. During considerable periods of the run, no bleeding occurred. Control of treating agent flow was not good. It was also noticed that the load on the centrifuge was intermittent (as judged by filtrate effluent and ammeter reading variations). These variations cannot be explained unless the pumps from the repulp vats to centrifuge lead tank were partially air bound. These surges probably caused a loss of color from the centrifuge, since the press cake is thixotropic.

Attempts were made to decrease bleeding of centrifuge by:

1. Studying repulp temperatures and volumes of water.
2. Alteration of point of entry of N-479 (increased time interval between N-390 and N-479 addition points.)
3. Variation in amount of N-479 used.
4. Ommission of T-58 in connection with point 2 above.

Complete control of bleeding was not obtained during this run which lasted only 11 hours. Longer time intervals between N-479 and N-390 addition points seem to improve both color properties and Vaposet flow as judged by evaluation of Fin Drum samples and plant grinds.

The treating stages of medium and light yellows were studied in the following runs to determine optimum methods of handling alum and "L" liquor so as to get maximum lightfastness and color stability with minimum use of agent.

4. In the interim period, while the results of IPI's evaluation of Y-662-D-6992 were awaited, work was concentrated on the known deficiencies of the product which involved:

1) A tendency to bleed while centrifuging the washed material.
2) Excessive reactivity in Socony Paint products vehicle.

These problems are inter-related since the known variable for correcting excessive reactivity (use of increased quantities of sodium pyrophosphate) is also the cause of thixotropy of precipitates and centrifuge bleed.

Laboratory studies revealed that thixotropy of the press cake can be eliminated by means of a treating agent Nopco 2125 (non ionic agent) and also by means of washing with dilute solutions of

electrolytes such as aluminum sulfate or magnesium chloride. Unfortunately the use of electrolytes seem to have detrimental effects upon reactivity and does not offer a practical solution to the problem. The use of Nopco 2125 in amounts from 1-3% does not appear ~~extensively~~ harmful to Vaposet viscosity. Reactivity in K-6 has not been evaluated.

A plant trial (Y-662-D - 7658) of the concept of recirculating the discharge of 68 centrifuge (wash centrifuge) back into the feed line to 66 and 67 centrifuges (dewatering centrifuges) has led to an apparently satisfactory solution to the bleeding problem. A tendency to the formation of a very high solids discharge from 68 centrifuge was noted. This results in a higher load on No. 68 centrifuge motor, and a tendency to hard stick formation on the fin drum. The dryer bed is only lightly loaded because of the high density of the solids. The process appears to be operable. Lower speeds on 68 centrifuge may result in a softer discharge cake. The products obtained as a result of this run which differs from lot 6992 in (1) addition point for use of sodium pyrophosphate (greater time period between $TiOSO_4$ entry point and pyrophosphate entry point); (2) use of recirculation of 68 centrifuge effluent have shown definite advantages in quality. Improvements have been made in -

1) resistance to darkening
2) lightfastness
3) Initial Vaposet Flow
4) Socony Paint Products Reactivity

SOCONY PAINT PRODUCTS REACTIVITY

| | INITIAL | 3 DAYS | 1 WEEK | 2 WEEKS | 4 WEEKS |
|---|---|---|---|---|---|
| Y-662-D A-6992 (not recirculated) | 68 | 77 | 80 | 86 | 94 |
| Y-662-D A-7665 (recirculated) | 65 | 70 | 72 | 75 | 77 |
| Y-662-D A-7667 (recirculated) | 67 | 69 | 71 | 73 | 76 |
| Imperial X-1810 C#54336 | 72 | 73 | 74 | 78 | 82 |
| " S-3268 C#54709 (Vaposet Special) | 70 | 71 | 72 | 75 | 77 |

Readings in KU by Stormer Viscosimeter.

K-6 REACTIVITY

| | INITIAL | 72 HRS/158°F |
|---|---|---|
| Imperial S-3268 C#54709 | 31 | 47 |
| Y-662-6992 | 30 | 86 |
| Y-662-D-7664 | 30 | 62 |
| 7665 | 29 | 45 |
| 7667 | 26 | 38 |

VAPOSET FLOW

| | INITIAL | 7 DAYS/120°F |
|---|---|---|
| Imp. 3268 - C#54709 | 10 | 21 |
| Y-662-6992 | 5 | 16 |
| Y-662-7664 | 15 | 16 |
| 7665 | 18 | 31 |
| 7667 | 22 | 40 |

A 15,000 lb. blend of Y-662-D production (95662-6) was then prepared for shipment to IPI. This material was a blend of the type made in the run (Y-662-D, A-7658) (recirculation of 68 centrifuge effluent to prevent bleed loss) and earlier materials to make up to the 15,000 lbs. required.

As expected, the blend was not fully equal in quality to the best material produced in Y-662-D-7658 run but was superior to Y-662-D, A-6992, and competition in rheological and tinctorial properties.

A sample of the material made in the run Y-662-D-7658 was also submitted to Dr. Larsen (IPI) for his comments. Our evaluation showed it to be superior to lot 6992 ( first sample submitted) in all respects. To date no comment has been received in the latter samples, and favorable comment was received on the 15,000 lb. mix.

5) In view of the good results from the Y-662-D-7658 run, it was decided to standardize on this type material. A final standardization run Y-662-D lot 7961 was made 10/13/52 duplicating Y-662-D 7658. The run did not show any particular difficulty and a mix for standard was chosen from the production of this run and run 7658.

Y-662-D SD-765 is comprised of Y-662-D lots 7665,6,7,9 7964,7, 7970,3,7, 7980, 7982,4 and 6.

Test data on this mix can be seen in the attached letter.

In addition to these studies work was also conducted on the Vaposet characteristics of Y-434-D. This code was found to respond to similar variables, in particular to the use of tetrasodium pyrophosphate, with or without a cut in alum. This work will be reported by J. E. Bishop in KN-52-20 and DSN-52-6. See also NB 1358-1437.

The standard formula for this new process coded Y-662-D will be issued by J. E. Bishop.

6) Recommendations for future study - The attached letter covers the recommendations of writer to those concerned with #12 Building operations.

J. E. Booge, Wilmington
D. B. Killian, "
F. E. Bolton, "
M. J. McLain, "
J. N. Tully, Newark
A. Siegel, "
B. H. Perkins, "
J. H. Bailey, "
C. W. Oskins, "
H. Hughes, "
H. Arnoul, "
R. Hageman, "
A. J. Hughes, "
J. E. Bishop, "

NEWARK, NEW JERSEY
NOVEMBER 12, 1952

W. F. STRENKMAN (2)

STANDARDIZATION OF Y-662-D MS-765

We are submitting, for your approval, Y-662-D MS-765. This material is a mix of a number of lots taken from two consecutive plant runs. It is felt that this lot is representative of what can be produced in the plant. The attached Summary gives the results of our testing on this lot.

J. E. BISHOP
CHEMICAL DIVISION

jc

# SUMMARY OF TESTING RESULTS ON Y-662-D NS 765

## Rubout Comparisons

3:1 P:V in varnish drier, tints 1:30 with ZnO

| Lot 765 versus | Mt wet | Mt dry | strength | tint | Mt Lightfastness |
|---|---|---|---|---|---|
| K-1810 (C#54336) | lt & grn | lt & grn | 97 | s grn, s int | s better |
| S-3268 (C#54709) | lt & grn | vs lt, vs grn | 97 | s grn, s int | vs-s better |
| Y-469-D lot 658 | vs lt, vs grn, vs int | vs drk, vs red | 99 | vvs red | much worse |
| Y-661-D, SD 95500 | lt, int | vs lt, s int | 95 | s grn, s int | s better |

## Solvent Bleed (TF-7201-2)

| Sample | Alcohol | Toluol | Water |
|---|---|---|---|
| Y-662-D, NS 765 | none | none | bad |
| Y-469-D, lot 658 | none | very slight | none |
| Imp. S-3268 (C#54709) | -- | --- | bad |

## Texture & Consistency

Texture and consistency of Y-662-D NS 765 is equal to that of the previous samples of Y-662-D submitted to IPI (lots 6992 and 7667) or Imperial's S-3268 (C#54709).

## Reactivtty

K-5 readings in MacMichael readings using #26 wire.

| Sample | P/V | Initial | Oven aged 72 hrs. at 158°F |
|---|---|---|---|
| Y-662-D NS-765 | 60/40 | 61 | 44 |
| Y-662-D, lot 6992 | 60/40 | 41 | 102 |
| Y-662-D, lot 7667 | 60/40 | 39 | 39 |
| Imp. S-3268 (C#54709) | 60/40 | 43 | 48 |

## Vaposet (YV-1304-4)

readings by inclined plane flow meter.

| Sample | P/V | Initial Flow | 7 days oven aging |
|---|---|---|---|
| Y-662-D NS 765 | 65/35 | 10-1/2 | 17 |
| Y-662-D Lot 6992 | 65/35 | 3-1/2 | 7 |
| Y-662-D lot 7667 | 65/35 | 16 | 25 |
| Imp. S-3268(C#54709) | 65/35 | 5 | 15 |

(Initial flow is the important reading in this test).

Socony Paint Products Vehicle (readings in KU by Stormer Viscosemeter)

(CONSISTANCY)

| Sample | Initial | 3Day | 2Weeks | 3Weeks | 4Weeks |
|---|---|---|---|---|---|
| Y-662-D NS 765 | 65 | 63 | 70 | 81 | 85 |
| Y-662-D lot 6992 | 69 | 69 | 77 | 97 | 104 |
| Y-662-D lot 7667 | 65 | 65 | 65 | 74 | 77 |
| Imp. S-3268(C#54709) | 70 | 74 | 75 | 73 | 73 |

3M Vehicle (readings in KU by Stormer Viscosemeter)

| Sample | Initial | 2Days | 5Days |
|---|---|---|---|
| Y-662-D NS 765 | 71 | 92 | 116 |
| Y-662-D lot 6992 | 65 | 104 | livered |
| Y-662-D lot 7667 | 65 | 101 | 118 |
| Imp. S-3268(C#54709) | 65 | 94 | 109 |

(Test officially ends at 2 days).