

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

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT
Final Report

Title: IRON OXIDE PULP F-4-P - YIELD STUDY.

Period Covered: April 1-30, 1946

FILE: 210.2

DATE: 5-14-46

Serial No. KN-46-20

Copy No. /

Copy to:

- #1 - Numerical File
- 2 - Research Office (210.2)
- 3 - Library File (210.2)
- 4 - #10 Building File
- 5 - # 6 " "
- 6 - Imperial Chemical Industries, Ltd.
- 7 - " " " "
- 8 - " " " "
- 9 - D. H. Dawson, Newark

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT
Final Report

Title: IRON OXIDE PULP F-4-P - YIELD STUDY.

Period Covered: April 1-30, 1946

SUBMITTED BY: R. R. Denslow *[Signature]* 5/1/46

DATE SUBMITTED: 4-30-46

APPROVED BY: V. Chalupski *[Signature]*

DATE ISSUED: 5-14-46

N41556.01

DUP050150537

INTRODUCTION

Initial manufacture in the plant indicated the yield given in K-2076 to be too high. Since little reliable information was available, three laboratory series were run for yield using different lots of copperas. Care was taken to avoid loss and the figures obtained should be reliable. Yields on pulps are of course open to error resulting from careless sampling; in the case of F-4-P there is the additional difficulty that the apparent dry content varies with the analytical procedure used for determining solids.

SUMMARY AND CONCLUSIONS

The results obtained are summarized below and in more detail in the accompanying table (Table II).

	Wt. dry pigment ex 7500# $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	Procedure for determining solids
K-2076 - Formula yield	2755#	-
Laboratory yield	2300	2 hrs/160°C (Brabender)
" "	2500	16 hrs/70°C
Approx. Plt. Performance	2400	"

Theoretically, 7500# $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ gives 2155# Fe_2O_3 or 2880# $\text{Fe}(\text{OH})_3$. The above laboratory yields correspond respectively to Fe_2O_3 . 5.596 H_2O and Fe_2O_3 . 1.42 H_2O .

For testing purposes (intercomparison of samples), the quick method (2 hrs/160°C) is more convenient. Plant shipments are analyzed by the other procedure (16 hrs/70°C) which is more in line with the temperatures at which the pulp is actually used.

DISCUSSION

Copperas liquor is analyzed for " $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ " by titrating the Fe^{++} with permanganate (KCR-138). If no Fe^{+++} is present, this also gives the total iron and the yield will correspond with the weight of copperas taken. If Fe^{+++} is present it will be precipitated during the process and the yield would be correspondingly higher. In practice, only a small amount of Fe^{+++} is present and it can be neglected without serious error. It was determined in Exp. 1214-1 and amounted to ca. 0.8-0.9 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in a 2 mole batch (556 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$).

Solids were determined as follows:-

- I. 2 hrs. - 160°C. Brabender
- II. 16 hrs. - 70°C. Oven (Plant method)
- III. Large sample (200 g) dried 18-20 hrs. in oven at 60°C. (140°F.)
- IV. Like 2 but run in Brabender. Additional heating showed that weight had become constant.

Methods II, III, & IV give substantially the same results; they are higher than what is obtained by method I and give correspondingly higher yields. (This confirms preliminary results obtained sometime ago. Ref. 1126-64). Apparently, the material obtained by drying at 60-70°C contains about 10% water which comes off if the temperature is raised to 160°C.

As shown in the table a few of the results were badly out of line for no known reason; these were omitted in determining the average yields.

As a further check on the yields by method I, the dry samples (ex 200 g. pulp) were placed in the Brabender at 160°C and from the "solids" obtained another set of yields were calculated. These are shown below.

TABLE I

	<u>Wt. of Pulp</u>	<u>Wt. Obtained from 200 g. pulp</u>	<u>% "Solids" 2 hr.-160°C.</u>	<u>Yield (2 mole)</u>
1126-81A	787	48	87.4	165.0
B	805	47	88.9	168.0
C	757	50	90.1	170.2
D	806	47	90.2	171.0
1126-82A	824	48	92.7	(183.1) (1)
B-	841	44	92.7	171.1
C	831	44	92.6	169.5
D	741	44	92.6	(151.0) (1)
1214-1A	866	42.5	92.1	169.6
B	801	43.5	90.0	164.0
C	865	43.0	91.7	170.4
D	796	46.0	90.4	165.5
Average				168.4/556 FeSO ₄ ·7H ₂ O

(1) Omitted in determining average.

The results obtained by this indirect method are in fair agreement with the average figure obtained by direct analysis (Method I), viz. 170.5. They are not included in Table II.

TABLE II

YIELD STUDY F-4-P
2 mole batches (556 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) (1) (3)

Expt. No.	Copper- as solu- tion(2)	% Fe++ as Fe $\text{SO}_4 \cdot 7\text{H}_2\text{O}$ (1)	% Fe+++ as Fe $\text{SO}_4 \cdot 7\text{H}_2\text{O}$ (3)	Wt. Pulp	S O L I D S				2 mole yield (1) (3)			
					I 2 hrs. - 16 hrs. 160°C. 70°C.	II Bra- bender	III Plt. Method	IV sample (4)	I(4)	II	III(4)	IV
1126-81A	#406	39.16%	not detd.	787g	21.5%	-	24.0%	-	171.6	-	188.6	-
B	"		"	805	21.0	-	23.5	-	169.0	-	189.0	-
C	"		"	757	22.6	-	25.0	-	171.0	-	189.2	-
D	"		"	806	21.0	-	25.5	-	169.1	-	189.3	-
1126-82A	404	36.22	not detd.	824	20.6	-	24.0	-	169.8	-	(198.1)	-
B	"		"	841	20.5	-	22.0	-	172.5	-	185.0	-
C	"		"	831	20.5	-	22.0	-	170.3	-	183.0	-
D	"		"	741	22.0	-	22.0	-	(163.0)	-	(163.0)	-
1214-1A	407	33.50	0.0479%	866	19.9	21.6%	21.25	21.6%	172.0	187.0	184.1	187.0
B	"			801	21.3	23.1	22.75	23.1	170.6	185.0	182.1	185.0
C	408	33.54	0.0531	865	19.7	21.4	21.50	21.6	168.5	185.0	186.0	186.9
D	"			796	21.5	23.2	23.0	23.3	171.0	184.9	183.0	185.3
Average (5)					170.5		185.5		185.9		186.0	
Yield 7500# $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$					2300		2500		2505		2505	

(1) Based on analysis for ferrous iron. KCR-138.

(2) Plant N-24.

(3) Based on KCR-138 but Fe+++ calculated as $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in copper's liquor. Ratio to Fe++(4) In Expt. 1214-1, the additional iron used (calculated as $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) was before ca. 0.8-0.9 g.

(5) Water (2 hr. - 160°C) was determined in the dry material; the average yield calculated from these figures (see Table I.) was 168.4 g as compared with 170.5 g shown in column I.

Figures in parenthesis omitted as obviously out of line for no known reason.