ICA CLASSIFICATION AND PLANT RUN HISTORY FOR "AFFLAIR" NF-132-D By: B. W. Cetnar 12/66 - 11/67

E. I. DU PONT DE NEMOURS & COMPANY

PIGMENTS DEPARTMENT

Serial No. KN-69-5

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PIGMENT COLORS RESEARCH REPORT

SUBJECT: MICA CLASSIFICATION AND PLANT RUN HISTORY FOR AFFLAIR® NF-132-D

PERIOD COVERED: December 1966--November 1967

Date: 2/10/69

SERIAL NO. KN-69-5

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SUBMITTED BY: B. W. CETNAR BWCetnar DATE SUBMITTED: 1/10/69

APPROVED BY: J. F. MAURER DATE RELEASED: 2/10/69

Abstract

The report summarizes the work performed toward the development of an Afflair® grade suitable for use in automotive finishes. Included is a process for classifying mica and the results obtained when the classified mica was converted into Afflair® in the plant.

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MICA CLASSIFICATION AND PLANT RUN HISTORY FOR AFFLAIR® NF-132-D

INTRODUCTION

The current automotive metallic finishes are susceptible to chemical spotting. Afflair® flake pigments, however, are inert in the environments that cause spotting. This opens a new potential market for Afflair® provided the flake size can be controlled and Afflair® quality upgraded.

Control of the mica flake size is essential for an automotive grade product. Large flakes would protrude through the paint film while very small flakes would scatter light excessively, contributing to a dull, lusterless product.* Since commercially available micas contain significant amounts of both large and fine flakes, a research program was undertaken to develop a classification process to remove them. Hydraulic cyclones were the classification devices selected for the process development.

SUMMARY AND CONCLUSIONS

An improved Afflair® can be produced when a highly classified mica is used as the substrate material. A flowsheet for classifying mica has been developed along with the plant process procedures for converting the mica into an Afflair® product. For automotive finishes, however, the problem of mica flakes protruding through the paint film remains to be overcome along with the attainment of adequate humidity resistance before a satisfactory automotive product can be realized.

The market potential for Afflair® in automotive finishes makes it attractive to continue research efforts on this specialized grade. With this as the incentive it is recommended that development work on an automotive-grade Afflair® be continued.

EXPERIMENTAL DETAILS

Four plant trial runs were made. The details for each run are as follows:

A. Run 32-2

1. Feed Mica

The feed mica for Run 32-2 was India scrap that was water-ground by the Diamond Mica Company.

* Large flakes are those in excess of 38 microns while the fines are estimated at -5 microns.

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2. Mica Classification

A two-step classification was used to remove oversize and undersize mica. Hydraulic cyclones (hydroclones) were employed in both steps.

The two-part classification consisted of first removing the oversize (coarse or grit). The product from this classification then became the feed for the second part or undersize (fines) removal. The flow diagrams for each classification are given in Figures 1 and 2.

a. Hydroclone Description

1. 3-inch Hydroclone (Clones A, B, C, D and E)

Bauer "Centri-Cleaner" Model 600-3
Nylon plastic body and underflow nozzle

Vendor: The Bauer Bros. Co.
Springfield, Ohio

2. 10 Millimeter Hydroclone

"Dorrclone" TM (Single Stage)
Size: 10 mm (32 units); nylon clones in stainless steel housing

Vendor: Dorr-Oliver Incorp.
Stamford, Connecticut

b. Hydroclone Operating Details

For Part 1 of the classification (coarse removal), a mica-water slurry was prepared that analyzed 17.1% solids. However, at this feed concentration, the underflow (UF) at clone "A" was too concentrated to pass out of the UF tip and the clone plugged. Dilution of the feed slurry with water to 15% solids was also too concentrated. A satisfactory concentration was reached at a solids content of 12.5% and below. The concentration range during the run was 8.7% to 12.5%.

The slurry concentration was only a problem in the first stage or "A" clone since the feed was progressively diluted in each subsequent stage.

Table 1 lists the slurry concentrations, flow rates, and pressure readings for each hydroclone.

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SUBJECT FLOWSHEET CALCULATIONS - COARSE CLASSIFICATION

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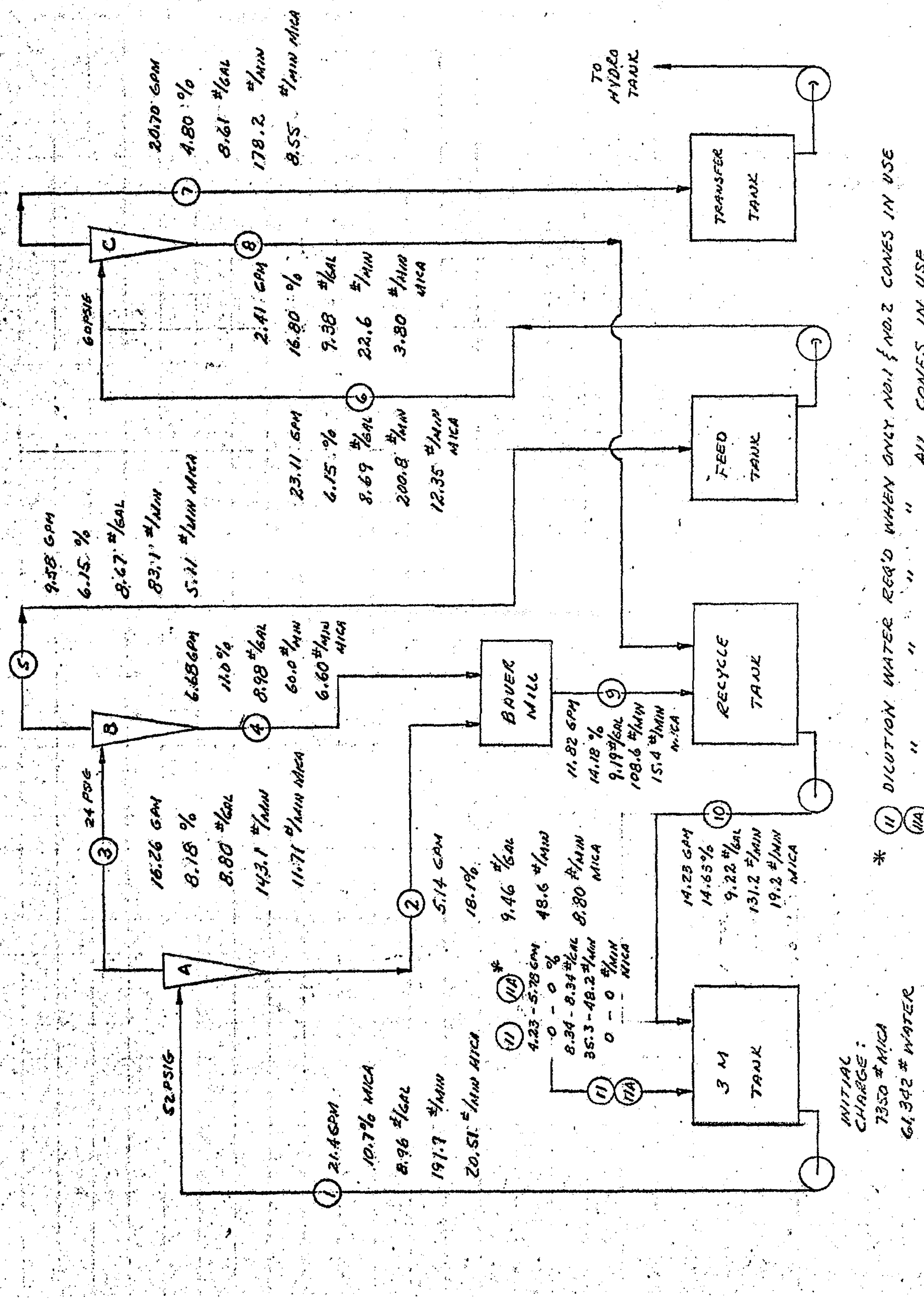
FIGURE 1



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TITLE OF PROJECT OR STUDY "AEFLAIR" RUN 132-2

SUBJECT FLOWSHEET CALCULATIONS - FINES CLASSIFICATION

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FIGURE 2

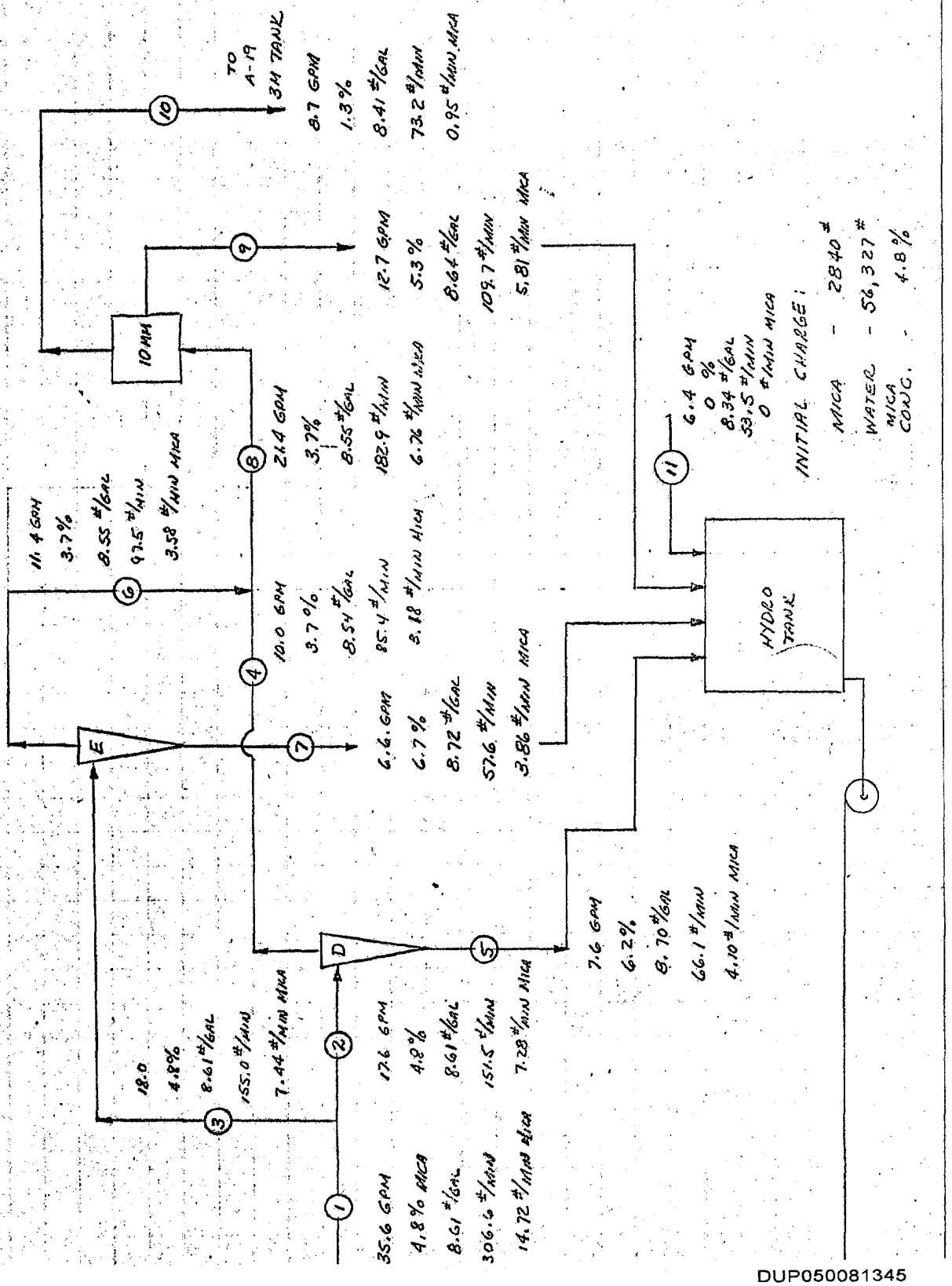

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TABLE 1

CLONE	INLET			OVERFLOW			UNDERFLOW		
	% Solids	GPM	PSIG	% Solids	GPM	PSIG	% Solids	GPM	PSIG
A	10.7	21.4	52	8.2	16.3	24	18.1	5.1	0
B	8.2	16.3	24	6.2	9.6	0	11.0	6.7	0
C	6.2	23.1	60	4.8	20.7	0	16.8	2.4	0
D	4.8	17.6	52	3.7	10.0	28	6.2	7.6	0
E	4.8	18.0	52	3.7	11.4	28	6.7	6.6	0
10 mm	3.7	21.4	28	1.3	8.7	4	5.3	12.7	0

Since the flowsheets were designed to recirculate the underflow from the hydroclones, the method used to determine the stopping point of the classification was by the number of volume changes made in the feed tank, i.e. the total volume pumped from the feed tank divided by the average volume in the feed tank. For oversize removal, 9.3 volume changes were made. During removal of the fines, 7.4 volume changes were made.

c. Classification Results

In order to evaluate the effectiveness of coarse and fine particle removal, samples of the product streams were wet-screened. The mica size distribution was determined as the percent retained on the screens. The screen sizes used are listed in Table 2 along with the screening results obtained on the feed and product streams.

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TABLE 2

CLASSIFICATION RESULTS

Screen Size	Particle Size Range	Mica Feed %	Product Coarse Removal[a] %	Product Fines Removal %
200 Mesh	+200 M	4	0.1	0.2
270 "	-200 +270	8	0.6	1.0
325 "	-270 +325	7	0.7	1.8
30 μ	-325 +30μ	26	17.6	26
20 "	-30 +20	17	21	22
10 "	-20 +10	17	10[b]	36
pan	-10	21	50	13
		100	100	100

a) Product from oversize removal became the feed for the fines removal.

b) The low value can be attributed to a faulty screen. A more nearly correct estimate would be 24% for this fraction and 36% for the -10μ fraction.

d. Classification Yield

The yields below were obtained when mica was classified on a semiworks scale. The value is the percent recovered of the mica charged. The overall yield is the product weight from the fines classification divided by the mica charged to the first hydroclone stage of the oversize classification.

Product from oversize removal	49%
Product from undersize removal	62%
Overall yield	30%

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3. Hydrolysis

The mica product from the classificaton was TiO_2-coated in the plant. The following is the formula used for the hydrolysis:

Mica	--1,590 Lbs.
Water	--2,179 Gal.
TiO_2	--1,590 Lbs.
TiO_2 concentrate	--1,053 Gal.
78% H_2SO_4	-- 243 Gal.

The strike was made with both slurry and concentrate at 60°C. The addition time for the concentrate was 12 minutes. The heat to boil period was 120 minutes. The hold time at boil was 2-1/2 hours.

The large TiO_2-to-mica ratio employed resulted in the hydrolysis exceeding the desired color endpoint. The hydrolysis product was deep red instead of the desired red rose color.

4. Hydrolysis Product Classification

At the completion of the hydrolysis the slurry was classified using the fines removal flowsheet. This was done in an effort to increase the luster of the final product by removing free TiO_2 that precipitated during the hydrolysis. Figure 3 is a material balance calculated from measurements taken while the classification was in process.

5. Calcination

The presscake for Batch 32-2 was processed in the Edgemoor kiln. The kiln temperature was adjusted so that the kiln discharge matched the color of a laboratory calcination made at 950°C. To obtain that color, the kiln temperature at the 1-foot thermocouple was controlled in the range of 1000-1025°C.

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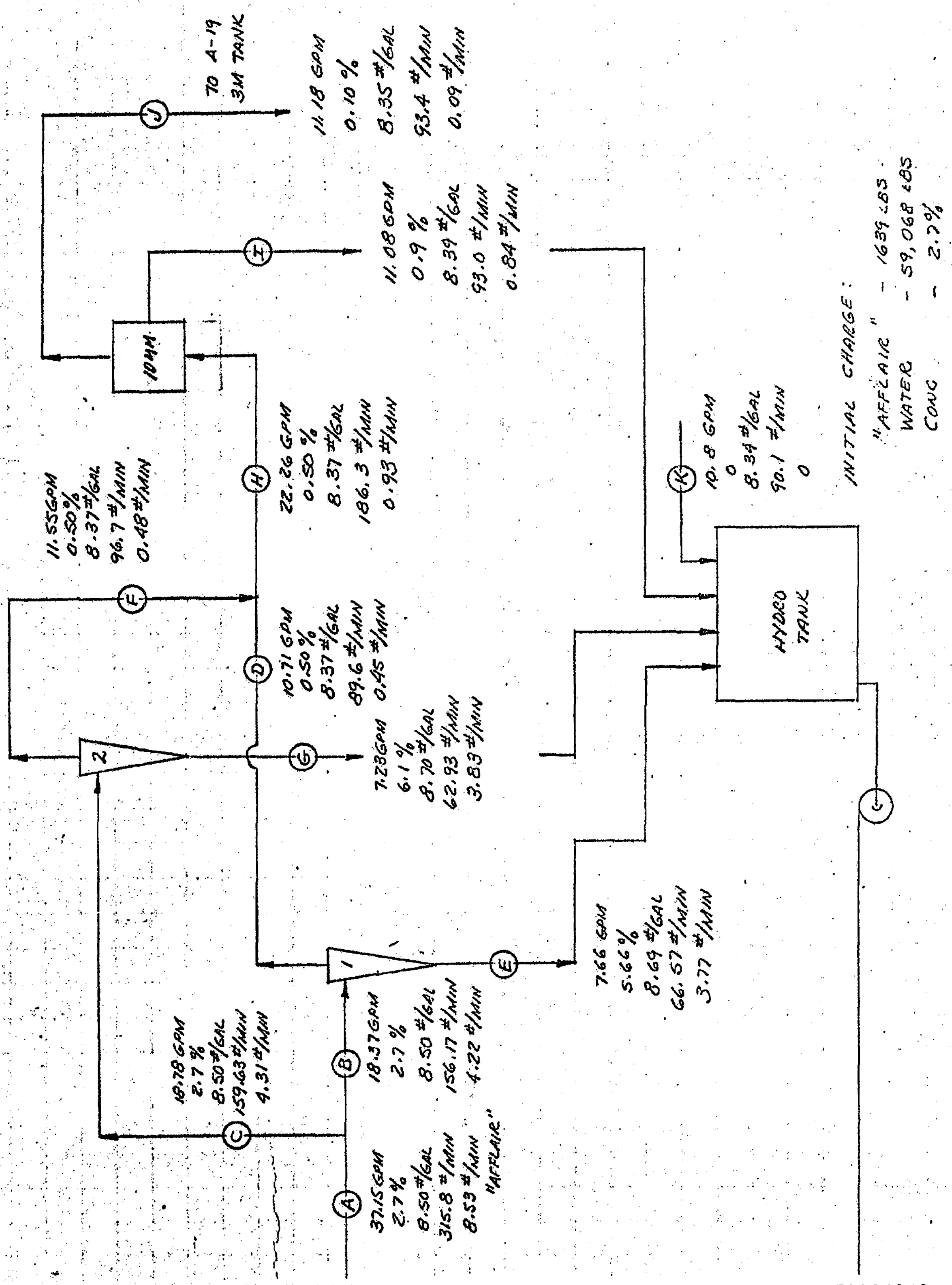
FIGURE 3



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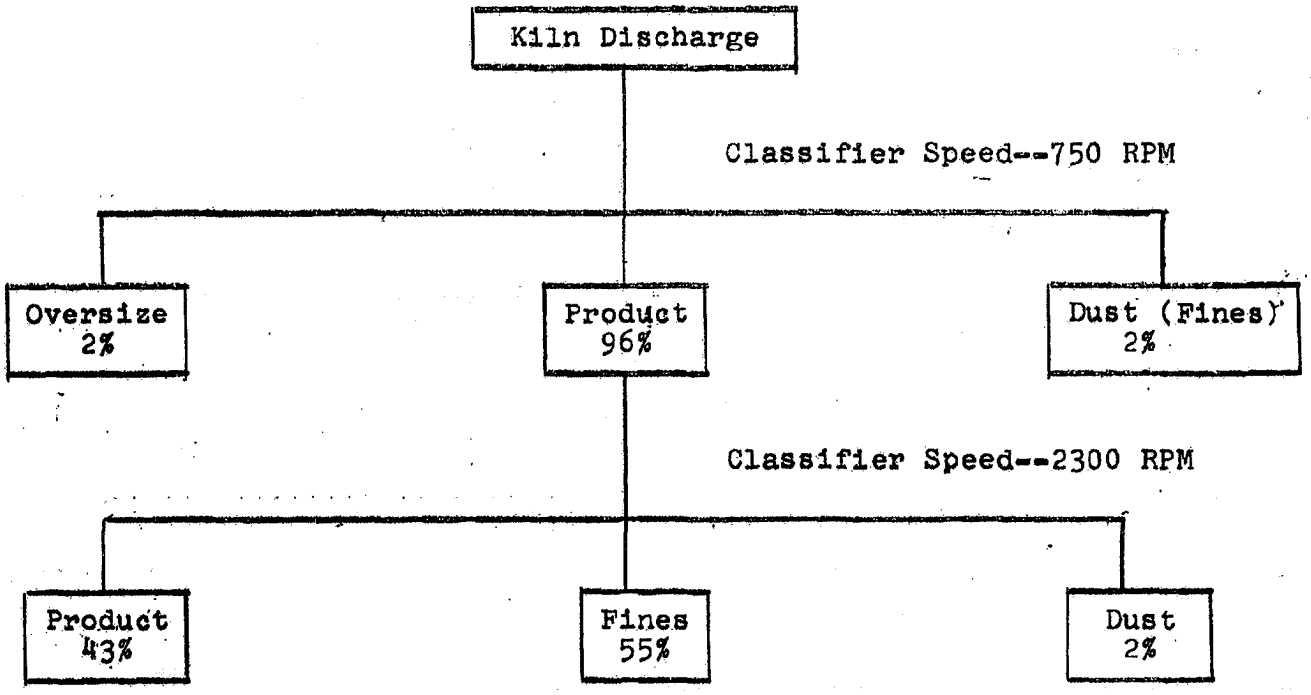
6. Air Classification

Prior to submitting the Afflair® product for evaluation in an automotive finish, the calcined flakes were given a final classification in the "Hurricane" classifier. This is normal procedure for all Afflair® grades. The classification for Batch 32-2 differed, however, in the speed setting at which the classifier was operated. Normally, a speed of 1600 rpm is used. For the experimental batch the classifier was operated at two speeds. On the first pass through the machine, the speed was set at 750 rpm. At this setting, the larger flakes were separated from the bulk of the material. A second pass was then made using the product fraction from the first pass. For this pass, a speed of 2300 rpm was used. At this speed, the smaller flakes were removed. Figure 4 shows the flowsheets and the yields obtained for each pass--

FIGURE 4



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7. Evaluation Results

The Afflair® product from Run 32-2 was evaluated in a medium Blue Teal automotive finish and was found to be deficient in color, gloss and surface quality.

The flake color was a red gold instead of the desired yellow gold. The red color was a direct result of not recognizing that a mica soaked in water for several hours causes a change in the hydrolysis rate. This was established and confirmed in subsequent runs.

While color was important to the evaluation, of greater significance was the surface quality of the paint film when the experimental flakes were used in the paint formulation. Earlier attempts to use the regular Afflair® grades in automotive finishes failed because the larger flakes protruded through the film. They also lacked sufficient intensity for automotive use and when incorporated in a finish depressed the gloss of the film below an acceptable level. The experimental pigment also exhibited these deficiencies but to a much less marked degree.

B. Run 32-3, 32-4 and 32-5

1. Feed Mica

The feed mica for runs 32-3, -4, and -5 was "Alsibronz" from the Franklin Mineral Company. The lot identification number and weights used were as follows:

Lot No.	Mica Weight (Lbs.)
48	30,150
55	2,000
57	10,000

2. Mica Classification

Mica for the trial runs was prepared by hydraulic cyclone classification. Two classifications were made, one to remove the oversize platelets and the other to remove the fines. Figure 5 shows in block diagram form the flowsheet used. Figure 6 is the same flowsheet presented as a line diagram.

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SUBJECT FLOWSHEET FOR RUNS 32-3, 4, & 5 WORK'S

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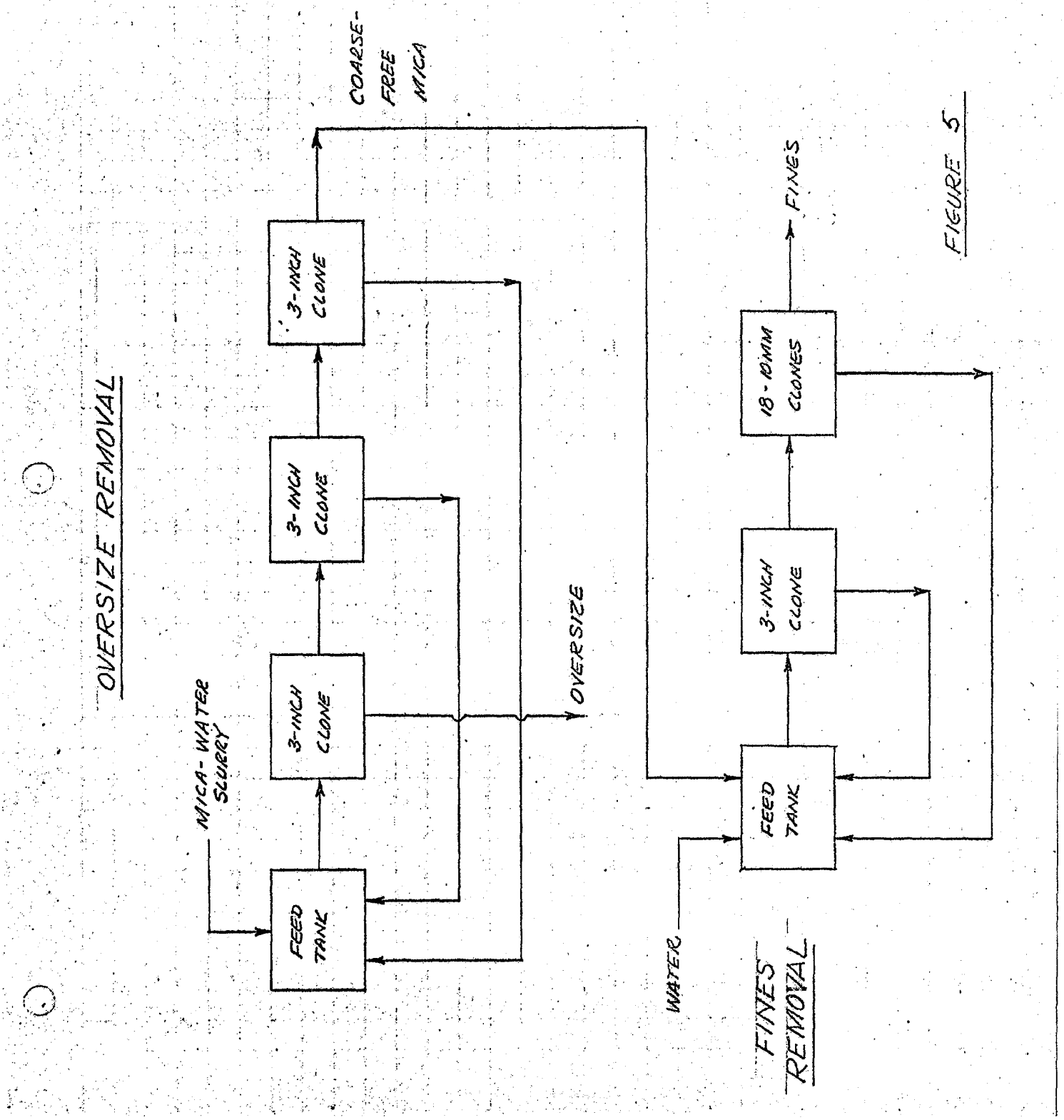

FIGURE 5

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MICA CLASSIFICATION

FLOWSHEET FOR RUNS 32-3,4,&5

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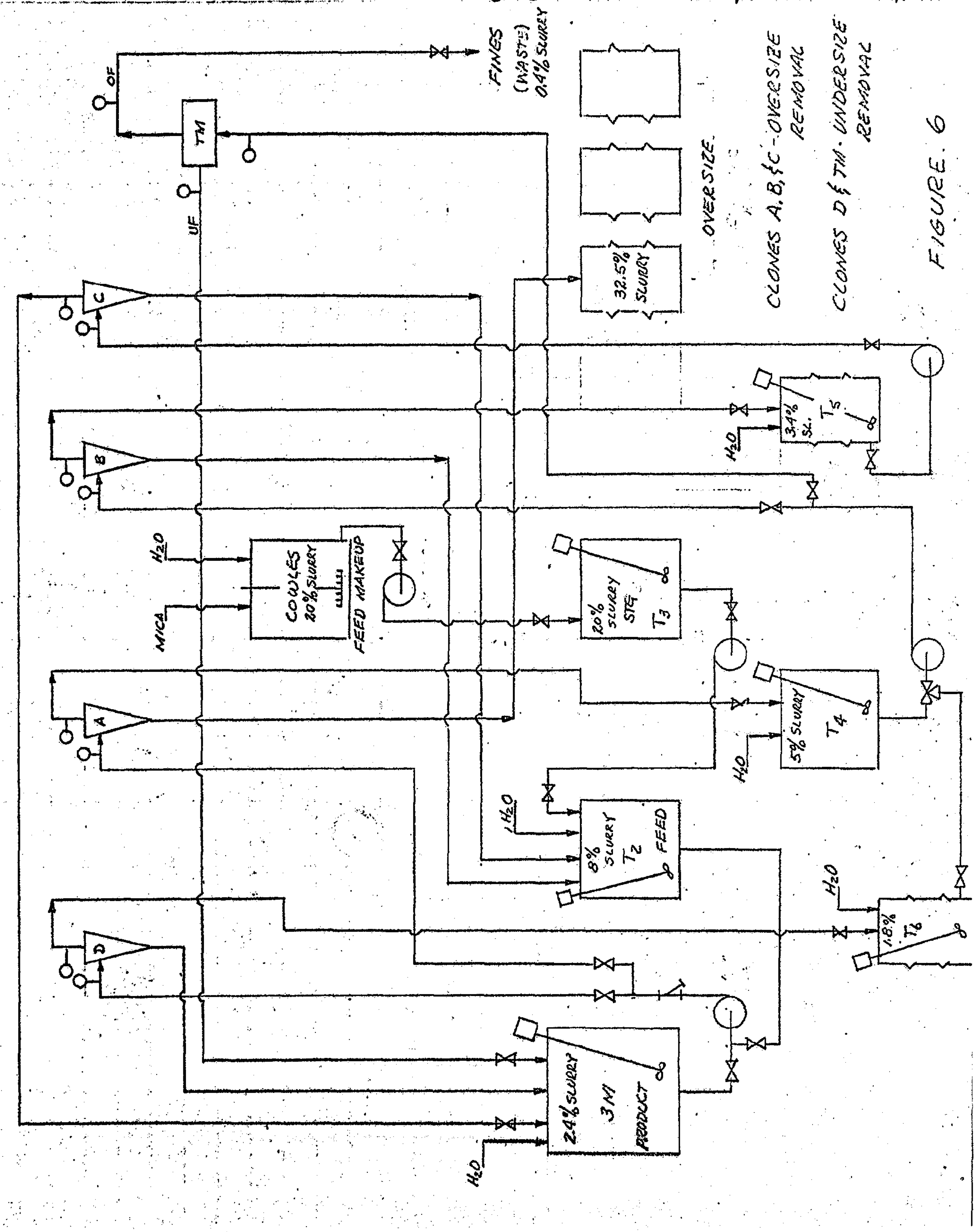

FIGURE. 6

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2. Mica Classification (Contd)

To remove the oversize, a mica-water slurry at 8% solids was pumped into a 3-inch Bauer hydroclone. The oversize platelets were rejected from the system in the hydroclone underflow. The overflow or product fraction was sent to a second clone where another separation was performed and finally to a third clone. The product fraction from the third separation stage, which was now free of large flakes, became the feed for the fines removal.

In contrast to the oversize removal, which employed continuous feed, the flowsheet for fines elimination was designed for recirculation (Figures 4 and 5). The oversize-free product was pumped through the clones and returned to the feed tank except for the overflow from the 10 mm clones which contained the fines. The recirculation was continued until the fines fracton in the feed dropped to the level desired. In generating classified mica for this Afflair® series, the volume in the feed tank was turned over approximately 7.5 times.

a. Hydroclone Operating Details

Since the flowsheet for the oversize removal required a continuous feed supply, a mica-water slurry was made up at a mica concentration of 20% by weight. A Cowles Dissolver was used to disperse the mica in the water. The 20% slurry was transferred to the feed tank at a controlled rate where it was combined with the underflow from hydroclones B and C. Sufficient water was metered into the feed tank to obtain an overall concentration of approximately 8% solids. The primary clone (hydroclone A) was fed at this concentration.

The fines removal was batch operated. The oversize-free product mica (overflow from hydroclone C) was the feed source for this part of the classification. When tank 3M was filled to capacity with the overflow from clone C, the oversize classification was stopped and removal of the fines begun. The volume in the feed tank (3M) was allowed to deplete until half the original volume remained. At this point, water was metered into the tank to maintain a constant level throughout the remainder of the run. The run time was calculated on the basis of 7.5 changes of the average volume in the tank.

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a. Hydroclone Operating Details (Contd)

The hydroclone operating conditions are shown in the following table.

TABLE 3

CLONE	INLET				OVERFLOW				UNDERFLOW			
	% Solids	GPM	Mica #/ min.	PSIG	% Solids	GPM	Mica #/ min.	PSIG	% Solids	GPM	Mica #/ min.	PSIG
A	7.5	20.6	12.8	28	5.0	19.3	8.4	0	32.5	1.3	4.4	0
B	5.0	19.3	8.4	33	3.4	18.1	5.3	3	26.0	1.2	3.1	0
C	2.8	19.0	5.3	34	2.4	18.0	3.8	4	16.1	1.0	1.5	0
D	2.4	19.6	4.1	48	1.8	16.8	2.7	23	5.7	2.8	1.4	0
10 mm	1.8	16.8	2.7	54	0.4	13.6	0.5	0	7.8	3.2	2.2	7

b. Classification Results

Wet screening was used to determine the effectiveness of the classification. The wet screen data in Table 4 are representative of that obtained in 29 semiworks runs.

The screen data show the feed mica contained 28% +400 mesh (38 microns) and 31% -10 microns. The product mica from the oversize removal contained only 3% +400 mesh. The final product after fines elimination contained 22% -10 micron material.

The assignment of -10 microns as fines was arbitrary and was employed only because the wet screen analyses were limited by the smallest screen used, i.e. 10 microns. However, classification effectiveness below 10 microns can be estimated from the data. Figure 7 shows a log probability plot of the particle size for the feed mica and product mica. Extrapolation of the screen data indicate a substantial reduction of small platelets in the product mica. For example at the 3 micron size, the -3 micron material was reduced from about 8% in the feed to less than 1/2% in the final product.

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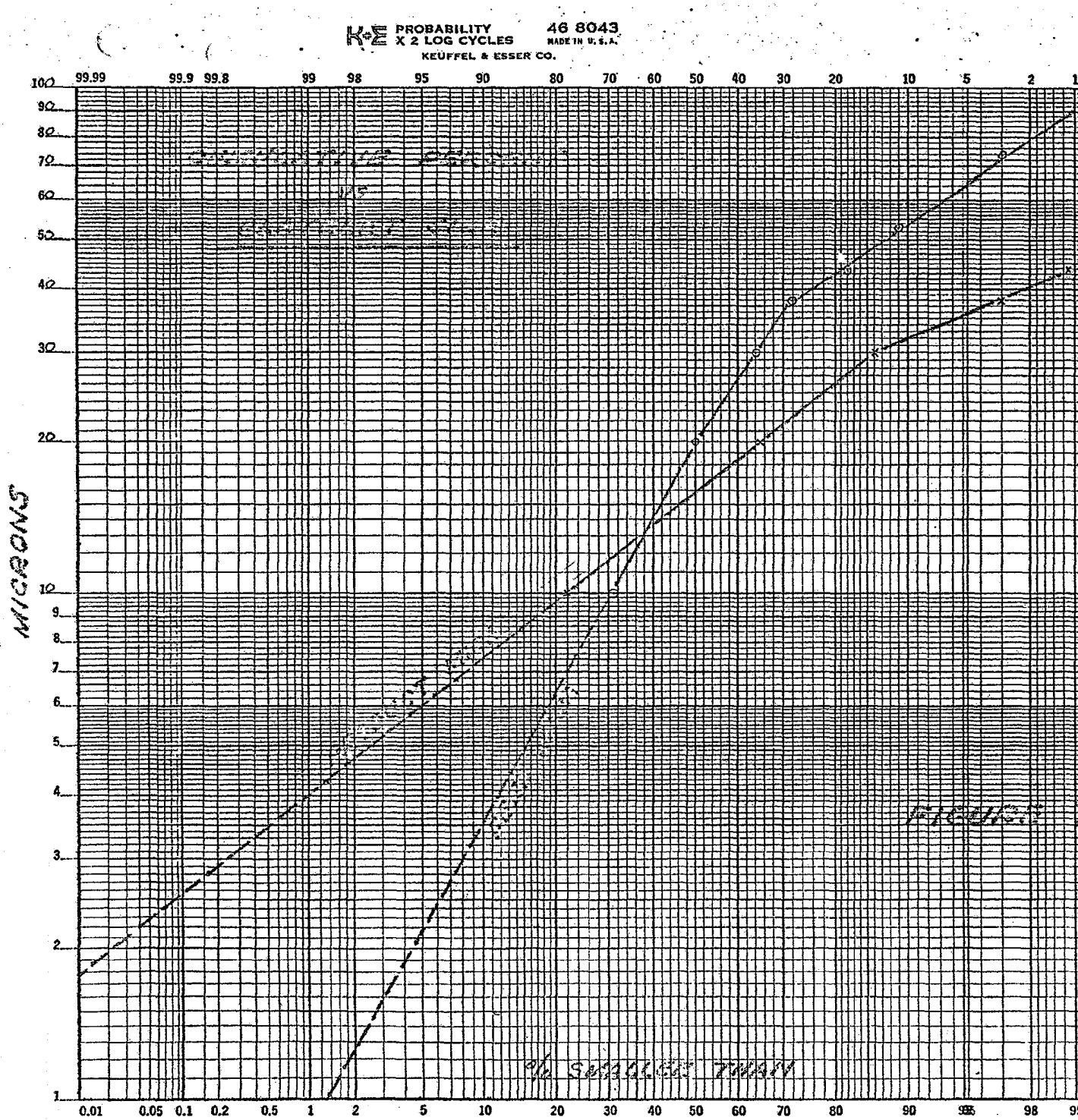

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b. Classification Results (Contd)

TABLE 4

PARTICLE SIZE DISTRIBUTION

		Feed Mica %	Oversize Fraction %	Product Oversize Removed %	Product Fines Removed %
	+200 Mesh	3	5	0.3	0.1
-200	+270 "	8	18	0.4	0.4
-270	+325 "	7	13	0.5	0.6
-325	+400 "	10	8	1.8	1.9
-400	+30 μ	8	25	7	11
-30	+20 "	14	14	16	21
-20	+10 "	19	11	37	43
-10		31	6	37	22
		100	100	100	100

c. Classification Yield

The following table summarizes the classification yields obtained in the semiworks.

TABLE 5

CLASSIFICATION YIELD

	(A) SEMIWORKS PROCESS	(B) REPROCESSED OVERSIZE
1. OVERSIZE REMOVAL		
a. Mica Charged:	41,367 lbs.	2,257 lbs.
b. Mica Recovered:	20,549 lbs.	816 lbs.
c. Yield:	50%	36%
2. FINES REMOVAL		
a. Mica Charged:	20,549 lbs.	816 lbs.
b. Mica Recovered:	11,207 lbs.	566 lbs.
c. Yield:	55%	69%

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TABLE 5

(Contd)

	(A) SEMIWORKS PROCESS	(B) REPROCESSED OVERSIZE
3. FILTRATION		
a. Mica Charged:	11,207 lbs.	566 lbs.
b. Mica Recovered:	10,654 lbs.	538 lbs.
c. Yield:	95%	95%
4. OVERALL YIELD		
a. Mica Charged:	41,367 lbs.	2,257 lbs.
b. Mica Recovered:	10,654 lbs.	538 lbs.
c. Yield:	26%	24%

In addition to the yield data above, two runs were made in which the oversize mica was returned to the system and reclassified. The yields obtained are shown in Column B of Table 5. While not demonstrated over an extended period, projection of the reprocessed mica data to the regular process indicates that the overall yield could be increased from 26 to about 38%.

3. Hydrolysis

The classified mica was stored as wet filter cake until the plant hydrolysis run was made.

The hydrolysis formula for Run 32-2 is given below:

Hydrolysis Formula

Classified Mica	--5,310 lbs.
Water	--to make mica-water slurry at 19.6% solids
TiO_2	--2,549 lbs.
TiO_2 Concentrate	--1,311 gal.
78% H_2SO_4	-- 287 gal.

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3. Hydrolysis (Contd)

The strike was made with both the slurry and concentrate at 60°C. The concentrate addition time was 15 minutes. The heat to boil period was 3 hrs. 10 min. The hold time at boil was scheduled for 3 hrs.; however, the hydrolysis proceeded much faster than anticipated with the result that the desired end-point was reached before the heat-to-boil period was completed. Consequently, the resulting hydrolysis product was too red to be of value.

In a subsequent laboratory investigation it was established that the hydrolysis reaction could be controlled to give a quality product by decreasing the time required to make the concentrate addition and by decreasing the heat-to-boil time. A 5-minute addition time and 30-minute heating period was found to give reproducible results. No further formula changes were found necessary.

Runs 32-4 and 32-5 were made using the modified times and both runs produced superior Afflair® quality. However, in order to achieve the revised time limits, it was necessary to reduce the batch size to 42% of the mica weight listed in the preceding formula. This was due to heating limitations of the plant equipment.

4. Calcination

The presscake for Run 32-4 and 32-5 was processed in the Edge Moor kiln. A feed rate of 70 lbs. wet cake per 15 minutes was used. The best color quality was obtained at 950°C. based on the 1-foot thermocouple.

5. Air Classification

The kiln product was air classified at Newport. Only a single pass through the classifier was made. The one pass was sufficient to remove many of the larger agglomerates formed during calcination and to remove some of the residual fines. Wet screen data shown in Figure 8 indicate that the final product contained about 3% +38 micron (+400 mesh) material and less than 3% -5 micron particles.

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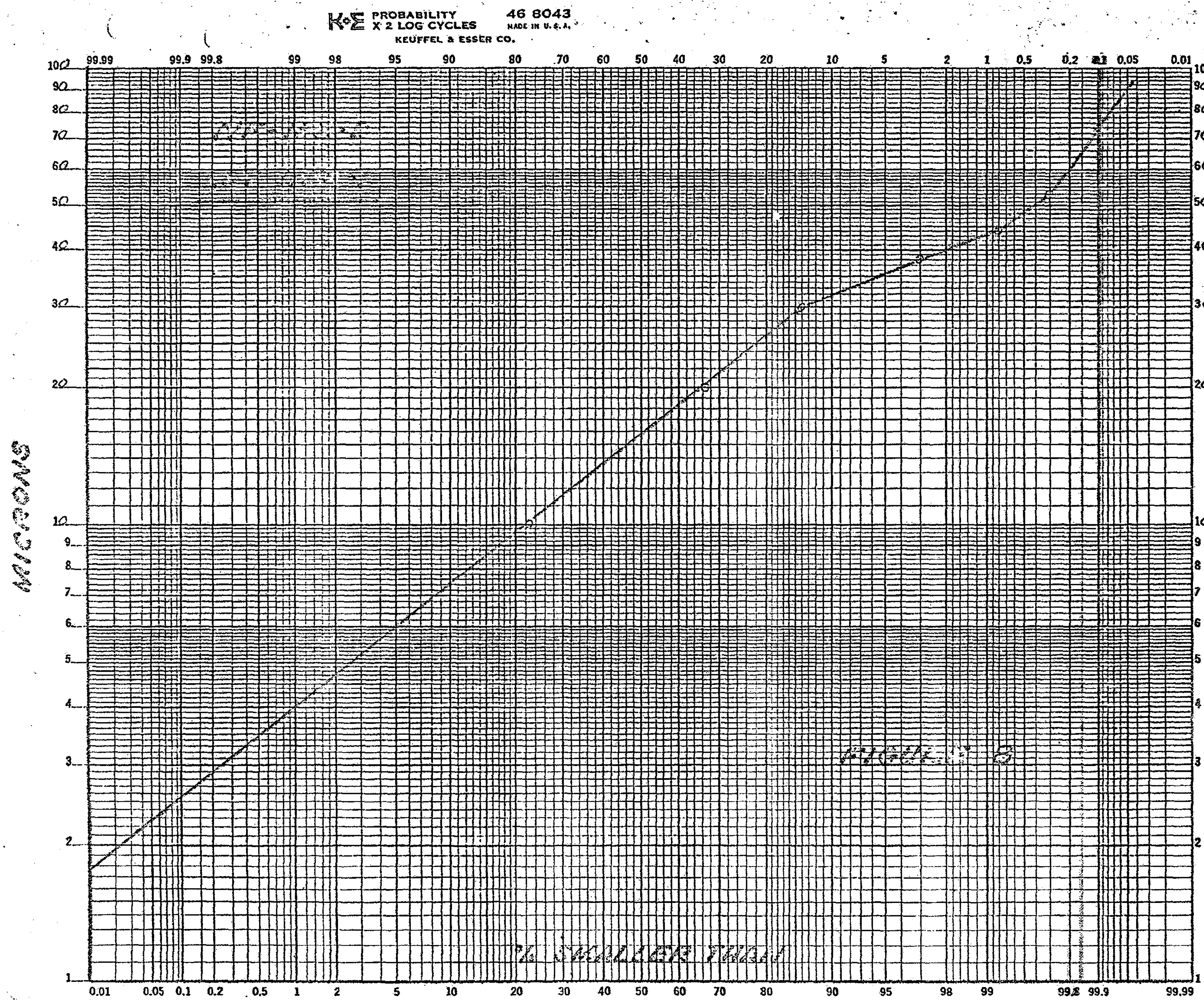

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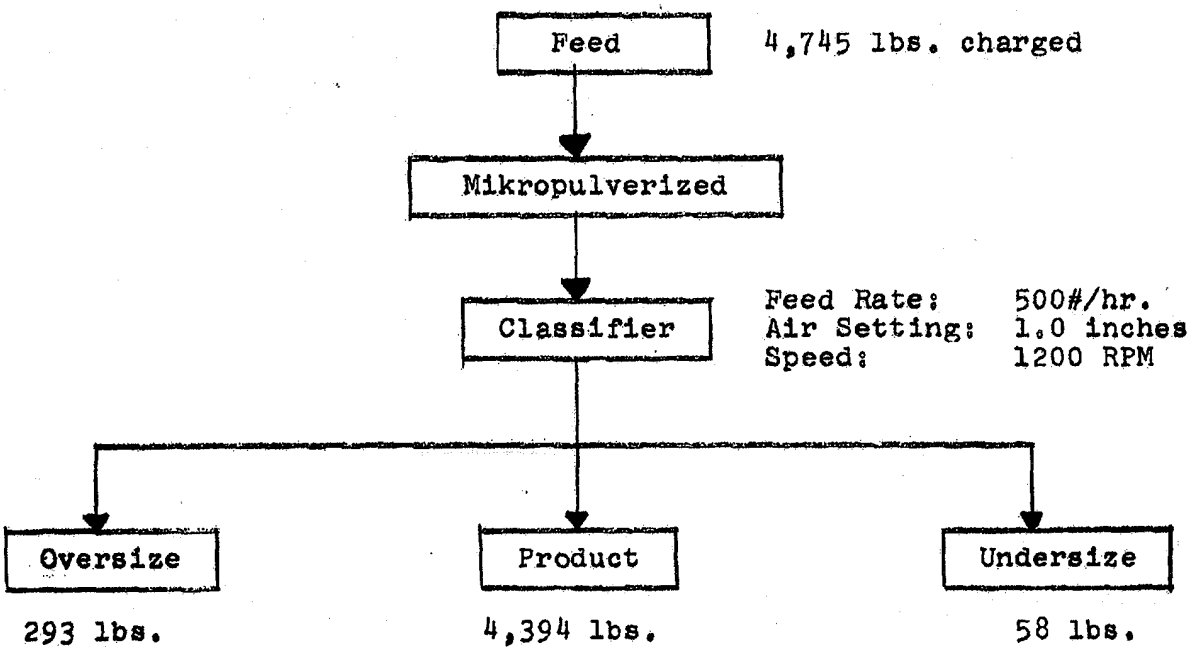
5. Air Classification (Contd)

Based on the weight charged to the classifier and product fraction recovered, a yield of 92.6% was obtained. Figure 9 shows a line diagram of the classification flowsheet.

FIGURE 9

AIR CLASSIFICATION FLOWSHEET
RUN 32-4 and 32-5



Yield $\frac{4394}{4745}$ x 100 = 92.6%

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6. Evaluation Results

The product from 32-4 and 32-5 contained significant improvements in Afflair® quality. The flakes were an attractive gold color and were more intense and lustrous than any of the current Afflair® grades. They also exhibited particularly good flip-flop and lacked much of the milkiness of the other Afflair® products. However, even with these improvements, the flakes were only marginally acceptable in an automotive finish.

When evaluated for automotive use, the quality criteria for automotives was met regarding aesthetic appeal. The deficiencies that were found were physical-chemical in character. These included protrusions in the film and unsatisfactory humidity resistance.

The protrusions were only evident when the film was viewed at the extreme grazing angle. They appeared as hair-like filaments (mica flake on end) sticking through the film. Catalyzed finishes gave poorer results than uncatalyzed finishes.

The principal failing, however, was humidity resistance. A significant loss in adhesion occurred after exposure to saturated air at temperatures ranging from 100°-150°F.* It is suspected that the rapid failure resulted from a number of contributory causes including residual soluble salts, inadequate dispersion, incomplete wetting and faulty application of the finish.

* The poor humidity resistance is by no means unique with NF-132-D. All other Afflair® grades, including Afflair®-like products from competitors also exhibited poor humidity resistance.

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