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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

MANUFACTURE OF MOLYBDATE ORANGE IN THE PLANT
(#12 BUILDING) BY A CONTINUOUS PROCESS

Period Covered: 11/21/47 to 6/1/49

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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Progress Report

Title: MANUFACTURE OF MOLYBDATE ORANGE IN THE PLANT
(#12 BUILDING) BY A CONTINUOUS PROCESS

Period Covered: 11/21/47 to 6/1/49

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INTRODUCTION

The original laboratory work on the manufacture of molybdate orange by a continuous process was completed in July 1944 after a brief study. The semi-works process was also established with little difficulty and with only minor changes from the laboratory formula. In view of the successful laboratory and semi-works study, it was decided not to make a trial in the larger #6 Bldg. continuous unit but to undertake a plant evaluation of the process upon completion of the full-scale #12 building unit. This decision was also influenced by the fact that equipment alterations would have been necessary in #6 building to accommodate the YE-421-D process.

The objective of the work summarized in this report was to develop a continuous process using the #12 building equipment to manufacture molybdate orange equal in all important respects to standard, YE-421-D SD-48416. With the setting up of the new improved texture standard, SD-89686, the preparation of such an improved product has been accepted as a objective.

CONCLUSIONS

The best molybdate orange made in the #12 building continuous unit to date is slightly light and slightly dull in masstone versus YE-421-D SD-48416, 5% weak, yellow in tint, and worse in lightfastness. Considerable improvement over earlier runs was effected by increasing agitation in the strike tank, increasing the lead excess after the strike from 1.8% to 2.7%, and decreasing the chromate/molybdate ratio.

SUMMARY

Two trial runs of YE-421-D were made in the #12 Building unit soon after its completion in 1947. Products from the first run were predominantly light and dull in masstone versus YE-421-D SD-48416, about 8% wk, and very yellow in tint. There was some improvement in strength and masstone depth during the second run. Both runs were troubled with mechanical difficulties.

In March 1949 a run was made essentially duplicating the previous work so that control samples could be taken to determine where the process was "going off". The only changes were an increase in the lead excess after the strike from 1.8% to 2.7% and an increase in the retention time before the acid addition. Most of the final product was light, dull, 15% weak and red and dull in tint. By removing samples at various points and finishing them in the laboratory, it was established that while the masstone color changed little from the beginning to the end of the process, considerable strength was lost. Lightfastness also suffered near the end of the process.

Laboratory work was immediately started to determine what changes to make in the #12 Building formula to equal standard. "Dump" strikes commonly employed to simulate continuous processes at first yielded very poor molybdate oranges. Great improvements were effected by increasing the agitation and using a slow "dumping" technique. By adjustment of the chromate/molybdate ratio products were obtained which were fairly close to standard. Throughout the laboratory work a lead excess of about 3% following the strike was employed. Efforts to reduce this excess yielded dull masstones and weak, yellow tints.

A plant run was made with increased agitation in the strike tank and with the laboratory chromate/molybdate ratio. The products were slightly light and dull in masstone, about 5% weak, and yellow in tint. In view of these encouraging results, another run was immediately made with a lower "chrome" pH

which had given a redder tint in the laboratory. Most of the products were light and slightly dull, about 5% weak and close in tint compared to the standard. Apparently the tint was corrected at the expense of masstone depth (a similar masstone/tint change is obtained in batch manufacture by lowering the lead pH). It was also apparent that masstone/tint relationship of the #12 process were different from that of the batch process. Control samples taken during the run indicated a loss of about 5% in strength following the acid addition. A loss in lightfastness was indicated following the alum and silicate addition and at the end of the drum of the dryer. Work on this problem by the Chemical Division was discontinued following completion of this last mentioned run and the responsibility for further work was transferred to the Production Division.

A flow sheet of the process and sample operating formula are included in the appendix of this report.

PATENT STATUS

Nothing of a patentable nature was uncovered during the study summarized in this report.

FUTURE WORK

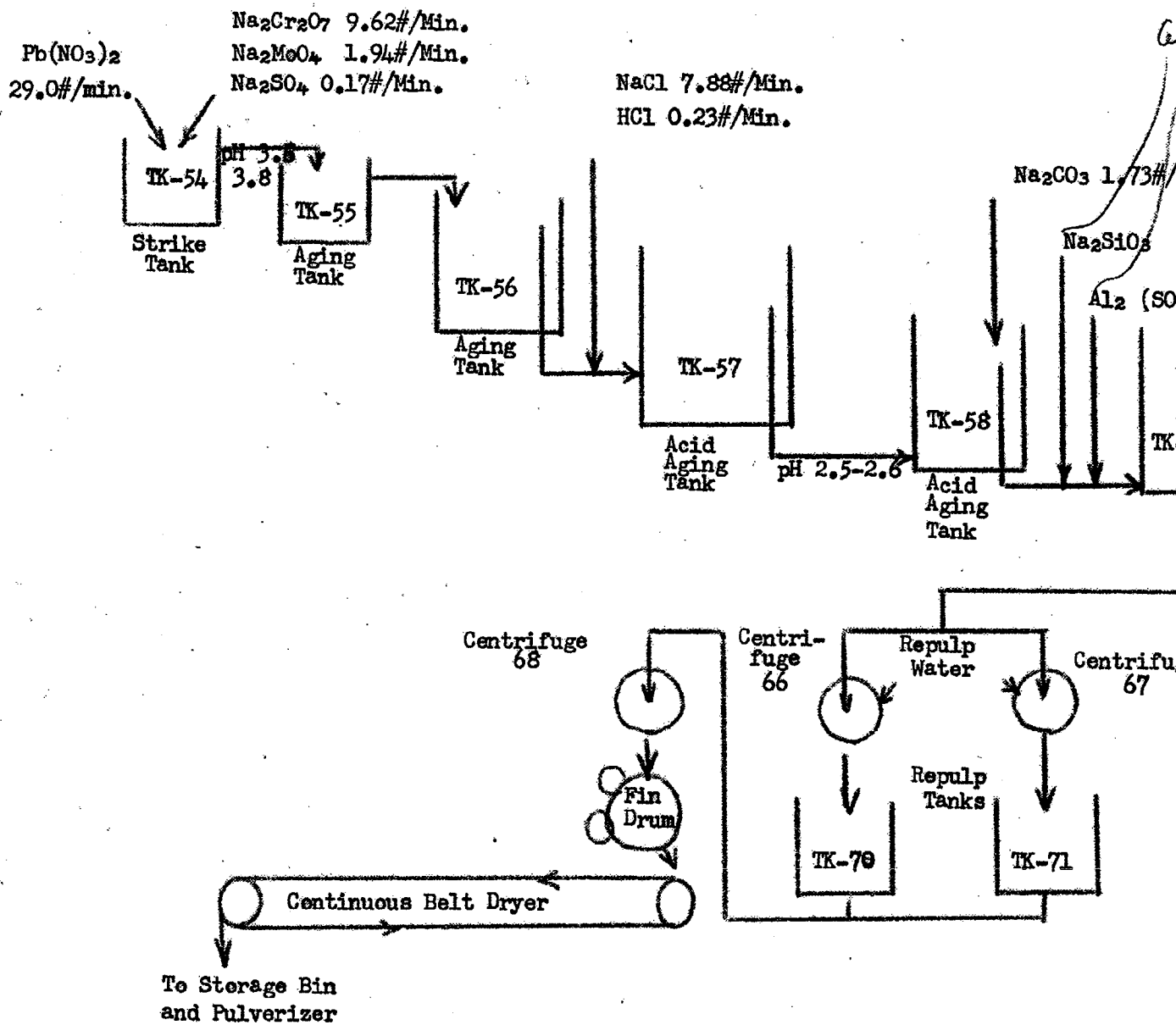
Further work on this problem has been turned over to the Production Division. It is recommended that the method of salt/acid addition be investigated with an aim to preventing the 5% loss in strength which occurs with the present pipeline addition. It is possible that addition directly to the first acid ageing vat would prevent this loss in strength. No work on lightfastness was done in the laboratory or the plant but this property must be improved to equal standard. Laboratory work has produced no leads which might improve the "out-of-line" masstone/tint relationship of recent products. It may be that more dilute solutions, as are used in batch manufacture, will have to be employed rather than the concentrated solutions now used in #12 building. The effect of concentration on the masstone/tint relationship should be investigated.

REFERENCES

N.B. 1111

DSN-46-9

Why not using the strike tank?



FLOW SHEET

Continuous Manufacture of Molybdate
Orange in 2 Building.

Coal Yield (Based on N-41) -
Product Rate = $\pm 1600\#/HR$

Color Code = YE-421-D
EK: 2-25-48

Standpipes	Liquid Level	Ret. Times	TK - 54 Agitator Speed at Maximum Water Temp. = 70°F	
TK - 54 - as is	23"	TK-54 = 0.89 min.	First Ht. Dev. = X	Second Ht. Dev. = X
TK - 55 - as is	23"	TK-55 = 0.89 min.		
TK - 56 - 22"	25"	TK-56 = 5.3 min.	First Ht. Dev. = X	Second Ht. Dev. = X
TK - 57 - 40"	43"	TK-57 = 8.5 min.		
TK - 58 - 57"	60"	TK-58 = 11.8 min.	First Ht. Dev. = X	Second Ht. Dev. = X
TK - 59 - 40"	43"	TK-59 = 7.8 min.		

Rotameter	Setting	#/Gal.	#/Min.	G.P.M.	Total G.P.M.	Added to	Solutions #/Gal. Sp.Gr.	
N-41		1.10	26.1		50.4	54 Tank		
Water								
N-10		1.00	9.62		28.8	54 Tank		
Water								

There should be a slight lead xs at the entrance to 56 Vat.

N-71		1.40	7.88			TK-56 to TK-57 Line		
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Maintain pH 2.5 - 2.6 in 58 Vat by regulating N-71 flow.

N-3		1.00	2.76			58 O.P.		
N-91		0.70	1.58			P.L. 58-59		
N-15		0.70	2.03			P.L. 58-59		

Maintain pH 5.8 - 6.0 leaving 59 Vat by regulating N-3 flow.
Dryer Temp. sec. #1 220°F Sec. #2 220°F Sec. #3 200°F Sec. #4 200°F