

Pigments Department

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Newport, Delaware
April 27, 1973

CPC--NEWPORTEXPERIMENTAL OPERATING PROCEDURE NO. 428

Title: BT-476-D, N-8730-A, Monastral® Blue G

Objective: Demonstration of plant-scale manufacture by preparation of 2,000# of BT-476-D.

Background: Sales has requested a Blue G pigment which can be dispersed by a Cowles milling, followed by one pass on a three-roll mill. Ciba-Geigy's Irgalite Blue LGLD has been promoted for oil-based inks on this basis.

BT-476-D prepared in the laboratory and in the Newark semiworks has been superior versus LGLD in dispersibility, transparency, gloss and viscosity in heat-set inks. A previous attempt to manufacture BT-476-D under XOP-424 in December of 1972 failed to produce acceptable material. Contamination with Arquad 16-50 resulted in a product of extremely poor dispersibility. In-process sampling indicated two major sources of contamination: (1) insufficient cleanout of the dispersion mill and (2) use of tote bins which contained substantial amounts of standard BGD mill powder. Several modifications of the procedure have been made to reduce the threat of contamination by Arquad 16-50 during the execution of this XOP.

Number of Tests: Ten (10) Dispersion Millings
Two (2) Extractions

Starting Date: April--May 1973

N35374

April 27, 1973

Procedure: This operation including cleanout should have special supervisory coverage on a 24-hour basis. No equipment is to be used without approval from supervision. BT-476-D should follow a BT-436-F campaign which utilized vinyl pyrrolidone but no Arquad 16-50. The details for equipment cleanout and for the process are attached.

Testing: Research will evaluate by Cowles milling in Thermex® A6E heat-set vehicle.

Newport Disposition: To be shipped to Newark for drying and pulverization.

Samples:

- 1 gallon of each milling at completion (10)
- 1 gallon of 4-hour milling from #6 milling (1)
- 1 gallon vat contents on route to filter (each extraction) (2)
- 1 gallon tote bin presscake (each extraction) (2)

Safety Aspects: No new hazards involved.

<u>Responsibilities:</u>	Scheduling & Batch Cards:	Production Control
	Samples:	Operations
	Testing:	Research
	Closing Report:	Research, E. W. Gillow

APPROVALS:

R. H. Hutzler 4/27/73
Research Supervisor

W. J. Hoffman 4/29/73
Area Supervisor

J. J. Laffer 4/30/73
Production Supervisor

J. C. Choney 4/27/73
Scheduling & Control

EWG/mja

April 27, 1973

CLEANOUT PROCEDURE FOR DISPERSION MILLING
N-8730-A, BT-476-D

(Use only mills cleaned as follows)

1. Flush with water until flushes are clear, probably 8-10 hours.
2. Dry mill with low water alum 2 or 3 flushes at 400 lbs. each until dry. About 1-2 hours required for this step.

1/2 min. turn max. w/ ^{dry} alum flush

This cleanout method was suggested by W. G. Hoffman, CPC Area II Supervisor. He should be consulted if clarification is needed.

The mill or mills used should be selected from those just completing an Arquad 16-50 free campaign of BT-436-P.

EWGillow/mja

CLEANOUT PROCEDURE FOR MILL DISCHARGE,
EXTRACTION, FILTRATION, PACKOUT
N-8730-A, BT-476-D

TOTE BINS FOR MILL POWDER*

1. Select 10 tote bins for mill powder discharge.
2. Chop out any encrusted mill powder, particularly in corners.
3. Flush profusely with water until foam is no longer detectable.

PUMPING STATION, VAT 47C

1. Fill 47C vat and empty several times.
2. Pass vat contents through lines to rotary filter.
3. Send water flush from clean tote bin through lines to 47C.

ROTARY FILTER

1. Drain bowls
2. Flush cloths and bowl with water until free of pigment from previous batches. A thorough job will take one shift.

TOTE BINS FOR PRESSCAKE*

1. Select four (4) tote bins.
2. Turn on side and flush with high pressure hose until no presscake clings to surfaces.

* If new tote bins are used, they must be cleaned and inspected in the same manner.

EWGillow/mja

OPR. _____

N-8730-A

BT-476-D

Mill _____

Chg # _____

DATE	TIME	OPR.	INSTRUCTIONS	QUANTITY	
				STD.	RECOR
			1. Check mill		
			a) Brake set tightly.		
			b) Stop rod in place.		
			c) Mill empty.		
			Internal mill temperature previous batch		
			65°C or below.		
			If not, cool mill until temperature is 40°C		
			or below.		
			2. Add aluminum sulfate.	1100#	
			3. Add CP-LC blue crude Lot No.	220#	
			Lot No.		
			4. Add		
			(a) Perclene	30#	
			(b) EVACITE 2046	7#	
			5. Install grinding head - make sure cooling water		
			is on full to mill jacket.		
			6. Remove stop rod, unlock brake, and start milling.		
			7. Record H ₂ O outlet temp. after milling for 2 hrs.		
			If temp. is above 20°C notify Supv.		
			8. Record H ₂ O outlet temp. immediately before dumping.		
			If temp is above 20°C notify Supv.		
			9. Milling finished (actual milling).	5 hrs.	
			Stop mill, set brake, insert stop rod. Remove		
			grinding head, and install dumping head.		
			10. Start dust collector.		
			11. Remove stop rod, unlock brake and start dumping		
			charge by rotating mill for 17 min. If discharge		
			weight is under 1350# min. inform supv.		
			12. Clean scale face and platform.		
			13. Drum charge to clean, dry Tote Bin		
			Take 1 qt. sample during drum out.	#/Bin	
			14. Shut off dust collector.		
			15. Position mill, set brake, insert stop rod and		
			remove dumping head.		
			Record: Sampled by:		
			Total Net Weight.	1357#	
			Stencil: Code, Lot, Net and Tare Weights		
			on each container - 5 Solids		
			Net Solids		
			MAKE OUT PACKOUT CARD		
			16. Record internal mill temperature.	65°C Max	
			(Record this temp. on next Charge - Item 1(c)		
			Same Mill)		

PROVALS:

BGD EXTRACTION-

BT-476-D,

N-8730-A

47C - ROTARY FILTERS

BATCH NO.

TIME	OPR.	INSTRUCTIONS	QUANTITY	
			STD.	RECO.
		1. Select 5 lots for extraction and store in area east of unloading station. Hold for step 9. Record total weight.		
		Lot No. Lbs. Gallons H ₂ O		
		Lot No. Lbs. Gallons H ₂ O		
		Lot No. Lbs. Gallons H ₂ O		
		Lot No. Lbs. Gallons H ₂ O		
		Lot No. Lbs. Gallons H ₂ O		
		Record Total Pounds	Lbs.	
		*Use water to mill powder chart for correct gallons H ₂ O.		
		2. Check 47C tank empty.	MT	
		3. Close 47C tank discharge valve - Very important that valve is closed. Acid solution will corrode magnet and pump internal parts.		
		4. Add 250 gallons water to 47C tank -	Gals.	
		5. Record time started water addition.		
		6. Start agitator. Fume scrubber must be operating before agitator will start.		
		7. Add 344 lbs (4.0") Sulfuric acid, 100% basis 255 lbs. (3.1")	4.0"	
		8. Heat 47C tank to 94-96°C - set control point at 95°C.	94-96	
		9. Slurry and pump 5 charges mill powder via tote bin unloading station. + 6785 lbs. mill powder (+ 1100 lbs. 100% CPC). Check 47C tank temperature before each tote bin unloading.		
		Extraction tank temperature must be 85°C or above before proceeding. If 47C tank is below 85°C, contact supervisor.	85°C	
		10. Record time completed tote bin unloading.		

BATCH NO.

DUP050109374

FILTRATION-WASHING-PACKING

N-8730-A BT-476-D

DATE	TIME	CPR.	INSTRUCTIONS	QUANTITY	
				STD.	REC.
			1. Start rotary vacuum filter and auxiliary equipment.		
			2. The following operating condition must be set before slurry from 47C is to be pumped to rotary vacuum filter bowl.		
			a. Drum speed set at RPM. Numbers on dial selector.		
			b. Vacuum to drum inches Hg.		
			c. Waterflow cover drum		
			#1 wash line gpm Valve setting		
			#2 wash line gpm Valve setting		
			#3 wash line gpm Valve setting		
			* Wash water temperature 90-95°C.		
			d. Primary filtrate receiver vacuum inches Hg.		
			e. Secondary filtrate receiver vacuum inches Hg.		
			f. Air to drum - adjust as needed.		
			3. Start filling #1 rotary vacuum filter bowl. Record time bowls starts filling.		
			4. Check #1 rotary vacuum filter primary and secondary filtrate pumps for effluent color. If color is present, stop filtration and investigate cause and make the necessary correction.		
			5. Check #1 rotary vacuum filter primary and secondary filtrate pumps effluent hourly for color.		
			6. Fill repulper with water manually.		
			7. Set water flow to repulper on automatic control. Record time #2 bowl starts filtering		
			8. Start #2 rotary vacuum filter and auxiliary equipment when level in bowl covers rake agitator.		
			9. The following operating condition must be set on the secondary rotary vacuum filter.		

FILTRATION-WASHING-PACKING

N-8730-A BT-476-D

DATE	TIME	OPR.	INSTRUCTIONS	QUANTITY	
				STD.	RECC
			b. Vacuum to drum _____ inches H ₂ O.		
			c. _____ water _____ to #1 wash line _____ gpm		
			H ₂ O temperature _____		
			d. Turn on NH ₃ flow to #2 wash line _____ gpm.		
			H ₂ O temperature _____		
			Water flow to #3 wash line _____ gpm		
			H ₂ O temperature _____		
			e. Primary filtrate receiver vacuum _____		
			_____ inches H ₂ O.		
			f. Secondary filtrate receiver vacuum _____		
			_____ inches H ₂ O.		
			g. Air to drum - adjust as needed.		
			10. Check #2 rotary vacuum filter primary and secondary filtrate receiver pumps effluent for color. If color is present, stop filtration and investigate cause and make necessary corrections.		
			11. Check #2 rotary vacuum filter primary and secondary filtrate pumps effluent hourly for color. Record on check sheet.		
			12. Packout presscake in Drums or Tote Bins. Take a 2 oz. presscake sample every 15 minutes from conveyer before dumping to shipping containers. Make composite sample of all 2 oz. samples taken during packing out lot.		
			13. Record Lot No. _____ Sampled by _____ Opr. _____		
			No. Containers _____ Net Weights _____		
			14. Identify each container with following information:		
			_____ N-Code		
			_____ Lot No.		
			_____ Gross Wt.		
			_____ Tare Wt.		
			_____ Net Wts.		
			15. Make out packout card and deliver to Area Office.		