COPY

cc: C. E. Rossee - Wilm.
V. Chalupski - Newport
C. J. Higgins - "
C. T. Axelberg - "
C. H. Collier - "
J. N. Tully/A. Siegel - Nwk.
J. H. Cooper
P. D. Graham - Newport

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MR. F. C. EVANS
NEWPORT

ACID FLUSHING PROCEDURE

Attached is a tentative operating procedure using an acid flushing ("AF") technique in place of kerosene flotation ("KF") to provide an extracted crude presscake, which is essentially kerosene-free and can be dried in available water-drying facilities. It is recommended that two batches of Blue LB be processed accordingly as soon as necessary minor equipment changes can be made.

It has been necessary to modify the Semi-works process for AF to adapt it to Newport plant equipment; nevertheless, it is felt desirable to proceed with plant trials at this time to actually determine the equipment and process requirements for sustained operation and accumulate crude acid-flushed CPC for quality evaluation. The effect of these necessary modifications upon quality can not be predicted; however, it is believed that the immediate goal of obtaining a substantially kerosene-free crude presscake will be realized.

Based on available information, the procedure as outlined is designed to minimize operating hazards arising from the use of equipment, which is obviously not designed for acid flushing.

The Chemical Division will provide complete coverage for the recommended plant trials.

E. F. KLENKE
CHEMICAL DIVISION

EFK:pr
jc

# ACID FLUSHING (AF) PROCEDURE

## Acid Flushing

1. Pump entire synthesis charge from 19A or 19B to tank 28 (steam still). Flush as usual and agitate in tank 28 for approximately 20 min.

2. Inspect 24 tank (sulfation tank) and make certain it is clean and dry.

3. Add 6300# of 98% $H_2SO_4$ (52-1/2" measured from acid head tank) to tank 24. Measure and record volume in 24 tank. (No agitation is required during the acid flushing in the sulfator).

4. Pump from 28 tank to 24 tank over a period of 60 minutes. CAUTION - A considerable volume of gas is evolved during this pumping operation, which could possibly cause an overflow from 24 tank if the pumping rate is too rapid. For this reason it is imperative that the area around 24 tank be roped off and operating personnel be kept out of the immediate vicinity.

5. When pumping is completed, allow the contents of 24 tank to settle for a period of 1 hr. Measure and record volume in 24 tank.

6. Decant supernatent kerosene to within 2 to 3 inches of the acid layer.

7. Discharge acid/pigment solution to 22 tank (hydrolysis tank) leaving in 24 tank a 6 inch heel which should be approximately half kerosene and half acid/pigment. No agitation is required in the hydrolysis tank. Measure and record level in 22 tank.

## Drowning Step

1. Run 2460 gallons of water into 31 tank (alk. extr. tank) 5 ft. 4 inches actual depth (not liquid level gage measurement).

2. Start the agitator.

3. Half the contents of 22 tank are to be pumped into 31 tank in not less than 1 hour. CAUTION - Rapid evolution of steam and gases may cause a foamover, therefore, the area around 31 tank should be roped off and operating personnel warned.

## Neutralization & Extraction

1. Upon completion of step 3 (above) 50% NaOH solution is to be added to 31 tank, using extreme caution. Probably a 2 to 3 hr. period will be required for the NaOH addition to prevent foamover due to excessive local heat generation during neutralization. Approximately 5200 lbs. of 50% NaOH soln. will be required (70 inches from head tank) to give the desired final pH of 9 to 11.

2. Following pH adjustment as outlined above, heat to 95°C and hold for 30 minutes.

3. Filter in 33A or 33B (this represents one half of synthesis charge, the second half to be filtered in the other press subsequent to processing by the same procedure as outlined above).

4. Wash sulfate free. The washing cycle will be appreciably larger than usual due to the greater amount of $H_2SO_4$, used.

5. Drum out entire charge sampling representatively for % solids and % kerosene. Hold charge for testing.