FOR COMPANY USE ONLY

DATE January 21, 1975

TEST AUTHORIZATION NO. TA-C- 75-51

OFFICIAL PRODUCT NAME 2-Amyl Anthraquinone Crude

PRODUCT CODE NO. 050 816 2000 POUNDS PRODUCT PROPOSED 42,000 lbs.

AREA "Ponsol" BUILDING NAME Intermediates

TITLE 2-Amyl Anthraquinone - Increase charge size and modification of neutralization and washing procedure.

PREPARED BY: D. S. James DSJ/JNL RESPONSIBLE FOR TEST AND TEST CONCLUSION: E. G. Leser

PURPOSE OF TEST: To increase the charge size in the reaction kettle by 25% in order to increase the pounds of AAQ produced; and to increase the salt content in the final wash from 4% to 8% to improve AAQ Crude recovery.

DURATION OF TEST: One month (20 lots).

STANDARD INVOLVED: TMR-C-71-61 (8/16/72); TA-C-74-565 (8/7/74); TA-C-73-683 (8/7/74); TA-C-74-471 (8/7/74); TA-C-74-1001 (11/5/74); TA-C-74-1105 (& conclusion 11/22/74).

ANALYTICAL CONTROL: ☒ No change ☐ Change described under Test Details

TECHNICAL BASIS FOR CHANGE: See attached.

TEST DETAILS:
(Include Safety and Waste Disposal) See attached.

SAFETY: No change.

WASTE DISPOSAL: No change in the nature of the waste products is anticipated.

DISTRIBUTION:

R. H. Terss	D. O. Miller (3)	E. G. Leser
J. L. Files	G. L. Porter	A. F. Burk
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APPROVED BY:

James E. Kelly 1/23/75 — CHIEF SUPVR., OPERATIONS — DATE

Clifton F. Burk 1/23/75 — CH. SUPVR., TECH. OR PLT. ASSIST. — DATE

DATE

SUPERINTENDENT, ANALYTICAL (When Required) DATE

SUPERINTENDENT, DYE QUALITY CONTROL (When Req'd) DATE

AUTHORIZED BY:

1/29/75 — SUPERINTENDENT, MANUFACTURING (Or Delegates) DATE

SUPERINTENDENT, TECHNICAL (If Any) DATE

/lu MU-1752-B-R6

Technical Basis for Charge

The current charge size for AAQ Crude utilizes only 67% of the capacity in the reaction vessel, K-7. Increasing the loading size in the reaction vessel to 84% of the capacity will permit a corresponding product charge size increase of 25%.

TA-C-74-1001 Part I Technical Basis cited poor recoveries of AAQ Crude due to emulsions and slow settling rates of the organic and aqueous layers through the wash steps as a source of product low yield. Settling rate improvements were observed for mixtures with up to 12% sodium sulfate content in the aqueous phase. Charges ran under Part I showed an 8% yield improvement (a solvent corrected weight out of 1700 lbs. versus 1575 lbs.) from mixtures with a calculated 3.8% salt content in the final wash. Calculations have been made to increase the salt concentration of the final wash to 7.5% in this test authorization.

Test Details

Oleum Preparation

1. Charge K-3 or K-7 with 5588 (± 100) lbs. of 8.5 to 9.5% oleum.

   If K-3 is used, oleum is subsequently transferred to K-7.

   For make-up of oleum directly into K-7 (most cases), use the indicated amounts below according to the daily orders. Daily orders are based on the % sulfuric acid in the " 98% Acid" (Summer Acid is 98.0% and Winter Acid is 96.5%).

   Summer Acid

   2128 lbs. 98% H_2SO_4 (33 inches from TM-68)
   3460 lbs. 20% oleum (29.5 inches from TM-12)

   Winter Acid

   1729 lbs. 98% H_2SO_4 (27 inches from TM-68)
   3859 lbs. 20% oleum (33 inches from TM-12)

2. Agitate acid make up for 10 to 15 minutes and then sample charge for "% oleum". Do not wait for results, proceed to addition of ABBA/TCB solution.

   If analysis begins running high or low charge after charge, supervision should check both the 98% acid and 20% oleum concentrations.

Addition of ABBA/TCB

3. With acid in K-7 at 25-30°C., charge 5000 lbs. of ABBA/TCB solution from TW-1 to K-7.

   Over a one-hour period, drop 5000 lbs. of ABBA/TCB solution in incremental shots keeping the temperature in K-7 less than 95°C. Add the ABBA/TCB solution in four shots (500 lbs., 1000 lbs., 1500 lbs., and 2000 lbs.) applying cooling water as needed to maintain temperature. The first three charges should be run with supervision.

   The charge should be on temperature, 87 to 93°C.; at the end of the addition; adjust by heating if necessary.

   Operator must remain at K-7 until charge is on temperature.

4. Hold charge in K-7 at 87-93°C. for four (4) hours.

5. Continue following standard process procedure as reflected in TA-C-74-1001 for drowning and pumping of charge to neutralization and wash tank, T-19.

Neutralization

6. When three charges are in T-19, without agitation, adjust temperature to 75 to 80°C. and allow the charge to settle for 1 to 2 hours.

7. Transfer the water layer to the ditch from the bottom side outlet of the upper set.

   This leaves about 1723 gal. in T-19 of which about 1347 gal. is AAQ/TCB solution. Check for presence of product before ditching.

8. Add zero to 10,000 lbs. of 55-60°C. process water.

   Preferred amount of water is zero.

9. With agitation, adjust the pH of the charge to 7.0 to 9.0 by addition of approximately 1800 lbs. of 30% caustic soda.

   Maximum temperature 90°C.

10. When pH has been properly adjusted, agitate the charge for an additional 5-15 minutes and adjust temperature to 55 to 80°C., then allow charge to settle for 2 to 3 hours.

    Preferred settling temperature range is 75 to 80°C.

11. Transfer the top, water layer to the ditch from the bottom side outlet of the upper set.

Washing

12. Add 5,000 to 15,000 lbs. of 75 to 80°C. water to T-19 and agitate for 5-15 minutes.

    Preferred amount of water is 7,000 lbs.

13. Allow charge to settle for 2-4 hours, preferred 3 hours, at 55 to 80°C., preferred 75-80°C.

14. Transfer the top, water layer to the ditch from the bottom side outlet of the upper set.

15. After water layer has been ditched, pump charge from T-19 to S-9 such that only the lower product layer is observed going through bull's eye. Discontinue pumping when water layer is observed in bull's eye.

    Operator must stand at bull's eye during this step to be sure no more water than is necessary is pumped to S-9. If an emulsion layer is observed, this layer must also be transferred to the still and only the clear, upper water layer should remain in T-19.

16. When the charge is in S-9, drain remaining water in T-19 to the ditch.

17. Continue with the standard process procedure for completion of process.

    Distill the charge in S-9 until a wet dip of 32 inches is obtained for drumming.