

FOR DU PONT USE ONLY

DATE January 23, 1976TEST CONCLUSION NO. TA-C- 75-130OFFICIAL PRODUCT NAME 1-Bromo-2,4,6-triethylbenzene CrudePRODUCT CODE NO. 770-113-020 POUNDS PRODUCT ACTUAL 9775 (100%)AREA "Ponsol" BUILDING NAME ColorsTITLE Modification of process to utilize carbon tetra-
chloride in full drum amounts.PREPARED BY: D. J. Vrencur

PURPOSE OF TEST: To simplify operating procedures by eliminating the charging of partial drums of carbon tetrachloride. A laboratory run incorporating the proposed changes showed no difference in yield or quality.

RESULTS OF TEST: Eight charges were made via the T.A. procedure. Yield (100% basis, before distillation) averaged 85% of standard. Reason for 85% is not known. When new production is scheduled, attention to this will be given.

RECOMMENDATIONS: Do not adopt the T.A. procedure.

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APPROVED BY:

W. L. Seal 1/27/76
SR. SUPV. MANUFACTURING DATE
M. L. Runko 1/28/76
CHIEF SUPV. MANUFACTURING DATE

J. L. Lancia 26 Jan 76
SMT./CH. SUPV. TECH./PLT. ASSIST. DATE
NOT REQUIRED.
QUALITY CONTROL OR ANALYTICAL* DATE

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MANUFACTURING MANAGER* DATE
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MARK "NOT REQUIRED" WHERE APPLICABLE

MO-1755 (Rev. 3/75)

DUP050467759

FOR COMPANY USE ONLY

DATE February 6, 1975TEST AUTHORIZATION NO. TA-C- 75-130

PROCESS FILE

OFFICIAL PRODUCT NAME 1-Bromo-2,4,6-triethylbenzene CrudePRODUCT CODE NO. 770-113-020 POUNDS PRODUCT PROPOSED 5800 lbs.AREA "Ponsol" BUILDING NAME ColorsTITLE Modification of process to utilize carbon tetra-
chloride in full drum amounts.PREPARED BY: D. J. Vrencur *DPV* RESPONSIBLE FOR TEST
AND TEST CONCLUSION: D. J. Vrencur
A. H. Pagano

PURPOSE OF TEST: To simplify operating procedures by eliminating the charging of partial drums of carbon tetrachloride. A laboratory run incorporating the proposed changes showed no difference in yield or quality.

DURATION OF TEST: Four charges.STANDARD INVOLVED: CWP 770-113-020 dated October 8, 1971.ANALYTICAL CONTROL: ☒ No change ☐ Change described under Test DetailsTECHNICAL BASIS FOR CHANGE: OCNB 2900-68.TEST DETAILS:
(Include Safety and Waste Disposal) See attached.SAFETY: Improved by minimizing potential exposure to carbon tetrachloride.WASTE DISPOSAL: The amount of carbon tetrachloride is increased from 2020 lbs. per charge to 2100 lbs. per charge.

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A. H. Pagano

R. T. McNally

APPROVED BY:

CHIEF SUPVR., OPERATIONS

DATE

CH. SUPVR., TECH. OR PLT. ASSIST.

DATE

SUPERINTENDENT, ANALYTICAL (When Required)

DATE

SUPERINTENDENT, DYE QUALITY CONTROL (When Req'd)

DATE

AUTHORIZED BY:

SUPERINTENDENT, MANUFACTURING (Or Delegates)

DATE

SUPERINTENDENT, TECHNICAL (If Any)

DATE

DUP050467760

N33002.01

Test Details:

Replace Steps 1 to 5 in the Chambers Works process with the following:

Process Steps and Limits

1. Charge the 500-gallon enamel-lined reaction kettle with 1,450 lbs. of 1,3,5-triethylbenzene (TEB) and 700 lbs. of carbon tetrachloride. Agitate and cool this mixture to 0°C.

The vessel must be vented to the fume-scrubber box.

Carbon tetrachloride is used as a diluent to minimize formation of by-products.

2. Into a measuring tank, charge 700 lbs. of carbon tetrachloride, add 683 lbs. of bromine and mix these materials.

Suck the solvent into the tank and then blow the bromine into it with nitrogen pressure.

3. Run this mixture into the reaction kettle over a 2- to 3-hour period, keeping the temperature of the bromination mass in the reactor at 0-5°C.

If fuming starts or if the temperature rises, slow down the addition of the bromine--carbon tetrachloride mixture. Vent fumes through a reflux condenser to minimize solvent loss.

4. When the mixture has been added, prepare a second mix in the measuring tank of 700 lbs. of carbon tetrachloride and 683 lbs. of bromine, as before.
5. Add this mixture to the reaction kettle over a 2- to 3-hour period, keeping the temperature at 0-5°C.

Two mixes are made up because available measuring tanks are too small for one mix. Technically, this is not necessary.