

FOR COMPANY USE ONLY

DATE July 15, 1974TEST CONCLUSION NO. TA-C- 72-261OFFICIAL PRODUCT NAME 2-Chloro-4-Nitroaniline (OCPNA)PRODUCT CODE NO. 142 160 POUNDS PRODUCT ACTUAL Approx. 500AREA "Ponsol" BUILDING NAME Bldg. 743 - "Ponsol" Dryer No. 2TITLE Spray Drying FeasibilityPREPARED BY: R. C. Franklin, A. C. Stein

PURPOSE OF TEST: To demonstrate the feasibility of spray drying OCPNA wet cake in the new Proctor & Schwartz dryer. A Whirlpool hopper will be used to prepare the feed slurry since TA-C-70-901 was unsuccessful due to poor homogenization of the feed slurry and subsequent plugging of piping and nozzles.

RESULTS OF TEST: Spray drying of OCPNA (Chg. 1) was carried out in the Proctor & Schwartz dryer using an internal mix P&S paste nozzle in April, 1972, according to the procedures of this TA. Slurries prepared in the Whirlpool hopper were pumped satisfactorily to the dryer, but problems were encountered with some plastering on the walls and build-up at the discharge valve. About 500 lb. of marginal quality OCPNA was collected before discontinuing the experiment. Attempts to vary drying conditions were not successful in eliminating the problems.

Data on the feed slurry and product are attached.

RECOMMENDATIONS:

Carry out further studies on spray drying OCPNA in "Ponsol" Dryer No. 1, and with different types of nozzles in No. 2 (i.e., pressure, 2 fluid, 3 fluid).

DISTRIBUTION:

J. F. Laucius

R. F. Stalzer

C. J. Slentz

R. J. Pohl

R. H. Terss

Jackson Lab. Files

C. H. Lake

T. E. Lewis

M. S. Moores

J. A. Moffett

D. O. Miller (3)

G. L. Porter

J. A. Oscar

D. Putnam (3)

R. C. Franklin

A. C. Stein

J. J. Meyer

M. L. Reinhart

APPROVED BY:

R. W. Anderson
CHIEF SUPERVISOR, OPERATIONS7/23/74
DATEJ. F. Laucius 22 July 74
CH. SUPVR., TECH. OR PLANT ASSIST.

DATE

AUTHORIZED BY:

S. J. [Signature]
SUPT., MANUFACTURING (OR DELEGATES)

DATE

[Signature] for R. H. Terss 7/24/74
SUPT., TECHNICAL (IF ANY)

DATE

TC-C-72-261

OCPNA Slurry from Whirlpool Hopper

Solids - 22%

Wet screen tests - 0.45%* retained on 20 mesh
- 1.14%* retained on 40 mesh

* Based on 100% OCPNA.

Product collected (approx. 500 lb.)

Appearance - brownish yellow powder (normally
bright yellow).

Moisture - nil

Freezing point - 107.0°C (Specif. 106.8°C)

Purity - 99.8% (Spec. 99.0%)

Diazo insolubles - 1.14% vs. 0.58% on a control sample.

Material Collected from Walls

Appearance - reddish brown, slightly tacky

Moisture - nil

Freezing point - 107.0°C

Purity - 99.1%

Diazo insolubles - not determined because of tackiness.

7/15/74

/grg

FOR COMPANY USE ONLY

File Copy

DATE April 3, 1972

TEST AUTHORIZATION NO. TA-C-72-261

OFFICIAL PRODUCT NAME 2-Chloro-4-nitroaniline (OCPNA)

PRODUCT CODE NO. 142 160 POUNDS PRODUCT PROPOSED Up to 5,000 lbs.

AREA "Ponsol" BUILDING NAME Bldg. 743 - "Ponsol" Dryer No. 2

TITLE Spray drying feasibility.

PREPARED BY: L. C. Hale *LC/H* RESPONSIBLE FOR TEST AND TEST CONCLUSION: L. C. Hale, A. C. Stein, R. C. Franklin

PURPOSE OF TEST: To demonstrate the feasibility of spray drying OCPNA wet cake in the new Proctor & Schwartz dryer. A Whirlpool hopper will be used to prepare the feed slurry since TA-C-70-901 was unsuccessful due to poor homogenization of the feed slurry and subsequent plugging of piping and nozzles.

DURATION OF TEST: One to three charges.

STANDARD INVOLVED: Chambers Works process for 2-Chloro-4-nitroaniline dated May 23, 1966.

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

TECHNICAL BASIS FOR CHANGE: TA-C-70-901 dated January 13, 1972.

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

Safety:

- No unusual safety hazards are expected.
- Refer to TA-C-70-901 for dust explosion properties of OCPNA. No explosion hazard exists.
- OCPNA is an aromatic amine that may cause cyanosis. Avoid contact with the skin or inhalation of the dust.

Waste Disposal:

1. Dust Emission - A negligible amount of dry product (~ .004 lb./100 lb. of produced product) is expected to be released to the atmosphere as a yellow dust.
2. Liquid Wastes - Two streams are involved:
 - (a) Dust scrubber liquor - This stream will contain approximately 0.4 lb. of OCPNA/100 lb. dry product produced + 3000 lbs. of water.
 - (b) Wash water used to clean tanks, dryer, dust collector, Whirlpool hopper, etc.

DISTRIBUTION:

J. Blackwell	A. J. Edison	J. F. Laucius	M. S. Moores	R. Wiederhorn
R. J. Pohl	R. C. Franklin	J. J. Meyer) In	G. L. Porter	
J. L. Files	J. F. Froning	C. J. Slentz) Turn	D. Putnam (3)	Bob Hollands
H. P. Landerl	R. T. Gilsdorf	R. T. McNally) In	R. B. Root	Ernie Porter
A. S. Brookes, Jr.	L. C. Hale	D. M. Dietz) Turn	R. F. Stalzer	3458
J. E. Cole, Jr. (3)	R. H. Kasch	D. O. Miller	A. C. Stein	
J. H. DeYoung	D. B. Kirk	J. A. Moffett	R. H. Terss	

APPROVED BY:

CHIEF SUPVR., OPERATIONS DATE 4/14/72 CH. SUPVR., TECH. OR PLT. ASSIST. DATE 4/14/72

SUPERINTENDENT, ANALYTICAL (When Required) DATE

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

AUTHORIZED BY: *[Signature]* 4/14/72
SUPERINTENDENT, MANUFACTURING (Or Delegates) DATE

[Signature] 4/14/72
SUPERINTENDENT, Dye Modernization DATE

DUP050467917

N33045.01

Page 2

TA-C-72-261

2-Chloro-4-nitroaniline (OCPNA)

Test Details:

1. The dryer feed slurry will be prepared in a Whirlpool hopper by Dye Modernization - Facilities personnel. Depending upon the solids content of the wet material, a sufficient quantity of water to produce a 20-25% solids slurry should be added to the hopper. This will yield a slurry with a viscosity of ~ 10,000 cp. The mass will be deagglomerated by high pressure drop (~ 80 psig) pumping across a valve such that 100% of the slurry will pass through a 20 mesh screen.
2. The OCPNA slurry will be pumped from the hopper by a Moyno pump and be fed through a 15 mesh riddle.

Note: This riddle will be unnecessary when a dis-integration system is available. Its primary purpose is to screen tramp material and agglomerates.

3. Spray dry the charge in D-10 using the paste nozzle consisting of a 1" diameter slurry tube and a 1/2" nozzle diameter at the following initial conditions:

Steam pressure = 70 psig

Ratio setting $\left(\frac{\text{lb. steam/hr.}}{2 \text{ gpm slurry} \times 10^2} \right) = 0.9$

Outlet temperature = 175°F. (80°C.)

FN-11 damper setting = 50%

A satisfactory product (> 99% purity, < 0.1% moisture) was obtained in the Azo dryer under TA-C-70-901 at 482°F. (250°C.) inlet and 175°F. (80°C.) outlet temperatures.

4. Adjust the outlet temperatures to maintain product moisture below 0.5%.
 - Any of the initial conditions should be adjusted at the discretion of spray dryer supervision.
5. Send a one-pint composite sample of each 10 drums or of each significant change in drying conditions to G.A.L. for determination of product moisture, purity and freezing point. Label samples "TOXIC". Send a similar sample to R. C. Franklin, Azo Laboratory for evaluation of diazotization properties.
6. Toward the last hour of the run, rinse the feed tank walls with water and dry the resultant dilute slurry.

Page 3

TA-C-72-261

2-Chloro-4-nitroaniline (OCPNA)

7. Clean and shut down the system per spray dryer supervision's instructions.
8. Weigh and label drums: OCPNA Spray Dried
TA-C-72-261
Toxic