

MONSANTO COMPANY
ORGANIC CHEMICALS DIVISION
W.G.K. PLANT

STANDARD

FOR

Supersedes: Process for Manufacture of Crude
2,4-Dichloro, dated April, 1965.

Prepared by Paul A. Erzer

Approved by EDM. John N. Calmar

E. R. Malone 8-14-68

M. A. Terpstra 8/23/68

H. C. Carder 1/2/69

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Issued: NOV.
September, 1968

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K031098

MONSANTO COMPANY
ORGANIC CHEMICALS DIVISION
W.G.KRUMMRICH PLANT

STANDARD MANUFACTURING PROCESS FOR
CRUDE 2,4 - DICHLOROPHENOL

DEPARTMENT 236

Supersedes: Process Description for Manufacture of Crude
2,4-Dichlorophenol, dated April, 1965.

Prepared by Paul A. Erzer

Approved by E. P. Malone 8-14-68

M. A. Terpstra 8/27/68

H. C. Carder 1/3/69

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AMENDING RECORD SHEET

K031101

Dept. 236 - Process Description

CRUDE 2,4 DICHLOROPHENOL

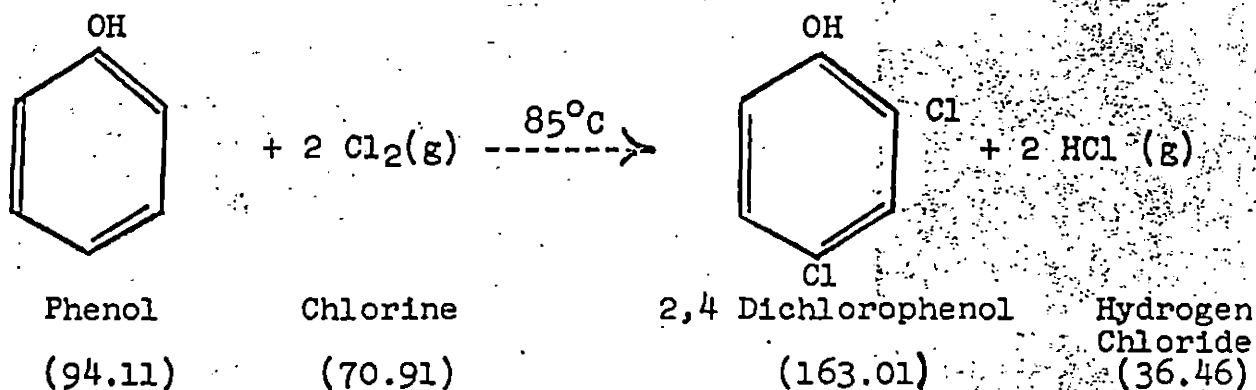
SECTION I. - SYNOPSIS OF THE PROCESS

Crude 2,4 Dichlorophenol is produced by the batchwise chlorination of phenol and high parachlorophenol content fractions (minimum ratio of phenol to ortho of 4:1) from Dept. 237 using swift gas chlorine at a pressure of 2-4 psig. The chlorination is carried out between 70-90°C. to a specific gravity of 1.400-1.404. The finished product contains approximately 88% 2,4-Dichlorophenol, 7.5% 2,6-Dichlorophenol, 2.5% 2,4,6-Trichlorophenol and 2% Monochlorophenol. The chlorinated product is transferred to a storage tank to await loading into tank cars.

The product is used exclusively for production of 2,4-Dichlorophenoxyacetic acid (2,4-D.) in Department 262.

By-product HCl gas is scrubbed with phenol, cooled to condense out chlorophenol vapor, and scrubbed with sulfuric acid. The HCl gas is then either transferred directly to Dept. 217 by pipeline or absorbed in water and sewerred. HCl gas is used in Dept. 217 for the manufacture of Chlorosulfonic Acid.

The basic reaction for this process is as follows:



YIELDS:

The theoretical usage of phenol is calculated as follows:

$$\frac{\text{Theo. Lbs. of Phenol Used}}{\text{Cwt. 2,4-DCP Produced}} = \frac{94.11}{163.01} \times 100 = \underline{57.73} \text{ lbs./cwt.}$$

The standard yield on phenol has been established as 96.99% of theory. The standard usage of phenol is calculated as follows:

$$\frac{\text{Std. Lbs. of Phenol Used}}{\text{Cwt. 2,4-DCP Produced}} = \frac{57.73}{0.9699} = \underline{59.52} \text{ Lbs./cwt.}$$

SECTION I - SYNOPSIS OF THE PROCESS

YIELDS: (contd.)

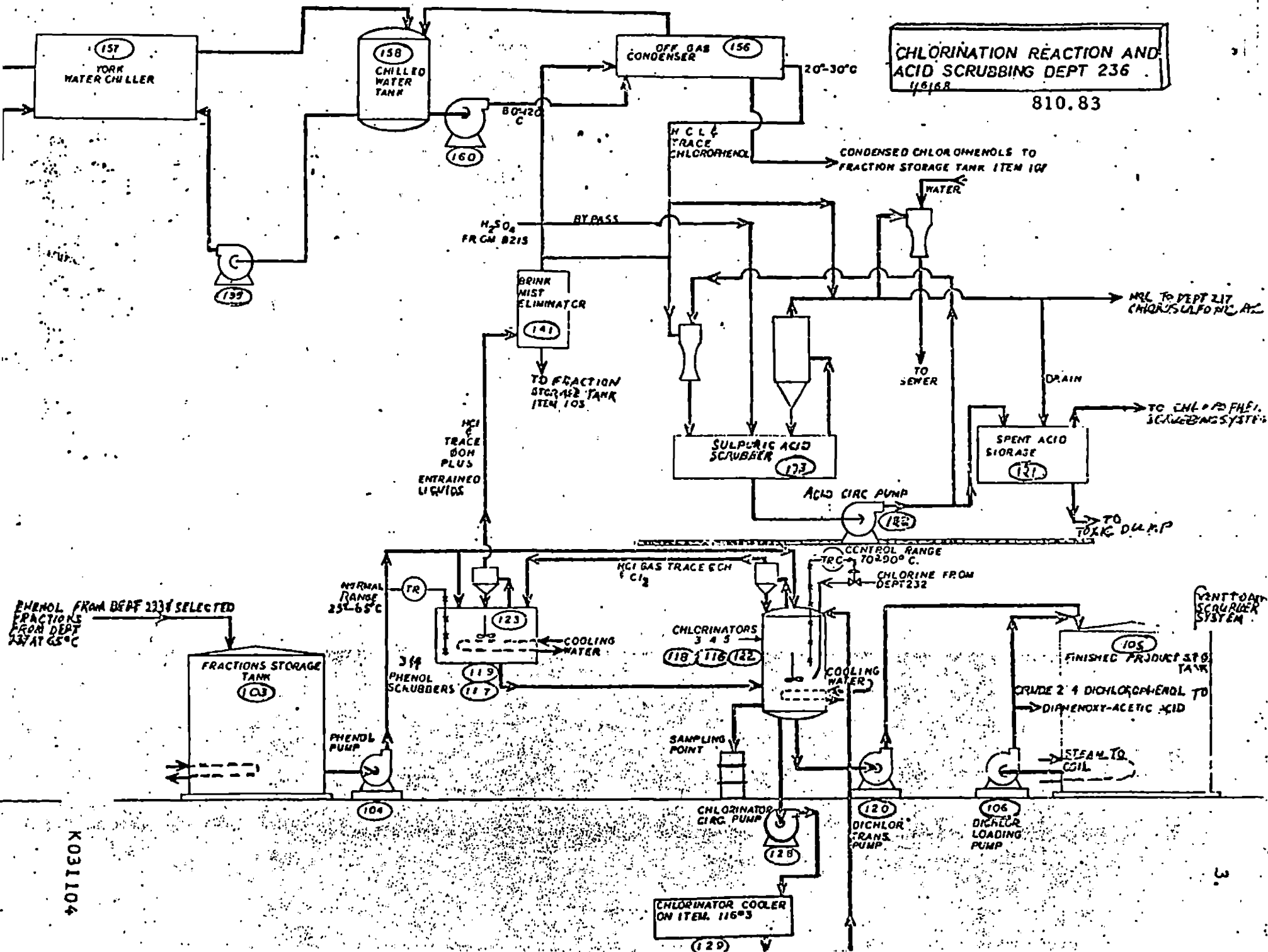
The theoretical usage of chlorine is calculated as follows:

$$\frac{\text{Theo. Lbs. of Chlorine Used}}{\text{Cwt. 2,4-DCP Produced}} = \frac{(2)(70.91)}{163.01} \times 100 = \underline{87.01 \text{ Lbs./cwt}}$$

The standard yield on chlorine has been established as 95.60% of theory.

The standard usage of chlorine is calculated as follows:

$$\frac{\text{Std. Lbs. of Chlorine Used}}{\text{Cwt. 2,4-DCP Produced}} = \frac{87.01}{0.956} = \underline{91.00 \text{ Lbs./Cwt.}}$$



K031104

Dept. 236 - Process Description

4.

CRUDE 2,4-DCP

SECTION III - PROCESS IN DETAIL

A. CHARGING CHLORINATOR AND SCRUBBER

The starting raw materials are U.S.P. Phenol plus fractions from distilled chlorophenols operation and "snift" gas chlorine. Phenol is transferred at 65°C. from the Phenol Department by steel pipe line into a 16,000 gallon steel fractions (DCP) storage tank. Fractions are transferred from Dept. 237 receivers into the Fractions (DCP) Storage Tank (Item 103). The ratio of phenol to orthochlorophenol is maintained at a 4:1 minimum in this tank. This is necessary in order to insure production of specification grade Crude DCP. Chlorine is delivered from the Chlorine Dept. directly by steel pipe line.

The batches are charged by transferring the Phenol Scrubber charge to the chlorinator. The phenol-chlorophenol fractions blend from the Fractions Storage Tank is then charged to the Phenol Scrubber to a 40" outage. Additional fractions are charged to the chlorinator to increase the quantity of material to 46" outage (#5 chlorinator) or 44-53" outage ("1,2,3 or 4 chlorinator dependent on gravity of Phenol Scrubber batch).

B. CHLORINATION

The U.S.P. Phenol is chlorinated batchwise at a rate of 1500-2500 lbs./hr. in a monel system which consists of a 1,300 gallon chlorinator and an 1,150 gallon phenol scrubber. The chlorinator is equipped with an internal cooling coil and a chlorine sparger.

In addition, #3 chlorinator is equipped with a steel shell and tube cooler. The contents of this chlorinator are continually circulated through this unit, where they are cooled with well water.

The Phenol Scrubber is a jacketed vessel equipped with a Turbo-Mixer agitator. This special agitator is used to increase the contact of the chlorinator off-gas with the contents of the Phenol Scrubber.

While charging the batch, well water is applied to the coils in the chlorinator and scrubber. After the batch is charged, chlorine flow is started. The chlorine rate is automatically controlled to give a chlorination temperature of 80-90°C. The phenol scrubber temperature is controlled at 35-45°C. by manual control of cooling water to the jacket. The chlorination is continued until the batch end-point gravity of 1.402 at 45/15.5°C. (for virgin phenol raw material) to 1.404 at 45/15.5°C. (for fractions and phenol as raw material) is reached. The specific gravity of the reaction mass is checked at two-hour intervals until the specific gravity reaches about 1.35. After this the gravity is checked more frequently.

CRUDE 2, 4 DCP

SECTION III - PROCESS IN DETAIL

C. FINISHED BATCH TRANSFER

At the end of the chlorination, the chlorinator batch is transferred to the 20,000 gallon steel Crude DCP storage tank (Item 105). From here the material is loaded into tank cars for transfer to the 2, 4-D Acid Dept. (Dept. 262).

D. HCl HANDLING

Throughout the chlorination, the by-product HCl from the chlorination process is scrubbed with phenol in the phenol scrubber by means of a draft tube and agitator. Here, unreacted chlorine from the chlorinator is removed by reaction with the phenol. Temperatures of 35 - 45°C. are maintained during this exothermic reaction by controlling the well water flow to the jacket.

Entrained chlorophenols in the cooled HCl off-gas leaving the scrubber are recovered in a Brink Mist Eliminator and returned to the Penta-chlorophenol process. The HCl off-gas is then cooled in the Monel off-gas condenser (Item 156) to a temperature of 20 - 30°C. The chlorophenols remaining in the HCl gas are removed by scrubbing with sulfuric acid in a Venturi contactor. The virgin sulfuric acid has a 98% assay. As the sulfuric acid is contacted with sulfuric acid, its assay and reactivity decrease and its corrosiveness increases. The sulfuric acid is transferred to the spent acid storage tank when its assay is reduced to 85% free H_2SO_4 . From here the spent acid is loaded into tank trucks for shipment to the toxic dump. One 8,000 gallon tank car of 98% H_2SO_4 is used every 3-1/2 to 4-1/2 weeks when scrubbing HCl from the Penta-chlorophenol, Crude DCP, and Crude Parachlorophenol (Dept. 237) chlorinations.

The cleaned HCl is piped to Dept. 217 for use in the production of chlorosulfonic acid. Excess HCl is absorbed in water at a drowning jet and sewer. Pressure drop through this system results in the chlorinator and phenol scrubber operating under 2 - 4 psig back pressure. The chlorinators are equipped with a 20 psig safety rupture disc. The phenol scrubbers are equipped with a 10 psig safety rupture disc.

CRUDE 2, 4-DCP

SECTION IV - COMMENTS ON PROCESS

A. RAW MATERIAL YIELDS

1. Phenol and Chlorine Yield

a) Chlorinator and Scrubber Entrainment

The Department 237 chlorinator (#5) is equipped with an improved 3-ring sparger. When this reactor is used for Crude DCP manufacture, the chlorine flow to the top ring is shut off when batch gravity reaches 1.300. This is necessary in order to reduce the carry-over of entrainment to and out of the phenol scrubber and the resultant yield loss. At present the Dept. 236 Chlorinator (#3) which is used primarily for Crude DCP, is not equipped with the improved sparger. This makes it unnecessary to use the procedure used on #5 Chlorinator on the #3 unit.

The following data shows the difference in losses when the chlorine flow to the top ring is shut off at a batch specific gravity of 1.300:

Total Loss of Equivalent Phenol
from #5 Chlorinator & Scrubber

Shutting off chlorine flow to top
ring at 1.300 batch specific gravity. 73 lbs./batch

Maintaining chlorine flow to all
sparger rings for entire batch. 772 lbs./batch

The table above is actual data based on material balance losses over 3-month periods, using operator stick measurements.

b) Vapor Losses from Chlorinator and Scrubber

In order to minimize vapor losses from the chlorinator and scrubber, it is necessary to control their temperatures at 90°C. and 50°C. respectively or less. If the temperatures increase above these maxima, vapor losses rise significantly. Normal total material balance losses of equivalent phenol from the chlorinator and scrubber range from 0 - 100 lbs./batch.

2. By-Product Hydrogen Chloride Gas Yield

K031107

The recovery of chlorophenol HCl gas is a function of the chlorosulfon acid department's ability to match their demand with the chlorophenol HCl gas generation rate. Major losses of this gas are incurred when it is necessary to drown and sewer the gas.

Pluggage of one or both HCl transmission lines with chlorophenols can result in major losses of HCl gas due to drowning and sewerage. The pressure drop for a flow of 450 CFM through one line should be in the 20-30 inch H₂O range. The pressure drops through the two trans-

CRUDE 2,4-DCP

SECTION IV - COMMENTS ON PROCESS (contd.)

2. By-Product Hydrogen Chloride Gas Yield ()

Improper setting of the Dept. 236 control valve to the HCl drowning jet can cause significant losses of HCl. This valve should be set to open when the HCl pressure in the transmission lines reaches 35 in H₂O. A lower setting will result in increased HCl losses. A higher setting may result in flow of chlorophenol HCl to Dept. 218 with the resultant chlorophenol contamination of their food grade muriatic acid production.

Leakage of HCl gas through the Dept. 236 control valve to the HCl drowning jet can also result in high losses. This can be checked by shutting off water to the jet when the valve position indicator shows the valve is closed. A blind flange on the catch tank (for the drowning jet discharge) should be removed to determine whether HCl is leaking through.

Chlorine assay also affects HCl recovery. The lower the chlorine assay (due to greater concentration of air) the lower the HCl recovery. This is because the HCl transmission lines are flow limiting at high HCl generation rates. Chlorine assay normally runs in the 80-90% range. This is based on measurements made in 1967 in Dept. 236, with a continuous in-line chlorine analyzer.

3. Sulfuric Acid Yield

The primary factor affecting sulfuric acid usage is operation of the HCl off-gas condenser. With the condenser out of operation the usage is about 8.0 lbs./cwt. Molten Penta and Crude DCP. With the condenser in operation, the usage is about 2.0 lbs./cwt. Molten Penta and Crude DCP. These usages are based on a 20-25°C. temperature of the gases leaving the condenser.

B. CAPACITY

Capacity of the Crude DCP chlorinators is a function of cooling capacity and chlorine flow rate.

The capacity limiting variable for the #5 chlorinator under normal conditions is chlorine flow. Under present conditions, with a clean chlorinator cooler, the chlorination time should be about 4 hours.

The capacity limiting variable for the #3 chlorinator is also chlorine flow. Under present conditions, the chlorination time should be about 5.5 hrs. However batches have been run with a 4-hour chlorination time, when the chlorine flow was adequate.

Dept. 236 - Process Description

8.

CRUDE 2,4-DCP

SECTION IV - COMMENTS ON PROCESS (contd.)

B. CAPACITY (contd.)

The capacity limiting variable for the #1, #2, or #4 chlorinator for Crude DCP is cooling capacity.

C. QUALITY

The primary quality consideration for Crude DCP is the 2,4-Dichlorophenol assay.

1. Chlorine Sparger Design

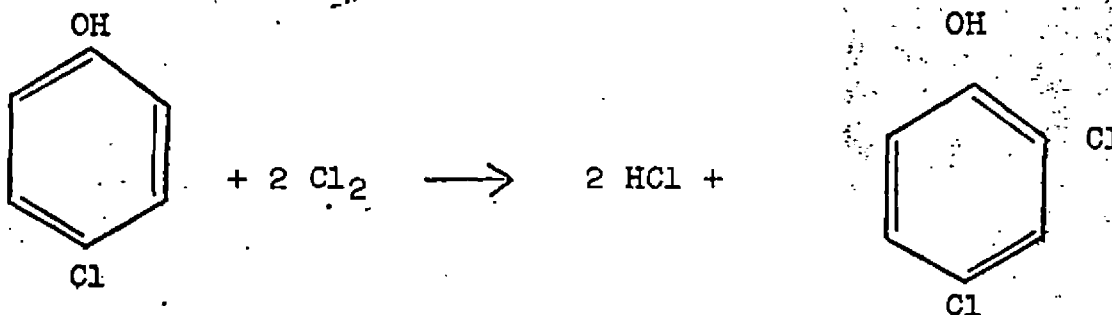
The improved sparger installed in the Dept. 237 chlorinator (#5) made possible an increase of 1-1½% in the Crude DCP assay (from 87.5% to 88.5-89%). Work has been initiated to install a similar sparger in the #3 chlorinator in Dept. 236.

2. Raw Material Composition

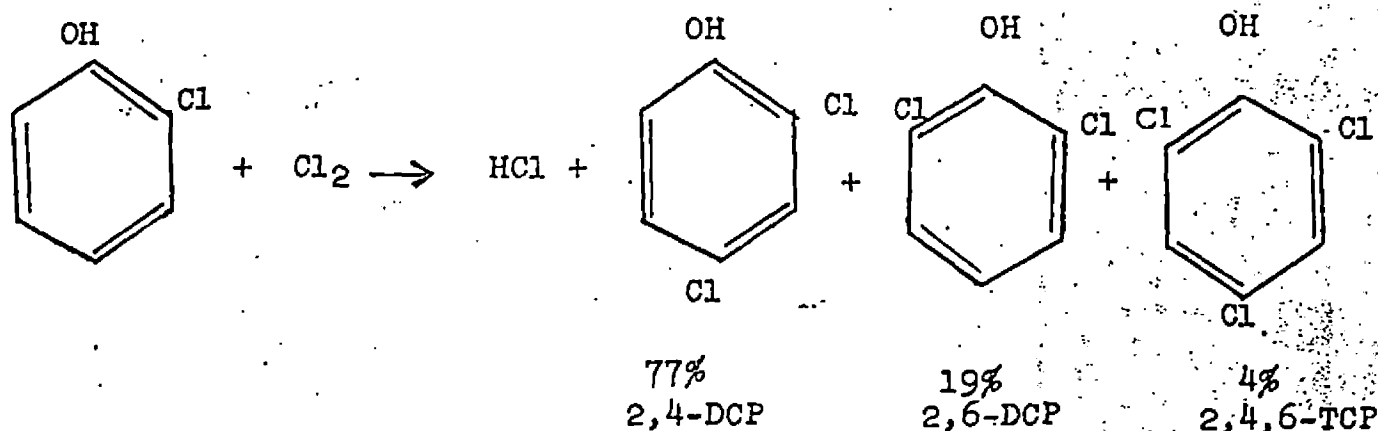
The present specification for Crude DCP calls for an 87.0% minimum assay.

The chemistry of the Crude DCP chlorinations is as follows:

- a) Parachlorophenol chlorinated to the Crude DCP stage gives 100% 2,4-Dichlorophenol. The mechanism for this reaction is as follows:

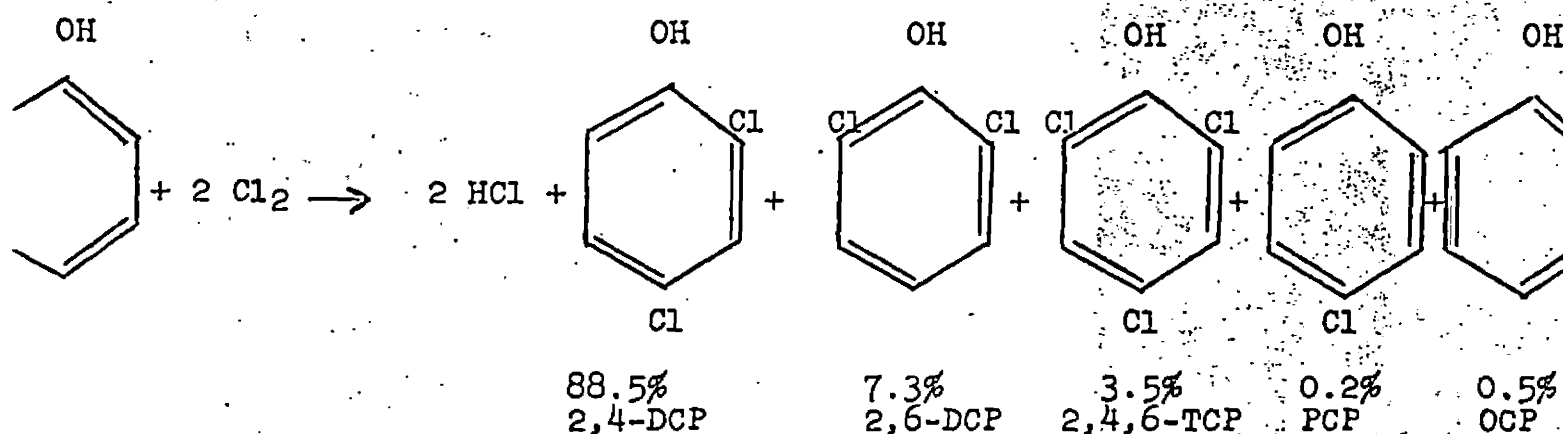


- b) Orthochlorophenol chlorinator to the Crude DCP stage (1.400-1.404 S.G.) gives material having the following composition:

CRUDE 2,4-DCPSECTION IV - COMMENTS ON PROCESS (contd.)c. QUALITY (contd.)

(equation not balanced)

- c) Phenol chlorinated to the Crude DCP stage yields material having the following composition:



Based on this work and actual experience, the following operating guidelines have been set:

- Fractions from the Parachlorophenol refining operation can be used as raw material for Crude DCP if they are blended with phenol to maintain a 4:1 phenol-ortho-chlorophenol weight ratio.
- The last 50% of the dry fraction cut from the Parachlorophenol refining operation can be used as raw material for producing specification grade Crude DCP, without blending with phenol. This is possible because of the high parachlorophenol content of these fractions.

CRUDE 2,4-DCP

SECTION IV - COMMENTS ON PROCESS (contd.)

C. QUALITY (contd.)

- c) Fractions from the Refined DCP refining operation cannot be used as raw material for producing specification grade Crude DCP.

3. Water Content of Raw Material

The present maximum control limit for water content of raw material used for Crude DCP is 0.1% H₂O. An increase in water content to 0.15% will result in a rise of approximately 0.5% in the 2,6-Dichlorophenol content of the finished Crude DCP. An increase in water content to 0.20% will result in about a 1% rise in the 2,6-DCP content of the finished product. This can be a good indicator of a cooling coil or cooler leak in the Crude DCP chlorinator.

4. Degree of Chlorination

Both over and under chlorination can result in the production of low assay Crude DCP. At the present time the endpoint control points for Crude DCP specific gravity are 1.402 (for phenol raw material) and 1.404 (for fractions and phenol raw material) at 45/15°C.

The graph in Figure 1 indicates the effect of the degree of chlorination on Crude DCP assay.

An experimental in-line Infra-Red (IR) analyzer has been installed on the #5 chlorinator for use in determining Monochlorophenol and Crude DCP endpoints (in place of the present specific gravity method). Successful use of this unit will result in a 0.5-1.0% increase in the average Crude DCP assay. The IR analyzer will enable chlorination termination at the maximum assay on each batch.

5. HCl Off-Gas Quality

HCl off-gas quality is a function of the quantity of chlorophenol remaining in the gas when it is transmitted to Dept. 217. This, in turn, is dependent on the efficiencies of the Phenol Scrubber, the Brinks Demister, the HCl Off-Gas Condenser, and the H₂SO₄ Scrubbing System. Since the total efficiency of these units approaches, but does not equal 100%, the HCl contains a small quantity of chlorophenols. For this reason, chlorophenol HCl gas cannot be used for making food grade muriatic acid in Dept. 218. Chlorophenol HCl gas is used only in the production of Chlorosulfonic Acid in Dept. 217.

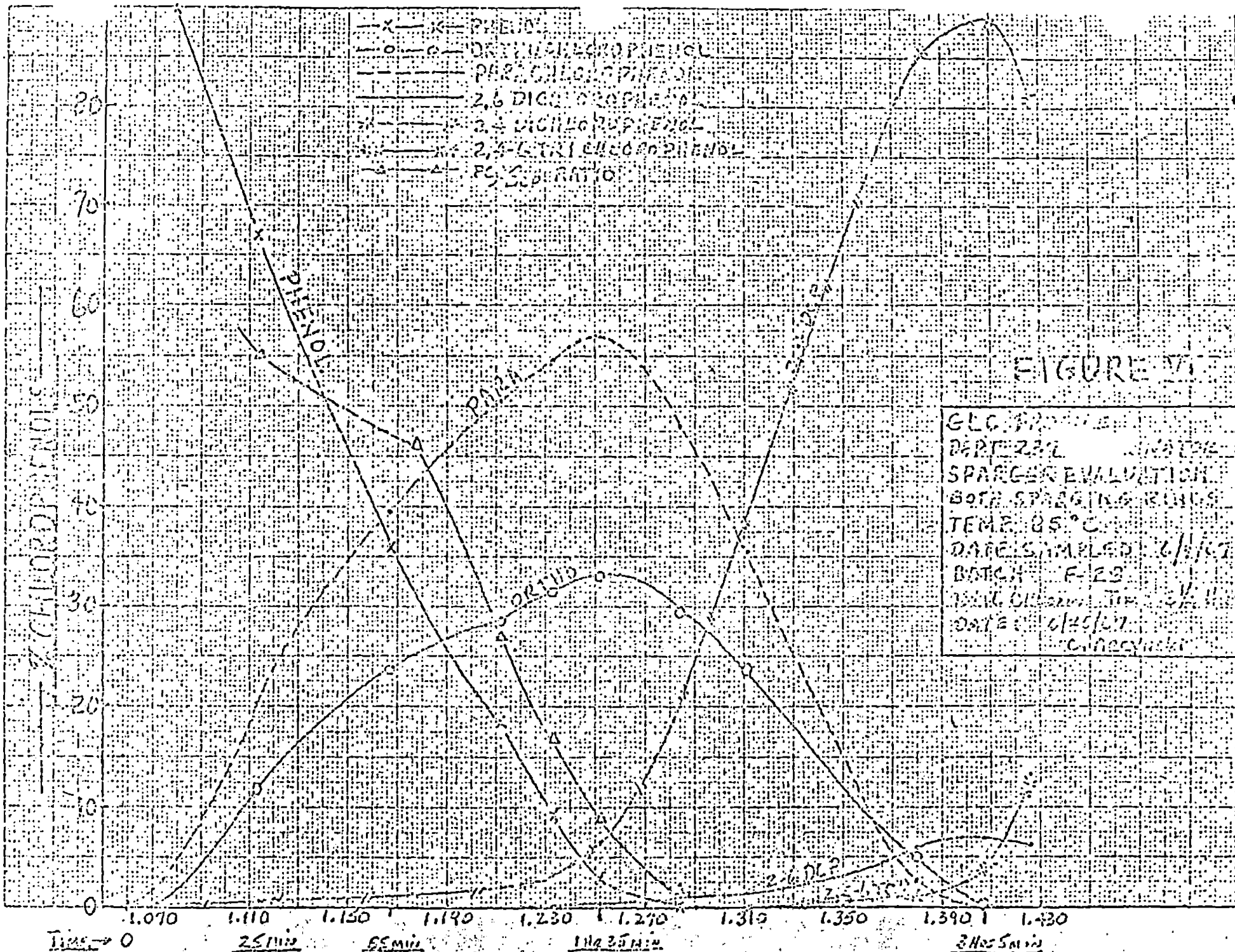


FIGURE VI

GLC PROFILE
 DEPT 232
 SPARGER EVALUATION
 BOTH SPARGING RINGS
 TEMP 45°C
 DATE SAMPLED 6/1/67
 BATCH F-23
 VAL. OF 232 100%
 DATE 6/25/67
 C. H. H. H.

Sp Gr. n 45°C

CRUDE 2,4-DCPAPPENDIX A - EQUIPMENT LIST

Both Crude DCP and Molten Pentachlorophenol are manufactured in the Dept. 236 equipment center. The number of Dept. 236 chlorinators used for Crude DCP manufacture varies depending on Crude DCP demand and refined chlorophenols production. The equipment list below is typical of the equipment used and shared by Crude DCP.

<u>NAME</u>	<u>ITEM NO.</u>	<u>EQUIPMENT NUMBER</u>	<u>EQUIPMENT DESCRIPTION</u>
Chlorophenol Fractions (Crude DCP) Storage Tank	103	21-3491	13' I.D. x 16' High 16,000 Gal. uninsulated vertical, steel storage tank. Tank equipped with heating coil.
Chlorophenol Fractions Tank Pump	104		
#3 Chlorinator	116	21-2015	4'-9" I.D. x 10' High 1300 Gal. vertical insulated monel tank, equipped with a monel cooling coil (area-133 ft ² .)
#4 Chlorinator	118	21-3096	
#5 Chlorinator	122	21-3058	Same as above but without coil Vessel, located in Dept. 237.
#3 Phenol Scrubber	117	21-2016	6' I.D. x 5' high vertical, 1200 Gal. jacketed monel uninsulated tank, equipped with a Turbo-Mixer Agitator system.
#4 Phenol Scrubber	119	21-3097	
#5 Phenol Scrubber	123	21-1914	Same as above except located in Dept. 237.
#3 Chlorinator Cooler.	129	38-2751	Steel shell and tube heat exchanger. Water on tube side and process on shell side. Area 480 ft. ²
#5 Chlorinator Cooler	127	38-5714	18" O.D. x 15' long steel shell and tube heat exchanger. Water on 8-pass tube side. Process on 2-pass shell side. Area 700 ft. ²
#3 Cooler Circulating Pump	128		3" x 1½" cast iron centrifugal pump equipped with mechanical seal, 1800 RPM, 10 H.P. motor.

Dept. 236 - Process Description

13.

CRUDE 2,4-DCP

APPENDIX A - EQUIPMENT LIST (contd.)

<u>NAME</u>	<u>ITEM NO.</u>	<u>EQUIPMENT NUMBER</u>	<u>EQUIPMENT DESCRIPTION</u>
#5 Cooler Circulating Pump	124		4" x 3" ductile cast iron centrifugal pump equipped with outside mechanical seal, 1800 RPM, 25 H.P. motor. Nominal capacity.
Crude DCP Transfer Pump	120		
Crude DCP Storage Tank Pump	106		
#5 Phenol Scrubber Transfer Pump	125		
Crude DCP Neutralizing tank.	107	21-2471	6' I.D. x 7' high vertical, uninsulated 1500 gal., steel tank.
Crude DCP Storage Tank	105	21-3519	13' I.D. x 19'-6" high, insulated, vertical; 20,000 gal. steel tank. Tank equipped with nickel coil.
HCl Brink Demister			See Molten Penta SMP
Chlorine Brink Demister			"
HCl Off-Gas Condenser System			"
Sulfuric Acid Scrubbing System			"
Chlorophenol Air Scrubbing System			"

CRUDE 2,4-DCP

APPENDIX B - FINISHED GOODS AND RAW MATERIAL SPECIFICATIONS

The following raw materials are used in the process:

<u>RAW MATERIAL</u>	<u>CODE NO.</u>
Phenol	810.40
Chlorine (Snift Gas)	810.59
Sulfuric Acid (98%)	815.03

The finished Product produced in the process is:

Crude 2,4-Dichlorophenol 810.83

Specifications for the materials follow on succeeding pages:

Monsanto
ORGANIC CHEMICALS DIVISION

**FINISHED PRODUCT
SPECIFICATION**

PRODUCT PHENOL (USP)		DEPT. 223
PLANT WGK	PRODUCT CODE 81040	USE SALES
DATE EFFECTIVE 1-21-66	SALES CODE 7100-100-03-003	

APPROVALS

PRODUCTION W. J. Wilson	RESEARCH J. F. Quinn
SALES W. E. Ault	QUALITY CONTROL (Chief Chemist) W. J. Gresham

GENERAL DESCRIPTION

CHEMICAL FORMULA

MOL WT. 94.11

SUPERSEDES SPECS. OF 9/30/65 & 10/31/62	SAMPLE FOR ANALYSIS 2 x 8 oz. GGS Bottle	ISSUED BY J. P. Prader
--	---	---------------------------

CHARACTERISTIC

LIMITS

METHOD

UNRESTRICTED INFORMATION

(R) Appearance	Crystalline solid	10,516
(R) Color of Melt:		10,323
Shipped in Nickel; Stainless Steel or Lined Containers	APHA 10, max. *	
Shipped in Steel Containers	APHA 50, max. *	
(R) Foreign Odor	None	11,734
(R) Turbidity of Melt	Standard No. 0, max. (None)	10,324
(R) Crystallizing Point	40.60°C., min.	10,322
Non-Volatile Residue @ 100% max.	0.01%, max.	10,325
Solution in:		
Water (1:15)	Clear and Colorless	
10% NaOH (1:10)	Clear and Colorless	10,326
Distilling Range: **		10,329
First Drop	180.0°C., min.	
100% Range	2.0°C., max.	
50% to Dry Point	0.2°C., max.	

* At time of loading

** To be determined on one car sample each day and all car and lot samples with a crystallizing point lower than 40.65°C

K031116

RAW MATERIAL

Date Approved OCT 14 1966

For Dept. 736, 7374

Monsanto
ORGANIC CHEMICALS DIVISION

**FINISHED PRODUCT
SPECIFICATION**

PRODUCT Chlorine (Snift Gas)		232
PLANT WGK	PRODUCT CODE 81059	USE Internal
DATE EFFECTIVE 10/18/66	SALES CODE	

APPROVALS

PRODUCTION T. W. Dalton	RESEARCH M. A. Terpstra
SALES	QUALITY CONTROL (Chief Chemist) W. J. Gresham

GENERAL DESCRIPTION

CHEMICAL FORMULA

Mixture

MOL. WT.	SUPERSEDES SPEC. OF 12/23/63	SAMPLE FOR ANALYSIS 100 c.c. Gas Burette	ISSUED BY J. P. Prader
CHARACTERISTIC	LIMITS		METHOD

UNRESTRICTED INFORMATION

	Typical Analyses
Chlorine (Cl ₂)	86% By Volume
Oxygen (O ₂)	3% " "
Carbon Dioxide (CO ₂)	1.5% " "
Carbon Monoxide (CO)	0.1% " "
Hydrogen (H ₂)	1.0% " "
Inert Gasses (By difference)	8.4% " "
Moisture (H ₂ O) @ Std. Conditions	2.4 x 10 ⁻⁵ lbs./ft. ³ (Average)

Direct pipeline transfers to using Depts.

233, 236, 237, 246

This specification is for internal use at WGK Plant only.

RAW MATERIAL
Date Approved JUN 05 1968
For Dept. _____

K031118

MORGANTO CHEMICAL COMPANY
INORGANIC CHEMICALS DIVISION
FINISHED PRODUCT
SPECIFICATION

PRODUCT

Sulfuric Acid

PLANT

WGK

CODE NO.

8300-098

GRADE

98%

DATE EFFECTIVE

6/1/66

ISSUE NO.

102

SUPERSEDES

4/15/63

APPROVALS**PRODUCTION (Plant Manager)**

E. L. Boehm

RESEARCH (Asst. Director)

E. L. Moore

SALES (Sales Manager)

A. R. Handleman

QUALITY CONTROL (Chief Chemist)

W. J. Gresham

AUTHORIZED (Quality Control Director)

J. S. Metcalf

GENERAL DESCRIPTION

An essentially clear colorless viscous liquid.

CHARACTERISTICS		UNITS	TYPICAL VALUES	METHOD NO.
(R)	Appearance	Water white to light yellow liquid.		10,215
(R)	Color	150 APHA, Max.	100 APHA	10,215
(R)	Turbidity	250 APHA, Max.	60 APHA	10,216
(R)	Assay	98.0 - 99.5%	99.2%	10,217
(AI)	Nitrate as HNO_3	0.001%, Max.		12,654
(AI)	Iron as Fe	0.020%, Max.		10,219

RAW MATERIAL

Date Approved NOV 17 1966

For Dept. 223, 232

236, B 25

MONSIEUR
ORGANIC CHEMICALS DIVISION

2,4-Dichlorophenol, Crude B-Grade B-25

FINISHED PRODUCT
SPECIFICATION

PLANT WGK	PRODUCT CODE 81083	USE Dept. 262
DATE EFFECTIVE JUN 23 1965	SALES CODE Internal Use	

APPROVALS

PRODUCTION W. J. Wilson	RESEARCH R. W. Bucknell
SALES	QUALITY CONTROL (Chief Chemist) W. J. Gresham

GENERAL DESCRIPTION

CHEMICAL FORMULA

MOL WT.

SUPERSEDES SPECS. OF 12-22-64	SAMPLE FOR ANALYSIS 2 x 16 oz. GGS Bottles	ISSUED BY M. D. Dealy
CHARACTERISTIC	LIMITS	METHOD

(R) Appearance and Color	Amber Liquid or Crystalline Mass	11308
(R) 2,4-DCP Isomer	87.0%, min. (1)	12782
(R) Water	0.10%, max.	11310
(R) Sp. Gr. @45/15.5°C Anhydrous Basis	Report	11311
(R) Acidity (as HCl)	0.10%, max.	11879

(1) 2,4-DCP Isomer content will average 88% minimum.

Approximate isomeric composition of this product is:

2,4-Dichlorophenol	87 - 90%
2,6-Dichlorophenol	6 - 8%
Trichlorophenol	1 - 3%
Monochlorophenol	0 - 1%

This specification is for internal use only.

K031120

(R) Routine Analysis, All others Analyzed by request Only

Dept. 236 - Process Description

15.

CRUDE 2,4-DCP

APPENDIX C - PHYSICAL CONSTANTS

K031121

CRUDE 2,4-DCP

APPENDIX D - UTILITIES

The following types and amounts of utilities are used in the process:

<u>TYPE</u>	<u>AMOUNT</u>	<u>USE</u>
Steam (80 psig)	1000 M#/mo.	Stg. tank coils, tracers.
Electricity	40 CKWH/mo.	Pump motors, instruments, lights.
Water { Purchased)	450 M Gal./mo.	Equipment washes.
(Plant)	6000 M Gal./mo.	Chlorinator cooling coils, Phenol Scrubber cooling jackets, HCl drowning jets, floor washes.

Dept. 236 - Process Description

17.

CRUDE 2,4-DCP

APPENDIX E - PERSONNEL

The following personnel are required to maintain operations:

<u>CLASSIFICATION</u>	<u>NUMBER OF MEN</u>	<u>JOB DESCRIPTION</u>
Premium Operator	1/shift	Complete control of chlorination to include charging raw material determining chlorination endpoint air blowing finished product, transferring product to finished product storage, procurement of necessary data and samples, and operation of sulfuric acid scrub
Chief Operator	As required	Receive phenol from Dept. 223, empty and recharge sulfuric acid scrubber.
"A" Operator	As required	Receive high para fractions from Dept. 237
"B" Operator	As required	Transfer of finished product from storage to tank cars.
237 Operator	As required	Transfer high para fractions to Dept. 236.

K031123

CRUDE 2,4-DCPAPPENDIX F - PACKAGING AND SHIPPING

Crude 2,4-Dichlorophenol is packaged exclusively in tank cars for shipment to Dept. 262. The following cars are currently used for storage and shipment of the product:

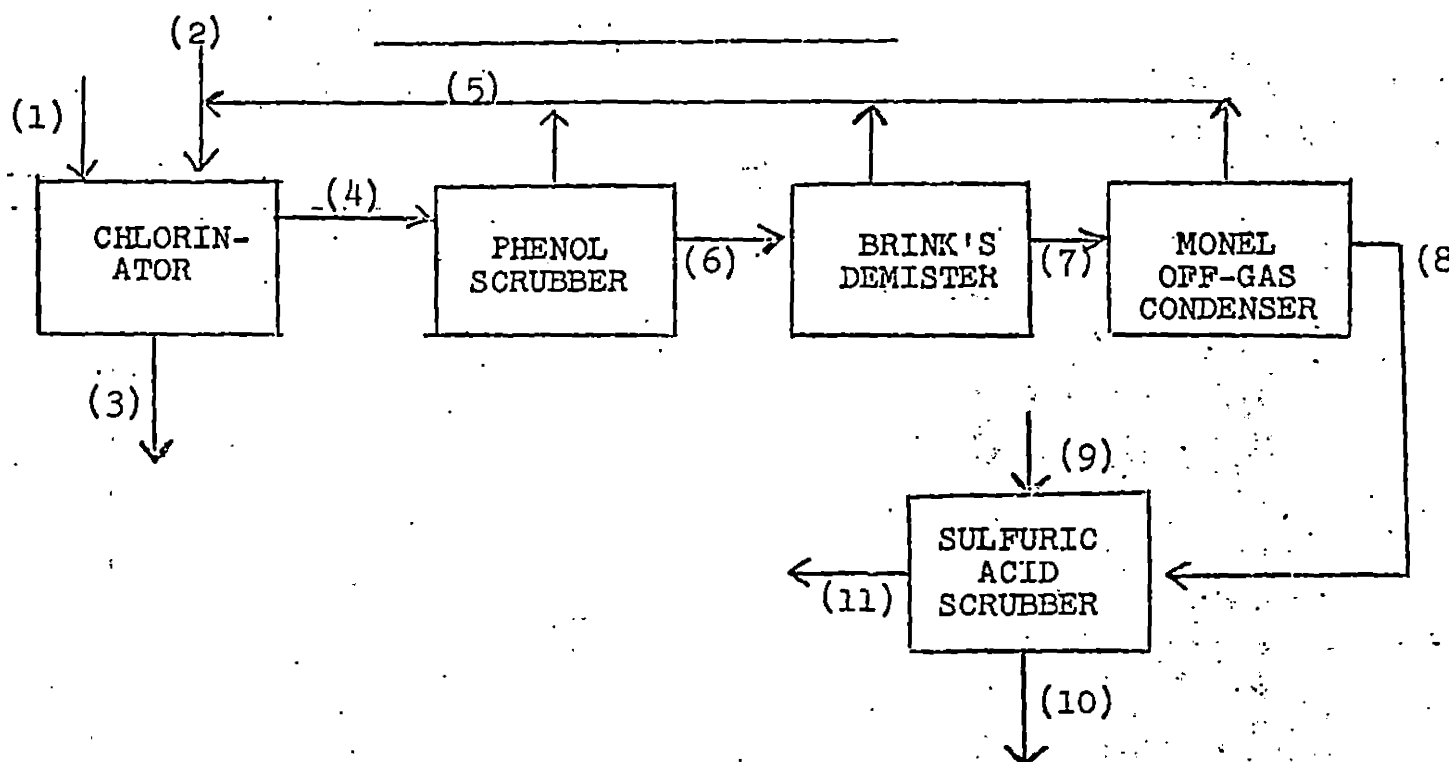
<u>CAR NUMBER</u>	<u>NOMINAL CAPACITY</u>	<u>MATERIAL OF CONSTRUCTIC</u>
GATX 95388	10,000 Gal.	Steel
NATX 18205	8,000 "	"
MONX 808	8,000 "	"
MONX 806	8,000 "	"
GATX 77006	8,000 "	"
MONX 824	8,000 "	"
NATX 18135	8,000 "	"
MONX 814	8,000 "	"
SHPX 22772	8,000 "	"
GATX 95286	10,000 "	"
SHPX 88727	10,000 "	"
NATX 18141	8,000 "	"
SHPX 23866	8,000 "	"
GATX 76573	8,000 "	"

CRUDE 2,4-DCP

APPENDIX G - MATERIAL BALANCE

The material balance is based on monthly inventory information, since 6/66.

Actual production observations were used when more formal information was not available.



Dept. 236 - Process Description

CRUDE 2,4-DCPAPPENDIX G - MATERIAL BALANCE (contd.)

RAW MATERIAL USAGES - LBS./CWT.

PROCESS STREAM RAW MATERIAL OR COMPONENT	M.W.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
		Chlorine	Phenol & Fractions	Crude DCP	HCl Off- Gas	Recovered Fractions	HCl Off- Gas	HCl Off- Gas	HCl Off- Gas	Sulfuric Acid	Spent Acid	HCl Off- Gas
Phenol	94.11		59.00			?	?	?	1.27		1.27	
Chlorine	70.90	91.00				?	?	?	3.40		2.00	1.40
H ₂ SO ₄	98.10									2.30	2.30	
2,4-DCP	163.01			88.5								
2,6-DCP	163.01			7.3								
2,4,6-TCP	197.46			3.5								
Orthochloro- phenol	128.56			0.5								
Para-chloro- phenol	120.56			0.2								
HCl	36.70				47.1		47.1	47.1	47.1			47.1
Total		91.00	59.00	100					51.77	2.30	5.57	48.

APPENDIX G - MATERIAL BALANCESUMMARY OF MATERIAL BALANCE RESULTS

<u>RAW MATERIAL</u>	<u>USAGES LBS./100 LBS. PROD.</u>		<u>MATERIAL BALANCE</u>	
	<u>THEORY</u>	<u>Material Bal.</u>	<u>Usages lbs./100 lbs. Product</u>	
1. Phenol	57.730	59.000	57.730	Process
			1.27	H ₂ SO ₄ Spent Aci
			59.000	
2. Chlorine	87.000	91.00	87.600	Process
			2.00	H ₂ SO ₄ Spent Aci
			1.4	To atmosphere
			91.000	

CRUDE 2,4 DCPAPPENDIX H - ATMOSPHERE - STREAM DISCHARGE

Summarized below are materials discharged from the Crude 2,4 DCP process to the atmosphere or sewers. The quantities listed are based on the material balance shown in the previous section. Existing problem areas are listed even though actual quantities are unknown.

Phenolic and HCl Discharge to Atmosphere	Lbs/100 lb. Product	Total lbs. per Month based on Production Rate of 1,700,000 lbs./mo.
<u>ITEM</u>		
1. Phenol storage tank vent (only phenolics)	Unknown	Unknown
2.. HCl drowning jet	Unknown	Unknown
3. Vent on DCP Storage	Unknown	Unknown
HCl and Phenolic Discharge to Sewer		
1. . a. HCl from drown- ing jet (under normal operations)	a. HCl 19.1	324,300
	b. Phenolics-trace	-----
b. HCl from drown- ing jet (when 217 shut down)	a. HCl 46.55	792,000
	b. Phenolics 0.20	3,400
2. Drain line to sewer on Dept. 217 HCl line.	Unknown	Unknown

CRUDE 2,4 DCPAPPENDIX I - PROCESS CONTROL

<u>SAMPLING POINT</u>	<u>FREQUENCY</u>	<u>REASON FOR SAMPLE</u>	<u>NORMAL RANGE</u>	<u>METHOD #</u>
1. Chlorinator	Every 2 hrs. & more toward end of batch.	Specific gravity is indication of degree of chlor- ination.	50 gravity points/hr. Finished product 1.402- 1.404 @ 45/15.5°C.	Operator test (1131
2: Tank Car	Once from every full tank car. 1-2 tank cars/day.	To compare against speci- fications for Dept. 262. (2,4-D Mfg.)		
		Appearance & Color	Amber liq. or crystalline mass.	11308
		Water	0.01-0.10%	11310
		Acidity	0.01-0.10%	11879
		2,4 DCP isomer	87.0-90.0%	12782

Process Area Or Step	Variable Or Parameter	Description of Parameter As It Affects Safety, Quality, Yield Rate Or Other	Range For Best Known Process Performance	Frequency Of Monitoring By Supervision	Measurement of Variable			Interaction With Other Variables	How Control Variables	Comments
					Frequency	Method	Accuracy			
C. CHLORINATION	d. Scrubber Off-Gas Temperature	Affects yield.	35 - 50°C	Hourly by operator (Not Logged)	Continuous	Temperature Recorder	± 1°C	High temperature indicates entrainment, hot scrubber, undercharged scrubber, or hole in draft tube.	Lower scrubber temperature by increasing cooling and reduce chlorine rate if needed. Check that scrubber is not undercharged.	
	e. Cooling Water	Affects rate, yields, quality.	Full water on coil if running w/o exchanger. Full water on exchanger if using #3 chlorinator	Hourly by operator (Not Logged)	Continuous	Valve position with exchanger. Flow indicator with coil.	Unknown	Chlorine added automatically to maintain temperature. Water rate determines chlorine rate and consequently process rate. High flow rates can cause excessive entrainment.	Maintain flow rate in range at maximum permissible by entrainment. Do not use coil when using external heat exchanger.	The need to reduce indicates separator error, chlorinator charge, or chlorine rate
	f. Entrainment	Affects quality, rate.	Low entrainment as defined by operator and foreman experience.	Hourly by operator	Continuous	Site glass		High entrainment can cause poor quality on next batch. Accompanied by high chlor. off gas temperature, and scrubber temp. Results from plugged separator, or too fast gas addition.	Control process within ranges. Determine deviation or problem if entrainment occurs. Reduce rate if necessary.	
	2. Batch Data									
	a. Batch Gravity	Indicates proper level to achieve quality	1.400-1.403 at 70°C	Final batch gravity by operator	Every hour until approach end point and then as needed	Operator gravity analysis	± 0.002	Low gravities mean low 2,4 assay and an excess monochloro. High gravities mean a trichlor excess, again with low 2,4 assay.	Take adequate hydrometer reading near end of batch to be able to control in range. Stop chlorination while analyzing if near end point.	
	b. Assay	Quality	87-90% 2,4DCP	For Routine	Non Routine	Laboratory Analysis		Correlates with batch gravity directly up to 1.402 and inversely after that, i.e. assay increases as gravity increases and then decreases after passing optimum point. High water in feed, low phenol/ortho feed ratio, and high entrainment can cause isomer shift so that low assays may occur even if gravity in control range.	Controlled by batch specific gravity.	

Process Area Or Step	Variable Or Parameter	Description of Parameter As It Affects Safety, Quality, Yield Rate Or Other	Range For Best Known Process Performance	Frequency Of Monitoring By Supervision	Measurement of Variable			Interaction With Other Variables	How Control Variables	Comments
					Frequency	Method	Accuracy			
RAW MATERIALS										
	1. Blended fractions									
	a. Phenol/Ortho Ratio	Affects isomer split causing low 2,4 isomer product.	At least 5 parts Phenol to 1 part Ortho	After blend at once per day	Daily by operator	GLC analysis by Lab	$\pm 0.1\%$	A high percentage of ortho present will convert to the undesirable 2-6 isomer resulting in a 2-4 isomer reduction. Extremely high ratio can cause a build-up of fractions inventory due to under-use.	Maintain at least a 5/1 ratio. Maximize fractions usage.	2-6 isomer usually 6-7% of batch. Higher, run GLC check of feedstock. Control of phenol and fractions is essential to good blended product quality.
	b. Water	Causes 2-6 shift, builds up nickel salts, and causes corrosion.	$< 0.08\%$	Daily by chief operator. Foreman by exception. ($> 0.10\%$)	Daily by operator	Laboratory Analysis	$\pm 0.1\%$	High water content causes an increase in 2-6 isomer.	Rely on phenol department to control H_2O , control fractions water content in 237.	
	c. Iron	Darkens product and also 2-4D acid in 262.	18 ppm max.	Non Routine	Non Routine	Laboratory Analysis		High iron darkens the color of all phenolics	Minimize recycle of washout batches. Dilute with water white phenol to lower color.	
	d. Color	Color of feed propagates to final product	< 500 APHA	Non Routine	Non Routine	Laboratory Analysis	± 10 APHA	Results in dark product. Usually caused by iron.	Control iron.	Visual check by chief operator daily basis. If dark, request to be run by 1
	2. Chlorine									
	a. Header Pressure	Indicates proper pressure at BD to maintain standard rates.	25-30 psig	Hourly by operator on each batch sheet	Continuous	Pressure Recorder	Unknown	Low pressure cuts chlorination rate since must lower cooling water flow to maintain temperature. Low pressure means low gas flow which can cause poor isomer distribution.	Contact chlorine dept. to determine cause of problem. Check demister inlet pressure to see pad is not plugged.	
	b. Demister Inlet Pressure	Indicates same as header pressure.	25-30 psig	Monthly by foreman	Continuous	Pressure Gage	Unknown	Interacts with header pressure directly. High pressure difference.	Clean or replace demister pad when ΔP exceeds 3 psi or once per year.	
1. BATCH CHARGING										
	1. Chlorinator Charge	Indicates proper charge to maintain rate, quality.	48-52" outage	Each batch by operator	Each batch by operator	Stick measurement	$\pm 1/2"$	Overcharge causes entrainment loss. Undercharge causes rate loss.	Charge to proper level.	If too much is left in scrubber after drop, it affects 2-4 isomer subsequent batch
	2. Chlorinator Charge Time	Time to drop scrubber to chlorinator affect rate.	30 min. ± 5 min.	Each batch by operator	Each Batch	Time		Long drops means line blockage	Check tracers if time of drop is too long.	

Process Area Or Step	Variable Or Parameter	Description of Parameter As It Affects Safety, Quality, Yield Rate Or Other	Range For Best Known Process Performance	Frequency Of Monitoring By Supervision	Measurement of Variable			Interaction With Other Variables	How Control Variables	Comments
					Frequency	Method	Accuracy			
<u>F. BATCH CHARGING</u>	3. Scrubber Specific Gravity	Gravity determines following batch quality and yield.	1.050 - 1.250 at 70°C	Each batch by operator	Each Batch	Hydrometer test by operator	± 0.001	High gravities can cause an isomer shift, reducing 2-4 isomer. Low gravities can result in high entrainment on subsequent batch.	Monitor gravities for each charge. With low gravity observe entrainment and reduce rate if needed. With high gravities, process the batch but reduce rate to improve quality of following scrubber.	Do not hold batch as it results in product when the chlorine is finally discharged.
	4. Scrubber Charge	Indicates proper level to prevent excessive entrainment or Cl ₂ emission.	40" ± 1" outage	Each batch by operator	Each Batch	Stick Measurement	± 1/2"	If outage high, off gas from chlorinator will not be scrubbed letting chlorine gas into the HCL system and eventually to the atmosphere. Low outage result in excessive phenol losses to the off gas system.	Add feedstock to proper outage.	
<u>G. CHLORINATION</u>										
	a. Gas Addition									
	a. Chlorinator Temp.	Affects yields and product quality.	90 - 100°C	Hourly by operator	Continuous	Temp. Recorder Controller & 12 pt Recorder	± 1°C ± 1°C	Low temperature results in slow rates since cooling becomes limiting at low batch/coil temp. differences. High temperatures result in excessive carry-over to the scrubber.	Position controller set point at proper temperature.	
	b. Scrubber Temperature	Affects yields, air pollution.	40 - 50°C	Hourly by operator	Continuous	Temp. Recorder	± 1°C	High temp give high off gas temperature and results in yield loss to HCL system. Indicative of poor chlorinator operation or low jacket cooling water flow. Low temperature reduces scrubbing efficiency, letting Cl ₂ into HCL system.	Manual adjust cooling water flow to jacket to maintain temperature.	
	c. Chlorinator Off-Gas Temperature	Affects quality.	85 - 100°C	Hourly by operator (Not logged)	Continuous	Temp. Recorder	± 1°C	Low temperature indicates low chlorinating flow and low chlorinator temp. High temperature indicates entrainment.	Check entrainment separator for pluggage.	

CRUDE 2,4-DCPAPPENDIX J - SAFETY AND TOXICITY DATA AND ACCIDENT REPORTS

The following precautions should be heeded when in the dichlorophenol area.

1. Finished Products Handling: (Reference: Sax Handbook of Dangerous Chemicals)

CRUDE 2,4 DICHLOROPHENOL is corrosive to the skin. Small amounts spilled on the skin should be washed off immediately with the alcohol located in the first aid stations located throughout the area. This alcohol wash should be followed by a thorough wash with soap and water. If a large amount is spilled on the skin, the contaminated area should be flushed immediately with a large volume of water and then treated at the dispensary. Safety showers are located throughout the area. A fresh change of clothes is provided daily for the operating work force. Wearing of safety glasses is an essential part of Dichlorophenol operations.

2. Raw Materials Handling: (Reference: Sax Handbook of Dangerous Chemicals)

PHENOL is extremely corrosive to the skin and should be removed as previously stated. Sulfuric acid is also corrosive and should be removed immediately by flushing the contaminated area with a large volume of water. CHLORINE and HCl gases are highly irritating and dangerous. If the area must be entered when the gases are present in large amounts, the protective gas masks located in the operating area should be worn.

3. FIRE Hazards

The flash and fire points for Dept. 236 materials are:

	<u>Flash Point</u>	<u>Fire Point</u>
Phenol	79°C.	85°C.
Crude 2,4 Dichlorophenol	> 200°C.	> 200°C.

While none of these materials are technically flammable, no burning or welding is allowed on equipment until it has been washed clean and then gas tested. This is due to the possibility that any residual HCl present may form Hydrogen by reacting with the metal of the vessel.

CRUDE DCP -APPENDIX K - Background of Process and Annual Remarks

Crude DCP is produced in chlorination equipment originally installed in Bldg. BD in 1947, for the production of Pentachlorophenol. Penta and Crude DCP requirements have steadily increased until a total of four chlorinators are now required. Most Crude DCP is produced in the #3 chlorinator which is equipped with an external steel cooler. Crude DCP is also produced in #1, #2, and #4 chlorinators and the Dept. 237 chlorinator.

Crude DCP is used exclusively by Dept. 262 for the production of 2,4-Dichlorophenoxyacetic Acid (2,4-D). The Bldg. BD chlorinators are scheduled to supply the Crude DCP. All other chlorination capacity is used for the production of Pentachlorophenol. Average monthly Crude DCP production in 1967 was 1,980,000#/mo. Requirements in 1968 are forecast at 2,140,000 lbs./mo. In order to satisfy increased demand for both Crude DCP and Penta a capacity expansion project was initiated in late 1967. Tentative plans are to install another batch chlorinator to be used exclusively for Crude DCP production.

APPENDIX K - Process Amendment Record Sheet

<u>Amendment No.</u>	<u>Date</u>	<u>Subject</u>	<u>Disposition</u>
"A" to SMP Dated 4/65	7/26/65	Use of essentially all Dept. 237 fractions for Crude DCP Raw Material.	Accepted 12/2/65

CRUDE DCPAppendix L - Time CycleA. Time Cycle

<u>Item</u>	<u>#3 or #5 Chlorinator Time (Hrs.)</u>	<u>#1,#2,#4 Chlorinator Time (Hrs.)</u>
Charge Chlorinator	0.5	0.5
Charge Scrubber	0.5	0.5
Chlorinate	3.78	5.59
Pump to Storage	<u>0.5</u>	<u>0.5</u>
Total	5.28	7.09

B. Capacity

#3 or #5 chlorinator

13,200 lbs. Crude DCP/batch (720 hrs./mo.) (78% OST)
5.28 hrs./batch

= 1,408,500 lbs./mo.

Chlorine flow is the capacity-limiting factor. With adequate flow the OST would increase to 87% and the capacity would increase correspondingly. Cooling capacity is the next capacity-limiting factor after chlorine flow.

#1, #2, or #4 Chlorinator

13,200 lbs./batch (720 hrs./mo.) (78% OST)
7.09 hrs./batch

= 1,041,500 lbs./mo.

Cooling capacity is the capacity-limiting factor. After cooling capacity, chlorine flow is the next capacity-limiting factor. Nos. 3 and 5 chlorinators are equipped with external coolers. Nos. 1, 2 and 4 chlorinators are equipped with lower capacity internal cooling coils.

CRUDE DCP

APPENDIX M - QUALITY CONSIDERATIONS

CRUDE DCP ASSAY

Maximum Crude DCP assay is desirable in order to maximize yields in the 2,4-D. operation. Recent calculations that each 1% increase in Crude DCP assay results in a \$36,100/year yield savings in the 2,4-D. operation.

HCl and WATER CONTENT

HCl and water concentrations in the finished product must be minimized in order to minimize tank car and storage tank corrosion. Normally this is no problem and no special steps are necessary to insure this.

Dept. 236 - Process Description

28.

CRUDE 2,4-D.

APPENDIX N - MISCELLANEOUS

Attached is the production Cost Standard for 1968.

K031140

PRODUCTION COST STANDARD

PLANT

116K

PAGE

1

OF

1

PRODUCT

2-1 DCP

63025

COST CENTER

2450.000

EFFECTIVE

8-1-62

PROD. CODE 8102

THEORY TO STD. ON

81000

96.99

%

CAPACITY

2450.000

INVT. UNIT COST

6.014

THEORY TO STD. ON

81059

95.10

%

COST UNIT

6.014

INVT. UNIT COST

6.014

MATERIAL CODE

NAME

THEORY

STANDARD

PRICE PER LB.

UNIT COST

81000

THEORY

57.73

59.520

0.000

2.571

81059

CHLORINE

87.00

91.000

0.270

1.975

81503

H₂SO₄

2.286

0.035

0.019

81055

NaOH

0.350

0.2176

0.03

RAW MATERIALS AND CONTAINERS

RAW MAT'L WAREHOUSING & HDLG.

GROSS TOTAL MAT'L'S

BY-PRODUCTS

22.851 116L 540

29.000

0.1625

4.711

0.13

5.591

TOTAL BY-PRODUCTS

NET TOTAL MAT'L'S

CONTAINERS:

USAGE/UNIT

PRICE PER CONT.

CONT. COST/LB

PKG. UNIT STD.

5.120

PRODUCTION CONTROL DATA

CLASSIFICATION

AMOUNT

COST OF SHUT-DOWN

COST OF START-UP

COST OF CHANGEOVER

PACKING CHANGEOVER COST

PACKAGING RATE LBS/HO.

PRODUCTION CODE

CAPACITY

REPAIR

DT

RL

TOTAL

#34 #5 2500

PROCESS

3.0

1.0

4.0

#4 18130

OTHER

4.0

9.0

13.0

0

INVENTORY

0

0

0

0

TOTAL

12.0

12.0

22.0

0

ON-STREAM

12.0

12.0

22.0

0

OTHER DATA

ON-STREAM RATE

43.43

43.43

43.43

0

TYPE PKG.

NET LB/PKG.

GROSS LB/PKG

NET LB/PKG

GROSS LB

OTHER PRODUCTS PRODUCED IN CONJUNCTION WITH THIS PROD.

K031142