1-30-52

TO: Mr. C. H. Kolker

cc: Mr. J. Burton
Mr. L. A. Kolker

FROM: T. M. Barna

SUBJECT: 2,4,5-TRICHLORPHENOL

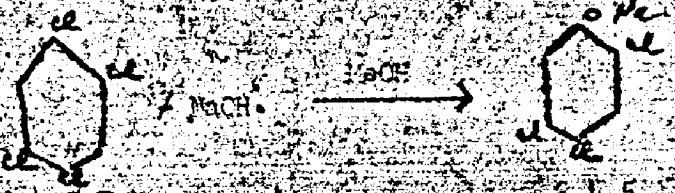
A flowsheet, equipment description and operating instructions for the manufacture of 2,4,5-trichlorphenol from 1,2,4,5-tetrachlorbenzene is attached.

T. M. Barna

2,4,5-TRICHLORPHENOL

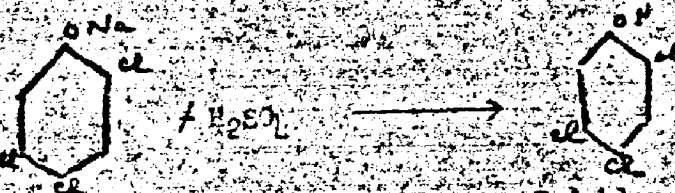
CHEMISTRY OF THE PROCESS:-

1,2,4,5-tetrachlorbenzene (settling point 137°C) is hydrolysed in methanolic NaOH in an autoclave at 170-180°C (pressure 350-400 p.s.i.)



1,2,4,5-Tetrachlorbenzene
Mol.Wt.216

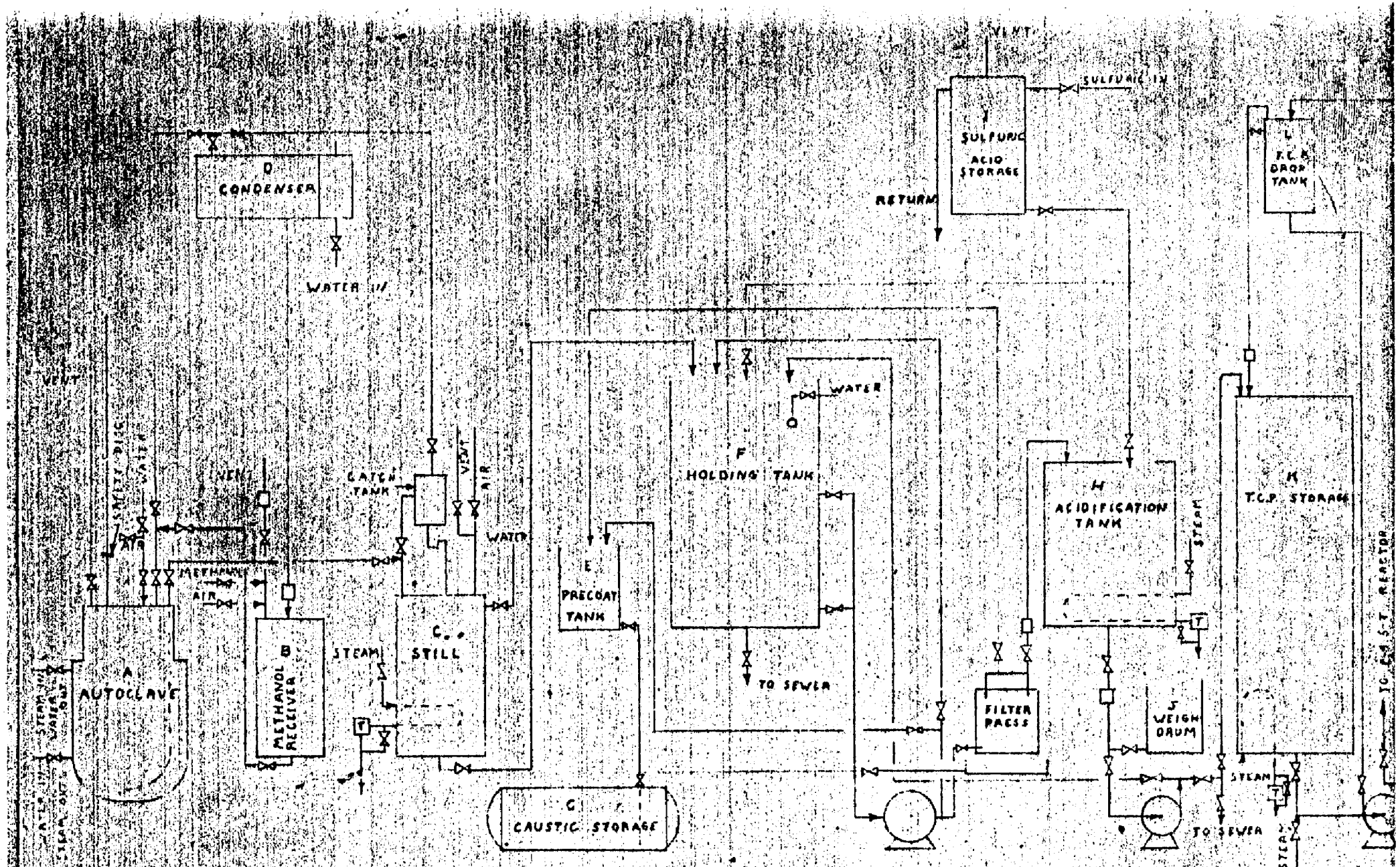
2,4,5 Sodium Trichlorphenate
Mol.Wt.220



2,4,5 Sodium Trichlorphenate

2,4,5 Trichlorphenol
Mol.Wt.197

The methanol is distilled off and the residue is diluted with water. The sodium trichlorphenol solution is filtered and treated with sulphuric acid to precipitate the free trichlorphenol. The free trichlorphenol is collected in a storage tank and used in the manufacture of 2,4,5-trichlorphenoxy acetic acid (2,4,5-T) without further purification.



2,4,5-Trichlorphenol

Pilot Plant Flow Sheet

DS 00008173

K. Aspinall

DESCRIPTION OF EQUIPMENT

AUTOCLAVE

AUTOCLAVE (A)

A 500 gallon Blaw-Knox high pressure autoclave. Pressure 660 p.s.i. at 650°F maximum. Internal test pressure 1320 p.s.i. hydrostatic. Jacket pressure 250 p.s.i., body built in accordance with the A.S.M.E. code par. U68 for unfired pressure vessels including X-Ray test. Jacket built in accordance with A.S.M.E. code par. 69 for unfired pressure vessels.

Drive - Falk 5 H.P. vertical all motor type Reducer, class 11 service, output speed 190 RPM.

Coupling - Falk 9F flexible coupling.

Stuffing Box - Cartridge type, grease lubricated, water cooled.

Agitator - 15½" diameter Blaw-Knox, Turbine type agitator-speed 190 RPM.

Discharge Blow Leg - A 2" steel line to bottom of autoclave (internal).

Rupture Disk - Black-Sival-Bryson stainless steel disc. 678 p.s.i. at 204°C. (945 p.s.i. at 72°F.)

Hand Hole - 6" quick opening closure using 2 rings of ½" 166S-Johns-Hansville packing.

METHANOL RECEIVER (B-)

A vertical 200 gallon steel calibrated tank. Sight glass.

STILL (C)

A 500 gallon vertical steel tank equipped with two bayonet heaters. 100# steam. The still was equipped with a 6" X 16" extrainment separator.

CONDENSER (D)

An all steel shell and tube water cooled condenser 8" diameter X 8' long. Single pass. Graham Manufacturing Company, Inc., Approximately 50 square feel cooling surface.

HOLDING TANK (F)

A 5' 6" diameter flat bottom steel vertical tank 8' high. Air agitated. Two side outlets (1) 12" from the bottom (2) 4½ feet from the bottom. Sewer connection at bottom. Sulphuric acid line connection. Steam connection to the air sparger.

FILTER PRESS

A 24" Shriver iron filter press 14 plates & frames. Cotton filter cloths. Sight glass in line from press to the acidification tank.

ACIDIFICATION TANK (H)

A 650 gallon cone bottom Monel tank. 6" turbine type agitator ca 200 RPM Monel steam coil. Acid tank connection. Bottom sight glass.

T.C.P. CRUDE STORAGE TANKS (K)

Two vertical steel tanks (1) 1000 gallon (2) 2000 gallon capacity. Steam heater, pump and heated lines to 2,4,5-T manufacture unit.

2,4,5-TRICHLORPHENOL

OPERATING INSTRUCTIONS #3

GENERAL INSTRUCTIONS

1. 2,4,5-Trichlorphencl is prepared from 1,2,4,5-Trichlorbenzene is an autoclave using NaOH in a methanol medium.

2. The autoclave pressure will rise to 350-450 p.s.i. during the heating period. It is important that the autoclave hand hole be tightly closed and that the valves do not leak.

3. No maintenance work is to be done on the autoclave when under pressure. If maintenance work is required, cool the autoclave to 60°C or lower and then vent the system. Check with the supervisor before starting any maintenance work on the autoclave.

4. The autoclave is equipped with a safety rupture disc which will rupture at 670 p.s.i. at 204°C. (945 p.s.i. at 72°F).

CHARGING THE AUTOCLAVE

A. The batch charge consists of the following:

800 (1) ~~700#~~ Hooker Tetrachlorben_ene. Setting point 137°C.

400 (2) ~~350~~ lbs. flake NaOH.

170 (3) ~~160-180~~ gallons methanol. (Recovered and/or new)

B. Method of Charging:

(1) Cool autoclave to below 80°C, vent, and open handhole.

(2) Connect link from hopper line to handhole and charge NaOH through the hopper.

(3) Charge tetrachlorobenzene through the hopper.

(4) Check volume of recovered methanol in the receiver. Use a total of 160-180 gallons of methanol per charge. This methanol can be either recovered or new methanol or a mixture of both.

(5) Open autoclave vent, close low-pressure autoclave valve to methanol condenser and blow recovered methanol from receiver until a total charge of 160-180 gallons is in autoclave. Use 6-9 p.s.i. air pressure on receiver to blow the methanol to autoclave.

NOTE: While charging methanol, fresh or recovered, keep water on autoclave jacket to hold down temperature rise.

OPERATION OF AUTOCLAVE

(1) Close valves on the autoclave.

(2) Tighten the hand hole closure.

(3) Add grease to the autoclave packing gland.

(a) Add grease to upper fitting until the old grease is forced up the shaft.

(b) Add grease to the lower fitting until grease is forced out of the fitting at the base of the gland. Replace plug in the fitting.

(4) Drain the jacket through the bypass on the trap. Turn on steam.

(5) Close bypass on the trap and heat autoclave at full steam pressure until temperature rises to 160°.

(6) Shut off the steam at 160, let temperature of autoclave rise to 170-180°C. The pressure will rise to 350-400 p.s.i.

(7) Maintain temperature of autoclave at 170-180°C and the pressure will rise to 350-400 p.s.i. for 4½ hours.

(8) Record hourly readings on log sheet.

DISTILLATION OF METHANOL

1. Cool autoclave to 100-130°C - pressure 25-35 p.s.i.
2. Check still - still to be empty.
3. Set autoclave high pressure valve to discharge the batch (slowly!) to the still. The still is vented thru the condenser and receiver.
4. Blow the charge to the still. Do not exceed 10 # p.s.i. on the still or the receiver.
5. Heat the still using 100-125 p.s.i. steam.
6. Distill off the methanol. This will require approximately 4 hours. The residue temperature will rise to 120°C. Leave steam on.
7. Measure the recovered alcohol and record.
8. Dilute the residues with 200 gallons of water. Agitate with air for 10-20 minutes.
9. Transfer the aqueous solution to the sodium salt holding tank.
10. Add 200-250 gallons of water to the still.
11. Agitate with air 20 minutes - steam on.

Transfer the washings to the sodium salt holding tank.

FILTRATION - SODIUM SALT, TCP

(1) Dilute batch to 18" outage. Agitate with air while diluting. Close valve to float valve before proceeding.

(2) Adjust p^H of batch to 85-9 (Hydrion Paper)

(3) Settle for one hour. Take off from top tap.

(4) Set valves above filter press to return first portion of filtrate to precoating drum.

(5) Filter into precoating drum until it is full with liquid drawn from upper half of settling tank

(6) Add one scoop of filter-aid to precoating drum and recirculate until filtrate is clear.

(7) Adjust valves over filter press to send filtrate to acidification tank.

(9) When batch has been acidified and acidification tank is empty, repeat above procedure (steps 4 to 8) for bottom half of settling tank.

RESIDUE - SODIUM SALT HOLDING TANK

1. Extract residue in sodium salt holding tank after each 4 batches.

2. Add warm water to salt tank to the level of the 1st tap.

3. Adjust ph to 10-12 on Hydrion paper.

4. Stir residue with air for ½ hour.

5. Shut off air. Let batch settle for 1 hour.

7. Filter the aqueous portion as directed above under "Filtration - Sodium Salt, TCP."

9. Discard the residue in the holding tank. Flush to sewer.

ACIDIFICATION OF TCP

1. Heat the filtered Sodium salt in acidification tank to 50-60°C.

2. Add 66° Be H_2SO_4 to a ph 3-4. (Blue or Congo Red paper.) Requires approximately 15 gallons.

3. Stop stirrer . Let batch settle for ½-3/4 hour.

4. Drop TCP to the measuring drum.

5. Measure TCP using 20.5# TCP per inch. Record yield. Test- see below.

6. Re Transfer TCP to the storage tank. Blow line clear with steam.

7. Repeat with second half of the batch.

TEST - TCP

Add 15 cc of TCP in 50 cc 38% NaOH diluted to 200 cc with water. If clear or slightly cloudy, the TCP is acceptable for use in 2,4,5-T.

If a heavy precipitate appears - transfer the TCP to drums and rework as directed by the supervisor.

RAW MATERIAL USAGE PER BATCH

Tetrachlorbenzene	700# as is Hooker Electrochemical Co.
Methanol	180 gallons total (120 gal. recovered, 60 gallons of new methanol.)
NaOH-Flake	350# flake.
Sulphuric Acid (66°Be)	15 gallons.

YIELD

The overall yield based on 1,2,4,5-Tetrachlorbenzene is 85% of theory or 550# of (dry basis) TCP per day. The TCP is used without further purification in the manufacture of 2,4,5-T.

T. M. Barna
1-31-52