

Copy for file

T. Barna
3/20/51

2,4,5-TRICHLORPHENOL

Operating Instructions #1

GENERAL INSTRUCTIONS:

1. 2,4,5-Trichlorophenol is prepared from 1,2,4,5-tetrachlorobenzene in an autoclave using methanol and NaOH.
2. The autoclave pressures will, at times, rise to 500 p.s.i. It is very important that all valves and the hand-hole closure be tight during the operations.
3. No maintenance work must be done on the autoclave while under pressure. If repairs are needed, the autoclave must be cooled to below 50° and vented. Check with supervisor before cooling down if repairs are required.
4. The autoclave is equipped with a safety rupture disk. The disc is installed in a flanged fitting which is connected to 2" pipe. The line is vented to the atmosphere through the roof. The rupture disc is set to break at 945 PSI at 72°F and 678 PSI at 400°F (204°C)

METHANOL - NaOH CHARGE:

7.1ms

1. To the NaOH dissolve~~r~~ add - 285# flake NaOH
2. Bolt down the cover securely.
3. Blow to the dissolver from the methanol receiver a total of 160-170 gal. methanol.
4. Add sufficient fresh methanol to the dissolver so that the total charge is 160-170 gal.
5. Heat the NaOH - methanol mixture and let reflux for 1 hour. Adjust the valves so that the methanol will reflux. The reflux condenser is connected to a horizontal condenser which is in turn connected to a receiver.
6. Shut off valve at the reflux condenser.
7. Blow batch to autoclave when T4CB has been charged.
8. Cool the dissolver to room temperature before loading next charge. Naoh.
NOTE: A residue of about 50# remains from batch to batch.
9. Record all above usages on log sheet.

DS 00008192

CHARGING THE AUTOCLAVE:

1. Check the autoclave for residue from last batch.
2. Grease the bearing using Quaker State pressure gun lubricant (black grease) from a Alemite pressure gun.
 - (a) Grease is to be added to the upper fitting until the old grease is forced up along the shaft.
 - (b) The grease is to be added to the lower fitting until grease is forced and if the 1/4" plug at the base of the bearing.
 - (c) Grease is to be added to the middle fitting to fill pressure which can be exerted on the grease gun.
- 2.7 mms 3. Hand feed 585# real tetrachlorbensene centrifuge cake.
4. Close handhole, set valves to vent through the 1" line to the condenser.
5. Close 2" high pressure valve to the blowleg.
6. Open 2" high pressure charging valve.
7. Close 2" plug valve above the charging valve which is connected to the horizontal condenser.
8. Blow the charge of methanol-NaOH from the dissolver.
9. Close the high pressure valves. Check the following valves:
 - (a) - High pressure charging valve 2"
 - (b) - " " Blowleg " 2"
 - (c) - " vent valve 1"
 - (d) - " " water valve 1"
10. Check handhole closure to be sure there are no leaks. Replace gasket when required.
11. Heat autoclave to 170-180°C - No higher
12. Hold 170-180° for 5½ hours after the temperature reaches 160°C.
13. Record hourly readings. Temperature and pressure

DISTILLATION OF METHANOL

Changed 6/23/51
TB

1. Cool Autoclave to 130°C.
2. Set valves for distillation of methanol.
3. Start water through the condenser.
4. Open the 2" high pressure valve slightly so that the autoclave is slowly vented through the condenser. Be sure the pressure on the receiver does not exceed 2-3 P.S.I.
5. Cool the autoclave with cooling water on jacket to an internal temperature of approximately 90°C.
6. Drain the jacket and heat the autoclave until the autoclave temperature reads 110-115°C.
7. Shut off the steam
8. Collect methanol in the receiver and measure the recovered methanol. Record on log sheet.
9. Add water to the residue in the autoclave until the free space above the liquid is approximately 20" when measured from the top lip of the hand hole.
10. Stir one-half hour to dissolve the sodium salt
11. Blow to the sodium salt holding tank.
12. Dilute to 12-16" from top rim of holding tank
13. Add approximately 20% filter aid
14. Agitate with air
15. Adjust to pH 9-10 using 66° Be H₂SO₄
16. Stir 15 minutes then let batch settle for 1-2 hours
17. Precoat filter press with a slurry of approximately 10% filter cel in 30 gallons of water. The press is dressed with filter cloth and paper.
18. Filter batch three press. Be sure the filtrate is clear. If cloudy, return the filtrate to the holding tank and refilter.
19. Hold residue in the holding tank until 4 batches have been processed.
20. Process the residue after 4 batches;
(A) Dilute the residue to 12-16" from top rim of the holding tank.

DS 00008194

DISTILLATION OF METHANOL CONTINUED

6/27/51
JP

- (B) Adjust pH to 9-10 - Stir with air
- (C) Let settle for 1 hour
- (D) Filter to holding tank
- (E) Discard final residue to sewer.
- (F) ACIDIFY the Washings
- (G) Let settle in acidification
- (H) Draw off TCP to drum
- (I) Discard aqueous layer.
- (J) Weigh the TCP and record weight.

DS 00008195

ACIDIFICATION

Charged 6/22/51

1. Add 40-50 gallons of Monochlorobenzene to batch.
2. Stir one-half hour
3. Let settle for 1 hour.
4. Draw off MCB layer. ^{use} Rinse MCB four times then hold for distillation.
5. Acidify the extracted water layer to pH 4 using H_2SO_4 while agitating batch
6. Stop stirrer, let batch settle for $\frac{1}{2}$ -1 hour
7. Draw off TCP to a tared drum
8. Weigh TCP- Take thief sample
9. Run aqueous layer to sewer.
10. Rinse kettle with water to remove acid.

DS 0008196

MAXUS1239007