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2,5-Dichlorophenol

cc: R. L. Annis  
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In accordance with our conversation yesterday afternoon, we attach information developed by our Process Development Section on a method for producing 2,5-dichlorophenol from 1,2,4-trichlorobenzene. You will note that information is given for a one gallon and for a 400 gallon batch. The latter, of course, will give us sufficient material for the foreseeable future.

Our Purchasing Department has told us that the Dow Chemical Company maintains a stock of 1,2,4-trichlorobenzene, technical grade, freezing point 16.5°C, at Port Newark, New Jersey.

Please advise if you require additional information.

G. F. Rugar

GFR:bjm

DS 00011682

## 2,5-Dichlorophenol

### Preliminary Operating Procedure

#### Materials required -

	<u>Batch Size</u>	
	<u>1 gal.</u>	<u>400 gal.</u>
1,2,4-trichlorobenzene	905 grams	800 lbs.
methanol	900 cc	95 gal. (630 lbs.)
water	100 cc	10.5 gal. (87.5 lbs.)
caustic soda, flake	600 grams	530 lbs.

#### Equipment required -

1. Autoclave - provided with agitation and controlled heating
2. Equipment for steam distillation
3. Suitable source of steam

#### Hazards - Safety Precautions

1. The usual precautions in autoclave operation.
2. The alkaline solution of chlorophenols is corrosive to the skin and mucous membranes. Wear rubber gloves and safety goggles (or face shield) when handling the reaction product.
3. Avoid all contact with the skin. In case of accidental contamination, wash thoroughly with soap and water.
4. The steam distillation should be done in a well ventilated area.

#### Operations

1. Mixing the chemicals
2. Assembling the autoclave
3. Heating the autoclave (with vigorous agitation)
4. Steam distillation of the reaction mixture for separation of the methanol and unreacted trichlorobenzene.

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## Procedure

### Preliminary

1. Make sure the autoclave is in good operating condition.

#### I. Mixing the chemicals and assembling the autoclave

1. Weigh or measure out the chemicals
2. Add them in the order given to the autoclave reaction chamber (starting with the trichlorobenzene).
3. Assemble the autoclave for operation.
4. Tighten all connections.

#### II. Heating the autoclave

1. Start the stirring and heating.
2. Hold the temperature at 320°F (160°C) for five hours.
3. Record the temperature and pressure at least every 15 minutes.
  - a) The pressure reaches about 450 psi.
4. Let the autoclave cool at least below 150°F (65°C) with continued stirring.
5. Open the autoclave.

#### III. First steam distillation (separation of methanol and unreacted trichlorobenzene)

1. Assemble the steam distillation apparatus in a good hood.
2. Transfer the reaction mixture from the autoclave to the steam distillation flask.
3. Use water to remove residual reaction mixture and add the washings to the distillation flask.
4. Pass steam through the mixture until most of the methanol has been removed.
5. Continue the operation with external heating until the distillate is clear and colorless (on cooling and dilution with water) for complete removal of unreacted trichlorobenzene.
  - a) Test small portions of the distillate frequently by cooling below room temperature and dilution with an equal volume of cold water.
6. Stop the distillation as soon as the distillate contains no organic material.
7. The trichlorobenzene may be separated (lower liquid phase) from the cold steam distillate and weighed for material balance.
8. Save the distillation residue (the contents of the distillation flask) for the next operation.

C. E. Entemann

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