

TRANSLATION

C. H. BOEHRINGER SOHN
Chemische Fabrik.

Ingelheim am Rhein 22b

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Diamond Alkali Company
Research Center
Painesville, Ohio
P. O. Box 348

Gentlemen:

Our Dr. Kudzus informs us that you are also engaged in the manufacture of trichlorophenol and that you too have had complaints about the occurrence of chloracne in the case of workers occupied in this process. Until one year ago we were confronted with the same problem, forcing us to initiate research on how the occurrence of chloracne coarsing agents is to be avoided. This research has been concluded. The recent installation of a plant with utilization of these experiences has reported a successful result. There have been no further cases among the workers in the new plant, which has now been in operation for about one year.

Our experiences are consolidated in a brief paper which we are enclosing for your files.

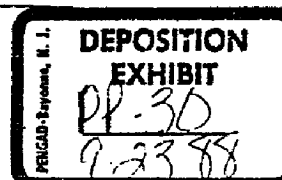
We remain,

Very truly yours,

C. H. BOEHRINGER SOHN

/s/ Dr. Kudzus W. Weyland

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TRANSLATION

C. H. Boehringer Sohn

A Method for the Preparation of Trichlorophenoxyacetic Acid Which Avoids Formation of Chloracne-Causing Agents

Our synthesis of 2,4,5-trichlorophenoxyacetic acid proceeds in the following steps:

1. trichlorobenzene $\xrightarrow{\text{Cl}_2}$ tetrachlorobenzene
2. tetrachlorobenzene $\xrightarrow{\text{NaOH}}$ sodium trichlorophenolate
 - a) autoclave reaction
 - b) evaporation of CH_3OH
 - c) steam distillation of anisol
 - d) isolation of trichlorophenol by acidification and subsequent distillation
3. trichlorophenol $\xrightarrow[\text{CH}_2\text{ClCOONa}]{\text{NaOH}}$ 2,4,5-trichlorophenoxy sodium acetate
4. further processing to pure, crystalline 2,4,5-T-acid

According to our experience, the chloracne coarsing effect is due to impurities and side reactions. These can arise in the conventional process only when sodium trichlorophenolate or other alkali salts of trichlorophenol are produced, purified, and further processed. In this case reaction conditions result which are close or tantamount to a salt force.

With this consideration in mind, the following holds with respect to the origin of chloracne agent at the various processing steps:

- At 1 and 4 - These steps are completely safe.
- At 2 - At this step the chloracne agent can originate.
- At 2a - In order to avoid this at the autoclave reaction, an overheating of the autoclave contents is to be avoided. (Maximum temperature 150°C). Further is to be observed that the work is carried on in the highest possible dilution with methanol.
- At 2b - In the methanol distillation the water formed should be retained in the distillation flask in order to avoid the dangers of heating to dryness of sodium trichlorophenolate. The temperature of the residue should not rise above 100°C .
- at 2c - The steam distillation for the separation of trichloroanisole has to be carried out in such a fashion that no change in the concentration of the sodium trichlorophenolate solution occurs. If necessary, water has to be added.

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At 2d

- When the crude phenol is distilled, it has to be observed that no sodium trichlorophenolate and no sodium chloride is carried over into the distilling flask and heated together with a phenol.

At 3

- The chloracne agent can also originate at this step. In order to avoid this, the concentration of sodium trichlorophenolate with sodium chloroacetate should not be carried out in non-aqueous medium. A concentration in excess of 40% is to be avoided in any case.

The following can be said briefly about the properties of chloracne agents: As a neutral body it is volatile with steam and thus can accumulate in the trichloroanisole which is distilled off in Step 2c. It is, therefore, advisable to forego further usage of the trichloroanisole and destroy it by burning.

The chloracne agent shows distinct sublimation phenomena already at a temperature above 100° C. Therefore, the reaction product should be processed from step 2 in a completely closed apparatus. Adequate ventilation of the working area should be provided.

/s/ M. Weyland

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