DS

<u>October 11, 1952</u>

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<u>Chloracne - Trichlorophenol</u>

PLAINTIFF'S EXHIBIT P-0105

cc: J. Burton-Newark
✓G. J. Lukas

Enclosed is a translation, thanks to Roland Steinkoenig, of a <u>letter and brief report on chloracne just received from C. H. Boehringer</u> Sohn, Ingelheim. This confirms in essence the comments passed on to me some time back by Dr. Kaiser, of this company.

In view of John's recent comments about Monsanto's policy of limiting carefully the amount of active material present at any time in an autoclave and in view of your recent communication pointing out that the product slurry should be fairly simple to handle, it seems to me there is a great deal of incentive in our trying to run an autoclave batch at <u>a controlled temperature.</u>

If you run into continued difficulties with the plant scale units, perhaps we could try a run here at Painesville where the much greater surface-to-volume ratio might very effectively prevent run away temperatures.

I am attaching a sketch which resulted from some doodling the other day about ways in which a continuous autoclave unit might be set up and controlled.

D. J. Porter

DJP:ajm

Attach.

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 $\xrightarrow{Cl_2}$ tetrachlorobenzene
2. tetrachlorobenzene $\xrightarrow{NaOH}$ sodium trichlorophenolate
    - a) autoclave reaction
    - b) evaporation of CH_3OH
    - c) steam distillation of anisol
    - d) isolation of trichlorophenol by acidication and subsequent distillation
3. trichlorophenol $\xrightarrow[CH_2ClCOONa]{NaOH}$ 2,4,5-trichlorophenoxy sodium acetate
4. further processing to pure, crystalline 2,4,5-T-acid

According to our experience, the chloracne causing 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 melt 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 - In 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.

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 10% 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. Wayland