

L0052310

FROM (ORIGINATOR) Agricultural Research - St. Louis

cc. W. P. DUNLAP
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D. B. SHARP

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TO E. F. Taka - Nitro

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243-T in
Controlled
Letter

The results of our analysis of four autoclave batches are summarized in the attached graphs (Figures 1-4). I believe the code is self-explanatory with the possible exception of DMEO. This refers to the total of the dimethoxydichlorobenzenes and the methoxydichlorophenols. The tetrachlorodibenzodioxan (TDD) concentration is read on the right abscissa. The TDD concentrations are based on the total of the other products (TCP, TCA, DMEO) and the TCB. We have previously shown that the concentrations of TDD in the TCP is approximately equal to the concentration of TDD in the T-acid produced from that TCP. With this background, certain conclusions can be drawn by a comparison of Figures 1-4 and a knowledge of the past history of TDD production (Figure 5). Some of these conclusions are:

1. During April, 1965, production of TDD rose to an all-time high and has remained much higher than the previous norm-current ~50 ppm., normal ~10 ppm.
2. The formation of TDD takes place almost entirely during the hold period.
3. At the end of the hold period the TCA content accounts for 2.0-2.5% of the yield. About 1/2 of this is removed by azeotropic distillation in the methanol still and recycled with the methanol.
4. The TCB is essentially gone at about one hour into the hold period.
5. Extending the hold period results in increased TDD formation (Batch 701).

The cause of the recent increase in TDD formation is not obvious from the data. However, since TDD formation has been relatively low in the past we feel that a detailed laboratory study of the autoclave reaction has a reasonably good chance for success. Such a study is now in the planning stage.

W. R. Udell

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Attachments

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