

C.F. #188

TO: W. E. HILL, JR.

FROM: R. F. LINDEMANN

SUBJECT: TCP - ANISOLE STRIPPING AND AUTOCLAVE CONDITIONS - REPORT I

The recent change in Autoclave conditions from temperature averages of about 185°C to lower temperatures (160-165°C) has increased the 2,4,5-TCP assay of the finished product. The chart below shows some of the assays which have been run.

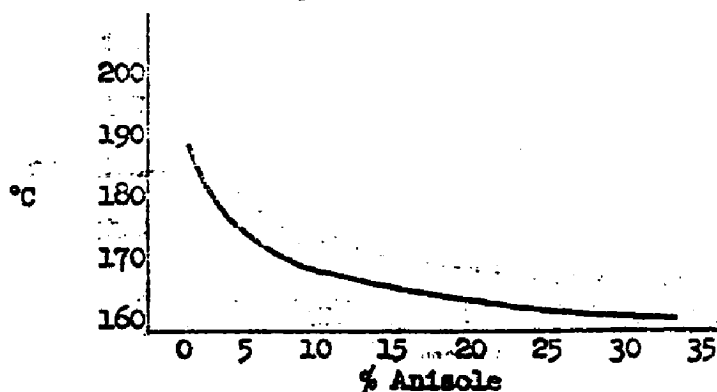
<u>Autoclave Temperature</u>	<u>% 2,4,5-TCP</u>
185°C	83.7
185	81.9
185	84.6
165	91.8
165	92.3
165	94.5
165	93.2
165	92.4

The change in conditions has also resulted in an increase in by-product anisole, thereby decreasing the capacity of both Autoclaves and anisole still. The chart below shows the approximate amount of anisole left in the reaction mixture at various temperatures.

<u>Autoclave Temperature</u>	<u>% Anisole</u>
185°C	1
165	16
163	26
160	33

These numbers are better illustrated in Figure 1. following.

FIGURE 1



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The curve shown in Figure 1 may be drawn several ways until more data is obtained; however, it suggests that the reaction at 170°C should be investigated.

It has also been established that increasing the digestion time from five to six to ten hours at 160°C does not significantly lower the amount of recycle anisole. This was not checked at 165°C, but will be considered in the future runs. Depending on the quality of product obtained at 170°C, digestion time will be studied at 170°C or 165°C, that is the highest temperature at which good quality TCP can be produced.

The stripping as a function of time was measured on two runs. The data from these runs is shown in the following tables.

1330	1 hr., 45 mins.	5 hrs., 5 mins.
1331	2 hrs., 35 mins.	5 hrs., 0 mins.
1332	1 hr., 55 mins.	5 hrs., 0 mins.
1333	1 hr., 30 mins.	5 hrs., 0 mins.
1334	1 hr., 45 mins.	5 hrs., 0 mins.
1335	2 hrs., 15 mins.	5 hrs., 0 mins.

24 hours	5
48 hours	2
Final (72 hours)	1

1336	1 hr., 45 mins.	5 hrs., 0 mins.
1337	1 hr., 40 mins.	5 hrs., 0 mins.
1338	1 hr., 40 mins.	4 hrs., 0 mins.
1339	1 hr., 45 mins.	4 hrs., 0 mins.
1340	1 hr., 30 mins.	4 hrs., 0 mins.

24 hours	3
Final (48 hours)	1

The curve shown in Figure 1 may be drawn several ways until more data is obtained; however, it suggests that the reaction at 170°C should be investigated.

It has also been established that increasing the digestion time from five to six to ten hours at 160°C does not significantly lower the amount of recycle anisole. This was not checked at 165°C, but will be considered in the future runs. Depending on the quality of product obtained at 170°C, digestion time will be studied at 170°C or 165°C, that is the highest temperature at which good quality TCP can be produced.

The stripping as a function of time was measured on two runs. The data from these runs is shown in the following tables.

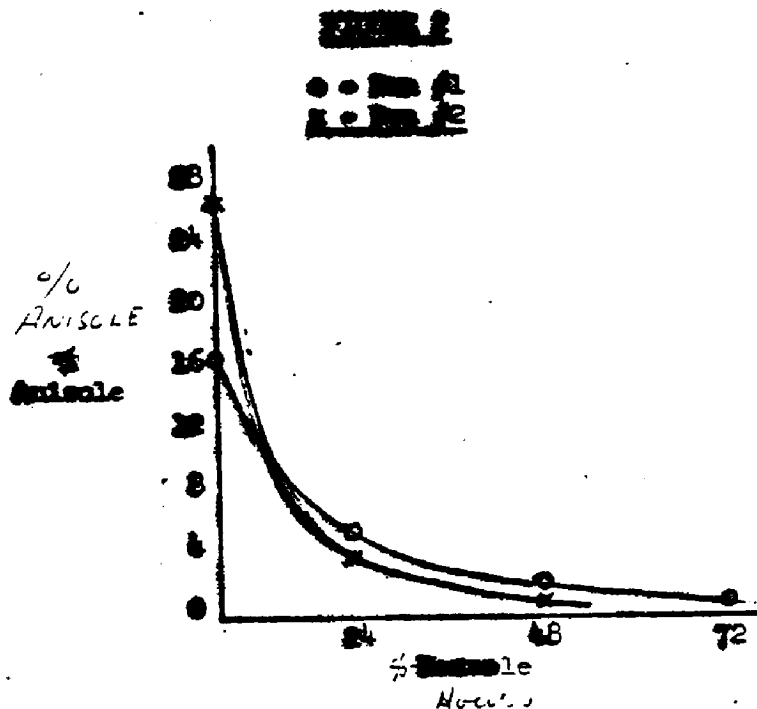
RUN #1 - 165°C		
Batch No.	Reaction Time	Digestion Time
1330	1 hr., 45 mins.	5 hrs., 5 mins.
1331	2 hrs., 35 mins.	5 hrs., 0 mins.
1332	1 hr., 55 mins.	5 hrs., 0 mins.
1333	1 hr., 30 mins.	5 hrs., 0 mins.
1334	1 hr., 45 mins.	5 hrs., 0 mins.
1335	2 hrs., 15 mins.	5 hrs., 0 mins.

Sample From Still	% Anisole
Original	16
24 hours	5
48 hours	2
Final (72 hours)	1

RUN #2 - 163°C		
Batch No.	Reaction Time	Digestion Time
1336	1 hr., 40 mins.	5 hrs., 0 mins.
1337	1 hr., 40 mins.	5 hrs., 0 mins.
1338	1 hr., 40 mins.	4 hrs., 0 mins.
1339	1 hr., 45 mins.	4 hrs., 0 mins.
1340	1 hr., 50 mins.	4 hrs., 0 mins.

Sample From Still	% Anisole
Original	26
24 hours	3
Final (48 hours)	1

Figure 2 illustrates the stripping as a function of time, considering, however, that there are many variables associated with the still.



It is now planned to investigate the reaction at 170°C. Should the quality remain good, then the digestion period will be varied to study the effect on anisole.

WFL/mc

4/15/63

cc: R. A. Giddi
F. R. Kennedy
W. Robin


R. F. LINDEMAN