

**Diamond Alkali Company**

INTER-OFFICE CORRESPONDENCE

DATE  
March 23, 1966

118-1A

FROM

Mr. F. G. Steward - Newark ✓

Mr. W. A. Goodloe - Newark

SUBJECT:

INTERIM REPORT - DIOXIN STUDY

REPLY REQUESTED BY (DATE)

DIOXIN REMOVAL

Preliminary Laboratory studies to determine dilution level necessary to remove dioxins by filtration of TCP solutions have been completed. It was found that dilution to 10% TCP concentration was necessary for removal of dioxins to a level of less than 1 ppm in the filtrate.

The stock solution used in this study was a 37% TCP solution from regular plant production. Approximately 2% supercell was added to each sample after dilution, and the solution filtered on a buchner funnel fitted with No. 1 filter paper. A weighed sample of filtrate was extracted with 2 x 25 cc portions of chloroform. The combined chloroform extracts were concentrated to a 10 cc volume and chromatographed. Comparable results were obtained when diluted sample of TCP solution was filtered through a supercell pre-coat, with no filter-aid added directly to the solution. No dioxin was detected in filtrate after filtering 800 grams of 10% solution through 3 grams of pre-coat.

The dioxin content of all samples analyzed is based on solution weights. In order to determine dioxin content on a 100% TCP basis, several samples of filtrate will be concentrated to approximately 40% TCP before chloroform extraction. Based on previous experience, approximately 95% TCP recovery from dilute filtrate can be expected.

DILUTION DATA

<u>TCP Content</u>	<u>p-dioxin content (ppm)</u>	<u>Unidentified impurity* as dioxin</u>
37%	7	7
20%	2	3
15%	None detected (<1)	3
13%	" "	<3
12%	" "	<3
11%	" "	<2
10%	" "	None detected (<1)
5%	" "	" "

\* This impurity is believed to be an isomer of p-dioxin and calculated as such because standard for it is not available.

DIOXIN CONTENT OF TCP PROCESS STREAMS

Recent samples from various stages of TCP manufacture have been analyzed for dioxin content. Results are as shown on the following page.

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<u>Source</u>	<u>% TCP</u>	<u>Date Sampled</u>	<u>Dioxin Content (100% TCP Basis)</u>	<u>Remarks</u>
Tank #130 before steam distillation	39	12/65	56 ppm	
Tank #137	34	12/65	30	
Tank #133	35	12/65	34	
Tank #131 after methanol distillation	40	2/66	40	Composite batches 24-29.
Tank #131 after methanol distillation	46	2/66	21	Composite of 8 batches using all flake NaOH
Autoclave (Batch #39)	30	2/66	23	
Recovered TCP*	93	3/66	10	

\* "T" Mother Liquor acidified in Lab and organic layer dissolved in alkaline solution prior to chloroform extraction.

WAG/nc

*W. A. Goodloe*  
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cc: F. R. Kennedy

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