

Diamond Alkali Company

INTER-OFFICE CORRESPONDENCE

DACO 118

DATE

September 12, 1956

TO Mr. J. Burton	FROM Mr. J. J. Browne
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SUBJECT: Chronological Listing of Process Development - 2,4,5-TCP cc: Mr. J. R. Trocki

A chronological list of the process changes that have been instituted is attached.

Of particular interest is the series of failures of the rupture disc on the large autoclave in March and April 1955 concurrent with the 32° low reading on the temperature recorder. This combination may have introduced extreme amounts of chloracne producing materials in TCP stock. The storage tanks were very rarely emptied in this period since it was a production season. The impurities would then be mixed with normal material resulting in contamination of TCP solutions for several months coincidental with the major outbreak of chloracne cases in May-June of 1955.

Change of caustic ratio was made based on results of Boehringer Report No. 593. Graph of data is attached.

Following are some notes on the material contained in the report:

1. Adjustment of the pH of sodium salt to 10.1 results in almost quantitative removal of anisole while stripping at higher pH results in retention of 1% anisole in TCP. Perhaps this pH adjustment would improve our product and assist in removal of other organic compounds during the steam distillation.

<u>DATE</u>	<u>DESCRIPTION</u>	<u>MOLS T₄CB</u>	<u>MOLS NaOH</u>	<u>RATIO T₄CB/NaOH</u>	<u>TEMP. RANGE</u>
	Smith report	1.0	2.50	2.50	
1st Op'n	Original plant operation	3.24	8.75	2.70	
1/2/52	Oper. inst. rev. T. Barna	3.71	10.0	2.70	
1952-54	Oper. Inst. large & small autoclaves	10.4	25.0	2.40	180-186*
6/54	Oper. temp. raised to conform with oper. inst.	10.4	25.0	2.40	180
7/13/54	First batch steam stripped in plant (L-84) following lab. check in June by Schoffman	10.4	25.0	2.40	180

*Survey of 31 autoclave bathes from 12/28/53 to 6/7/54 indicated a plant practice of operating about 170°C. with some batches so low as 160°C. Operating instructions called for 180-186°C. so this temp. range was maintained from 6/54 to 9/54 lacking any other information.

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<u>DATE</u>	<u>DESCRIPTION</u>	<u>MOLS T₄CB</u>	<u>MOLS NaOH</u>	<u>RATIO T₄CB/NaOH</u>	<u>TEMP. RANGE</u>
9/54	Caustic ratio changed from 2.4/1 to 2.7/1 temp. 170-180° Steam stripping all production in small autoclave-acidifying.	11.1	30	2.70	170-180
10/54	First 2 plant 2,4,5-T batches made from direct sodium salt. 50% of sodium salt made by direct process.				
11/54	No production.				
12/54	All sodium salt by direct method.				
1/55	Changed to crystalline caustic replacing flake.				
1/55	Badger still used as steam stripper. Production increased to 1 large-1 small batch/shift.				
3/55	<u>Rupture disc failed in large autoclave</u> , at 452 psi. Temp. recorder found to be reading <u>32° low</u> .				
4/55	Rupture disc failures. Large autoclave. 4/8 4/22 4/23. Standards on T ₄ CB. Lowered from 1.25 to 1.20 due to high conversions.				
7/55	Test Ba L-101-L-123 for process evaluation.				
7/29/55	Process changes to improve color-kerosene insolubles-caustic insoluble.				
	1. Autoclave temp. 175-180°C. batch to be held as close to 180°C. as possible but no higher.	11.1	30	2.70	175-180
	2. MeOH dist. 120°C. max. (formerly 130°C.)				
	3. Steam dist. 120°C. max.				
	4. City water substituted for well water in dilutions.				
8/29/55	Lowered caustic ratio and temp. result of test batches.	12.1	30.2	2.5	170° max.
8/28/56	No major process changes to date.				
9/4/56	Caustic/T ₄ CB ratio changed from 2.5/1 to 2.7/1.	12.1	32.7	2.7	170° max.

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