

December 23, 1969

Mr. P. B. Kennedy - Cleveland

Mr. P. G. Steward - Newark

p-DIOXIN REVIEW

As you well know, the problem of chloracne in the manufacture of TCP has been known for quite a few years. During the late 50's, and particularly during the research program in 1960, information was developed both at Newark and at Concord that the causative agent might be a diphenyl ether. This belief was confirmed by Dow on March 24, 1965 in a meeting attended by Ed Chandler and yourself, together with representatives of Hercules and Hooker, when they said that though there are 26 or 27 suspect materials, the most active causative agent is "2,3,7,8 tetrachloro-dibenzo-p-dioxin (symmetrical)" --- see attached reports on meetings.

Following the meeting with Dow, an extensive series of studies was started directed toward: the identification of p-dioxin, determination as to how much was present in our products, and finally, means of removal. The first substantive results were reported in May of 1965 when, following perfection of analytical techniques (see attached), analysis of our product showed:

<u>Sample</u>	<u>Dioxin conc., ppm</u>
TCP Solution, 40%	25-40
TCP Solution, 40% (diluted and filtered)	None
Recovered Trichloroanisole	73
Recovered Methanol	None
"T" Acid (7 lots)	<10 (3 lots) to 40

Work in June 1965 was directed toward additional tests to determine levels present in plant streams and test removal by filtration. Samples were found with p-dioxin levels (on a 100% TCP basis) ranging from 80 ppm to a high of 140 ppm. Filtration was shown to markedly reduce or eliminate the p-dioxin. Samples of "T" acid assayed in September 1965 showed varying p-dioxin concentrations as follows:

<u>Lot Number</u>	<u>p-Dioxin Content, ppm</u>
123	13
124	26
281	7
282	7

Because of the start-up of the "D" Malt Unit, tests were not resumed until November, when efforts primarily were directed to removal of the p-dioxin by filtration. This information is briefly summarized in W. A. Goodloe's interim report of March 23, 1966.

Efforts during the rest of 1966 were directed toward getting a commercially practical means of removing the p-dioxin by filtration. Many problems were encountered as we attempted to scale-up our process. Alternate purification procedures were

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examined and removal by absorption on activated carbon appeared promising. Work continued in 1967 both on filtering and carbon absorption techniques, with the latter becoming the favored approach as we neared the design deadline for the commercial purification unit. Significant summary reports of lab work, written in January and July 1967, are attached. Purification of all TCP produced commenced in September 1967, initially in the 12" pilot column. A report of the operation of the pilot column is attached, but the important item to note is that the p-dioxin level in the treated TCP averaged 1.4 ppm (100% TCP basis) for the first 20,000 gallons treated.

Treatment of TCP continued until plant operations were suspended in August 1969. Though some variation in p-dioxin levels occurred, levels in our TCP and "T" acid were significantly reduced from those experienced prior to treatment. TCP composition reported by quarters for 1968 and 1969 is as follows:

	<u>2,4,5-TCP</u> %	<u>2,4,6-TCP</u> %	<u>DCP</u> %	<u>UNKNOWNES</u> %	<u>p-DIOXIN AND RELATED COMPOUNDS</u> ppm(1)
Jan.-March 1968	90.5	0.6	0.8	8.0	3.8
Apr.-June 1968	93.6	0.6	0.4	5.4	0.9
July-Sept. 1968	93.6	NA(2)	NA	NA	0.2
Oct.-Dec. 1968	91.9	NA	NA	NA	9.0(3)
Jan.-March 1969	95.0	0.6	1.0	3.2	3.2
Apr.-June 1969	NA	NA	NA	NA	1.7

- (1) 100% TCP basis
- (2) NA - Not Available
- (3) Very high p-dioxin level resulted when a labor-management dispute delayed necessary cleaning of the carbon tower.

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Attachments

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