

Monsanto

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NOV 2 1982

FROM
(NAME-LOCATION-PHONE) M. E. Whelan - WGK

DATE : November 1, 1982

cc E. A. Fay
F. B. Matthews
D. J. Metherd

SUBJECT : Chlorophenol Trace Impurity Levels

REFERENCE :

TO : ~~H. H. Kibbourne - NSA~~

Attached are results of our recent Dayton Laboratory analysis of Chlorophenol Products for dioxin content. As you are aware, process modifications in 1979 thru 1980 cleared all chlorophenol products of dioxins. The only continuing levels were found in the still pot residue which is currently stored pending incineration. A continuing sampling program was initiated which consisted of accumulating various product samples and shipping these to Dayton for analysis on a quarterly basis.

A recent calendar of events is really necessary to put these numbers in perspective.

- 6/81 Analysis continued to show process streams below the detection limits (1 ppb)
- 7/81 Residue in the chlorine supply line was beginning to restrict chlorine flow to the chlorinator.
- 8/81 The chlorine system was pressure jetted and caustic washed to remove the residue without success.
- 11/81 Higher levels of dioxin appeared in unrefined 93% 2-4 DCP that was shipped directly from the chlorinator.
- 1/82 Refined PCP samples began showing low level of dioxins in the lower chlorination ranges. The results of these samples were not received until July of 1982.
- 7/82 A new process for direct chlorination of PCP to 2-4 DCP was demonstrated. This was the first material made directly from the chlorinator since the November 93% 2-4 DCP. Subsequent analysis showed this material very high in dioxin.

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H. W. Kilbourne
Chlorophenol Trace Impurity Levels

Page 2
WGK
11/1/82

- 8/82 The chlorinator chlorine supply line was changed out and modifications made to prevent further build-up. Subsequent analysis confirmed that the build-up material was high in chlorinated phenol compounds, especially Penta Chlorophenol and very high in dioxin.
- 9/82 2-4 DCP manufactured showed much lower dioxin levels but still well above the detection level. The actual sparge ring in the chlorinator was changed completely to eliminate any potential for contamination.
- 10/82 Current 2-4 DCP production shows no dioxin at the detection limits of 1 ppb. Other ion interference make actual quantification of some chlorination isomers impossible since they could possibly hide dioxin peaks. For this reason, some results are reported as less than the interference peak height.

Summary

Current chlorophenol products including 2-4 DCP are free of dioxins at the detection limits of the mass spec. analysis. The early problems with the current 2-4 DCP production was a function of a physical problem in the chlorinator system and not of function of the new manufacturing process. The only known continuing source of dioxin in the chlorophenol process is the still pot residue which is being accumulated for future incineration.

If I can provide any other clarification or explanation of this problem, please let me know.

Michael J. Whelan
M. E. Whelan

/bb

Att.

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Date	Batch/Lot	Name	C1 ₁	C1 ₂	C1 ₃	C1 ₄	C1 ₅	C1 ₆	C1 ₇	C
<u>PCP Samples</u>										
1/82	5002	PCP Lot	5	5	3	8	4	ND	ND	
2/82	5007	"	2	4	3	6	4	ND	ND	
3/82	5012	"	ND	←						
4/82	5022	"	ND	←						
7/82	5043	"	ND	ND	ND	3	ND	→		
8/82	5044	"	ND	ND	ND	3	ND	→		
8/82	5047	"	ND	←						
9/82	BI 105	PCP From Final Distillation Cut	ND	←						
10/82		PCP From Tank 165 (Storage)	ND	ND	< 2		< 6	ND	ND	
<u>2-4 DCP Analysis (Unrefined)</u>										
4/81	7005	93% 2-4DCP	ND	ND	ND	5	18	14	8	
6/81	7006	93% 2-4 DCP	ND	←						
8/81	7009	93% 2-4 DCP	ND	←		ND	3	2	ND	
11/81	7010	93% 2-4 DCP	ND	< 7	14	29	61	81	58	
2/82	6018	96% 2-4 DCP (New Process)	ND	ND	27	44	87	79	110	
9/82	1101	"	ND	ND	ND	26	68	89	93	
9/82	1104	"	ND	ND	3	11	18	5	11	
10/82	J101	"	ND	ND	ND	< 7	< 6	6	2	
10/82	J103	"	ND	ND	ND	< 2	< 17	5	2	
10/82	J107	"	2	2	ND	4	< 3	ND	ND	
10/82	J110	"	ND	< 1	ND	< 2	ND	ND	< 1	
<u>OCP Crude</u>										
10/81	8005	OCP	ND	←						
6/82	8023	OCP	ND	←						
10/82		OCP Tank 163	ND	←						
10/82	8026	OCP	ND	←						
10/81		Still Bottoms Residue	5800	5000	1200	1770	4200	3940	3140	
Legend ND - None Detectable I - Interference Makes Value Non-Quantifiable < - Height of an Interference Peak Which Could be Hiding a Possible Dioxin Peak										

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