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TITLE: Chlorinated Dibenzo-p-dioxins in
Chlorophenols, Santophen®-1 and Process Samples

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ABSTRACT: Analyses of various chlorophenols for trace
amounts of chlorodibenzodioxin are presented.

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INTRODUCTION:

On Jan. 11, 1979 a derailed tank car spilled 20,000 gallons of crude o-chlorophenol near Sturgeon, Mo. Analysis of a retained sample of this material indicated the presence of chlorinated dibenzo-p-dioxins, including at least one tetrachlorodibenzo-p-dioxin (TCDD) isomer. Subsequently, a program was initiated to determine the levels of chlorinated dibenzo-p-dioxins in process samples and products and to establish the source of the dioxin contamination. Further, process changes were evaluated at the W.G. Krummrich Plant to determine the effect on the impurity levels.

SUMMARY:

Product and process samples from the chlorophenol and Santophen[®]1 processes at the W. G. Krummrich plant were collected by plant and D&P research personnel. These samples were submitted to MIC Environmental Sciences (MIC-ES), who coordinated analytical work with Monsanto Research Corporation (MRC), Dayton, Ohio. The results have shown the presence of chlorinated dibenzo-p-dioxins in many of the process samples and chlorinated phenol products and in several Santophen 1 samples. A process change was initiated which eliminated a caustic distillation from the chlorophenol process. Early indications are that this change has reduced the level of chlorinated dibenzo-p-dioxins to below the detection limit of the current methodology.

ANALYTICAL METHODOLOGY

The method used for the determination of the chlorinated dibenzo-p-dioxins in the samples has been previously described (Mieure, J. P. et al, J. Chrom. Science, 15, 275 (1977)). (Attached) Modifications were as follows:

- A) Apparatus - step 5 - the GC column was packed with 3% OV-101.
- B) Procedure - step 1 - the 5 g sample was dissolved in 50 ml of 10% methylene chloride in hexane. A 5 ml aliquot of this solution was added to the macro-column and eluted with 10% methylene chloride in hexane. 10% methylene chloride in hexane was used as the rinse solvent when transferring the sample to the concentrator. The eluate was concentrated to approximately 0.3 ml.
- C) Procedure - step 2 - the entire 0.3 ml concentrate was transferred quantitatively to the micro-column.

- D) Procedure - step 3.A. - the 50% methylene chloride in hexane fraction was evaporated to approximately 0.2 ml for GC/MS analysis. No chlorobenzene was added.

Detection of the chlorinated dibenzo-p-dioxins was by gas chromatography - mass spectrometry. The method was developed for the analysis of pentachlorophenol. However, its suitability for analysis of mono- and dichlorophenols and Santophen 1 was established by spiking TCDD into various samples. Details of the accuracy and precision are in a subsequent section.

CHLORINATED DIBENZO-P-DIOXINS IN SPILLED O-CHLOROPHENOL

A reserve sample of the crude o-chlorophenol spilled at Sturgeon, Mo., was analyzed for chlorodioxins by MIC-ES. The sample was identified by the following designations:

GATX 12932
KL 2 8001-1 1/7/79
MIC-ES Log 900701

A sample of o-chlorophenol from Fisher Scientific Co., Lot 776410 was analyzed concurrently as a blank. In addition GATX 12932 and the Fisher material were submitted to MRC as ES-11 and ES-12, respectively. The results are in Table 1.

DETERMINATION OF CHLORINATED DIBENZO-P-DIOXINS IN CHLOROPHENOL AND SANTOPHEN 1 PROCESS SAMPLES

Following detection of TCDD and other chlorinated dibenzo-p-dioxins in the reserve sample from the Sturgeon spill, samples were collected from the chlorophenol process to identify the source and level of contamination. These samples are identified as MB 655-696 and ES-1-51 on the sample identifications in Table II.

Results of the above analyses indicated the source of the chlorinated dibenzo-p-dioxins was the caustic neutralization and distillation in the chlorophenol process. A process change eliminating this step was made and further samples were submitted. These are identified as MB 704-43 and ES-52-90. Preliminary results suggest the process change has reduced or eliminated the chlorinated dibenzo-p-dioxins in the chlorophenol process.

Although some samples were analyzed by MIC-ES, most were coded by MIC-ES and forwarded to MRC for analysis. The results of those analyses are tabulated in Table III. (Analyses performed by MIC-ES are designated by *.)

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The data for TCDD in Tables I and III are total concentration of all isomers in each sample. The only isomers for which MIC-ES has standards are the 1,3,6,8- and 2,3,7,8-TCDD. These materials have different GC retention times under the conditions of analysis, so discrimination is possible. Figure I shows the separation of the two materials in a standard mixture. Table IV shows results for analyses by MIC-ES for which "isomer distribution" has been done. TCDD was reported as two groups: 1) isomers which elute at the retention time of 2,3,7,8-TCDD and 2) those which elute at other retention times, including 1,3,6,8-TCDD. These levels do not imply that the compounds have been identified as the isomer in question, only that there is retention time agreement. It is evident from these results that isomer distribution does vary from product to product.

Quantitation was by an external standard method using a mixed standard of available chlorinated dibenzo-p-dioxins. No standards were available for the mono- and pentachlorodibenzo-p-dioxins, so response factors for these materials were estimated. Chlorophenols from outside Monsanto were analyzed as "blanks" during this study. The results for these materials are in Table V. The material designated Fisher OCP is probably the best material for a blank for these analyses.

During the course of the study the samples were also analyzed for chlorinated dibenzofurans. However, recoveries of spiked tri- and tetrachlorodibenzofurans by MRC were low, so the data for the chlorinated dibenzofurans is not valid. No results are reported in this study.

ACCURACY AND PRECISION

The accuracy of the determinations reported in this study was determined by fortification (spiking) of selected samples with known amounts of chlorinated dibenzo-p-dioxins. In almost all cases 2,3,7,8-TCDD was the material used for spiking. These sets of data relevant to this study were divided as shown here:

<u>Spiking Lab</u>	<u>Analysis Lab</u>	<u>Table #</u>
MIC-ES	MIC-ES	VI-A
MIC-ES	MRC	VI-B
MRC	MRC	VI-C

The recovery data are reported in Table VI as indicated above. Table VI-D contains recoveries for one sample (MB 654) which MRC spiked with a mixed standard.

Determination of Trace Amounts of Chlorodibenzo-p-Dioxins and Chlorodibenzofurans in Technical Grade Pentachlorophenol

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Experimental

Abstract

A method has been developed for determining selected impurities in pentachlorophenol. Chlorodibenzo-p-dioxins and chlorodibenzofurans are isolated from the sample matrix with a two-step column chromatographic procedure. Measurement of these components is by gas chromatography using electron capture, flame ionization, or mass spectrometric detection. The method can be used as a quality control procedure for monitoring these components at ppb-ppm concentrations in technical pentachlorophenol.

Introduction

Pentachlorophenol (PCP) has wide application as a wood preservative and fungicide. Technical grade PCP contains low levels of several impurities, including chlorodiphenyl ethers (CDPEs), chlorodibenzo-p-dioxins (CDDs), chlorodibenzofurans (CDFs), and hydroxychlorodiphenyl ethers. Analytical methods for measuring hexa-, hepta- and octa-CDD impurities in PCP were recently reviewed (1). The authors concluded the method of Crummett and Stehl (2) provided greatest accuracy and convenience. Newer methods by Pfeiffer (3) and Buser (4) and a revision of (2) by Blaser et al. (5) have also been published.

These methods have limitations which preclude their use for routine CDD monitoring of technical grade PCP. The HPLC method of Pfeiffer is applicable to highly purified PCP. However, it is subject to interference from non-CDD impurities in technical grade PCP.

The method described by Buser provides adequate cleanup, but sample pretreatment may lead to positive or negative errors. For example, exposure of technical PCP to strong alkali has been shown to cyclize certain *o*-hydroxychlorodiphenyl ethers, yielding anomalously high concentrations of CDD (6). Also, petroleum ether does not quantitatively extract octa-CDD from alkali solution at the 500-1500 ppm levels typically found in technical PCP.

Blaser's method is suitable for determining CDD in technical PCP, but it requires use of GC/MS as the measurement technique. GC/MS is beyond the scope of many laboratories, including most plant quality control laboratories.

This paper describes a method which is amenable to routine monitoring of CDDs in technical PCP. CDD components are isolated from PCP by a two-step column chromatographic procedure. The first step removes phenolic components and the second step removes chlorobenzenes, chlorobiphenyls and CDPEs. The CDD-CDF fraction is analyzed by gas chromatography with electron capture, flame ionization or mass spectrometric detection. Detection limits depend on the detector and the component being measured. The lower detection limit for 2,3,7,8-tetra-CDD, which we have not detected in technical PCP, is 10 ppb when measured by GC/MS using selected ion monitoring.

Reagents

1. Benzene—Pesticide grade.
2. Hexane—Pesticide grade.
3. Methylene Chloride—Pesticide grade. Solutions containing 2 and 50% (V/V) methylene chloride are prepared by transferring 20 and 500 ml methylene chloride to one-liter volumetric flasks and diluting to the mark with hexane.
4. Sodium sulfate—anhydrous granular, Analytical Reagent grade.
5. Alumina
 - A. Fisher Adsorption A-540, 80-200 mesh.
190 g is heated to 400°C for 4 hours, cooled, and poured into a pint bottle. Ten ml of distilled water is pipetted into the bottle and the bottle capped and shaken at least ten minutes. (The cap should have an aluminum liner.) Absorption of water by alumina is exothermic, so the bottle will get hot to the touch. Alumina deactivated with 5% water in this manner can be stored on a laboratory shelf for several weeks without deterioration of quality.
 - B. ICN Aluminum Oxide W 200 basic, activity grade Super 1, used as received.
6. Chlorodioxin Standards—as available. We use concentrations of 0.01 - 0.1 µg/ml tetra-CDD and 1-10 µg/ml of a mixture of hexa-, hepta- and octa-CDD in benzene.

Apparatus

1. Liquid Chromatography Columns
 - A. Macro—30 cm × 1.8 cm i.d. Pyrex with Teflon stopcock.
A small amount of glass wool is placed in the bottom of the column, which is then half filled with benzene. Next, 40 ml of Fisher deactivated alumina is poured into the column and allowed to settle while tapping the column wall. The alumina is topped with 1 inch of sodium sulfate. The stopcock at the bottom of the column is opened and the level of benzene adjusted to the top of the packing. Caution: To minimize exposure to benzene, the entire macro-column procedure (column preparation, separation and solvent evaporation) should be carried out in a well-ventilated hood!
 - B. Micro—15 cm × 0.5 cm i.d. disposable Pasteur pipette.
A small glass wool plug is placed in the bottom of the pipette and 1.0 g of dry ICN aluminum oxide is added. The packing is settled by gently tapping 15-20 times with a small spatula (4).
2. Sample Concentrator
500 ml Kuderna-Danish evaporative concentrator with a 3-ball Snyder column and a 15 ml receiver (Ace Glassware). Further concentration is accomplished with a micro-Kuderna-Danish concentrator with a 4 ml receiver.
3. Flame Ionization Gas Chromatograph
A Hewlett-Packard 5731A or equivalent. The column is 3 mm i.d. × 4 m glass packed with 3% XE-60 or OV-1 on

XB665	90J0811	ES-16	1/1/79	Color	C-101	Chlorinated Mass - C Series	Yes
XB666	90J0812	ES-17	1/2/79	101	C101-103	Blown Crude PCP - C Series	Yes
XB667	90J0813	ES-18	1/2/ (4cc)	152	C-2	* Mono Crude - Neutralized - C Series	Yes
XB668	90J0814	ES-19	1/2/79	103	C-2	Dry Fractions Cut - C Series	Yes
XB669	90J0815	ES-20	1/1/79	P. Flow	C-2	Phenol Cut - C Series	Yes
XB670	90J0816	ES-21	1/1/79	104	C-2	Para-Rich Cut - C Series	Yes
XB671	90J0817	ES-22 (Spiked) ES-23 (Duplicate) ES-24 (Duplicate)	1/1/79	160	C-2	Refined PCP - C Series	Yes
XB672	90J0818	ES-25	1/1/79	still	C-2	Residue - C Series	Yes
XB673	90J0819	ES-26	2/27/79 (acc)	Color	B-123	Color Charge - B Series	Yes
XB674	90J0820	ES-27	2/27/79	123	B-123	Crude PCP Chlorinator Batch - B Series	Yes
XB675	90J0821	ES-28	2/28/79	101	B-22	Blown PCP Crude - B Series	Yes
XB676	90J0822	ES-29	1/1/79	152	B-123-B	Neutralized Chlor Mass - B Series	Yes
XB677	90J0823	ES-30 ES-31 (Spiked) ES-32 (Duplicate)	1/1/79	For. Flow	C-1	DCP Cut - B Series	Yes
XB678	90J0824	ES-33	1/1/79		C-1	Para Rich	Yes
XB679	90J0825	ES-34	1/2/79		C-1	Residue Sample	Yes
XB680	90J0826	ES-35	1/1/79	668	C-103	Para-Rich - Chlorinator Charge for 93V 2.4 DCP	Yes
XB681	90J0827	ES-36	1/1/79	668	C-103	93V 2.4 DCP before aeration	Yes
XB682	90J0828	ES-37	1/2/79	668	C-103	93V 2.4 DCP after aeration	Yes
XB683	90J0829	ES-38	2/29/79	129	B-10	still Residue + water	Yes
XB684	90J0830	ES-39	2/29/79		B-120	Para-Rich (prior to chlorination) 93V 2.4 DCP raw material	Yes
XB685	90J0831	ES-40	2/28/79		B-120	93V 2.4 DCP (acc sample) before aeration	Yes

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- 7 -

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TABLE 12

SAMPLE IDENTIFICATION MATRIX

REF TEST/ACK NUMBER	NIC-ES LOG NUMBER	NRC CODE	DATE OF SAMPLE	TANK NUMBER	SATCH NUMBER	DESCRIPTION OR SAMPLE LABEL	STATUS
2541					EE-12-8003	Orthochlorophenol - Crude	—
2553	9030103				KL02-15	Sandophen 1 Flakes	Yes
2554	9030104	ES-10			3-14	Para-chlorophenol (Lot KL02-5021)	Yes
2555	9030801	ES-1	2/26/79	123	B-117	Chlorinacor Charge (PCZ)	Yes
2556	9030802	ES-2	2/26/79	123	B-117	Flashed Chlorinacor Batch - A Series	Yes
2557	9030803	ES-3	2/26/79	1 Pac.		Crude MODO - Blown - A Series	Yes
2558	9030804	ES-4	2/27-79	152	B-21	Neutralized Crude PCZ - A Series	Yes
2559	9030805	ES-5	2/28/79	4		Forward Flow - Dry Fractions	Yes
2560	9030806	ES-6	2/28/79	105	B-21	Recycle (etc) BOK CUC - A Series	Yes
2561	9030807	ES-7 ES-8 (Split-1) ES-9 (Duplicate)	2/1/79	112	B-21	Still Para-Mich CUC - A Series	Yes
2562	9030808	ES-13	2/1/79	160	B-21	PCZ CUC - A Series	Yes
2563	9030809	ES-14	2/1/79	129	B-21	Still Residue - A Series	Yes
2564	9030810	ES-15	2/1/79		C-101	Chlorinacor Charge	Yes

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TABLE 11 (Cont'd)

SAMPLE IDENTIFICATION MATRIX

DCP RESEARCH NUMBER	MIC-ES LOG NUMBER	MPC CODE	DATE OF SAMPLE	TANK NUMBER	BATCH NUMBER	DESCRIPTION OF SAMPLE LABEL	CASSTIC
MB686	9030832	ES-41	3/1/79		B-120	93% 2,4 DCP Finished Product KL-03-7009	Yes
MB691	9030833	ES-42	3/11/79	129	C-9	Still Residue	Yes
MB692	9030834	ES-43	3/11/79	129	C-101	Still Pot Charge - DCP	Yes
MB693	9030835	ES-44	3/10/79	42	C-101	Forcut (sic) 2-4 Dry Fraction	Yes
MB694	9030836	ES-45 ES-46 (Spiked) ES-47 (Duplicate)	3/11/79	3	C-101	2,4 DCP Cut	Yes
MB695	9030837	ES-48	3/12/79	812	C-101	2,4 Fractions (Heavy)	Yes
MB696	9030838	ES-49	3/12/79	Still	C-101	Residue Off C-101 2-4	Yes
MB697	9030839	ES-50				(Relabeled MB686)	Yes
MB698	9031501		3/2/79		KL-03-41	S-1 Flakes	Yes
MB699	9031502				KL-01-06	S-1 Flakes	Yes
MB701	9032701				Lot 12302-78	Rhodia PCP	—
MB702	9032702					S-1 Still Bottom Residue	Yes
MB704	9042401	ES-52	4/16/79	152	D-10	Blown Chlor Crude	No
MB705	9042402	ES-53	4/16/79	82	D-10	Dry Fractions Cut	No
MB706	9042403	ES-54	4/17/79	83	D-10	Phenol Cut	No
MB707	9042404	ES-55	4/17/79	11/12	D-10	Para-Rich Cut	No
MB708	9042405	ES-56 ES-57 (Duplicate)	4/17/79	160	D-10	PCP Product Cut	No
MB709	9042406	ES-58	4/17/79	Still	D-10	Residue	No
MB710	9042407	ES-59	4/16/79	Chlor		DCP Chlor Feed	No
MB711	9042408	ES-60	4/16/79	Chlor		DCP Finished Chlor Mass	No
MB712	9042409	ES-61	4/16/79	Chlor		Blown DCP	No
MB713	9042410	ES-62 ES-63 (Duplicate)	4/18/79	42		2-4-D Refined Aerated (Still Charge)	No
MB714	9042411	ES-64	4/19/79	12	D-102	First Cut - 2,4 - DCP Batch	No
MB715	9042412	ES-65	4/20/79	2	D-102	2,4-DCP	No
MB716	9042413	ES-66	4/19/79	12	D-102	High Boiler Cut	No
MB717	9042414	ES-67	4/19/79		D-102	2,4 Batch Residue	No
MB721	9050901	ES-68	4/25/79	152	D-19	Still Charge	No
MB722	9050902	ES-69	4/25/79	Chlor	D-110	Chlorinator Crude	No
MB723	9050903	ES-70	4/25/79	101	D-108-110	Aerated Crude	No
MB724	9050904	ES-71	4/26/79	103	D-19	Dry Fractions Cut	No
MB725	9050905	ES-72	4/27/79	103	D-19	Recycle Phenol	No
MB726	9050906	ES-73	4/27/79	112	D-19	Para Rich Cut	No
MB727	9050907	ES-74 ES-75 (Duplicate)	4/27/79	160	D-19	Parachlorophenol	No
MB728	9050908	ES-76 ES-77 (Duplicate)	4/27/79	Still	D-19	Residue	No
MB729	9050909	ES-78				Relabeled MB541	—
MB730	9050910	ES-79			KI-06-124	S-1 Flake	—
MB736	9051701	ES-80	5/7/79	101	—	Fresh PCP	No
MB737	9051702	ES-81	5/7/79	251		Reactor Mass	No
MB738	9051703	ES-82	5/7/79		10	S-1 PCP Cut	No
MB739	9051704	ES-83 ES-90 (Duplicate)	5/7/79		10	S-1 Intermediate Cut	No
MB740	9051705	ES-84 ES-85 (Duplicate)	5/7/79 (sic)		10	S-1 Santophen Cut	No
MB741	9051706	ES-86	5/7/79		10	S-1 Last Fraction	No
MB742	9051707	ES-87	5/7/79		10	S-1 Residue Still Residue	No

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- 9 -

TABLE III

PPS Levels of Chlorinated Dibenzo-p-dioxins in
Chloronaphenol and Santophen Process Samples

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	Cl ₁	Cl ₂	Cl ₃	Cl ₄	Cl ₅	Cl ₆	Cl ₇	Cl ₈
MS41*	200	50	ND<20	ND<30	ND<200	ND<700	5000	ND<3000
SANTOPHEN → MS653*	7100	9800	3000	65	ND<50	ND<50	ND<50	ND<50
MS653*	5100	8600	1800	35	ND<50	ND<50	ND<50	ND<50
MS653*	—	10000	—	70	—	ND<50	—	ND<50
PARACALD → MS654*	9800	8100	3800	130	ND<50	ND<50	ND<50	ND<50 X
MS654*	6100	7000	3100	100	ND<50	ND<50	ND<50	ND<50
MS654*	—	7000	—	110	—	ND<50	—	ND<50
MS654	18000	5100	2100	110	ND<50	ND<40	ND<20	ND<30
MS654	11000	5600	2800	100	ND<20	ND<40	ND<40	ND<30
MS655	ND<50	ND<20	ND<30	ND<10	ND<50	ND<40	ND<20	ND<30
MS656	ND<50	ND<20	ND<30	ND<10	ND<50	ND<40	ND<20	ND<30
MS657	ND<50	ND<20	ND<30	ND<10	ND<50	ND<40	ND<20	ND<30
MS658	ND<50	ND<20	ND<30	ND<10	ND<50	ND<40	ND<20	ND<30
MS659	890	180	45	ND<10	ND<50	ND<40	ND<20	ND<30
MS660	ND<50	ND<20	ND<30	ND<10	ND<50	ND<40	ND<20	ND<30
STILL P. 41 → MS661	180000	91000	21000	180	ND<50	ND<40	ND<20	ND<30
MS661	180000	79000	12000	270	ND<50	ND<40	ND<20	ND<30
MS662	1800	970	180	ND<10	ND<50	ND<40	ND<20	ND<30
RESIDUE → MS663 (1st Ext)	64000	59000	15000	3100	6700	1300	2200	440
(2nd Ext)	4500	5600	2400	250	170	350	220	87
MS664	ND<30	120	81	ND<10	ND<20	ND<40	ND<40	ND<30
MS665	ND<30	ND<30	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS666	ND<30	ND<30	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS667	ND<30	ND<30	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS668	ND<30	ND<30	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS669	ND<30	ND<30	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS669	ND<30	39	28	ND<10	ND<20	ND<40	ND<40	ND<30
MS670	250	100	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS671	12000	2600	160	ND<10	ND<20	ND<40	ND<40	ND<30
MS671	10000	2000	140	ND<10	ND<50	ND<40	ND<40	ND<30
MS671*	8500	2200	300	ND<5	ND<40	ND<20	ND<20	ND<20
RESIDUE → MS672 (1st Ext)	280000	200000	35000	5900	1900	5200	1300	160
(2nd Ext)	11000	6400	2300	240	220	220	ND<40	ND<30
MS673	73	87	66	ND<10	ND<20	ND<40	ND<40	ND<30
MS674	74	83	55	ND<10	ND<20	ND<40	ND<40	ND<30
MS675	75	65	39	ND<10	ND<20	ND<40	ND<40	ND<30
MS676	52	ND<30	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS677	ND<30	ND<30	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS677	ND<30	55	ND<20	ND<10	ND<20	ND<40	ND<40	ND<30
MS677	ND<30	57	58	ND<10	ND<20	ND<40	ND<40	ND<30
PARACALD → MS678	22000	5400	1400	21	ND<20	ND<40	ND<40	ND<30
RESIDUE → MS679	240000	190000	64000	6200	100	450	280	140
PARACALD → MS680	22000	7800	2700	69	ND<20	ND<40	ND<40	ND<30
24DCP-BE/OP MS681	3400	14000	13000	340	ND<20	ND<40	ND<40	ND<30 X
24DCP-C/OP MS682	3500	29000	13000	190	ND<20	ND<40	ND<40	ND<30 X

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TABLE IV
PPB Levels of TCDD in Selected Samples
(MIC-ES)

<u>Sample</u>		<u>1,3,6,8- TCDD et.al.</u>	<u>2,3,7,8- TCDD</u>
Santolun —	MB653	65	ND<20
	MB653 (Replicate)	35	ND<20
	MB653 (Replicate)	70	ND<20
parachlorophenol —	MB654	110	23
	MB654 (Replicate)	88	25
	MB654 (Replicate)	110	ND<60
2,4-DCP —	MB697	47	180
	MB697 (Replicate)	85	240
SANTOMER —	MB698	20	ND<10
	MB699	33	ND<10
PCP —	MB701	ND<20	ND<20
RESIDUE —	MB702	170	ND<150
RESIDUE —	MB709	2200	190
2,4-DCP —	MB715	ND<20	ND<20
2,4-DPP RESIDUE —	MB717	7500	660
237 RESIDUE —	MB741	25	ND<20
Eastman PCP		ND<20	ND<20
Lab 2,4-DCP (unknown source)		ND<20	ND<20

TABLE IV
PPB Levels of TCDD in Selected Samples
(MIC-ES)

<u>Sample</u>	<u>1,3,6,8- TCDD et. al.</u>	<u>2,3,7,8- TCDD</u>
MB653	65	ND<20
MB653 (Replicate)	35	ND<20
MB653 (Replicate)	70	ND<20
MB654	110	23
MB654 (Replicate)	88	25
MB654 (Replicate)	110	ND<60
MB697	47	180
MB697 (Replicate)	85	240
MB698	20	ND<10
MB699	33	ND<10
MB701	ND<20	ND<20
MB702	170	ND<150
MB709	2200	190
MB715	ND<20	ND<20
MB717	7500	660
MB741	25	ND<20
Eastman PCP	ND<20	ND<20
Lab 2,4-DCP (unknown source)	ND<20	ND<20

TABLE IV

PPB Levels of TCDD in Selected Samples
(MIC-ES)

<u>Sample</u>	<u>1,3,6,8- TCDD et. al.</u>	<u>2,3,7,8- TCDD</u>
MB653	65	ND<20
MB653 (Replicate)	35	ND<20
MB653 (Replicate)	70	ND<20
MB654	110	23
MB654 (Replicate)	88	25
MB654 (Replicate)	110	ND<60
MB697	47	180
MB697 (Replicate)	85	240
MB698	20	ND<10
MB699	33	ND<10
MB701	ND<20	ND<20
MB702	170	ND<150
MB709	2200	190
MB715	ND<20	ND<20
MB717	7500	660
MB741	25	

Eastman PCP

HA 1135
in Dated
6-26-79

F120 ac 71135(C)
4-14 Para CP
Lot KL 02-5021
(Feb 1979)
7-14 24 DCP
56- March 1979