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TITLE: ANALYSIS OF 2,4-DICHLOROPHENOLS FOR
CHLORINATED DIBENZO-p-DIOXINS

AUTHORS: Frederick D. Hileman

ABSTRACT: A sample of 2,4-dichlorophenol from the Krummrich Plant was analyzed at the Dayton Laboratory in the Ultratrace Facility. The samples were analyzed for the presence of chlorinated dibenzo-p-dioxins.

Technical Approval: Frederick D. Hileman
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Approval date: 2-25-82

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AUTHORS: Frederick D. Hileman
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Analyses: D. E. Kirk

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SAMPLE PREPARATION AND ANALYSIS

One sample of 2,4-dichlorophenol was received for the analysis of chlorinated dibenzo-p-dioxins (CDDS). In addition a sample from a previous analysis, A-102 and 103 was to be reanalyzed. The samples were assigned MRC log number 1-82-02-11-02.

The samples were prepared for analysis by dissolving five grams of dichlorophenol in 1N KOH. The basic solution was extracted with 40 mL of petroleum ether by shaking the mixture for 15 minutes. The base layer was removed and the petroleum ether was washed with 50 mL of organic free water. This water wash was removed and the petroleum ether was dried over anhydrous sodium sulfate.

The petroleum ether was concentrated to 0.5 mL and applied to a chromatography column containing 4 grams of Woelm basic alumina. Due to some problems in lower recoveries for the monochloro and octachlorodibenzo-p-dioxin in the previous study the 10% dichloromethane/hexane elution was changed to 2% dichloromethane/hexane. The column was first eluted with 10 mL of this 2% dichloromethane/hexane and this fraction discarded. An additional elution was made with 15 mL of 50% dichloromethane/hexane and 300 μ L of dodecane was added to this eluant as a keeper. The elutant was concentrated to 300 μ L and submitted for GC/MS analysis.

The GC/MS analysis was carried out on a Hewlett Packard 5985B GC/MS system. A 30 meter SE-54 capillary column was used for all gas chromatographic separations. The mass spectrometer was used in the selected ion monitoring mode analyzing for ions characteristic of monochloro through octachlorodibenzo-p-dioxin.

The results of the analyses are shown in Table 1. In the case for sample A-102 and 103 the results are given as the sum total of all isomers of a given degree of chlorination of dibenzo-p-dioxin. The spiked samples were readily detected with the exception of the low level monochlorodibenzo-p-dioxin (MCDD) spike. The change in column elution conditions did not improve the recoveries from the last analyses and we are currently trying to resolve this problem. Note that for the low level MCDD spike, the "found" level was below the "Method detection limit". This "found" level reflects the instruments sensitivity while the "Method detection limit" takes into account the poor recovery for the MCDDs.

TABLE 1. ANALYSIS OF 2,4-DICHLOROPHENOL FOR
CHLORINATED DIBENZO-p-DIOXINS

ng CHLORINATED DIBENZO-p-DIOXIN/gm PHENOL (ppb)

<u>Sample</u>	<u>Cl₁</u>	<u>Cl₂</u>	<u>Cl₃</u>	<u>Cl₄</u>	<u>Cl₅</u>	<u>Cl₆</u>	<u>Cl₇</u>	<u>Cl₈</u>
2-7-82 Tank 163	ND*	ND	ND	ND	ND	ND	ND	ND
2-7-82 Tank 163 (Rep)	ND	ND	ND	ND	ND	ND	ND	ND
2-1-82 A-102 and 103	ND	ND	ND	2.1	4.9	5.4	3.6	5.7
2-2-82 B-101 (Blank)	ND	ND	ND	ND	ND	ND	ND	ND
Method Detection Limit	10	1	1	1	1	1	1	1
Spike Level	3.9	1.7	2.0	1.8	3.8	6.4	3.6	7.6
2-7-82 Tank 163 (found)	ND	1.0	1.9	1.6	3.6	5.7	3.3	6.1
Recovery %	-	59	95	89	95	89	92	80
Spike Level	39	17	20	18	38	64	36	76
2-7-82 Tank 163 (found)	.6	14	18	15	34	59	36	55
Recovery %	2	82	90	83	89	92	100	72

* ND=none detected at the method detection limit

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REPORT NO.: MDA-330

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DATE: October 12, 1982

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TITLE: ANALYSES OF PARACHLOROPHENOLS FOR CHLORINATED
DIBENZO-p-DIOXINS

AUTHORS: ~~Environmental~~ Frederick D. Hileman

ABSTRACT:

Five samples of parachlorophenol from the Krummrich Plant were analyzed at the Dayton Laboratory in the Ultratrace Facility. The samples were analyzed for the presence of chlorinated dibenzo-p-dioxins.

Technical Approval:

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10/12/82

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REPT. NO.: MDA-330
AUTHORS: Frederick D. Hileman
TITLE: ANALYSES OF PARACHLOROPHENOLS FOR CHLORINATED
DIBENZO-p-DIOXINS

100

COPY NO.: 100

SAMPLE PREPARATION AND ANALYSIS

Five samples of parachlorophenol were received for the analysis of chlorinated dibenzo-p-dioxins (CDD). The samples were assigned MRC log number 1-82-09-14-02.

The samples were prepared for analysis by dissolving five grams of the phenol in 1N KOH. The basic solution was extracted with 40 mL of petroleum ether by shaking the mixture for 15 minutes. The base layer was removed and the petroleum ether was washed with 50 mL of organic free water. This water wash was removed and the petroleum ether was dried over anhydrous sodium sulfate.

The petroleum ether was concentrated to 0.5 mL and applied to a chromatography column containing 2.5 grams of Woelm basic alumina. This column was first eluted with 10 mL of 2% dichloromethane/hexane and this fraction discarded. An additional elution was made with 15 mL of 50% dichloromethane/hexane and 300 μ L of dodecane was added to this eluant as a keeper. The eluant was concentrated to 300 μ L and submitted for GC/MS analysis.

The GC/MS analysis was carried out on a Hewlett Packard 5985B GC/MS system. A 30 meter Se-54 capillary column was used for all gas chromatographic separations. The mass spectrometer was used in the selected ion monitoring mode analyzing for ions characteristic of various CDDs.

The results of the analyses are given in Table 1. In all cases the results are given as the sum total of all isomers of a given degree of chlorination.

In the case for the tetrachlorodibenzo-p-dioxins (TCDDs), the 2,3,7,8-TCDD has been listed separately. Thus for KB-5043 there was a total of 2.6 ppb TCDDs, however less than 1 ppb coeluted with the 2,3,7,8-TCDD. In most instances the TCDDs consisted of the 1,3,6,8-TCDD and 1,3,7,9-TCDD.

TABLE 1. ANALYSIS OF PARACHLOROPHENOLS FOR CHLORINATED DIBENZO-p-DIOXINS
ng/gm (ppb)

Sample ID	Cl ₁	Cl ₂	Cl ₃	Cl ₄	Cl ₄ *	Cl ₅	Cl ₆	Cl ₇	Cl ₈
KB-5043	ND	ND	ND	2.6	ND	ND	ND	ND	ND
KB-5043 Replicate	ND	ND	ND	2.4	ND	ND	ND	ND	ND
KB-5044	ND	ND	ND	3.2	ND	ND	ND	ND	ND
KB-5045	ND	ND	ND	ND	ND	ND	ND	ND	ND
KB-5046	ND	ND	ND	2.7	ND	ND	ND	ND	ND
KB-5047	ND	ND	ND	ND	ND	ND	ND	ND	ND
Blank	ND	ND	ND	ND	ND	ND	ND	ND	ND
Method Detection Limit	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Spike Level	2.5	2.5	2.7	---	2.0	2.4	2.3	2.5	2.0
KB-5043	2.2	2.5	2.6	---	1.7	1.7	1.9	2.4	1.9
Recovery %	88	100	96	---	85	71	83	96	95
Spike Level	25	25	27	---	20	24	23	25	20
KB-5043	18	20	22	---	16	22	21	22	17
Recovery %	72	81	81	---	80	92	91	88	85

* = analysis of the 2,3,7,8-tetrachlorodibenzo-p-dioxin.

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