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

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ABSTRACT:

Three samples of 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:

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10-12-82

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ANALYSES OF DICHLOROPHENOLS FOR CHLORINATED
DIBENZO-p-DIOXINS

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Analyses: D. E. Kirk

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Three chlorophenols were received for the analysis of chlorinated dibenzo-p-dioxins. They were assigned MRC Log. Number 1-82-10-05-02.

The samples were prepared for analysis by dissolving five grams of the phenol in 1N KOH. The dioxins were then extracted with 40 mL of petroleum ether by shaking for 15 minutes. The base layer was removed and the petroleum ether was washed a second time with base followed by 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 a 50% dichloromethane/hexane and 300 µL of dodecane was added to this eluant as a keeper. The eluant was concentrated to 300 µL 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 in the selected ion monitoring mode analyzing for ions characteristic of various CDDs.

The results of the analyses are given in Table 1. Note that the results are reported as the sum of all isomers for a given degree of chlorination. Quality control results have been reported for both the 2,4-dichlorophenols and for the crude monochlorophenol.

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

Sample	Cl ₁	Cl ₂	Cl ₃	Cl ₄	Cl ₅	Cl ₆	Cl ₇	Cl ₈
Refined 2,4-D 10-3-82 J-101	ND*	ND	ND	ND	ND	ND	ND	ND
Refined 2,4-D 10-2-82 I-102	ND	ND	ND	ND	ND	ND	ND	ND
Refined 2,4-D (Rep) 10-2-82 I-102	ND	ND	ND	ND	ND	ND	ND	ND
Crude Mono 10-3-82 J-1	ND	ND	ND	1.4	3.0	3.8	4.5	5.9
Crude Mono (Rep) 10-3-82 J-1	ND	ND	ND	1.6	3.0	4.3	4.9	5.1
Blank	ND	ND	ND	ND	ND	ND	ND	ND
Method Detection Limit	1	1	1	1	1	1	1	1
Low Level Spike	2.5	2.5	2.7	2.0	2.4	2.3	2.5	2.0
Refined 2,4-D 10-2-82 I-102	1.7	2.1	2.3	2.0	2.3	2.4	2.8	1.9
Recovery %	68	84	84	100	96	104	112	95
Crude Mono 10-3-82 J-1	2.1	1.8	3	3.1	4.8	5.3	5.8	6.4
Recovery %	84	72	111	80	75	-	-	-
High Spike Level	25	25	27	20	24	23	25	20
Refined 2,4-D 10-2-82 I-102	18	18	22	17	22	21	22	19
Recovery %	72	72	81	85	92	91	88	95
Crude Mono 10-3-82 J-1	22	20	24	19	24	26	27	28
Recovery %	88	80	89	90	88	96	89	110

*ND = none detected at the method detection limit.

- Spike levels were not sufficiently larger than the amount of dioxin originally in the sample to be considered valid.

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