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TITLE: ANALYSIS OF TEXAS CITY SOIL SAMPLES FOR POLY-
CHLORINATED DIBENZO-P-DIOXINS (PCDDs) and POLY-
CHLORINATED BIPHENYLS (PCBs)

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ABSTRACT: Three soil samples were received from Monsanto
Texas City for the analysis of polychlorinated
dibenzo-p-dioxins (PCDDs) and polychlorinated
biphenyls (PCBs). The results of these analyses
are discussed in this report.

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AUTHORS: Frederick D. Hileman, B. Mason Hughes
TITLE: ANALYSES OF TEXAS CITY SOIL SAMPLES FOR POLYCHLORINATED
DIBENZO-P-DIOXINS (PCDDs) AND POLYCHLORINATED BIPHENYLS

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Sample Preparation: D. Kirk

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Three soil samples labeled TP-1 (Sample A); TP-S (Sample B), and TP-S;SW (Sample C) were received for the analysis of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated biphenyls (PCBs). These samples were assigned MRC Log number 1-82-05-07-01. Table 1 shows the results of the dioxin analyses, while Table 2 shows the results of the analyses for PCBs.

SAMPLE PREPARATION

The samples were prepared for analyses by weighing 5 grams of soil into a 250 mL bottle. Forty mL of petroleum ether was added to the bottle which was then sealed and shaken for 15 minutes. The petroleum ether was decanted out of the bottle into a centrifuge tube. Fifteen mL of methylene chloride was added to the bottle and then shaken for 5 minutes. The methylene chloride was also decanted into the centrifuge tube with the petroleum ether and these extracts were concentrated to 2 mL.

The extract was applied to a column containing 4 grams of silica gel which had been preeluted with 15 mL of hexane. This extract was then eluted through the column with 45 mL of methylene chloride and the eluant solvent exchanged to hexane concentrate and this concentrate was then placed onto a combination column containing silica gel, silica gel plus H_2SO_4 , and silica gel plus KOH. This column was eluted with 45 mL of hexane and this eluant was concentrated to 1mL.

The final chromatographic separation was carried out on a 2.5 gram column of Noelm B alumina. The first elution consisted of 10 mL of 2% methylene chloride/hexane (v/v) which was concentrated to 1 mL and submitted for PCB analysis. A second elution of this column was made using 15 mL of 50% methylene chloride/hexane (v/v) to which was added 300 μ L of dodecane keeper and the eluant was then concentrated to 300 μ L and submitted for GC/MS analysis for PCDDs.

Quality control was carried out by spiking Sample B with both PCBs and PCDDs. This sample was selected for spiking since it appeared to be the most contaminated and would thus give a worst case situation for the recovery studies. A replicate and a method blank were also prepared and analyzed.

DISCUSSION OF PCDD ANALYSIS

The GC/MS analyses for PCDDs were carried out on a Hewlett Packard 5985B GC/MS system using a 30 meter SE-54 capillary column for the separation prior to mass spectral detection. The mass spectrometer was operated in the selected ion monitoring mode following m/z values characteristic of monochloro through octachlorodibenzo-p-dioxin.

No PCDDs were detected in any of the samples at the detection limits given in Table 1. Results from the quality control samples are also given. Note that the detection limit for dichlorodibenzo-p-dioxin were higher than the spike levels in both the low level and even the high level spike. Therefore no recovery information could be given for these dichlorodibenzo-p-dioxins. For the other PCDDs the recoveries are not particularly good but in light of how contaminated the samples were and the short time constraints, these results were considered adequate.

DISCUSSION OF PCB ANALYSIS

Three sample extracts, one replicate and two spiked samples were analysed for Aroclor formulations. Eight PCB isomers were quantified and from these levels of Aroclors 1248, 1254, and 1262 were determined.

The low Aroclor 1248 and 1254 concentrations reported for Sample A may be due to instrument contamination from the the higher concentrations of these Aroclor formulations present in Samples B and C. The Sample B plus 0.16 $\mu\text{g/g}$ can be treated as a replicate of Sample B, since the spiking level is ten times less the amount of Aroclor 1248 present in Sample B. Recovery of Aroclor 1248, added to Sample B at a concentration of 1.6 $\mu\text{g/g}$, was measured to be 75%. The levels of Aroclor 1254 and 1262 are only order of magnitude estimates, due to the large number of interfering compounds present.

TABLE 1. ANALYSIS OF SOIL SAMPLES FOR CHLORINATED
DIBENZO-P-DIOXINS

Sample ID	ng Dioxin/gm Soil (ppb)							
	Cl ₁	Cl ₂	Cl ₃	Cl ₄	Cl ₅	Cl ₆	Cl ₇	Cl ₈
Sample A	ND*	ND	ND	ND	ND	ND	ND	ND
Sample B	ND	ND	ND	ND	ND	ND	ND	ND
Sample B (rep)	ND	ND	ND	ND	ND	ND	ND	ND
Sample C	ND	ND	ND	ND	ND	ND	ND	ND
Blank	ND	ND	ND	ND	ND	ND	ND	ND
Method Detection Limit	2	50	2	2	2	2	2	2
Spike Level	4	3	4	3	3	3	4	3
B (spiked)	3	ND	2	3	2	2	3	2
Recovery %	75	-	50	100	67	67	75	67
Spike Level	35	34	38	28	33	32	35	28
B (spiked)	22	ND	15	20	15	20	23	12
Recovery %	63	-	39	71	45	63	66	43

*ND = none detected at the method detection limit

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TABLE 2. LEVELS OF AROCLOR FORMULATIONS MEASURED
IN SAMPLE EXTRACTS

Sample ID	Concentrations ^a (ug/g)		
	Aroclor 1248	Aroclor 1254	Aroclor 1262
Sample A	0.058 ^{a,h}	0.028 ^h	<0.02
Sample B	2.2	0.069	0.075
Sample B (rep)	2.2	0.047	0.045
B + 0.16 ug/g Aroclor 1248	2.2	0.062	0.061
Sample C	1.6	0.15	0.37
B + 1.6 ug/g Aroclor 1248	3.4 ^c	0.10	0.09

^a ug Aroclor formulation/g sample. Eight PCB isomers were used to identify the Aroclor formulation and estimate the relative amounts.

^b Possible instrument contamination from analysis of Samples B & C.

^c This value for Aroclor 1248 concentration represents a recovery of 75% of Aroclor 1248 added to Sample B.

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