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TITLE: ANALYSIS OF LANDFILL WELLS

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ABSTRACT: Eight landfill well water samples from the MCI Krumrich plant were analyzed to determine the presence or absence of phenols, PCBs and TCDD. The analytical results are presented in this report.

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ANALYSIS OF LANDFILL WELL WATER SAMPLES FOR TOTAL PHENOLS,
TETRACHLORODIBENZO-p-DIOXIN AND POLYCHLORONATED BIPHENYLS

Eight (8) water samples were submitted to the Dayton Laboratory for analyses to determine the possible presence and concentrations of total phenols, polychlorinated biphenyls (PCB) and tetrachlorodibenzo-p-dioxins (TCDD).

Total Phenols

The presence and concentration of phenol in each of the water samples was determined and measured by typical wet chemistry techniques [1]. An independent phenol standard was also analyzed for quality assurance. The results of these analyses are shown in Table 1.

TABLE 1. ANALYSIS OF LANDFILL WELL
WATER FOR TOTAL PHENOL

SAMPLE ID	PHENOL, mg/L
Well 1	170
Well 2A	323
Well 2B	1,294
Well 9A	1,506
Well 15A	11 ^a
Well 15B	294 ^a
Well 2C	131

^aAverage of duplicate analyses.

QUALITY CONTROL DATA

Reference standard	Observed value, mg/L	True value, mg/L	Percent recovery
EPA 2767	0.86	0.82	105

[1] Standard Methods for Examination of Water and Wastewater, 14th Edition, American Public Health Association, Washington, DC, 1976. 1193 pp.

Polychlorinated Biphenyls (PCB)

500 mL of each water sample and its container were extracted with three (3) 50-mL aliquots of methylene chloride. The combined extracts (sample and container) of each sample were dried and evaporated to 1-mL. Each 1-mL sample was analyzed by a capillary gas chromatography/mass spectrometry (GC/MS) technique. The MS was operated in the Selected Ion Monitor mode (SIM) to enhance sensitivity and decrease possible interferences. The SIM mode monitors two ions for each isomer class of PCB's (number of chlorine atoms on the biphenyl molecule). Known PCB standards were analyzed to quantitate sample concentrations. Spikes and duplicates of the samples were run under similar analytical conditions for quality assurance. Results of these analyses are shown in Table 2.

Tetrachlorodibenzo-p-dioxins (TCDDs)

Eight water samples were received for the analysis of tetrachlorodibenzo-p-dioxins (TCDD). Two separate 1-liter samples were provided from each site. One of the 1-liter bottles for each site was used for the analysis with the other bottle being set aside in the event that some problem or difficulty occurred in the original analysis. The samples were analyzed by taking one of the 1-liter bottles and placing 500 mL of the water into each of two 1-liter bottles. One of the 500 mL aliquots was then used for the analysis with the other aliquot used as a replicate or for spiking with native (unlabeled) 2,3,7,8-TCDD. 200 mL of petroleum ether was then added to the original sample bottle and the contents were shaken for 30 seconds to extract any TCDDs which may adhere to the glass. During this time, all the 500 mL aliquots were spiked with $^{14}\text{C}_{12}$ -labeled 2,3,7,8-TCDD and various 500 mL aliquots were spiked with native 2,3,7,8-TCDD. 100 mL of the petroleum ether from the original sample bottle was then placed into each of the bottles containing the 500 mL aliquots.

TABLE 2. ANALYSIS OF LANDFILL WELL WATER SAMPLES FOR PCBs

Sample ID	Number of chlorines, ug/L										Total PCB, ug/L
	1	2	3	4	5	6	7	8	9	10	
Well 1	BDL ^a	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Well 1 duplicate	"	"	"	"	"	"	"	"	"	"	"
Well 2A	"	"	"	"	"	"	"	"	"	"	"
Well 2B	"	"	"	"	"	"	"	"	"	"	"
Well 2B duplicate	"	"	"	"	"	"	"	"	"	"	"
Well 9A	"	"	0.5	0.5	"	2.2	0.7	"	"	"	3.9
Well 9B	"	"	0.5	1.7	"	11.2	BDL	"	"	"	13.4
Well 15A	"	0.1	BDL	BDL	"	BDL	0.1	"	"	"	0.2
Well 15B	"	BDL	0.6	"	"	"	BDL	"	"	"	0.6
Well 20	"	"	BDL	"	"	"	"	"	"	"	BDL

^aBDL = below detection limit of 0.1 ug/L (ppb).

QUALITY CONTROL DATA

Well #2A spiked with 118 ppb of Aroclor 1254 = 80% recovery
 Well #20 spiked with 118 ppb of Aroclor 1254 = 68% recovery

The bottles were then shaken for five minutes and the water layer removed. The petroleum ether layer was then washed with base followed by sulfuric acid. Column chromatography of the concentrated petroleum ether was carried out on a Woelm Basic alumina column collecting the fraction that elutes with the 50% CH_2Cl_2 /Hexane fraction, concentrating to 25 μL and submitting this sample for GC/MS analysis.

The GC/MS analysis was carried out on a Hewlett-Packard 5985B GC/MS system. The chromatograph was equipped with a J and W 30 meter SE-54 capillary column. The mass spectrometer was operated in the selected ion monitoring mode following one ion characteristic of the labeled TCDD (m/z 334) and three ions characteristic of native TCDD (m/z 257, 320, 322).

Quantitation was carried out by ratioing the response of the native TCDD to the labeled TCDD. The results of the analyses are given in Table 3. Sample A15 showed very serious interferences so a different workup of this sample was carried out using 100 mL of water at the outset rather than the 500 mL and also using an initial alumina column clean up step prior to the base and acid washes. The results given as "ND" mean none detected at a detection limit given in parenthesis. This detection limit depends on the interferences present and sample recovery and thus varies from sample to sample. The blank is representative of the lower detection limit if there were no interferences. A TCDD was found in the samples from Wells 2A and 2B and is the 1,3,5,8-TCDD. The chromatographic traces for the analysis of Well 2A is given in Figure 1. The ions being monitored are characteristic of the molecular ions of the labeled and unlabeled TCDD and the COCl loss for the unlabeled TCDD. As a point of reference, the chromatographic trace for the 2,3,7,8-TCDD is given in Figure 2 showing comparable ions. The quantitation for the 1,3,5,8-TCDD is based on the calibration curve generated for the 2,3,7,8-TCDD and may be subject to some degree of error.

TABLE 3. RESULTS FOR THE ANALYSIS OF
TETRACHLOROBENZO-P-DIOXINS (TCDDs)

Sample ID	2,3,7,8-TCDD Found	2,3,7,8-TCDD Added	Other TCDDs Found
	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)
Landfill Well #1	ND (<2)	-	ND (<20)
" " #2A	ND (<7)	-	69
" " #2A (replicate)	ND (<8)	-	66
" " #2B	ND (<22)	-	290
" " A9	ND (<35)	-	- ^a
" " A9 (spike)	4.8	1.9	- ^a
" " 9B	ND (<9)	-	- ^a
" " A15	ND (<5)	-	ND (<5)
" " A15 (replicate)	ND (<5)	-	ND (<5)
" " A15 (spike)	13	19	ND (<5)
" " A15 (spike)	170	192	ND (<1)
" " B15	ND (<1)	-	ND (<1)
" " 20	ND (<2)	-	ND (<2)
Blank	ND (<1)	-	ND (<1)

^aSerious interferences prevented the analysis of isomers other than 2,3,7,8-TCDD while maintaining good sensitivity for 2,3,7,8-TCDD.

An additional comment should also be made about the spiking experiments. In sample #A9 an interference peak limited the detection limit to 3.5 ppt. When 1.9 ppt of 2,3,7,8-TCDD was spiked into a replicate sample the peak grew to 4.8 ppt for a difference of 1.3 ppt which is reasonable in view of the uncertainty in these analyses at these levels. For sample A15 the 5 ppt detection limit was determined by the ultimate sensitivity of the GC/MS and there were no interferences in the second workup of this sample. Therefore the 19 ppt spike should be compared directly to the 13 ppt sample recovered. Again this result is reasonable in light of the difficulties encountered in analyzing this particular sample.

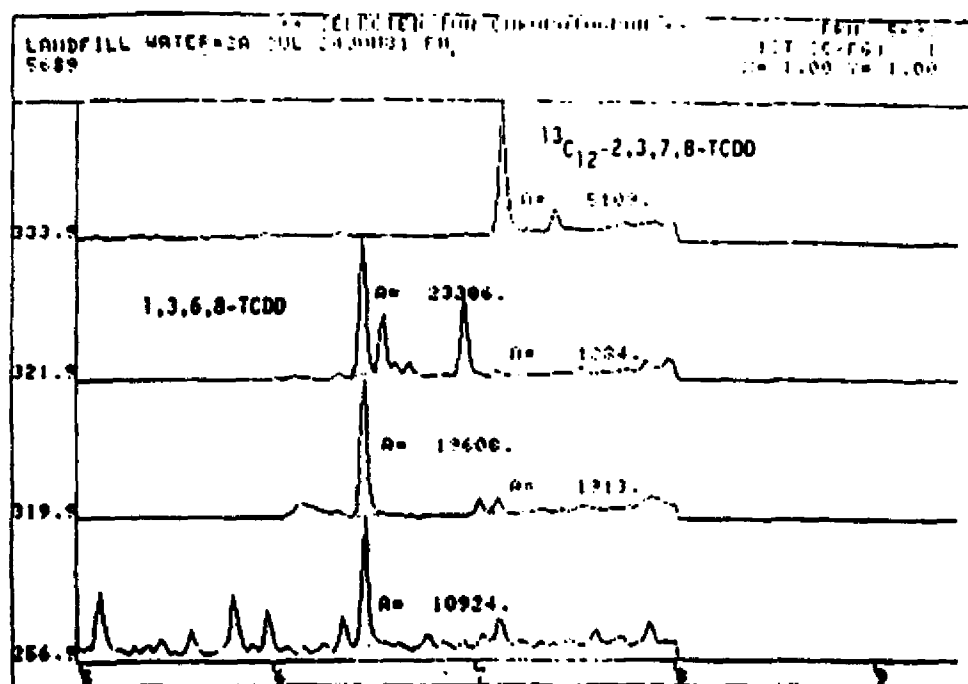


Figure 1. GC/MS analysis of Landfill Water #2A.

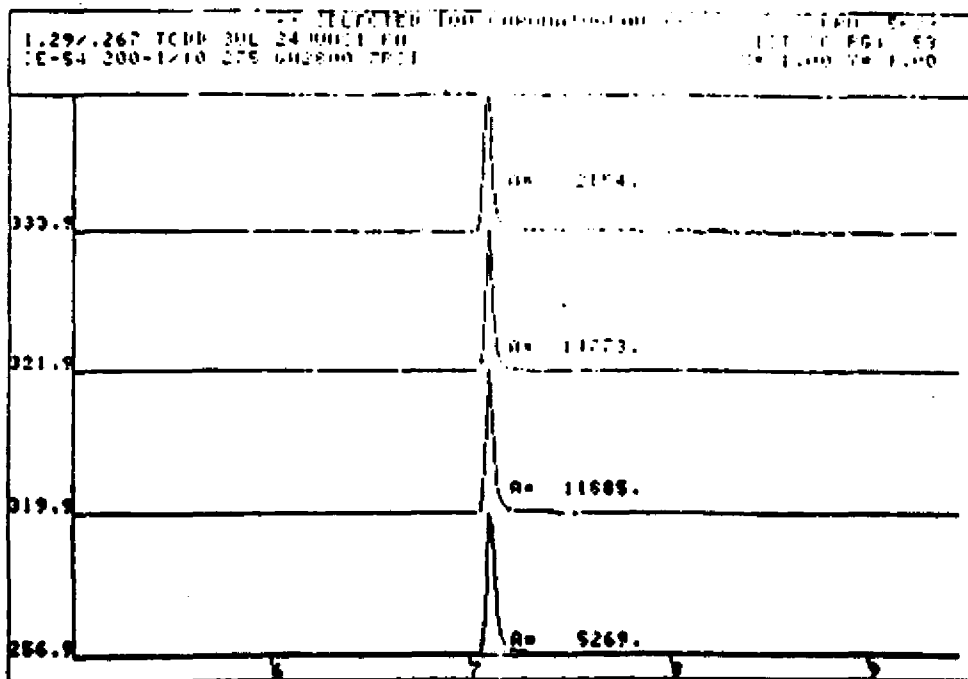


Figure 2. GC/MS analysis of 2,3,7,8-TCDD.