

Mansanto

NAME & LOCATION: R. F. Ivory - EASC - Dayton Lab - 1250

DATE: October 10, 1980

D. Dahm

SUBJECT: Analysis of Chlorophenols for
Dioxin Content

F. Hileman

REFERENCE: MRC Job #320.3010

T. Maser

TO: J. A. Gloeckner
Krummrich Plant

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Eighty one samples (fifty-five samples, thirteen spikes, seven duplicates, and six method blanks) were analyzed for the presence of chlorinated dibenzo-p-dioxins. The samples were analyzed on a Hewlett-Packard 5985A GC/MS system using MRC Method #01-GCMS-R2, with the exception that a 1% Dexsil 300 column was used instead of the 3% Dexsil 400.

An extensive clean up procedure was used to prepare the extracts for GC/MS analysis. Five gram samples of the phenols were dissolved by shaking with 75 ml of 20% KOH for five minutes. Forty ml of petroleum ether were added and shaken with the base for several minutes to extract the dioxins into the organic layer. The base was then removed, leaving the organic layer which was subsequently subjected to 20% KOH, concentrated sulfuric acid and water washes to remove various impurities and matrix components. The organic layer was then dried over sodium sulfate, followed by concentration to about 0.5 ml. This extract was then quantitatively transferred to a mini-chromatographic column packed with 3.0 g of Woelm alumina and eluted with 2% dichloromethane in hexane for orthochlorophenol and 10% dichloromethane in hexane for the para-chloro and 2,4-dichlorophenol. These fractions were discarded. The column was then eluted with 50% dichloromethane in hexane and this fraction collected, concentrated to dryness and submitted for analysis.

The results of the analyses are given in Tables 1 through 3. Detection limits achieved are lower than previously attained; in particular for TCDD the detection limit has been reduced by an order of magnitude. Footnotes included with these tables cover the fact that in certain cases an interference peak was observed which interfered in the analysis of a given dioxin and subsequently raised the lower detection limit. In addition, for those cases where the standard curves were prepared using one isomer of a given chlorodioxin and the unknown was a different isomer (as noted by a different retention time), these samples have been flagged to show that there could be some degree of error in the quantitation.

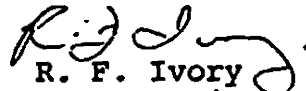
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J. A. Fletcher
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MRC Method #7 was followed for QA/QC with the exception; that one low spike for the 2,4-dichlorophenol was spilled in processing.

If you have any questions contact me at Ext. 417.



R. F. Ivory
EASC Service Project Manager

RFI:dem

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TABLE 1. NANOGRAMS PCDD/GRAM MATERIAL (ORTHOCHLOROPHENOLS)

Sample	Mono	Di	Tri	Tetra	Penta	Hexa	Hepta	Octa
8005	6.7	-	-	-	-	-	-	-
Blank	-	-	-	-	-	-	-	-
8015	-	-	-	-	-	-	-	2
8011	-	-	-	-	2.2	-	-	-
8023	-	-	-	-	-	-	-	-
8026	-	-	-	-	-	-	-	-
8025	-	-	-	-	-	-	-	-
8005 replicate	3.8	-	-	-	-	-	-	-
8001	-	-	-	-	-	-	-	-
8001 replicate	-	-	-	-	-	-	-	-
8018	-	-	-	-	-	-	-	-
8022	-	-	-	-	-	-	-	-
8027	-	-	-	-	-	-	-	-
Blank	-	-	-	-	-	-	-	-
8029	-	-	-	-	-	-	-	-
8031	-	-	-	-	-	-	-	-
8032	-	-	-	-	-	-	-	-
8015	-	-	-	-	-	-	-	2
8015 low spike	34%	a	66%	78%	a	74%	69%	85%
8015 high spike	21%	99%	72%	63%	77%	75%	74%	83%
8018	-	-	-	-	-	-	-	-
8018 low spike	56%	84%	73%	78%	a	76%	98%	84%
8018 high spike	23%	88%	70%	57%	71%	64%	62%	66%
Lower detection limit	2	1	1	0.9	2	3	3	2

^aCould not be calculated due to interference at this mass.

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TABLE 2. NANOGRAMS PCDD/GRAM MATERIAL (DICHLOROPHENOLS)

Sample	Mono	Di	Tri	Tetra	Penta	Hexa	Hepta	Octa
7000	-	-	2	2.6 ^{a*}	-	-	-	-
7003	15.6	553	{108a} {12.9}	{4.5 ^{a*} } {9.5}	13.2*	-	-	-
7007	-	0.9	4 ^a	2.1*	-	-	-	5
7010	-	-	6.6	3.0*	15*	-	-	2.6
7013	-	11.6	-	2.5*	-	-	-	10.6
Blank	-	-	-	-	-	-	-	-
7017	-	-	3.1	2.5 ^{a*}	-	-	-	-
7017 replicate	-	-	2.0	2.4 ^{a*}	-	-	-	-
7022	-	-	3	0.9 ^{a*}	-	-	-	-
7023	-	-	-	2.7 ^{a*}	-	-	-	6
7024	-	-	-	0.9 ^{a*}	-	13	-	8
7025	-	-	6.8 ^a	3.3 ^{a*}	0.6 ND 2.6 ND	19	-	-
7027	-	-	-	1.1 ^{a*}	6.6	-	-	-
7029	-	-	4.4	1.2 ^{a*}	-	-	-	-
Blank	-	-	-	-	-	-	-	-
7032	-	-	-	-	-	1.9*	-	-
7032 replicate	-	-	-	1.2*	-	1.8*	-	-
Lower detection limit	3	1.0	1.0	0.9	3	3	3	25
7007	-	0.9	4 ^a	-	-	-	-	5
7007 low spike	26%	73%	45%	58%	90%	64%	79%	68%
7007 high spike	23%	37%	31%	43%	48%	49%	44%	53%
7023	-	-	-	-	3.8	-	-	5.7
7023 low spike	19%	99%	59%	114%	131%	84%	89%	119%
7023 high spike	21%	51%	56%	89%	85%	67%	74%	93%

^aQuantitation was carried out on an isomer other than that in question.
 *Interference present which raise the lower detection limit.

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TABLE 3. NANOGRAMS PCDD/GRAM MATERIAL (PARACHLOROPHENOLS)

Sample	Mono	Di	Tri	Tetra	Penta	Hexa	Hepta	Octa
5002	-	-	-	-	-	-	-	-
5006	-	-	-	-	-	-	-	-
5011	34	18	-	-	-	-	-	-
5011 replicate	20	14	-	-	-	-	-	-
5014	-	-	-	-	-	-	-	-
5019	-	4	-	-	-	-	-	-
Blank	-	-	-	-	-	-	-	-
5020	-	-	-	-	-	-	-	-
5024	-	-	1*	-	-	-	-	-
5025	-	-	-	-	-	-	-	-
5028	26	-	1*	-	-	-	-	-
5030	-	-	-	-	-	-	-	-
5031	-	-	2*	-	-	-	-	-
Blank	-	-	-	-	-	-	-	-
5035	-	-	4*	-	-	-	-	-
5037	-	-	-	-	-	-	-	-
5040	-	-	2*	-	-	-	-	-
5040 replicate	-	-	2*	-	-	-	-	-
5044	-	-	1*	-	-	-	-	-
5049	-	-	1*	-	-	-	-	-
5050	-	-	-	-	-	-	-	-
5051	-	-	-	-	-	-	-	-
5053	-	-	1*	-	-	-	-	-
5056	-	-	1*	-	-	-	-	-
5058	-	-	-	-	-	-	-	-
5058 replicate	-	-	-	-	-	-	-	-
5063	-	-	-	-	-	-	-	-
5064	-	-	-	-	-	-	-	-
Blank	-	-	-	-	-	-	-	-
5065	-	-	-	-	-	-	-	-
5068	-	-	-	-	-	-	-	-
5070	-	-	-	-	-	-	-	-
5072	-	-	-	-	-	-	-	-

(continued)

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TABLE 3 (continued)

Sample	Mono	Di	Tri	Tetra	Penta	Hexa	Hepta	Octa
5014	-	-	-	-	-	-	-	-
5014 high spike	28%	36%	47%	54%	48%	57%	51%	61%
5030	-	-	-	-	-	-	-	-
5030 low spike	-	53%	45%	64%	69%	96%	48%	40%
5030 high spike	65%	71%	66%	78%	87%	75%	71%	74%
5056	-	-	1*	-	-	-	-	-
5056 low spike	-	42%	50	59%	59%	56%	44%	92%
5056 high spike	-	30%	56%	51%	68%	52%	59%	57%
Lower detection limit	6	3	1	0.9	3	3	3	2.5

*Interferences were present.

^aInterferences would not allow recovery calculation to be made.

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