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TITLE: ANALYSIS OF ADIPOSE TISSUE FOR 2,3,7,8-TCDD

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ABSTRACT: Human adipose tissue samples were analyzed for 2,3,7,8-TCDD concentrations. This report shows the results of the analyses.

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1-13-84

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AUTHORS: F. D. Hileman
TITLE: ANALYSIS OF ADIPOSE TISSUE FOR 2,3,7,8-TCDD
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Eight human adipose samples were received for the analysis of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2378-TCDD). The samples were assigned MRC log number 1-83-06-06-02.

Each sample was run in triplicate, a sample, a replicate and a low level spike (7 ppt). This spike level was selected because others have reported finding 1 to 10 ppt of 2378-TCDD in human tissue. Thus a 7 ppt spike could be readily detected in these samples in spite of background levels of 2378-TCDD. In addition, one of the samples was also spiked at a higher level (70 ppt) and a pork fat sample previously found to contain less than 0.7 ppt of 2378-TCDD was included as a blank.

The samples were prepared according to the diagram given in Figure 1. The final extracts were analyzed on a Finnigan MAT 212 GC/MS system. The gas chromatographic column that was used was a 60 m SP-2330 capillary column. This column was checked daily during this work to ensure that it uniquely resolved 2378-TCDD from the other 21-TCDDs. The mass spectrometer was operated at a resolution of 1000 in the selected ion monitoring mode following two ions characteristic of TCDD (320, 322) and one ion for each of the isotopically labeled TCDDs $^{37}\text{Cl}_4$ -2378 TCDD (328) and $^{13}\text{C}_{12}$ -2378 TCDD (334). Quantitation was achieved by comparing the area of unlabeled (naturally occurring) TCDD to the $^{13}\text{C}_{12}$ -2378-TCDD. The standards used in this work have been cross validated against two separate EPA quality control standards. Qualitative identification was made by requiring the compound in question to coelute with the $^{13}\text{C}_{12}$ -2378-TCDD and to have the correct 320/322 ratio of 0.79 ± 0.10 .

The results of the analyses are given in Table 1. Note that the increase observed for the spikes all agree reasonably well with the unspiked samples with the exception of sample Number 8. The cause of the problem may be sample inhomogeneity but the good precision obtained on the other samples would cast some doubt on this explanation.

It should be noted that an additional replicate had been analyzed for sample 7. In the initial work this particular sample had the highest level of interferences and this sample was singly carried through the entire workup and analyzed. What is most important is that it was weighed out and worked up by a different individual than the one who did the work up on the rest of the samples. Thus it gives some measure of the operator independence of the method.

Mention should also be given to detection limits. The GC/MS system was capable of detecting the equivalent of 0.5 ppt in a 25 gram initial sample. This assumes 100% recovery of the TCDD and no interfering compounds. The absolute recoveries of the $^{13}\text{C}_{12}$ -2378-TCDD varied from 35-85% which when combined with any interferences would raise the nominal detection limit to approximately 1.0 ppt for these samples. Slightly better detection limits could be achieved for the pork fat and the solvent blank due to lower levels of interferences present.

Analysis of Human Adipose Tissue for the Presence of 2378-TCDD

<u>Sample</u>	<u>ppt 2378-TCDD found</u>	<u>ppt 2378-TCDD added</u>
866-83 2275519-1*	1.7	-
(Rep)	1.9	-
(Spike)	8.8	6.8
881-83 2275519-2	4.1	-
(Rep)	5.1	-
(Spike)	11.8	6.6
890-83 2275519-3	3.3	-
(Rep)	3.1	-
(Spike)	9.8	6.8
928-83 2275519-4	4.8	-
(Rep)	4.8	-
(Spike)	11.1	6.6
959-83 2275519-5	5.4	-
(Rep)	6.2	-
(Spike)	13.0	6.9
(Spike)	67.9	67
971-83 2275519-6	3.5	-
(Rep)	3.8	-
(Spike)	10.3	6.8
989-83 2275519-7	9.2	-
(Rep)	8.6	-
(Spike)	14.9	6.9
(2nd Rep) *	9.5	-
1009-83 2275519-8	10.6	-
(Rep)	9.5	-
(Spike)	12.6	6.3
Pork fat	0.6	-
(Rep)	1.0	-
(Spike)	6.3	6.7
Solvent Blank	0.8	-

*Number to which samples will be referred.

*See report for a description of the replicate.

Adipose Preparation

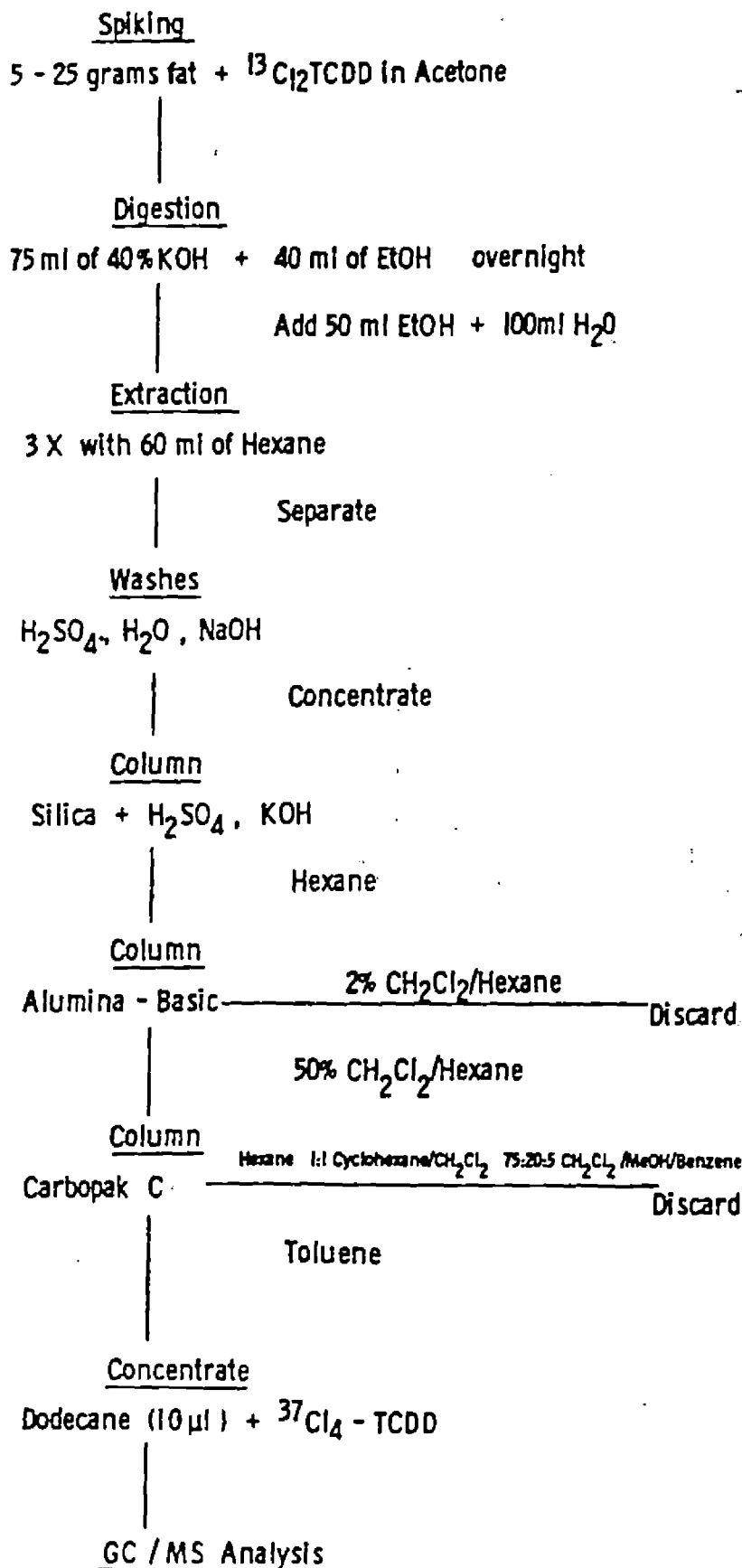


Figure 1. Scheme for the preparation of adipose for the analysis of 2378-TCDD.