United States
Environmental Protection
Agency

Office of
Toxic Substances
Washington DC 20460

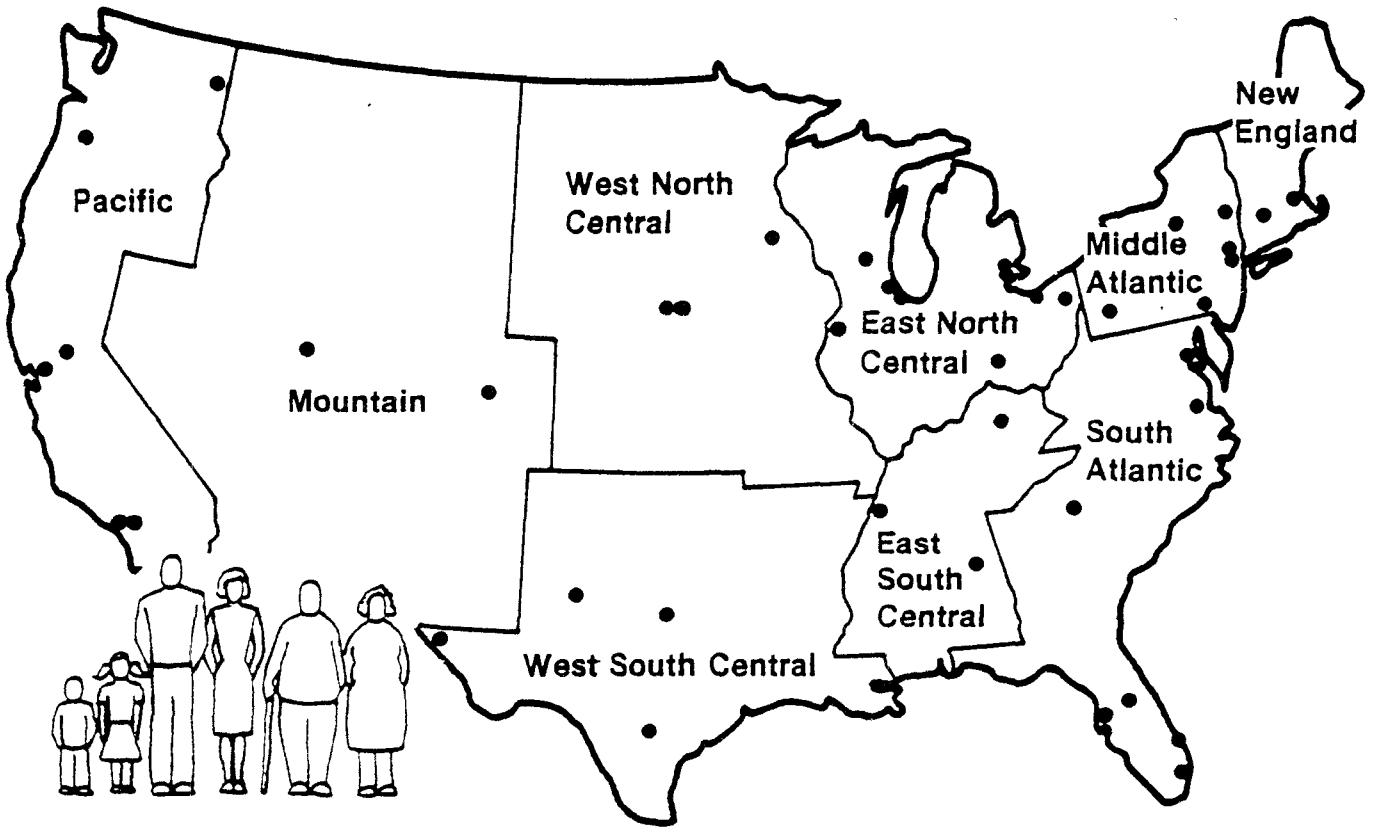
EPA-560/5-86-038
December, 1986

Toxic Substances

EPA

BROAD SCAN ANALYSIS OF THE FY82 NATIONAL HUMAN ADIPOSE TISSUE SURVEY SPECIMENS

VOLUME IV - POLYCHLORINATED DIBENZO-p-DIOXINS (PCDD) AND POLYCHLORINATED DIBENZOFURANS (PCDF)



BROAD SCAN ANALYSIS OF HUMAN ADIPOSE TISSUE
VOLUME IV POLYCHLORINATED DIBENZO-p-DIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs)

By

John S. Stanley

FINAL REPORT

EPA Prime Contract No. 68-02-4252
Work Assignment 21
MRI Project No. 8821-A(01)

December 31, 1986

Prepared for

National Human Monitoring Program
Field Studies Branch (TS-798)
Design and Development Branch
Office of Toxic Substances
U.S. Environmental Protection Agency
401 M Street, S.W.
Washington, DC 20460

Attn: Ms. Janet Remmers and Mr. Philip Robinson, Work Assignment Managers
Dr. Joseph Breen and Ms. Cindy Stroup, Program Managers

DISCLAIMER

This document has been reviewed and approved for publication by the Office of Toxic Substances, Office of Pesticides and Toxic Substances, U.S. Environmental Protection Agency. The use of trade names or commercial products does not constitute Agency endorsement or recommendation for use.

PREFACE

This report is the fourth of a five-volume series that details the broad scan chemical analysis of composite adipose tissue samples. These composite samples were prepared from individual specimens obtained from the Environmental Protection Agency's (EPA) National Human Adipose Tissue Survey (NHATS) fiscal year 1982 (FY82) repository.

This volume summarizes data generated from the analysis of the composited samples for polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF). Volume I, the executive summary, presents a synopsis of all analysis efforts completed under the broad scan program. Volumes II, III, and V deal specifically with the chemical analysis of the NHATS composites, volatile organics, semivolatile organics, and trace elements, respectively. The statistical analyses of the data reported in these volumes will be reported separately by the EPA's Office of Toxic Substances (OTS) Design and Development Branch contractor, Battelle Columbus Laboratories.

The entire series of reports are referenced as follows:

- Stanley JS. 1986. Broad scan analysis of human adipose tissue: Volume I: Executive summary. EPA 560/5-86-035.
- Stanley JS. 1986. Broad scan analysis of human adipose tissue: Volume II: Volatile organic compounds. EPA 560/5-86-036.
- Stanley JS. 1986. Broad scan analysis of human adipose tissue: Volume III: Semivolatile organic compounds. EPA 560/5-86-037.
- Stanley JS. 1986. Broad scan analysis of human adipose tissue: Volume IV: Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). EPA 560/5-86-038.
- Stanley JS, Stockton RA. 1986. Broad scan analysis of human adipose tissue: Volume V: Trace elements. EPA-560/5-86-039.

These method development, sample analyses, and reporting activities were completed for the EPA/OTS Field Studies Branch (FSB) broad scan analysis of human adipose tissue program (EPA Prime Contract Nos. 68-02-3938 and 68-02-4252, Work Assignments 8 and 21, respectively, Ms. Janet Remmers, Work Assignment Manager, and Dr. Joseph Breen, Project Officer).

The samples were prepared with the assistance of Ms. Leslie Moody and Mr. Steven Turner. The HRGC/MS methods development and sample analyses were conducted by Ms. Kathy Boggess, Mr. Jon Onstot, and Dr. Thomas Sack. The compositing scheme used to prepare the samples from the NHATS repository was provided by Dr. Gregory Mack, Battelle Columbus Laboratories, under contract to the EPA/OTS Design and Development Branch (Mr. Philip Robinson, Task Manager, and Ms. Cindy Stroup, Program Manager).

Approved:

John E. Going, Director
Chemical Sciences Department

MIDWEST RESEARCH INSTITUTE

Paul C. Constant
Program Manager

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF TABLES (concluded)

EXECUTIVE SUMMARY

The U. S. Environmental Protection Agency's Office of Toxic Substances (EPA/OTS) maintains a unique program for monitoring human exposure to potentially toxic substances. The National Human Adipose Tissue Survey (NHATS) is a statistically designed annual program to collect and analyze a nationwide sample of adipose tissue specimens for toxic compounds. The primary focus for NHATS has been to document trends in human exposure to environmentally persistent contaminants, specifically, organochlorine pesticides and polychlorinated biphenyls (PCBs).

EPA/OTS has recognized the need to provide a more comprehensive assessment of toxic substances that accumulate in adipose tissues. The NHATS specimens collected during fiscal year 1982 (FY82) were designated for "broad scan analysis" to detect volatile and semivolatile organic compounds and trace elements.

This volume of the final report deals with the measurement of polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) in composited adipose tissue samples from the FY82 NHATS repository. The objective of this study was (1) to identify analytical methods based on high resolution gas chromatography/mass spectrometry (HRGC/MS) detection that are capable of achieving detection limits in the low parts per trillion (picogram/gram, pg/g) concentration range for individual PCDD and PCDF congeners and (2) to complete the analysis of composite adipose tissue samples representing the general U.S. population for the tetra- through octachloro-PCDD and PCDF congeners.

Forty-six composite samples were prepared from the FY82 NHATS repository according to a study design prepared by the EPA/OTS Design and Development Branch contractor, Battelle Columbus Laboratories. The composite samples represent the nine U.S. census division stratified by three age groups (0-14, 15-44, and 45 plus).

The sample preparation was completed using techniques that isolate PCDD and PCDF congeners from potential interferences. The isolation of the PCDD and PCDF was achieved using carbon-based chromatography columns. Two different carbon materials were used to complete the analysis for the full range of tetra- through octachloro-PCDD and PCDF congeners. The HRGC/MS operated in the selected ion monitoring (SIM) mode was required for determination of the compounds at concentrations ranging from less than 5 pg/g (for tetra- and pentachloro congeners) to greater than 1,000 pg/g for the octachloro dibenzo-p-dioxin.

The results of this study demonstrate that the EPA NHATS program is an effective vehicle for documenting the exposure of the general U.S. population to PCDD and PCDF. The analysis of the 46 composite samples establishes the presence of the 2,3,7,8-substituted tetra- through octachloro-PCDD and PCDF congeners in the adipose tissues collected from the general U.S. population.

The data from this study are comparable to work that has been reported for other studies on adipose tissue samples from the United States (specifically, upstate New York), Sweden, and Canada. Specifically, 2,3,7,8-TCDD was detected in 35 of the 46 composites with an average lipid-adjusted concentration of 6.2 ± 3.3 pg/g. The average concentration of the other PCDD compounds ranged from 33.5 pg/g for pentachlorodibenzo-p-dioxin (detected in 91% of the composites) up to 554 pg/g for octachlorodibenzo-p-dioxins which was detected in all samples.

The data presented in this volume demonstrate some differences in PCDD levels for the three age groups (0-15, 15-44, and 45 plus) evaluated. The PCDF congeners were generally detected less frequently and were present at lower concentration than the PCDD congeners. Obvious trends in the levels of the PCDF congeners with respect to age were not observed. The PCDD and PCDF congeners were detected in the composites representing each of the nine U.S. census divisions.

The quantitative data for the PCDD and PCDF congeners presented in this report have been submitted along with all supporting quality control data to Battelle Columbus Laboratories for statistical analysis. This data will be analyzed to determine the significance of differences in PCDD and PCDF levels based on various demographic factors.

I. INTRODUCTION

The National Human Adipose Tissue Survey (NHATS) is the main operative program of the National Human Monitoring Program (NHMP). The NHMP was first established by the U.S. Public Health Service in 1967 and was subsequently transferred to the U. S. Environmental Protection Agency in 1970. During 1979 the program was transferred within EPA to the Exposure Evaluation Division (EED) of the Office of Toxic Substances (OTS).

NHATS is an annual program to collect a nationwide sample of adipose tissue specimens and to chemically analyze them for the presence of toxic compounds. The objective of the NHATS program is to detect and quantify the prevalences of the compounds in the general population. The NHATS data are used to address part of OTS's mandate under the Toxic Substances Control Act (TSCA) to assess chemical risk to the U.S. population. The specimens are collected from autopsied cadavers and surgical patients according to a statistical survey design (Lucas, Handy 1981). The survey design ensures that specified geographical regions and demographic categories are appropriately represented to permit valid and precise estimates of baseline levels, time trends, and comparisons across subpopulations. Historically, organochlorine pesticides and PCB residues have been selected for evaluation.

A. Broad Scan Analysis Strategy

EPA/OTS has recognized the need to provide a more comprehensive assessment of the toxic substances that accumulate in adipose tissue. An aggressive strategy to assess TSCA-related substances that persist in the adipose tissue of the general U.S. population has been developed by EED. The NHATS specimens collected during fiscal year 1982 (FY82) were selected for a broad scan analysis of volatile and semivolatile organic TSCA-related chemicals and trace elements (Mack, Stanley 1984).

The initiative to achieve a more comprehensive assessment necessitated either the development of new methods or the modification of the existing analytical procedures, specifically high resolution gas chromatography/mass spectrometry (HRGC/MS). Data on organochlorine pesticides and PCBs reported for the NHATS specimens up to the FY82 collection are based on packed column gas chromatography/electron capture detector (PGC/ECD) analysis.

B. Work Assignment Objectives

Growing concern about exposure to polychlorinated dibenzo-p-dioxins (PCDD) and dibenzofurans (PCDF) and reports of endogenous levels in human adipose tissues from upstate New York, (Schecter et al. 1985; Schecter, Ryan 1985) Canada, (Ryan et al. 1985; Ryan et al. 1985), and Sweden (Nygren et al. 1985) prompted analysis for these compounds using composite adipose tissue specimens from the FY82 repository.

The objectives of this phase of the work assignment were (a) to identify appropriate analytical methods to determine the presence of PCDD and PCDF congeners in human adipose tissue based on HRGC/MS detection; (b) to

conduct preliminary evaluation of the analytical procedures; and (c) to complete the sample workup and HRGC/MS analysis of 46 composite samples prepared from the NHATS specimens collected during FY82.

Following this introductory section, recommendations for improving the analytical method are presented in Section II. Experimental procedures, detailed results of sample analysis, and a summary of QA/QC activities are presented in Sections III to V. Pertinent references are cited in Section VI. Appendix A provides a glossary of terms used throughout this text.

II. RECOMMENDATIONS

The methods described for PCDD and PCDF analysis were developed in conjunction with the HRGC/MS broad scan analysis method (Stanley 1986c) for detecting general semivolatile organic compounds in human adipose tissues. A continued effort in following the trends of PCDD and PCDF will require that the analytical method with the modifications discussed below be fully validated through intra- and interlaboratory studies.

Certified standards other than the 2,3,7,8-TCDD are not currently available. It is imperative that the additional 2,3,7,8-substituted PCDD and PCDF congeners be made available as certified materials for future studies to make accurate comparisons of residue levels in the general population.

The analytical method also should be modified to include additional carbon-13 labeled internal standards to improve the accuracy of the quantitation of the tetra- through octachloro-PCDDs and PCDFs.

The time required for preparation of 10- to 20-g tissue samples by the method described in this report is time intensive as a result of bulk lipid removal by gel permeation chromatography (GPC). This procedure was necessary to achieve the objective of the overall broad scan analysis program. However, future studies that focus on PCDD and PCDF levels will require developing techniques that result in more expedient sample preparation.

III. EXPERIMENTAL

This section describes the procedures used to qualitatively and quantitatively determine the PCDD and PCDF compounds present in human adipose tissue. Figure 1 presents a schematic of the analytical methods for the broad scan analysis for semivolatile organic compounds that are present in human adipose tissue at concentrations of greater than 10 ng/g (parts per billion). The method required compositing specified adipose tissue specimens from the NHATS repository. The compositing scheme was prepared by Battelle Columbus Laboratories under contract to EPA/OTS Exposure Evaluation Division, Design and Development Branch.

The collection, handling, and storage of the FY82 NHATS specimens, as well as the composite design and compositing procedures have been described

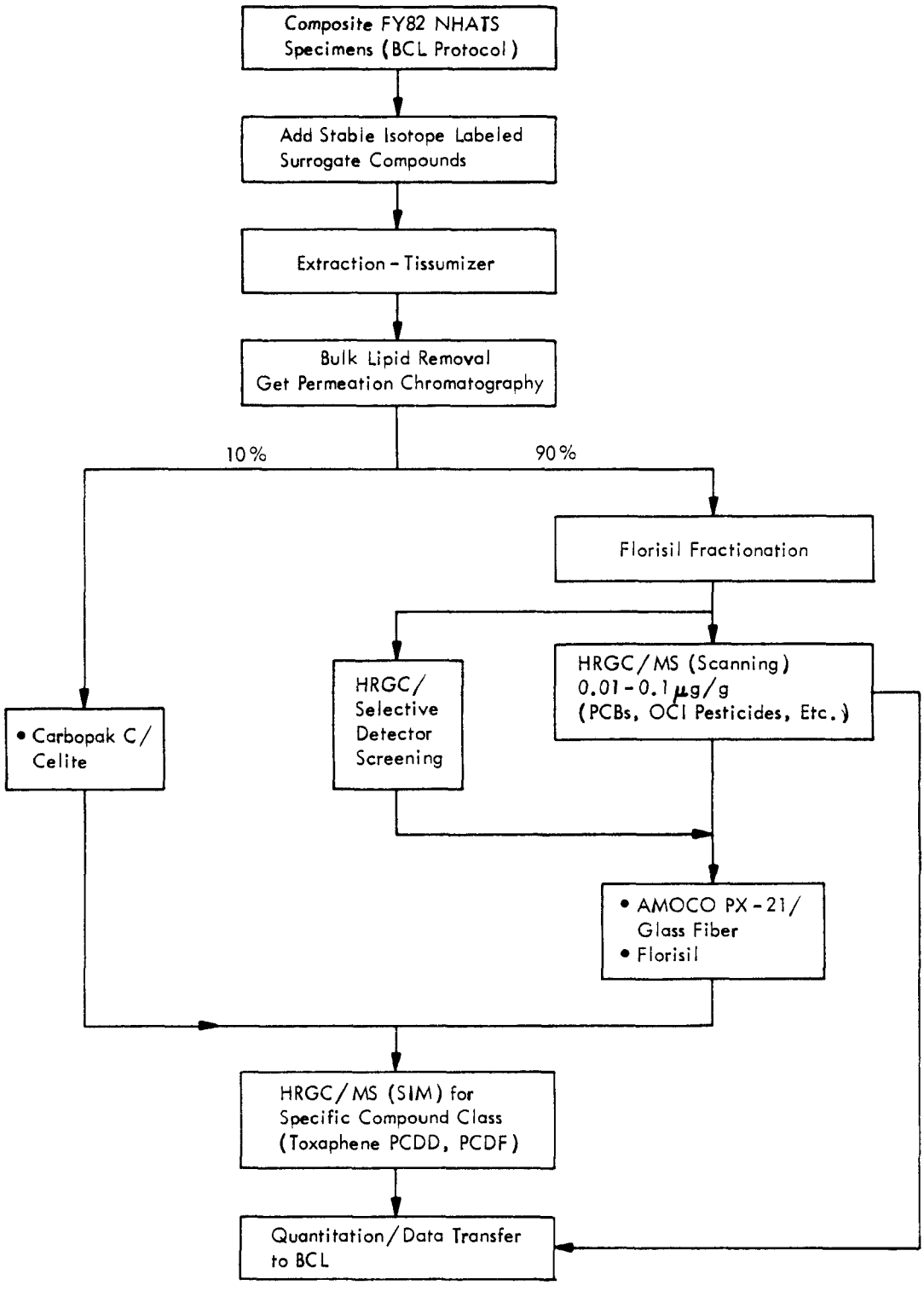


Figure 1 - Flow scheme for analysis of semivolatile organic compounds in human adipose tissue.

previously. (Stanley 1986a; Stanley 1986b). The composite samples described in this report consisted of aliquots of 5 to 26 individual adipose tissue specimens. The composites were prepared to represent the general vs. population stratified by the nine U.S. census divisions and three age groups.

Several stable isotope labeled compounds were added to the tissue as surrogates. The spiked adipose tissue sample was extracted with methylene chloride using a Tekmar® Tissumizer. The extracts were filtered through anhydrous sodium sulfate. Extractable lipid was determined using approximately 1% of the resulting extract. The extract was concentrated and the lipid was separated from organic analytes using GPC. Approximately 10% of each sample extract (1.0 to 2.0 g) was reserved for additional cleanup on a carbon based (Carbopak C/Celite) chromatography column. The GPC-cleaned extracts (90% of the original sample) were concentrated and then fractionated using Florisil. The Florisil fractions were concentrated, spiked with an internal quantitation standard, and analyzed by HRGC/MS. Before proceeding with the analysis of PCDD and PCDF, the fractions were combined and subjected to further cleanup on a charcoal/glass fiber column.

A. Extraction

Frozen composited adipose tissue samples (~ 20 g) were placed in a 2.2 x 15 cm culture tube. Each composite was spiked with several surrogate compounds including naphthalene-d_8 (2 µg), chrysene-d_{12} (2 µg), 1,2,4,5-tetrachlorobenzene-$^{13}C_6$ (2 µg), 3,3',4,4'-tetrachlorobiphenyl-$^{13}C_{12}$ (10 µg), 2,2',3,3',5,5',6,6'-octachlorobiphenyl-$^{13}C_{12}$ (8 µg), decachlorobiphenyl-$^{13}C_{12}$ (10 µg), 2,3,7,8-tetrachlorodibenzo-p-dioxin-$^{13}C_{12}$ (1 ng), and octachlorodibenzo-p-dioxin-$^{13}C_{12}$ (5 ng). The spiked adipose tissues were brought to room temperature and homogenized for approximately 1 min with a Tekmar® Tissumizer (Tekmar 18-EN probe) with successive aliquots (10 mL) of methylene chloride (Burdick & Jackson, distilled in glass). The methylene chloride extracts were dried by passage through anhydrous sodium sulfate. The sodium sulfate column was rinsed with enough methylene chloride to bring the final extract volume to 100 mL. The extractable lipids were determined by removing a 1-mL aliquot from the final extract. This aliquot was placed in a preweighed 2-dram vial, and solvent was removed using purified nitrogen. The vial was reweighed and the lipid content was determined using the weight difference.

B. Cleanup

1. Gel Permeation Chromatography

The sample extracts were concentrated by Kuderna-Danish evaporation. The final volumes were adjusted such that the solutions contained approximately 0.3 g of lipid per milliliter. An ABC autoprep GPC with an automated sampling valve was used for all bulk lipid separations. GPC columns were prepared with approximately 60 g of Biobeads SX-3 swelled in methylene chloride and packed as a slurry. The GPC was operated using methylene chloride at 5 mL/min under a pressure of 7 to 15 psi. The GPC columns were calibrated using a solution of vitamin E-acetate. Collection of the GPC effluent for the semivolatile organic compounds was initiated as the response to the vitamin E-acetate returned to baseline. Approximately 1 g of lipid material

was added to each sampling loop of the GPC system. A total of 160 mL of GPC effluent was collected after elution. The GPC effluents for a single sample (3,200 mL/20 g of lipid) were combined, concentrated, and taken through the GPC procedure a second time to remove residual lipid materials.

2. Florisil Fractionation

Florisil columns (12.5 g, 60/100 mesh, activated at 130°C) were packed in hexane. Anhydrous sodium sulfate was added to the top of each column. The GPC extracts were concentrated and exchanged to hexane (final volume approximately 5 mL). This extract was added to the top of the Florisil column and eluted with 200 mL each of 6%, 15%, and 50% diethyl ether in hexane. The 6% fraction was collected separately from the 15% and 50% diethyl ether fractions which were combined. The fractions were concentrated and solvent exchanged to hexane using Kuderna-Danish evaporation. When the eluents had concentrated to approximately 5 mL, they were further concentrated to 1 mL under a gentle stream of dry nitrogen. The fractions were transferred to 1-mL conical vials and concentrated again to a final volume of 200 µL using nitrogen. All extracts were stored in a refrigerator until analyzed by HRGC/MS. The 6% Florisil fractions were analyzed for general semivolatile organics (particularly organochlorine pesticides, PCBs, chlorobenzenes, polynuclear aromatic hydrocarbons, etc.). The more polar Florisil fractions were analyzed for compounds such as phthalates, phosphate triestes and additional organochlorine pesticide residues (Stanley 1986c).

C. Isolation of PCDD and PCDF

Following the broad scan HRGC/MS analysis for general semivolatile organics, the Florisil column fractions were recombined and the sample extracts were taken through additional cleanup for isolation of the PCDDs and PCDFs prior to analysis by HRGC/MS selected ion monitoring (SIM) techniques. It was necessary to combine the Florisil fractions due to partial separation of the PCDD and PCDF congeners in that chromatographic.

The isolation of the PCDD and PCDF congeners was accomplished using one of two carbon-based (Amoco PX-21 or Carbopak C) chromatography procedures. These cleanup techniques are modified procedures that isolate polychlorinated aromatics from biological samples (Smith 1984) and soils. (USEPA 1983) The Amoco PX-21 was used for samples that had been taken through the GPC and Florisil fractionation procedures. Analysis of these samples yielded information only for the tetra- and pentachloro dioxins and furans. The higher chlorinated PCDD and PCDF congeners, especially OCDD and OCDF were retained on Florisil using the elution procedure designated for the general semivolatile organic analysis.

The Carbopak C/Celite column was selected over the Amoco PX-21/glass fiber column for the cleanup of the smaller sample aliquots since it resulted in a less labor intensive procedure. The Carbopak C/Celite column was used for the aliquot (1.0-2.0 g) of the original sample that had been taken only through the GPC cleanup steps. These aliquots were used for analysis of the hexa- through octachloro congeners.

1. Amoco PX-21/Glass Fiber Adsorbent

This carbon based chromatography column was used to prepare the extracts that were analyzed for the tetra- and pentachloro PCDD and PCDF congeners. Whatman GF/D fiber filters (600 mg) were cut into small pieces, suspended in approximately 70 mL of methylene chloride, and shredded with a Tekmar® Tissumizer. Amoco PX-21 carbon (50 mg), provided by Dr. L. Smith, U.S. Fisheries and Wildlife, Columbia, Missouri, was added to this mixture. The grinding was continued until the carbon was uniformly distributed on the fibers. This mixture yielded the packing required for a single adsorbent column.

a. Preparation of the Adsorbent Column

Thick-walled, 1.0 cm i.d. precision bore glass tubes (6-cm lengths) were custom fit with Teflon® plugs. These plugs were bored to accommodate 1/16 in. o.d. stainless steel tubing. This stainless steel tubing was used to connect the columns to the solvent reservoir. Both ends of the column were equipped with stainless steel tubing to allow disconnection of the column and inversion to change the direction of the solvent flow. To pack the column, one end was fitted with a Teflon® plug. Four disks of glass fiber filters (Whatman GF/D 1.0 cm diameters) were placed flush against the Teflon® plug. The carbon/glass fiber mixture consisting of 600 mg of glass fibers and 50 mg of carbon was added to the column in methylene chloride. A glass rod was used to pack the mixture. When this mixture was added to the column, four disks of Whatman GF/D 1.0 cm diameter glass fiber filters were gently packed on top of the carbon/glass fiber adsorbent. The second Teflon® plug was pushed into place, compressing the adsorbent. The column bed height measured 3 to 4 cm.

b. Column Cleanup

Prior to sample cleanup, the column was washed with 100 mL of toluene, then 100 mL of methanol, and then 100 mL of toluene again. The residual toluene was displaced with 150 mL of cyclohexane/methylene chloride, 50/50. After this solvent had eluted through the column in reverse flow, the column was inverted for forward flow. Immediately before sample application, an additional 50 mL of the 50/50 solvent was eluted through the column with nitrogen pressure to remove any air pockets. To maintain a flow of 3 to 5 mL/min, a slight nitrogen pressure was necessary.

c. Cleanup of Composite Sample Extracts

Following the broad scan HRGC/MS analysis, Florisil fraction extracts for the selected composites were combined and diluted with 5 mL of the cyclohexane/methylene chloride, 50/50 (v/v), solvent. This sample was added to the column reservoir and allowed to drain onto the column. The sample vial and column reservoir were rinsed with two 5-mL portions of the cyclohexane/methylene chloride (50/50) solvent. The flow rate was adjusted to 3 to 5 mL/min. After the last rinse, 75 mL of the cyclohexane/methylene chloride (50/50) solvent was added to the reservoir. This was followed by 50 mL of methylene chloride/methanol/benzene (75/20/5). The flow of the column was

reversed by inversion of the column. The reservoir was filled with 40 mL of toluene. This fraction was collected from the column at a rate no greater than 3 to 4 mL/min. A positive pressure on the system with nitrogen was necessary to achieve this flow rate.

Each solvent that eluted through the carbon/glass fiber adsorbent was collected separately. The toluene fraction collected in the reverse elution sequence was reserved for analysis of PCDD and PCDF.

Concentration of the toluene fraction was achieved using a gentle stream of prepurified nitrogen. The extract was transferred to a 1.0-mL conical vial and concentrated just to dryness. The collection tube was rinsed with additional 1.0-mL aliquots of toluene that were also concentrated to dryness in the conical vial.

2. Carbopak C/Celite Adsorbent Column Cleanup

An alternate cleanup procedure was necessary to achieve analytical data for the hexa- through octachloro-PCDD and PCDF. The method evaluation studies for the broad scan analysis procedure of general semivolatile organics demonstrated that the higher chlorinated PCDD and PCDF, especially the octachloro congeners were inefficiently recovered from the Florisil fractionation column. (Stanley 1986) To overcome this problem approximately 10% (1 to 2 g original weight) of each sample was reserved following the GPC-cleanup of the original extract. This aliquot was taken through a carbon cleanup column consisting of 18% Carbopak C on Celite 545®.

a. Preparation of the Carbon Adsorbent Column

This material was prepared by mixing 3.6 g of Carbopak C (Supelco, 80/100 mesh) and 16.4 g of Celite 545® (Fisher Scientific). The mixture was activated at 150°C for at least 6 h and then stored in a desiccator. Chromatographic columns were prepared using 5-mL disposable pipettes fitted with small plugs of glass wool. The Carbopak C/Celite mixture was packed using a vacuum aspirator until a 2-cm (340 mg) length of packing was obtained.

b. Column Cleanup

The columns were preeluted with 2 mL of toluene followed by 1 mL of methylene chloride/methanol/benzene (75/20/5), 1 mL of 1/1 cyclohexane in methylene chloride, and 2 mL of hexane.

c. Cleanup of Composite Sample Extracts

The sample extracts were added to the Carbopak C/Celite columns with several rinses of hexane. The columns were eluted with 1 mL of cyclohexane/methylene chloride (50/50), 1 mL of methylene chloride/methanol/benzene (75/20/5), and 20 mL of toluene. The toluene fraction was collected in a culture tube and concentrated under flowing prepurified nitrogen. Final concentration was achieved in a 1-mL conical vial. The sample was taken just to dryness and submitted for HRGC/MS-SIM analysis for PCDDs and PCDFs.

D. Instrumental Analysis

The PCDD and PCDF analyses for the tetra- through octachloro congeners were completed using a Kratos MS-50 double focusing mass spectrometer. The ion source of the MS-50 was interfaced with a Carlo Erbra gas chromatograph equipped with a Grob type split/splitless injector. Analysis of the PCDD and PCDF by congener group was achieved using either a 15 m or 60 m x 0.25 Durabond DB-5 fused silica column. Twenty microliters of isooctane was added to the conical vials containing the sample extract from the Amoco PX-21/glass fiber adsorbent column. Ten microliters of isooctane was added to the residues from the Carbopak C/Celite column. The resulting extract was sonicated for at least 30 s in an ultrasonic bath before proceeding with the analysis.

The analyses for the tetra- to octachloro-PCDD and PCDF congeners for the composite of the 45-plus age category was achieved using two injections. Two ions characteristic of the molecular cluster for each PCDD and PCDF congener, the internal standards, and a reference compound (perfluorokerosene, PFK) were monitored. Tables 1 and 2 summarize the masses monitored. The two values for each entry indicate the narrow mass ranges that were monitored for a specified time at a mass resolution of 2500. All other sample analyses (0-14 and 15-44 age composites) were completed in a single injection using the data acquisition parameters listed in Table 3. The differences in the instrumental analyses for the 45 plus versus the 0-14 and 15-44 age groups resulted from modification of the data acquisition programs. This modification allowed the MS operator to automatically switch acquisition parameters to monitor first the tetra- and pentachloro congeners and then the hexa- through octachloro congeners.

One-microliter aliquots were injected and the HRGC column was held isothermally at 100°C for 4 min, programmed rapidly to 270°C, held for 15 min, and then programmed at 10°C/min to a final temperature of 325°C and held for 20 min. Table 4 summarizes the analytical standards used for calibration standards and isomer specific measurements. Quantitation was achieved by the internal standard method using 2,3,7,8-TCDD-$^{13}C_{12}$ for the tetra- and pentachloro congeners and OCDD-$^{13}C_{12}$ for the hexa- through octachloro congeners.

E. Quality Assurance/Quality Control

The QA/QC procedures included the daily verification of response factors and instrument sensitivities, analysis of method blanks, and estimating recoveries of the internal quantitation standards. Additional QA/QC procedures included establishing criteria for qualitative identification and quantitation. These criteria, including the relative retention time of specific congeners, ion ratios, and definition of limits of detection are described below in the discussion on data interpretation.

Table 1. Mass Ranges and Dwell Times for Analysis of Tetra- and Pentachloro PCDD and PCDF at 2500 Resolution

Analyte	Mass range (amu)	Dwell time (s)
TCDF	303.801 - 304.001	0.069
TCDF	305.798 - 305.998	0.069
TCDD	319.796 - 319.996	0.062
TCDD	321.793 - 321.993	0.062
$^{13}C_{12}$-TCDD	331.836 - 332.036	0.059
$^{13}C_{12}$-TCDD	333.833 - 334.033	0.057
PeCDF	337.762 - 337.962	0.056
PeCDF	339.759 - 339.959	0.056
PeCDD	353.757 - 353.957	0.051
PeCDD	355.754 - 355.954	0.051
PFK	330.879 - 331.079	0.059

Table 2. Mass Ranges and Dwell Times for Analysis of Hexa-, Hepta-, and Octachloro PCDD and PCDF at 2500 Resolution

Analyte	Mass range (amu)	Dwell time (s)
HxCDF	373.720 - 373.920	0.056
HxCDF	375.717 - 375.917	0.056
HxCDD	389.715 - 389.915	0.051
HxCDD	391.712 - 391.912	0.052
HpCDF	407.682 - 407.882	0.048
HpCDF	409.678 - 409.878	0.046
HpCDD	423.676 - 423.876	0.043
HpCDD	425.674 - 425.874	0.043
OCDF	441.593 - 441.893	0.061
OCDF	443.590 - 443.890	0.059
OCDD	457.587 - 457.887	0.056
OCDD	459.642 - 459.826	0.069
$^{13}C_{12}$-OCDD	469.627 - 469.927	0.052
$^{13}C_{12}$-OCDD	471.624 - 471.925	0.052
PFK	430.875 - 431.075	0.043

Table 3. Mass Ranges and Dwell Times for Analysis of Tetra- Through Octachloro PCDD and PCDF in a Single Determination at 2500 Resolution

Analyte	Mass range (amu)	Dwell time (s)
Descriptor 1		
TCDF	303.806 - 303.998	0.033
TCDF	305.801 - 305.995	0.033
$^{13}C_{12}$-TCDF	315.802 - 316.001	0.033
$^{13}C_{12}$-TCDF	317.798 - 317.999	0.032
TCDD	319.795 - 319.997	0.031
TCDD	321.791 - 321.995	0.031
$^{13}C_{12}$-TCDD	331.832 - 332.041	0.030
$^{13}C_{12}$-TCDD	333.820 - 334.039	0.030
PeCDF	337.756 - 337.969	0.029
PeCDF	339.752 - 339.967	0.030
PeCDD	335.742 - 355.967	0.029
PeCDD	357.738 - 357.964	0.056
PFK	380.855 - 381.095	0.052
Descriptor 2		
HxCDF	373.702 - 373.938	0.014
HxCDF	375.698 - 375.936	0.014
PFK	380.855 - 381.095	0.013
HxCDD	389.692 - 389.938	0.013
HxCDD	391.689 - 391.936	0.013
HpCDF	407.653 - 407.910	0.025
HpCDF	409.649 - 409.907	0.025
HpCDD	423.643 - 423.910	0.024
HpCDD	425.639 - 425.907	0.025
$^{37}Cl_4$-HpCDD	429.632 - 429.903	0.024
$^{37}Cl_4$-HpCDD	431.628 - 431.900	0.023
OCDF	441.603 - 441.882	0.023
OCDF	443.600 - 443.879	0.023
OCDD	457.593 - 457.881	0.022
OCDD	459.590 - 459.879	0.022
$^{13}C_{12}$-OCDD	469.589 - 469.885	0.022
$^{13}C_{12}$-OCDD	471.586 - 471.883	0.022

Table 4. Specific PCDD and PCDF Congeners Available for Calibration and Isomer Specific Measurements

Compound	Source	Lot/code	Use
2,3,7,8-TCDF	Cambridge Isotope Laboratories	EF-903	quantitative
2,3,4,8-TCDF	C Rappe, Univ. Umea, Sweden	T16	qualitative
2,3,7,8-TCDD	EPA QA Materials Branch	20603	quantitative
1,2,3,7,8-PeCDD	KOR Isotopes	AA-8-185	quantitative
1,2,3,7,8-PeCDF	C Rappe, Univ. Umea, Sweden	P8	qualitative
2,3,4,7,8-PeCDF	C Rappe, Univ. Umea, Sweden	P26	qualitative
1,2,3,4,7,8-HxCDD	KOR Isotopes	JB-II-65	quantitative
1,2,3,4,7,8-HxCDF	C Rappe, Univ. Umea, Sweden	Hx4	qualitative
1,2,3,4,6,7,8-HpCDD	KOR Isotopes	JB-II-64	quantitative
1,2,3,4,7,8,9-HpCDF	C Rappe, Univ. Umea, Sweden	Hp4	qualitative
OCDD	Ultra Scientific		quantitative
OCDF	Ultra Scientific		quantitative
2,3,7,8-TCDD-$^{13}C_{12}$	Cambridge Isotope Laboratories	R00208	quantitative
2,3,7,8-TCDF-$^{13}C_{12}$	Cambridge Isotope Laboratories	AWN-1203-T2	quantitative
1,2,3,4,6,7,8-HpCDD-$^{37}Cl_4$	KOR Isotopes	SSY-4-32	quantitative
$^{13}C_{12}$-OCDD	Cambridge Isotope Laboratories		quantitative

F. Data Interpretation

1. Qualitative

The HRGC/MS elution profiles of the tetra- through octachloro-PCDD and PCDF congeners were established through the analysis of environmental sample extract (fly ash from a municipal waste incinerator). The characteristic ions for each homolog were plotted within the retention window established using this mixture. The coincidental response of the characteristic ions monitored within the established retention window and within ± 20% of the theoretical ion ratio were the qualitative criteria that were used to identify a response as a PCDD or PCDF congener. The identification of the specific congeners required the response to be within ± 5 s of the retention of the authentic standard relative to the specific internal standard.

2. Quantitation

Quantitation of the PCDD and PCDF congeners was achieved using the carbon-13 (^{13}C) labeled internal standards, $^{13}C_{12}$-2,3,7,8-TCDD ($^{13}C_{12}$-TCDD), and $^{13}C_{12}$-OCDD. The tetra- and pentachloro homologs were quantitated versus $^{13}C_{12}$-TCDD, while the levels of the hexa- through octachloro compounds were calculated versus the $^{13}C_{12}$-OCDD response. The recoveries of the internal quantitation standards $^{13}C_{12}$-2,3,7,8-TCDD and $^{13}C_{12}$-OCDD were achieved by comparing the relative responses to the internal recovery standards, $^{13}C_{12}$-2,3,7,8-TCDF and $^{37}Cl_4$-1,2,3,4,6,7,8-HpCDD, respectively. These internal recovery standards were added only to the extracts from the 0-14 and 15-44 age groups.

Relative response factors (RRF) were calculated for each homolog using the PCDD compounds listed in Table 4. Except for 2,3,7,8-TCDF and OCDF, the PCDF congeners were available as qualitative standards only. The RRF values for the PCDF homologs, therefore, were assumed to be the same as the respective quantitative PCDD congener. The RRF values were calculated as shown in Equation 1.

$$RRF = \frac{A_{STD} \times C_{IS}}{A_{IS} \times C_{STD}} \qquad \text{Eq. 1}$$

where A_{STD} = the sum of the area responses for the two characteristic ions of the standard compound;

A_{IS} = the sum of the area responses for the two characteristic ions of the internal standard;

C_{IS} = concentration of the internal standard (pg/μL); and

C_{STD} = Concentration of the standard compound (pg/μL).

A calibration curve was established using three concentration levels of standards; for example, the calibration curve for 2,3,7,8-TCDD was initially established with standards at concentrations of 1, 10, and 100 pg/μL. The 1 and/or 10 pg/μL standards were analyzed daily to verify response factors and method sensitivity.

The concentration of a PCDD or PCDF congener in a composite sample was calculated as shown in Equation 2.

$$C_{WT} = \frac{A_{sample} \times C_{IS}}{A_{IS} \times RRF \times Wt} \qquad \text{Eq. 2}$$

where C_{WT} = wet tissue concentration of the PCDD or PCDF congener in each tissue (pg/g);

A_{sample} = sum of the area responses for the two characteristic ions of of the PCDD or PCDF congener;

A_{IS} = sum of the area responses for the two characteristic ions of the respective internal standard;

C_{IS} = concentration of the internal standard added to the sample (1,000 pg of $^{13}C_{12}$-TCDD or 5,000 pg of $^{13}C_{12}$-OCDD);

RRF = the relative response factor for the PCDD or PCDF congener from Equation 1; and

Wt = mass of the composited FY82 NHATS specimens (grams).

The lipid-adjusted concentration was calculated by dividing the wet tissue weight concentration by the extractable lipid (%) value.

All data were qualified to reflect whether the compound was a positive quantifiable parameter, present as a trace value only, or was not detected. Positive quantifiable values were identified for responses greater than 10 times the average background signal-to-noise. Trace (Tr) values were assigned to responses that were in the range of 2.5 to 10 times the average background signal-to-noise. A value of not detected (ND) was used to reflect that a response was not detected at greater than 2.5 times the average signal-to-noise. The definition of 2.5 times the average background signal-to-noise for the LOD measurement was selected to maintain consistency with the existing protocols for the determination of 2,3,7,8-TCDD (EPA 1983). A limit of detection (LOD) was calculated for all trace and not detected values using the peak height response of the respective internal standard and the average measured signal-to-noise for the characteristic ions of the PCDD and PCDF congeners.

IV. RESULTS

A qualitative summary of the HRGC/MS-SIM analyses of the sample extracts of FY82 NHATS composite samples is presented in Figures 2 to 5. Identification of compounds at trace or positive quantifiable values are designated by a plus. Compounds that were not detected are designated by a minus. The number of plus and minus symbols under each age group represents the total number of composites analyzed. As noted, the predominant compounds in the adipose tissue composites were the 2,3,7,8-substituted congeners. Congener designations are presented based on the corresponding retention times of the observed responses with the available standards and on the basis of information of the specific congeners present in adipose tissue from previous studies (Graham et al. 1985; Nygren et al. 1985; Patterson et al. 1985; Rappe et al. 1985; Ryan et al. 1985a; Ryan et al. 1985b; Shecter, Ryan 1985). Although the elution patterns of the HxCDD and HxCDF congeners were similar to those reported in other studies (Schecter et al. 1985; Schecter, Ryan 1985; Ryan et al. 1985a; Ryan et al 1985b; Nygren et al. 1985; Rappe et al. 1985; Graham et al. 1985; Patterson et al 1985; Ryan 1985), the exact congeners cannot be assigned due to availability of standards at the time of analysis.

Figure 6 provides examples of the HRGC/MS chromatograms observed for the two different extract cleanup procedures. The lower reconstructed ion chromatogram (RIC) illustrates the response noted for an extract taken through the Amoco PX-21/glass fiber column. The upper RIC was achieved for the analysis of an aliquot of the same sample taken through the Carbopak C/Celite column. As noted from the chromatogram (Figure 6) of the Amoco PX-21/glass fiber column, there was no response noted for the OCDD and $^{13}C_{12}$-OCDD. This is a result of the retention of the higher chlorinated compounds on the Florisil column used for preparation of the samples for broad scan analysis of semivolatile organic compounds by HRGC/MS scanning techniques. (Stanley 1986). This is the reason it was necessary to use a second aliquot of the extract that was taken only through GPC and a carbon based column to determine the hexa- through octachloro congeners. The abrupt change in baseline observed for each chromatogram at approximately 1,750 scans ($\sim$ 35 min) is due to the change in descriptors from monitoring the tetra- and pentachloro congeners to the hexa- through octachloro congeners.

The large response at approximately 1300 scans can be attributed to $^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl. This compound was added as a surrogate to the initial sample to monitor method recoveries in the broad scan analysis. Retention of this particular PCB on the charcoal column is a result of non-ortho,ortho'-substitution. (Stalling et al. 1979) The responses for the other peaks in the HRGC/MS chromatograms can be assigned to PCDD congeners and internal standards. The responses to PCDF congeners are obvious when looking at the extracted ion current plots of the characteristic ions.

Census Region	Northeast					
Census Division	New England			Middle Atlantic		
Age Group	0-14	15-44	45+	0-14	15-44	45+
Compound						
2,3,7,8-TCDD	-	+	-	+-	+-	-+
1,2,3,7,8-PeCDD	+	+	-	++	++	++
HxCDD	+	+	+	++	++	++
1,2,3,4,7,8,9-HpCDD	+	+	+	++	++	++
OCDD	+	+	+	++	++	++
2,3,7,8-TCDF	+	-	-	+-	+-	+-
2,3,4,7,8-PeCDF	-	+	-	++	++	++
HxCDF	-	-	+	-+	++	++
1,2,3,4,6,7,8-HpCDF	-	+	+	++	++	++
OCDF	-	+	+	+-	+-	+-

Figure 2. PCDD and PCDF detected in the NHATS FY82 composite specimens from the Northeast Census Region.

Census Region	West					
Census Division	Mountain			Pacific		
Age Group	0-14	15-44	45+	0-14	15-44	45+
Compound						
2,3,7,8-TCDD	-	+	+	-	+	+
1,2,3,7,8-PeCDD	+	+	+	+	+	+
HxCDD	+	+	+	+	+	+
1,2,3,4,7,8,9-HpCDD	+	+	+	+	+	-
OCDD	+	+	+	+	+	+
2,3,7,8-TCDF	+	-	-	-	+	-
2,3,4,7,8-PeCDF	+	+	+	+	+	+
HxCDF	-	+	+	-	+	-
1,2,3,4,6,7,8-HpCDF	+	+	+	+	+	-
OCDF	+	-	-	+	+	-

Figure 3. PCDD and PCDF detected in the NHATS FY82 composite specimens from the West Census Region.

Census Region	South								
Census Division	South Atlantic			East South Central			West South Central		
Age Group	0-14	15-44	45+	0-14	15-44	45+	0-14	15-44	45+
Compound									
2,3,7,8-TCDD	++	+-+-	+-++	+	++	+-	+	+-	+
1,2,3,7,8-PeCDD	++	++-+	+-+-	+	++	+-	+	++	+
HxCDD	++	++++	++++	+	++	++	+	++	+
1,2,3,4,7,8,9-HpCDD	++	++++	++++	+	++	++	+	++	+
OCDD	++	++++	++++	+	++	++	+	++	+
2,3,7,8-TCDF	--	-+--	+---	-	--	--	-	--	-
2,3,4,7,8-PeCDF	++	+++-	+-+-	+	++	+-	+	+-	+
HxCDF	+-	++--	++++	+	++	++	+	+-	+
1,2,3,4,6,7,8-HpCDF	++	++++	+-++	+	++	++	+	++	+
OCDF	--	++--	----	+	+-	--	-	--	-

Figure 4. PCDD and PCDF detected in the NHATS FY82 composite specimens from the South Census Region.

Census Region	North Central					
Census Division	East North Central			West North Central		
Age Group	0-14	15-44	45+	0-14	15-44	45+
Compound						
2,3,7,8-TCDD	-+	+++	-++	+	+	++
1,2,3,7,8-PeCDD	++	+++	-++	+	+	++
HxCDD	++	+++	+++	-	+	++
1,2,3,4,7,8,9-HpCDD	++	+++	+++	+	+	++
OCDD	++	+++	+++	+	+	++
2,3,7,8-TCDF	++	+--	---	-	-	--
2,3,4,7,8-PeCDF	++	+++	-++	+	+	++
HxCDF	-+	+++	+++	-	-	++
1,2,3,4,6,7,8-HpCDF	++	+++	+++	+	+	++
OCDF	++	+++	---	-	-	+-

Figure 5. PCDD and PCDF detected in the NHATS FY82 composite specimens from the North Central Census Region.

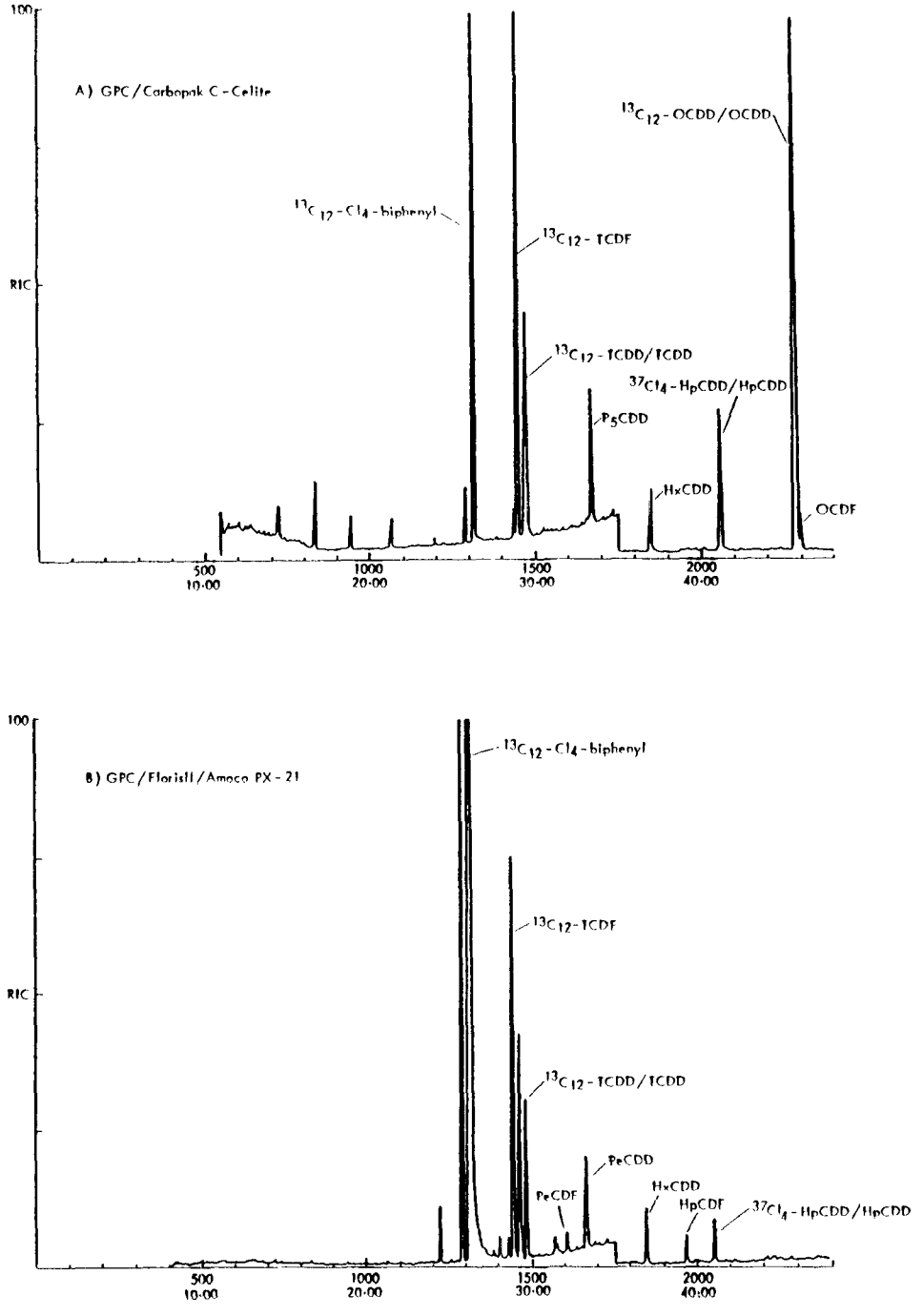


Figure 6. HRGC/MS chromatograms of a sample extract (composite 1 of the 15-44 age group, Middle Atlantic (MA) Census division) taken through (a) GPC/Carbopak C cleanup (equivalent to 2.0 g tissue sample) and (b) GPC/Florisil/Amocol PX-21 cleanup (equivalent to 20 g tissue sample).

The results of the PCDD and PCDF analyses for the specific congeners or homologs are summarized in Tables 5 to 14. These data are reported as both wet tissue weight and lipid-adjusted concentrations. As discussed in the experimental section of this report, the data for the tetra- and pentachloro-PCDD and PCDF compounds were determined in the sample aliquot (approximately 20 g), that was taken through the entire cleanup procedure (GPC, Florisil, and Amoco PX-21). However, as noted in Tables 5, 6, 10, and 11, the data for the tetra- and pentachloro congeners for several of the composites were reported for sample aliquots (1.0 to 2.0 g) that were taken only through the GPC separation and Carbopak C/Celite cleanup. This was done because of reanalysis requirements of the larger sample aliquot for general semivolatile organics or as a result of irrecoverable loss of the larger sample aliquot during final cleanup. The analysis of the smaller aliquot for the tetra- and pentachloro homologs was compromised only with respect to the achievable limit of detection.

The data for the hexa- through octachloro congeners (Tables 7-9 and 12-15) are generally reported for sample aliquots of 1.0 to 2.0 g that had been taken through the GPC and Carbopak C/Celite procedures. However, the results of the analysis of the larger sample aliquot for two composites (samples 82042 and 82083) are reported in the data tables. Quantitative data are reported for sample 82042 while entries for 82083 indicate that the hexa- through octachloro congeners were detected but not quantitated due to the low recovery of the internal standard. Quantitative data have been reported for the hexa- through octachloro-PCDDs and PCDFs for sample 82042 but the data may be considered suspect based on the possible differences in the recoveries of these compounds in the GPC, Florisil and Amoco PX-21 cleanup steps as compared to the GPC and Carbopak C/Celite procedure used for the smaller sample aliquots.

Figure 7 summarizes the average PCDD and PCDF congener levels in the composite specimens based on the age categories. The data are plotted to indicate both the average wet tissue weight concentrations and the lipid-adjusted concentrations. The average concentrations were determined from composites that resulted in trace or positive quantifiable values for each PCDD or PCDF congener. The estimated limits of detection for the not detected responses were not included in the calculation of these averages. A simple Q-test was used to determine data points that were qualified as outliers for each age group. The values determined to be outliers by this test were not included in the values for the average PCDD and PCDF levels given in Figure 7. These data points are also identified by a footnote in Tables 5-14.

As noted in Figure 7, the average concentrations of specific PCDD congeners, with the exception of PeCDD, generally show an increase with respect to age. But no trends are noted for the PCDF data with respect to age. This possibly resulted from the low levels of PCDFs in the composite samples, indicating that potential exposure to these compounds is considerably less than that of PCDDs. The large difference noted for the OCDF concentration in the 45-plus age category is that a positive response was detected in only 3 of the 16 composites. The OCDF concentration of these samples ranged from 240 to 360 pg/g (wet tissue weight concentration).

TABLE 5. DATA SUMMARY FOR 2,3,7,8-TETRACHLORODIBENZO-p-DIOXIN [1746-01-6] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	23.0	77.5		4.6		5.9		3/15/85
MA	82050	1	2	2.0	80.7		8.6		11		3/22/85
MA	82051	1	3	16.2	82.5	ND		20		24	10/30/84
MA	82052	2	1	2.0	84.7	ND		6.1		7.2	3/19/85
MA	82053	2	2	2.0	78.2	ND		6.0		7.7	3/19/85
MA	82054	2	3	18.0	83.9		6.6		7.9		10/31/84
MO	82036	1	1	1.0	62.3	ND		4.0		6.4	3/20/85
MO	82037	1	2	18.3	74.1		4.0		5.4		3/15/85
MO	82041	1	3	21.0	84.3		12		14		10/26/84
NE	82042	1	1	19.1	55.1	ND		1.0		1.8	3/18/85
NE	82043	1	2	21.9	87.6		4.4		5.0		3/18/85
NE	82044	1	3	26.7	79.2	NA					
PA	82046	1	1	19.7	62.9	ND		2.2		3.5	3/15/85
PA	82047	1	2	2.0	68.0	TR	3.0	1.0	4.4	1.5	3/20/85
PA	82048	1	3	22.0	87.1		0.4		0.5		10/31/84
WN	82063	1	1	23.4	63.1		1.4		2.2		3/13/85
WN	82064	1	2	20.6	76.2		10		13		3/11/85
WN	82065	1	3	22.5	89.8		2.4		2.7		10/30/84
WN	82066	2	3	21.4	80.4		2.6		3.2		10/30/84
SA	82067	1	1	20.7	83.9		2.5		3.0		3/18/85
SA	82068	1	2	26.4	90.3		4.6		5.1		3/15/85
SA	82069	1	3	20.0	82.5		2.9		3.5		10/31/84
SA	82070	2	1	2.0	69.1		1.5		2.2		3/13/85
SA	82071	2	2	19.5	89.3	ND		6.6		7.4	3/19/85
SA	82072	2	3	26.1	83.9	NA					
SA	82073	3	2	17.9	70.4	TR	6.8	2.5	9.7	3.6	3/20/85
SA	82074	3	3	18.0	86.7		7.3		8.4		10/31/85
SA	82075	4	2	2.0	72.0	ND		9.0		12.5	3/20/85
SA	82076	4	3	17.6	70.5		1.9		2.7		10/31/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NA = NOT ANALYZED--SUFFICIENT SAMPLE EXTRACT NOT AVAILABLE FOR ANALYSIS
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 5. DATA SUMMARY FOR 2,3,7,8-TETRACHLORODIBENZO-p-DIOXIN [1746-01-6] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	28.1	77.2		4.7		6.1		3/14/85
ES	82078	1	2	19.9	83.2		6.5		7.8		3/20/85
ES	82079	1	3	20.7	88.4		5.8		6.6		10/31/84
ES	82080	2	2	25.7	87.5		8.9		10		3/11/85
ES	82081	2	3	21.1	78.2	ND		1.0		1.3	10/31/84
WS	82082	1	1	11.1	67.6		2.7		4.0		3/18/85
WS	82083	1	2	22.7	86.7		6.8		7.8		3/18/85
WS	82084	1	3	22.4	94.6		2.4		2.5		10/26/84
WS	82085	2	2	2.0	86.3	ND		7.4		8.6	3/19/85
EN	82055	1	1	2.0	48.6	ND		4.2		8.6	3/22/85
EN	82056	1	2	21.6	75.5		3.1		4.1		3/14/85
EN	82057	1	3	19.8	80.6	NA					
EN	82058	2	1	2.0	69.3		7.1		10		3/19/85
EN	82059	2	2	21.4	83.2		7.6		9.1		3/13/85
EN	82060	2	3	26.2	83.2		4.5		5.4		11/1/84
EN	82061	3	2	2.0	83.2		6.0		7.2		3/20/85
EN	82062	3	3	23.2	85.3		6.0		7.0		12/5/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NA = NOT ANALYZED--SUFFICIENT SAMPLE EXTRACT NOT AVAILABLE FOR ANALYSIS
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 6. DATA SUMMARY FOR 1,2,3,7,8-PENTACHLORODIBENZO-p-DIOXIN [40321-76-4] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	TISSUE Weight g	% LIPID	Analysis	Wet Weight Conc. (PG/G)	Wet Weight LOD (pg/g)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	23.0	77.5		29		37		3/15/85
MA	82050	1	2	2.0	80.7		660[a]		820[a]		3/22/85
MA	82051	1	3	16.2	82.5		4300[a]		5200[a]		10/30/84
MA	82052	2	1	2.0	84.7		26		30		3/19/85
MA	82053	2	2	2.0	78.2	TR	37	22	47	28	3/19/85
MA	82054	2	3	18.0	83.9		7.9		9.4		10/31/84
MO	82036	1	1	1.0	62.3	TR	65	62	100	100	3/20/85
MO	82037	1	2	18.3	74.1		35		47		3/15/85
MO	82041	1	3	21.0	84.3		12		14		10/26/84
NE	82042	1	1	19.1	55.1		81[a]		150[a]		3/18/85
NE	82043	1	2	21.9	87.6		39		44		3/18/85
NE	82044	1	3	26.7	79.2	NA					
PA	82046	1	1	19.7	62.9	TR	22	9.9	34	16	3/15/85
PA	82047	1	2	2.0	68.0	TR	45	17	66	25	3/20/85
PA	82048	1	3	22.0	87.1		1.0		1.1		10/31/84
WN	82063	1	1	23.4	63.1		5.7		9.0		3/13/85
WN	82064	1	2	20.6	76.2		46		60		3/11/85
WN	82065	1	3	22.5	89.8		5.5		6.1		10/30/84
WN	82066	2	3	21.4	80.4		8.6		11		10/30/84
SA	82067	1	1	20.7	83.9	TR	10	6.2	12	7.4	3/18/85
SA	82068	1	2	26.4	86.4		36		42		3/15/85
SA	82069	1	3	20.0	82.5		15		18		10/31/84
SA	82070	2	1	2.0	69.1	TR	7.7	3.6	11	5.2	3/13/85
SA	82071	2	2	19.5	89.3	TR	150	50	170	56	3/19/85
SA	82072	2	3	26.1	83.9	NA					
SA	82073	3	2	17.9	70.4	ND		54		77	3/20/85
SA	82074	3	3	18.0	86.7		8.9		10		10/31/85
SA	82075	4	2	2.0	72.0	TR	60	50	83	69	3/20/85
SA	82076	4	3	17.6	70.5	ND		1.0		1.4	10/31/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NA = NOT ANALYZED--SUFFICIENT SAMPLE EXTRACT NOT AVAILABLE FOR ANALYSIS
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 6. DATA SUMMARY FOR 1,2,3,7,8-PENTACHLORODIBENZO-p-DIOXIN [40321-76-4] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	TISSUE Weight g	% LIPID	Analysis	Wet Weight Conc. (PG/G)	Wet Weight LOD (pg/g)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	28.1	77.2		20		26		3/14/85
ES	82078	1	2	19.9	83.2		47		56		3/20/85
ES	82079	1	3	20.7	88.4		15		17		10/31/84
ES	82080	2	2	25.7	87.5	TR	17	9.1	19	10	3/11/85
ES	82081	2	3	21.1	78.2	ND		1.0		1.3	10/31/84
WS	82082	1	1	11.1	67.6		11		16		3/18/85
WS	82083	1	2	22.7	86.7		29		33		3/18/85
WS	82084	1	3	22.4	94.6		1.9		2.0		10/26/84
WS	82085	2	2	2.0	86.3	TR	180	130	210	140	3/19/85
EN	82055	1	1	2.0	48.6	TR	20	11	41	23	3/22/85
EN	82056	1	2	21.6	75.5		17		23		3/14/85
EN	82057	1	3	19.8	80.6	NA					
EN	82058	2	1	2.0	69.3		3700 [a]		5300 [a]		3/19/85
EN	82059	2	2	21.4	83.2		51		61		3/13/85
EN	82060	2	3	26.2	83.2		8.2		9.9		11/1/84
EN	82061	3	2	2.0	83.2	TR	59	25	70	30	3/20/85
EN	82062	3	3	23.2	85.3		9.5		11		12/5/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NA = NOT ANALYZED--SUFFICIENT SAMPLE EXTRACT NOT AVAILABLE FOR ANALYSIS
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 7. DATA SUMMARY HEXACHLORODIBENZO-p-DIOXIN [34465-46-8] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	No. of Peaks	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	2.0	77.5	3		72		93		3/22/85
MA	82050	1	2	2.0	80.7	3		500 a		620 a		3/22/85
MA	82051	1	3	1.6	82.5	1		330		400		11/7/84
MA	82052	2	1	2.0	84.7	3		52		61		3/19/85
MA	82053	2	2	2.0	78.2	2		12		15		3/19/85
MA	82054	2	3	2.0	83.9	2		55		66		11/7/84
MO	82036	1	1	1.0	62.3	1	TR	36	24	58	39	3/20/85
MO	82037	1	2	2.0	74.1	3		83		110		3/22/85
MO	82041	1	3	2.0	84.3	1		30		36		11/8/84
NE	82042	1	1	19.1	55.1	1		6.2		11		5/1/85
NE	82043	1	2	2.0	87.6	3		50		57		3/22/85
NE	82044	1	3	2.0	79.2	1		330		420		11/8/84
PA	82046	1	1	2.0	62.9	2		38		60		3/21/85
PA	82047	1	2	2.0	68.0	2		76		110		3/20/85
PA	82048	1	3	2.0	87.1	1		28		32		11/6/84
WN	82063	1	1	2.0	63.1	0	ND		10		16	4/29/85
WN	82064	1	2	2.0	76.2	1		19		25		3/11/85
WN	82065	1	3	2.0	89.8	1		80		89		11/6/84
WN	82066	2	3	2.0	80.4	1		60		75		11/8/84
SA	82067	1	1	2.0	83.9	2		65		78		3/21/85
SA	82068	1	2	2.1	86.4	2		68		78		3/22/85
SA	82069	1	3	2.0	82.5	2		31		37		11/6/84
SA	82070	2	1	2.0	69.1	1		27		38		3/21/84
SA	82071	2	2	2.0	89.3	2		53		59		3/19/85
SA	82072	2	3	2.0	83.9	1		210		240		11/8/84
SA	82073	3	2	2.0	70.4	1		40		57		3/20/85
SA	82074	3	3	2.0	86.7	1		70		81		11/7/84
SA	82075	4	2	2.0	72.0	1		52		72		3/20/85
SA	82076	4	3	2.0	70.5	1		50		71		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N
NC = NOT CALCULATED THREE ISOMERS OF HxCDD WERE OBSERVED BUT NOT CALCULATED
DUE TO THE LOW RESPONSE OBSERVED FOR THE INTERNAL STANDARD, 13C12-OCDD.

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 7. DATA SUMMARY HEXACHLORODIBENZO-p-DIOXIN [34465-46-8] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	No. of Peaks	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	2.0	77.2	2		120		160		3/21/85
ES	82078	1	2	2.0	83.2	2		60		72		3/20/85
ES	82079	1	3	2.0	88.4	2		31		35		11/8/84
ES	82080	2	2	2.0	87.5	1	TR	140	43	160	49	3/21/85
ES	82081	2	3	2.0	78.2	2		50		63		11/8/84
WS	82082	1	1	1.0	67.6	1		13		19		4/11/85
WS	82083	1	2	22.7	86.7	3	NC					3/18/85
WS	82084	1	3	2.0	94.6	2		41		43		11/6/84
WS	82085	2	2	2.0	86.3	1	TR	29	11	33	13	3/19/85
EN	82055	1	1	2.0	46.6	2		26		53		3/22/85
EN	82056	1	2	2.0	75.5	2		91		120		3/21/85
EN	82057	1	3	2.0	80.6	1		70		87		11/8/84
EN	82058	2	1	2.0	69.3	2		350a		510a		3/19/84
EN	82059	2	2	2.0	83.2	3		75		90		3/21/85
EN	82060	2	3	2.0	83.2	1		22		26		11/8/84
EN	82061	3	2	2.0	83.2	2		93		110		3/20/85
EN	82062	3	3	2.0	85.3	1		42		49		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N
NC = NOT CALCULATED THREE ISOMERS OF HxCDD WERE OBSERVED BUT NOT CALCULATED DUE TO THE LOW RESPONSE OBSERVED FOR THE INTERNAL STANDARD, 13C12-OCDD.

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 8. DATA SUMMARY FOR 1,2,3,4,7,8,9-HEPTACHLORODIBENZO-p-DIOXIN [35822-46-9] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	2.0	77.5		43		55		3/22/85
MA	82050	1	2	2.0	80.7		360[a]		440[a]		3/22/85
MA	82051	1	3	1.6	82.5		340		410		11/7/84
MA	82052	2	1	2.0	84.7		31		36		3/19/85
MA	82053	2	2	2.0	78.2		63		81		3/19/85
MA	82054	2	3	2.0	83.9		90		110		11/7/84
MO	82036	1	1	1.0	62.3		44		71		3/20/85
MO	82037	1	2	2.0	74.1		83		110		3/22/85
MO	82041	1	3	2.0	84.3		45		53		11/8/84
NE	82042	1	1	19.1	55.1		2.8		5.1		5/1/85
NE	82043	1	2	2.0	87.6		45		51		3/22/85
NE	82044	1	3	2.0	79.2		390		490		11/8/84
PA	82046	1	1	2.0	62.9		30		48		3/21/85
PA	82047	1	2	2.0	68.0		84		120		3/20/85
PA	82048	1	3	2.0	87.1	ND		23		26	11/6/84
WN	82063	1	1	2.0	63.1		25		40		4/29/85
WN	82064	1	2	2.0	76.2		26		34		3/11/85
WN	82065	1	3	2.0	89.8		240		270		11/6/84
WN	82066	2	3	2.0	80.4		85		110		11/8/84
SA	82067	1	1	2.0	83.9		75		89		3/21/85
SA	82068	1	2	2.1	86.4		58		67		3/22/85
SA	82069	1	3	2.0	82.5		60		73		11/6/84
SA	82070	2	1	2.0	69.1		25		36		3/21/84
SA	82071	2	2	2.0	89.3		78		87		3/19/85
SA	82072	2	3	2.0	83.9		80		95		11/8/84
SA	82073	3	2	2.0	70.4		42		59		3/20/85
SA	82074	3	3	2.0	86.7		180		210		11/7/84
SA	82075	4	2	2.0	72.0		55		76		3/20/85
SA	82076	4	3	2.0	70.5		85		120		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED.
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N.
NC = NOT CALCULATED THE 1,2,3,4,7,8,9-HPCDD WAS DETECTED BUT NOT CALCULATED DUE TO LOW RECOVERY OBSERVED FOR THE INTERNAL STANDARD, 13C12-OCDD.

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 8. DATA SUMMARY FOR 1,2,3,4,7,8,9-HEPTACHLORODIBENZO-p-DIOXIN [35822-46-9] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	2.0	77.2		50		65		3/21/85
ES	82078	1	2	2.0	83.2		61		73		3/20/85
ES	82079	1	3	2.0	88.4		22		25		11/8/84
ES	82080	2	2	2.0	87.5		64		73		3/21/85
ES	82081	2	3	2.0	78.2		44		56		11/8/84
WS	82082	1	1	1.0	67.6		16		23		4/11/85
WS	82083	1	2	22.7	86.7	NC					3/18/85
WS	82084	1	3	2.0	94.6		140		150		11/6/84
WS	82085	2	2	2.0	86.3		83		96		3/19/85
EN	82055	1	1	2.0	48.6		33		68		3/22/85
EN	82056	1	2	2.0	75.5		96		130		3/21/85
EN	82057	1	3	2.0	80.6		75		93		11/8/84
EN	82058	2	1	2.0	69.3		900[a]		1300[a]		3/19/84
EN	82059	2	2	2.0	83.2		160		190		3/21/85
EN	82060	2	3	2.0	83.2		35		42		11/8/84
EN	82061	3	2	2.0	83.2		110		130		3/20/85
EN	82062	3	3	2.0	85.3		50		59		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED.
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N.
NC = NOT CALCULATED THE 1,2,3,4,7,8,9-HPCDD WAS DETECTED BUT NOT CALCULATED DUE TO LOW RECOVERY OBSERVED FOR THE INTERNAL STANDARD, 13C12-OCDD.

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 9. DATA SUMMARY FOR OCTACHLORODIBENZO-p-DIOXIN [3268-87-9] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	2.0	77.5		330		430		3/22/85
MA	82050	1	2	2.0	80.7		760		940		3/22/85
MA	82051	1	3	1.6	82.5		550		670		11/7/84
MA	82052	2	1	2.0	84.7		680		800		3/19/85
MA	82053	2	2	2.0	78.2		370		470		3/19/85
MA	82054	2	3	2.0	83.9		650		770		11/7/84
MO	82036	1	1	1.0	62.3		190		300		3/20/85
MO	82037	1	2	2.0	74.1		610		820		3/22/85
MO	82041	1	3	2.0	84.3		1000		1200		11/8/84
NE	82042	1	1	19.1	55.1		10		19		5/1/85
NE	82043	1	2	2.0	87.6		350		390		3/22/85
NE	82044	1	3	2.0	79.2		3000[a]		3700[a]		11/8/84
PA	82046	1	1	2.0	62.9		130		200		3/21/85
PA	82047	1	2	2.0	68.0		680		1000		3/20/85
PA	82048	1	3	2.0	87.1		500		570		11/6/84
WN	82063	1	1	2.0	63.1		150		240		4/29/85
WN	82064	1	2	2.0	76.2		390		510		3/11/85
WN	82065	1	3	2.0	89.8		1300		1400		11/6/84
WN	82066	2	3	2.0	80.4		650		810		11/8/84
SA	82067	1	1	2.0	83.9		270		320		3/21/85
SA	82068	1	2	2.1	86.4		560		650		3/22/85
SA	82069	1	3	2.0	82.5		600		730		11/6/84
SA	82070	2	1	2.0	69.1		160		240		3/21/84
SA	82071	2	2	2.0	89.3		650		720		3/19/85
SA	82072	2	3	2.0	83.9		190		230		11/8/84
SA	82073	3	2	2.0	70.4		360		510		3/20/85
SA	82074	3	3	2.0	86.7		750		870		11/7/84
SA	82075	4	2	2.0	72.0		510		700		3/20/85
SA	82076	4	3	2.0	70.5		1000		1400		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N
NC = NOT CALCULATED OCDD WAS DETECTED IN SAMPLE BUT WAS NOT CALCULATED
DUE TO LOW RECOVERY OBSERVED FOR THE INTERNAL STANDARD, 13C12-OCDD

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 9. DATA SUMMARY FOR OCTACHLORODIBENZO-p-DIOXIN [3268-87-9] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	2.0	77.2		900		1200		3/21/85
ES	82078	1	2	2.0	83.2		1000		1200		3/20/85
ES	82079	1	3	2.0	88.4		450		510		11/8/84
ES	82080	2	2	2.0	87.5		660		750		3/21/85
ES	82081	2	3	2.0	78.2		750		960		11/8/84
WS	82082	1	1	1.0	67.6		130		190		4/11/85
WS	82083	1	2	22.7	86.7	NC					3/18/85
WS	82084	1	3	2.0	94.6		600		630		11/6/84
WS	82085	2	2	2.0	86.3		650		750		3/19/85
EN	82055	1	1	2.0	48.6		220		450		3/22/85
EN	82056	1	2	2.0	75.5		360		470		3/21/85
EN	82057	1	3	2.0	80.6		650		810		11/8/84
EN	82058	2	1	2.0	69.3		1000		1400		3/19/84
EN	82059	2	2	2.0	83.2		1100		1300		3/21/85
EN	82060	2	3	2.0	83.2		420		500		11/8/84
EN	82061	3	2	2.0	83.2		810		980		3/20/85
EN	82062	3	3	2.0	85.3		400		470		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N
NC = NOT CALCULATED OCDD WAS DETECTED IN SAMPLE BUT WAS NOT CALCULATED
DUE TO LOW RECOVERY OBSERVED FOR THE INTERNAL STANDARD, 13C12-OCDD

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 10. DATA SUMMARY FOR 2,3,7,8-TETRACHLORODIBENZOFURAN [51207-31-9] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	23.0	77.5		3.6		4.6		3/15/85
MA	82050	1	2	2.0	80.7		400[a]		500[a]		3/22/85
MA	82051	1	3	16.2	82.5		250		300		10/30/84
MA	82052	2	1	2.0	84.7	ND		2.4		2.8	3/19/85
MA	82053	2	2	2.0	78.2	ND		3.6		4.6	3/19/85
MA	82054	2	3	18.0	83.9	ND		38		45	10/31/84
MO	82036	1	1	1.0	62.3	TR	4.0	2.0	6.4	3.2	3/20/85
MO	82037	1	2	18.3	74.1	ND		12		17	3/15/85
MO	82041	1	3	21.0	84.3	ND		13		15	10/26/84
NE	82042	1	1	19.1	55.1		32		58		3/18/85
NE	82043	1	2	21.9	87.6	ND		15		17	3/18/85
NE	82044	1	3	26.7	79.2	NA					
PA	82046	1	1	19.7	62.9	ND		10		16	3/15/85
PA	82047	1	2	2.0	68.0	TR	2.3	1.0	3.4	1.5	3/20/85
PA	82048	1	3	22.0	87.1	ND	1.5		1.7		10/31/84
WN	82063	1	1	23.4	63.1	ND		8.1		13	3/13/85
WN	82064	1	2	20.6	76.2	ND		18		23	3/11/85
WN	82065	1	3	22.5	89.8	ND		4.6		5.1	10/30/84
WN	82066	2	3	21.4	80.4	ND		5.4		6.7	10/30/84
SA	82067	1	1	20.7	83.9	ND		8.8		10	3/18/85
SA	82068	1	2	26.4	86.4	ND		8.3		9.6	3/15/85
SA	82069	1	3	20.0	82.5		17		21		10/31/84
SA	82070	2	1	2.0	69.1	ND		9.2		13	3/13/85
SA	82071	2	2	19.5	89.3		7.9		8.8		3/19/85
SA	82072	2	3	26.1	83.9	NA					
SA	82073	3	2	17.9	70.4	ND		2.0		2.8	3/20/85
SA	82074	3	3	18.0	86.7	ND		14		16	10/31/85
SA	82075	4	2	2.0	72.0	ND		2.5		3.5	3/20/85
SA	82076	4	3	17.6	70.5	ND		4.0		5.7	10/31/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NA = NOT ANALYZED--SUFFICIENT SAMPLE EXTRACT NOT AVAILABLE FOR ANALYSIS
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N
a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 10. DATA SUMMARY FOR 2,3,7,8-TETRACHLORODIBENZOFURAN (51207-31-9) - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	28.1	77.2	ND		11		14	3/14/85
ES	82078	1	2	19.9	83.2	ND		1.6		1.9	3/20/85
ES	82079	1	3	20.7	88.4	ND		6.8		7.7	10/31/84
ES	82080	2	2	25.7	87.5	ND		5.8		6.6	3/11/85
ES	82081	2	3	21.1	78.2	ND		1.0		1.3	10/31/84
WS	82082	1	1	11.1	67.6	ND		5.3		7.8	3/18/85
WS	82083	1	2	22.7	86.7	ND		10		12	3/18/85
WS	82084	1	3	22.4	94.6	ND		5.1		5.4	10/26/84
WS	82085	2	2	2.0	86.3	ND		3.1		3.6	3/19/85
EN	82055	1	1	2.0	48.6		9.6		20		3/22/85
EN	82056	1	2	21.6	75.5		13		17		3/14/85
EN	82057	1	3	19.8	80.6	NA					
EN	82058	2	1	2.0	69.3		460 a		660 a		3/19/85
EN	82059	2	2	21.4	83.2	ND		12		14	3/13/85
EN	82060	2	3	26.2	83.2	ND		17		20	11/1/84
EN	82061	3	2	2.0	83.2	ND		1.7		2.0	3/20/85
EN	82062	3	3	23.2	85.3	ND		6.5		7.6	12/5/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NA = NOT ANALYZED--SUFFICIENT SAMPLE EXTRACT NOT AVAILABLE FOR ANALYSIS
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 11. DATA SUMMARY FOR 2,3,4,7,8-PENTACHLORODIBENZOFURAN [57117-31-4] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	23.0	77.5		26		34		3/15/85
MA	82050	1	2	2.0	80.7		28		35		3/22/85
MA	82051	1	3	16.2	82.5		72		87		10/30/84
MA	82052	2	1	2.0	84.7		77		90		3/19/85
MA	82053	2	2	2.0	78.2		29		37		3/19/85
MA	82054	2	3	18.0	83.9		24		29		10/31/84
MO	82036	1	1	1.0	62.3	TR	20	14	32	22	3/20/85
MO	82037	1	2	18.3	74.1		32		43		3/15/85
MO	82041	1	3	21.0	84.3		22		26		10/26/84
NE	82042	1	1	19.1	55.1	ND		5.3		9.6	3/18/85
NE	82043	1	2	21.9	87.6		34		38		3/18/85
NE	82044	1	3	26.7	79.2	NA					
PA	82046	1	1	19.7	62.9		18		28		3/15/85
PA	82047	1	2	2.0	68.0		40		59		3/20/85
PA	82048	1	3	22.0	87.1		18		21		10/31/84
WN	82063	1	1	23.4	63.1		7.7		12		3/13/85
WN	82064	1	2	20.6	76.2		48		63		3/11/85
WN	82065	1	3	22.5	89.8		11		12		10/30/84
WN	82066	2	3	21.4	80.4		18		22		10/30/84
SA	82067	1	1	20.7	83.9		55		66		3/18/85
SA	82068	1	2	26.4	86.4		35		40		3/15/85
SA	82069	1	3	20.0	82.5		11		13		10/31/84
SA	82070	2	1	2.0	69.1		6.4		9.3		3/13/85
SA	82071	2	2	19.5	89.3	TR	43	11	48	12	3/19/85
SA	82072	2	3	26.1	83.9	NA					
SA	82073	3	2	17.9	70.4	TR	40	22	57	31	3/20/85
SA	82074	3	3	18.0	86.7		22		25		10/31/85
SA	82075	4	2	2.0	72.0	ND		18		25	3/20/85
SA	82076	4	3	17.6	70.5	ND		6.7		9.5	10/31/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NA = NOT ANALYZED--SUFFICIENT SAMPLE EXTRACT NOT AVAILABLE FOR ANALYSIS
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 11. DATA SUMMARY FOR 2,3,4,7,8-PENTACHLORODIBENZOFURAN [57117-31-4] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	28.1	77.2		25		32		3/14/85
ES	82078	1	2	19.9	83.2		26		31		3/20/85
ES	82079	1	3	20.7	88.4		15		17		10/31/84
ES	82080	2	2	25.7	87.5		34		38		3/11/85
ES	82081	2	3	21.1	78.2	ND		2.0		2.6	10/31/84
WS	82082	1	1	11.1	67.6		8.5		13		3/18/85
WS	82083	1	2	22.7	86.7		26		30		3/18/85
WS	82084	1	3	22.4	94.6		2.2		2.3		10/26/84
WS	82085	2	2	2.0	86.3	ND		40		46	3/19/85
EN	82055	1	1	2.0	48.6		12		25		3/22/85
EN	82056	1	2	21.6	75.5	TR	1.8 [a]	1.0	2.4 [a]	1.3	3/14/85
EN	82057	1	3	19.8	80.6	NA					
EN	82058	2	1	2.0	69.3		25		36		3/19/85
EN	82059	2	2	21.4	83.2		56		68		3/13/85
EN	82060	2	3	26.2	83.2		17		20		11/1/84
EN	82061	3	2	2.0	83.2		33		40		3/20/85
EN	82062	3	3	23.2	85.3		47		55		12/5/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NA = NOT ANALYZED--SUFFICIENT SAMPLE EXTRACT NOT AVAILABLE FOR ANALYSIS
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 12. DATA SUMMARY FOR HEXACHLORODIBENZOFURAN [55684-94-1] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	No. of PEAKS	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	2.0	77.5	0	ND		11		14	3/22/85
MA	82050	1	2	2.0	80.7	2		13		16		3/22/85
MA	82051	1	3	1.6	82.5	1		35		42		11/7/84
MA	82052	2	1	2.0	84.7	2		8.5		10		3/19/85
MA	82053	2	2	2.0	78.2	2		18		23		3/19/85
MA	82054	2	3	2.0	83.9	1		23		27		11/7/84
MO	82036	1	1	1.0	62.3	0	ND		32		51	3/20/85
MO	82037	1	2	2.0	74.1	2	TR	12	6.3	16	8.5	3/22/85
MO	82041	1	3	2.0	84.3	1		15		18		11/8/84
NE	82042	1	1	19.1	55.1	0	ND		4.6		8.3	5/1/85
NE	82043	1	2	2.0	87.6	0	ND		6.6		7.5	3/22/85
NE	82044	1	3	2.0	79.2	1		21		27		11/8/84
PA	82046	1	1	2.0	62.9	0	ND		4.8		7.6	3/21/85
PA	82047	1	2	2.0	68.0	2		27		39		3/20/85
PA	82048	1	3	2.0	87.1	0	ND		10		11	11/6/84
WN	82063	1	1	2.0	63.1	0	ND		19		30	4/29/85
WN	82064	1	2	2.0	76.2	0	ND		7.9		10	3/11/85
WN	82065	1	3	2.0	89.8	2		16		18		11/6/84
WN	82066	2	3	2.0	80.4	1		21		26		11/8/84
SA	82067	1	1	2.0	83.9	2		51[a]		60 [a]		3/21/85
SA	82068	1	2	2.1	86.4	2		11		12		3/22/85
SA	82069	1	3	2.0	82.5	1		10		12		11/6/84
SA	82070	2	1	2.0	69.1	0	ND		9.5		14	3/21/84
SA	82071	2	2	2.0	89.3	2	TR	22	9.1	24	10	3/19/85
SA	82072	2	3	2.0	83.9	1		22		26		11/8/84
SA	82073	3	2	2.0	70.4	0	ND		12		17	3/20/85
SA	82074	3	3	2.0	86.7	1		8.5		9.8		11/7/84
SA	82075	4	2	2.0	72.0	0	ND		29		40	3/20/85
SA	82076	4	3	2.0	70.5	1		17		23		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NC = NOT CALCULATED--TWO ISOMERS OF HxCDF WERE DETECTED IN THIS SAMPLE BUT WERE NOT CALCULATED DUE TO THE LOW RECOVERY OF THE INTERNAL STANDARD, 13C12-OCDD.
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 12. DATA SUMMARY FOR HEXACHLORODIBENZOFURAN [55684-94-1] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	No. of PEAKS	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	2.0	77.2	2		17		22		3/21/85
ES	82078	1	2	2.0	83.2	2		25		30		3/20/85
ES	82079	1	3	2.0	88.4	1		3.7		4.2		11/8/84
ES	82080	2	2	2.0	87.5	2	TR	28	20	32	23	3/21/85
ES	82081	2	3	2.0	78.2	1		14		18		11/8/84
WS	82082	1	1	1.0	67.6	1	TR	5.8	2.0	8.6	3.0	4/11/85
WS	82083	1	2	22.7	86.7	2	NC					3/18/85
WS	82084	1	3	2.0	94.6	1		12		13		11/6/84
WS	82085	2	2	2.0	86.3	0	ND		16		19	3/19/85
EN	82055	1	1	2.0	48.6	0	ND		11		23	3/22/85
EN	82056	1	2	2.0	75.5	2	TR	19	11	25	15	3/21/85
EN	82057	1	3	2.0	80.6	1		24		30		11/8/84
EN	82058	2	1	2.0	69.3	1	TR	11	6.9	16	10	3/19/84
EN	82059	2	2	2.0	83.2	2		29		35		3/21/85
EN	82060	2	3	2.0	83.2	1		9.5		11		11/8/84
EN	82061	3	2	2.0	83.2	2		29		35		3/20/85
EN	82062	3	3	2.0	85.3	1		23		27		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NC = NOT CALCULATED--TWO ISOMERS OF HxCDF WERE DETECTED IN THIS SAMPLE BUT WERE NOT CALCULATED DUE TO THE LOW RECOVERY OF THE INTERNAL STANDARD, 13C12-OCDD.
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 13. DATA SUMMARY FOR 1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN [67562-39-4] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	2.0	77.5		6.5		8.4		3/22/85
MA	82050	1	2	2.0	80.7		15		18		3/22/85
MA	82051	1	3	1.6	82.5		26		32		11/7/84
MA	82052	2	1	2.0	84.7		6.2		7.3		3/19/85
MA	82053	2	2	2.0	78.2		9.7		12		3/19/85
MA	82054	2	3	2.0	83.9		23		27		11/7/84
MO	82036	1	1	1.0	62.3	TR	8.6	5.0	14	8.0	3/20/85
MO	82037	1	2	2.0	74.1		9.0		12		3/22/85
MO	82041	1	3	2.0	84.3		8.5		10		11/8/84
NE	82042	1	1	19.1	55.1	ND		4.6		8.3	
NE	82043	1	2	2.0	87.6		7.0		8.0		3/22/85
NE	82044	1	3	2.0	79.2		31		39		11/8/84
PA	82046	1	1	2.0	62.9	TR	8.0	2.2	13	3.5	3/21/85
PA	82047	1	2	2.0	68.0		11		17		3/20/85
PA	82048	1	3	2.0	87.1	ND		6.0		6.9	11/6/84
WN	82063	1	1	2.0	63.1	TR	11	3.4	17	5.4	4/29/85
WN	82064	1	2	2.0	76.2		3.0		3.9		3/11/85
WN	82065	1	3	2.0	89.8		17		18		11/6/84
WN	82066	2	3	2.0	80.4		15		18		11/8/84
SA	82067	1	1	2.0	83.9		55		65		3/21/85
SA	82068	1	2	2.1	86.4		11		13		3/22/85
SA	82069	1	3	2.0	82.5		11		13		11/6/84
SA	82070	2	1	2.0	69.1		9.7		14		3/21/84
SA	82071	2	2	2.0	89.3		11		12		3/19/85
SA	82072	2	3	2.0	83.9	ND	10		12		11/8/84
SA	82073	3	2	2.0	70.4		15		21		3/20/85
SA	82074	3	3	2.0	86.7		21		24		11/7/84
SA	82075	4	2	2.0	72.0		15		20		3/20/85
SA	82076	4	3	2.0	70.5		14		19		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NC = NOT CALCULATED THE 1,2,3,4,6,7,8-HpCDF WAS DETECTED IN THIS SAMPLE BUT WAS NOT CALCULATED DUE TO THE LOW RECOVERY OF THE INTERNAL STANDARD, 13C12-OCDD.
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 13. DATA SUMMARY FOR 1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN [67562-39-4] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	2.0	77.2		16		20		3/21/85
ES	82078	1	2	2.0	83.2		27		32		3/20/85
ES	82079	1	3	2.0	88.4		7.5		8.5		11/8/84
ES	82080	2	2	2.0	87.5	TR	25	17	28	19	3/21/85
ES	82081	2	3	2.0	78.2		23		29		11/8/84
WS	82082	1	1	1.0	67.6		6.4		9.5		4/11/85
WS	82083	1	2	22.7	86.7	NC					3/18/85
WS	82084	1	3	2.0	94.6		10		11		11/6/84
WS	82085	2	2	2.0	86.3		12		14		3/19/85
EN	82055	1	1	2.0	48.6		4.4		9.1		3/22/85
EN	82056	1	2	2.0	75.5		16		21		3/21/85
EN	82057	1	3	2.0	80.6		30		37		11/8/84
EN	82058	2	1	2.0	69.3		55		79		3/19/84
EN	82059	2	2	2.0	83.2		29		35		3/21/85
EN	82060	2	3	2.0	83.2		9.5		11		11/8/84
EN	82061	3	2	2.0	83.2		43		51		3/20/85
EN	82062	3	3	2.0	85.3		14		16		11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NC = NOT CALCULATED THE 1,2,3,4,6,7,8-HpCDF WAS DETECTED IN THIS SAMPLE BUT WAS NOT CALCULATED DUE TO THE LOW RECOVERY OF THE INTERNAL STANDARD, 13C12-OCDD.
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 14. DATA SUMMARY FOR OCTACHLORODIBENZOFURAN [39001-02-01] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
MA	82049	1	1	2.0	77.5		7.9		10		3/22/85
MA	82050	1	2	2.0	80.7		270[a]		330[a]		3/22/85
MA	82051	1	3	1.6	82.5		240		290		11/7/84
MA	82052	2	1	2.0	84.7	ND		6.5		7.7	3/19/85
MA	82053	2	2	2.0	78.2	ND		1.0		1.3	3/19/85
MA	82054	2	3	2.0	83.9	ND		5.0		6.0	11/7/84
MO	82036	1	1	1.0	62.3	TR	6.6	2.0	11	3.2	3/20/85
MO	82037	1	2	2.0	74.1	ND		2.0		2.7	3/22/85
MO	82041	1	3	2.0	84.3	ND		38		45	11/8/84
NE	82042	1	1	19.1	55.1	ND		3.5		6.4	5/1/85
NE	82043	1	2	2.0	87.6	TR	6.8	23	7.8	26	3/22/85
NE	82044	1	3	2.0	79.2		360		450		11/8/84
PA	82046	1	1	2.0	62.9		13		21		3/21/85
PA	82047	1	2	2.0	68.0	TR	3.0	1.0	4.4	1.5	3/20/85
PA	82048	1	3	2.0	87.1	ND		29		33	11/6/84
WN	82063	1	1	2.0	63.1	ND		23		36	4/29/85
WN	82064	1	2	2.0	76.2	ND		2.0		2.6	3/11/85
WN	82065	1	3	2.0	89.8		250		280		11/6/84
WN	82066	2	3	2.0	80.4	ND		21		26	11/8/84
SA	82067	1	1	2.0	83.9	ND		4.0		4.8	3/21/85
SA	82068	1	2	2.1	86.4		5.0		5.8		3/22/85
SA	82069	1	3	2.0	82.5	ND		34		41	11/6/84
SA	82070	2	1	2.0	69.1	ND		6.0		8.7	3/21/84
SA	82071	2	2	2.0	89.3		10		11		3/19/85
SA	82072	2	3	2.0	83.9	ND		8.0		9.5	11/8/84
SA	82073	3	2	2.0	70.4	ND		3.5		5.0	3/20/85
SA	82074	3	3	2.0	86.7	ND		11		13	11/7/84
SA	82075	4	2	2.0	72.0	ND		6.0		8.3	3/20/85
SA	82076	4	3	2.0	70.5	ND		140		200	11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NC = NOT CALCULATED OCDF WAS NOT DETECTED IN THIS SAMPLE. A DETECTION LIMIT WAS NOT CALCULATED DUE TO THE LOW RECOVERY OF THE INTERNAL STANDARD, 13C12-OCDD
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

TABLE 14. DATA SUMMARY FOR OCTACHLORODIBENZOFURAN [39001-02-01] - FY82 COMPOSITE ADIPOSE TISSUE SAMPLES

Census	MRI NO	Comp No	AGE	Tissue Weight g	% LIPID	Analysis Code	Wet Weight Conc. (PG/G)	Wet Weight LOD (PG/G)	Lipid Weight Conc. (PG/G)	Lipid Weight LOD (PG/G)	Date Analyzed
ES	82077	1	1	2.0	77.2	TR	3.0	1.0	3.9	1.3	3/21/85
ES	82078	1	2	2.0	83.2	TR	3.0	1.0	3.6	1.2	3/20/85
ES	82079	1	3	2.0	88.4	ND		16		18	11/8/84
ES	82080	2	2	2.0	87.5	ND		4.0		4.6	3/21/85
ES	82081	2	3	2.0	78.2	ND		5.0		6.4	11/8/84
WS	82082	1	1	1.0	67.6	ND		4.2		6.2	4/11/85
WS	82083	1	2	22.7	86.7	NC					3/18/85
WS	82084	1	3	2.0	94.6	ND		34		36	11/6/84
WS	82085	2	2	2.0	86.3	ND		8.5		9.8	3/19/85
EN	82055	1	1	2.0	48.6	TR	9.4		19		3/22/85
EN	82056	1	2	2.0	75.5	TR	6.5	2.5	8.6	3.3	3/21/85
EN	82057	1	3	2.0	80.6	ND		22		27	11/8/84
EN	82058	2	1	2.0	69.3		620 a		890 a		3/19/84
EN	82059	2	2	2.0	83.2		22		26		3/21/85
EN	82060	2	3	2.0	83.2	ND		5.0		6.0	11/8/84
EN	82061	3	2	2.0	83.2		15		18		3/20/85
EN	82062	3	3	2.0	85.3	ND		15		18	11/8/84

AGE GROUP 1 = 0-14; AGE GROUP 2 = 15-44 YEARS;
AGE GROUP 3 = 45 PLUS YEARS

ND = NOT DETECTED
NC = NOT CALCULATED OCDF WAS NOT DETECTED IN THIS SAMPLE. A DETECTION LIMIT WAS NOT CALCULATED DUE TO THE LOW RECOVERY OF THE INTERNAL STANDARD, 13C12-OCDD
TR = TRACE RESPONSE > 2.5 BUT < 10 TIMES S/N

a Value determined to be an outlier for the respective age group based on Q-test.

The composite number reflects that more than one composite was analyzed for a specific age group within a specific census division.

Table 15. Wet Tissue Weight Concentration of PCDDs and PCDFs in the NHATS FY82 Composite Specimens

Compound	Frequency of detection (%)[a]	Mean concentration[b] (pg/g)	Range of detection (pg/g)
2,3,7,8-TCDD	76	5.0 ± 2.7	ND - 12
1,2,3,7,8-PeCDD	91	33.5 ± 37.4	ND - 4,300
HxCDD[c]	98	69.4 ± 68.5	ND - 500
1,2,3,4,7,8,9-HpCDD	98	81.9 ± 77.8	ND - 900
OCDD	100	554 ± 291	10 - 2,950
2,3,7,8-TCDF	26	10.1 ± 9.2	ND - 460
2,3,4,7,8-PeCDF	89	28.7 ± 17.1	ND - 77
HxCDF[b]	72	19.1 ± 9.5	ND - 51
1,2,3,4,6,7,8-HpCDF	93	16.5 ± 11.8	ND - 55
OCDF	39	60.1 ± 110	ND - 620

[a]Percentage of composites in which the compound(s) was detected.
[b]Mean concentration (± one standard deviation) calculated using trace and positive quantifiable values.
[c]Reference compounds not available for specific isomers.

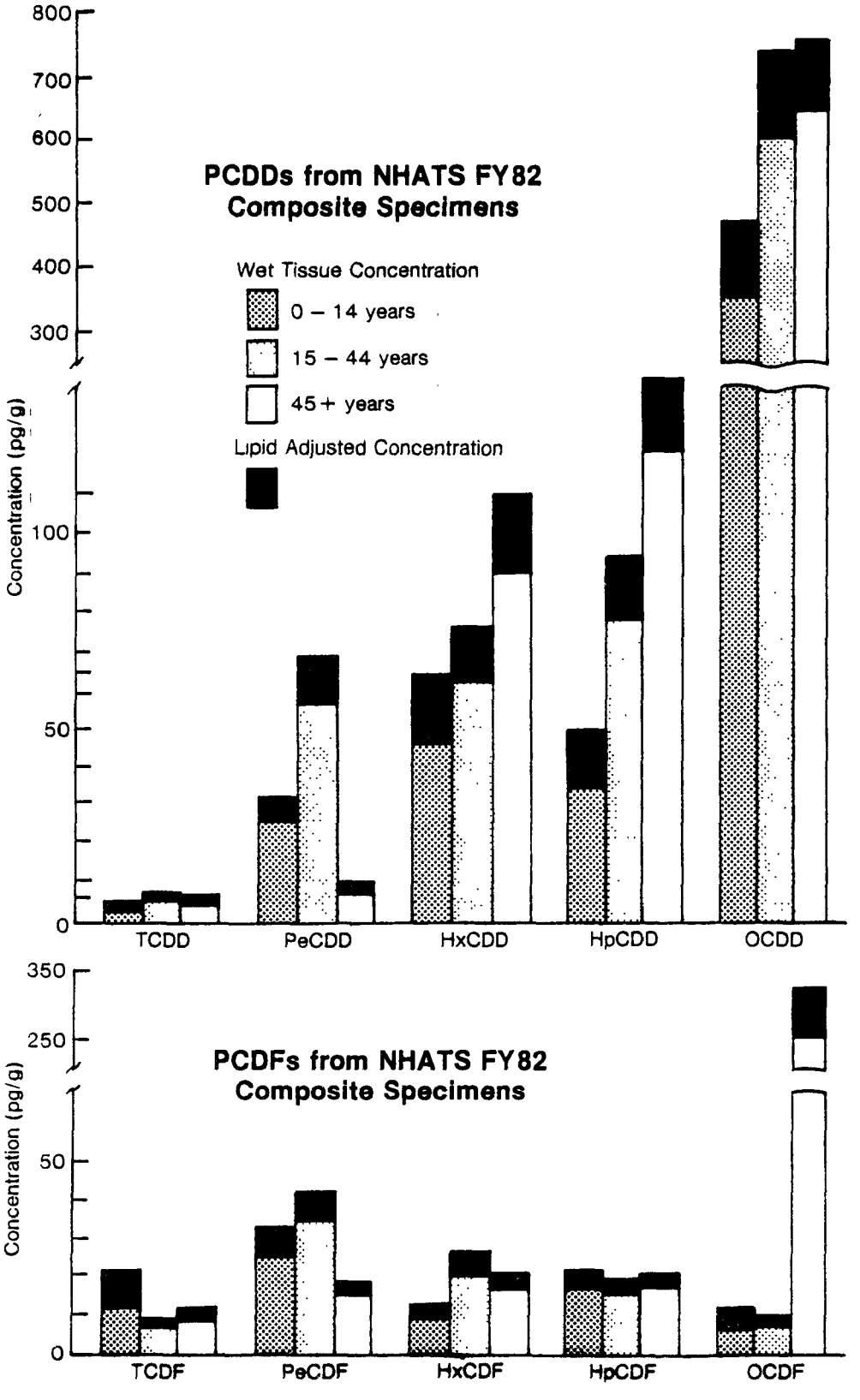


Figure 7. PCDD and PCDF distribution in the general U.S. population by age group.

The data used to prepare the illustration in Figure 7 were also used to calculate an average concentration across all age groups. Tables 15 and 16 provide these average concentrations based on wet weight and lipid bases, respectively. These tables indicate the number of samples for which positive responses were noted as well as the range of concentrations observed.

Figure 8 graphically demonstrates the average PCDD and PCDF data (based on wet tissue weight) for the FY82 NHATS composite samples as compared to the data reported for Swedish (Nygren et al. 1985; Rappe et al. 1985) and upstate New York human adipose tissue samples. (Schecter et al. 1985; Schecter, Ryan 1984) The profiles observed from each of these studies are very similar with the exception of the OCDF response in the NHATS specimens. This increase in the OCDF average value, as explained previously, is the result of high concentrations observed for a minimum number of samples observed as positive values. These data reported for the FY82 NHATS composite specimens also are consistent with the general trends reported for human adipose samples collected in Canada (Ryan et al. 1985a; Ryan et al. 1985b; Ryan 1985) and other specific regions within the United States. (Graham et al 1985; Peterson et al. 1985)

V. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

As discussed in the experimental section of this report, the analysis of the composite samples was completed with various QA/QC efforts. These included the analysis of method blanks, verification of column resolution for 2,3,7,8-TCDD from other TCDD isomers, daily verification of response factors and method sensitivity, estimation of the absolute recovery of the internal standards, and verification of 2,3,7,8-TCDD using fragment ions and high resolution MS.

A. Method Blanks

Method blanks were handled exactly as samples and were analyzed along with the actual composite sample extracts. The PCDD and PCDF congeners were not detected in the method blanks. The analysis of these method blanks documented that the response to the PCDD and PCDF congeners noted in sample extracts were, in fact, due to the endogenous levels in the composited specimens from the FY82 NHATS repository.

B. Column Resolution

The resolution of the 60-m DB-5 column for separation of 2,3,7,8-TCDD from the other TCDD isomers, specifically the 1,4,7,8-, 1,2,3,4-, 1,2,3,7-, and 1,2,3,8- isomers, was verified on a daily basis. Figure 9 is an example of a daily column resolution check using the DB-5 column. Resolution is defined as the height of the valley (x) between the 2,3,7,8-TCDD isomer and the closest eluting TCDD isomers divided by the height of the 2,3,7,8-TCDD response (y) times 100%. As noted in this figure, the analysis of the isomer mixture demonstrated that the column achieved a resolution of 29% using the 322 response from the other TCDD isomers. The column performance varied up to 50% through the analysis of these samples but was considered acceptable.

Table 16. Lipid-Adjusted Concentration of PCDDs and PCDFs in the NHATS FY82 Composite Specimens

Compound	Frequency of detection (%)[a]	Mean concentration[b] (pg/g)	Range of detection (pg/g)
2,3,7,8-TCDD	76	6.2 ± 3.3	ND - 14.2
1,2,3,7,8-PeCDD	91	43.5 ± 46.5	ND - 5,000
HxCDD[c]	98	86.9 ± 83.8	ND - 620
1,2,3,4,7,8,9-HpCDD	98	102 ± 93.5	ND - 1,300
OCDD	100	694 ± 355	19 - 3,700
2,3,7,8-TCDF	26	15.6 ± 16.5	ND - 660
2,3,4,7,8-PeCDF	89	36.1 ± 20.4	ND - 90
HxCDF[b]	72	23.5 ± 11.6	ND - 60
1,2,3,4,6,7,8-HpCDF	93	20.9 ± 15.0	ND - 79
OCDF	39	73.4 ± 134	ND - 890

[a]Percentage of composites in which the compound(s) was detected.
[b]Mean concentration (± one standard deviation) calculated using trace and positive quantifiable values.
[c]Reference compounds not available for specific isomers.

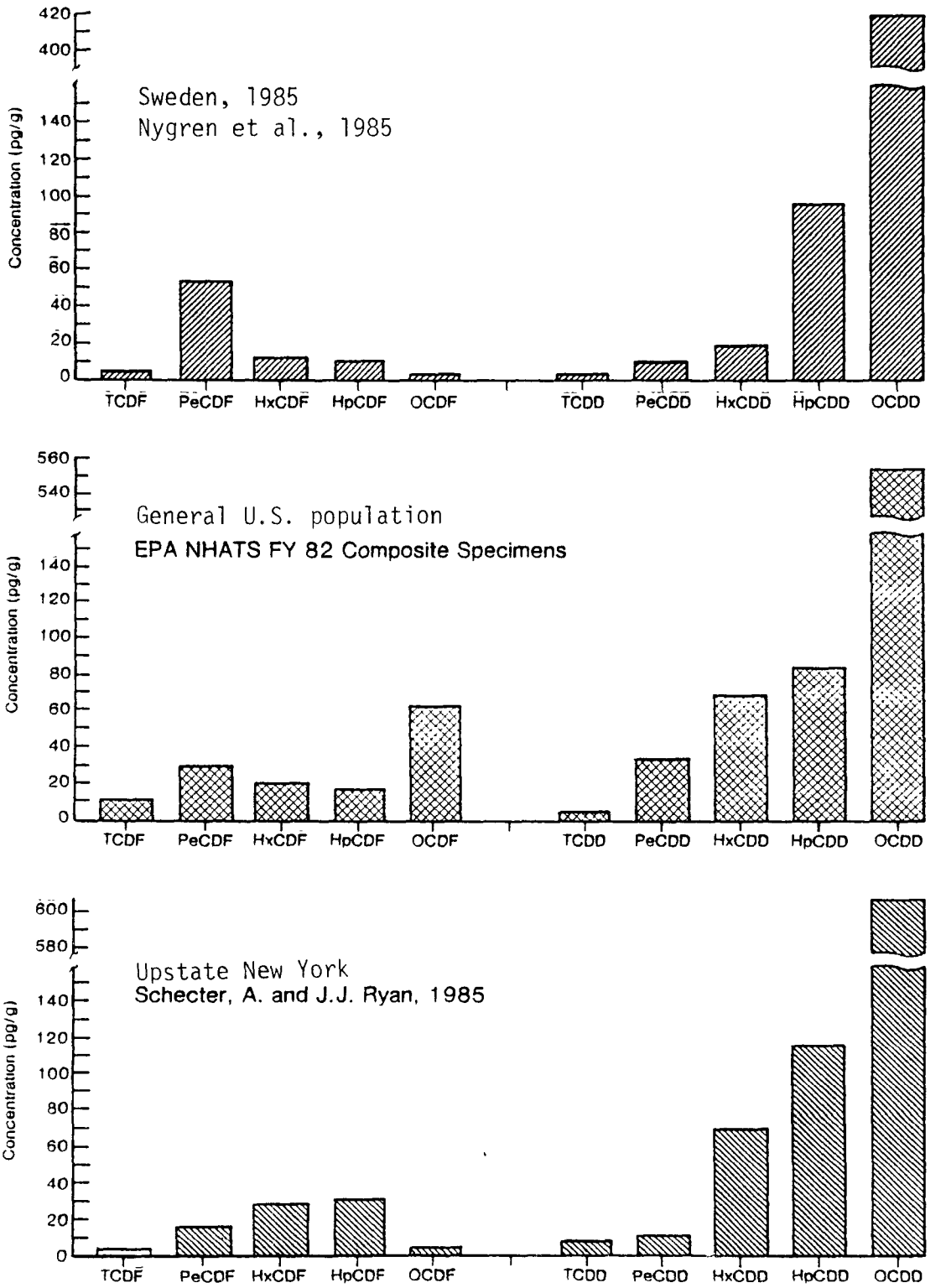


Figure 8. Comparison of PCDD and PCDF concentration (based on wet tissue weight) profiles for Sweden, the general U.S. population, and upstate New York.

(Source: Schecter, Ryan 1985; Nygren et al. 1985.)

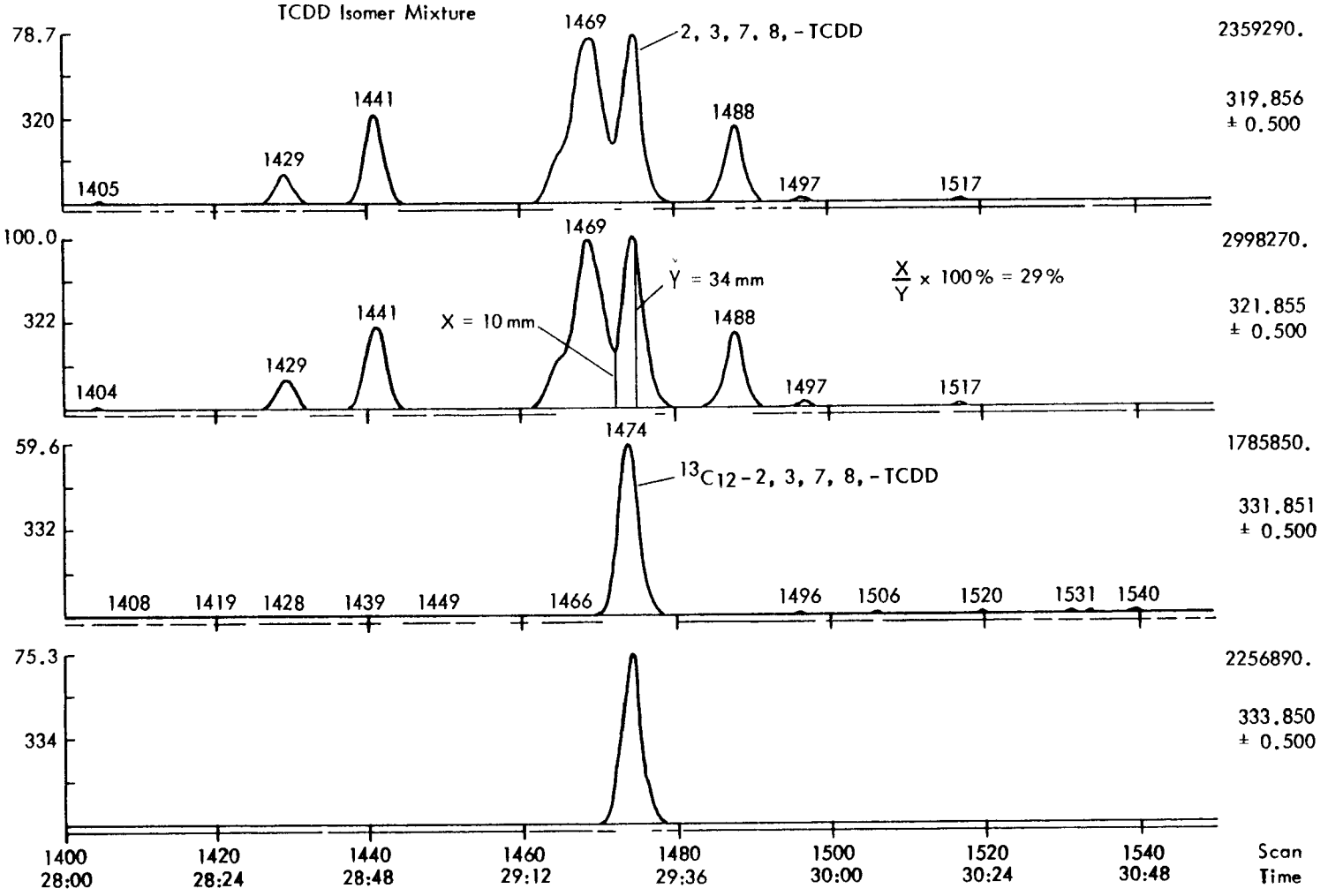


Figure 9. Selected ion current profile from the analysis of the TCDD column performance mixture on a 60 m DB-5 column.

However, it is recommended that future studies focusing on the analysis of 2,3,7,8-TCDD in human adipose tissue require a minimum column resolution of 25% to maintain conformance with other protocols developed during the course of this study. (USEPA 1983) This will require a specific analysis for 2,3,7,8-TCDD using a polar column such as 60 m SP2330, SP2340, or a 50 m CP-Sil 88.

C. Instrument Performance

The instrument sensitivity and consistency of response factors were documented through the routine analysis of calibration standards. Figure 10 provides a plot of the response factors for 2,3,7,8-TCDD and 2,3,7,8-TCDF over the course of the analysis of the 0-14 and 15-44 age categories. The target limits on the variability of the average relative response factors were ± 20% for TCDD and TCDF and ± 30% for the penta- through octachloro-PCDD and PCDF.

Tables 17 and 18 present the summaries of the actual response factors measured for calibration standards ranging from 1 to 100 pg/μL for 2,3,7,8-TCDD and 2,3,7,8-TCDF. The data represent the calibration summaries completed during the analysis of the 0-14 and 15-44 age group composites. The average relative response factor (RRF) was updated daily, and this mean value was used to calculate the residue levels in the composite samples on each analysis day. The composites representing the 45-plus age group were analyzed several months before the 0-14 and 15-44 age groups. Hence, a calibration curve for each analyte was prepared at the start of each analysis.

Table 19 presents a summary of the daily calibration data that was generated during the analysis of the 0-14 and 15-44 age groups. This table includes the calibration range, the average RRF, and the observed variability for each of the PCDD and PCDF congeners included in the calibration standards. The ratio of the average RRF values for 2,3,7,8-TCDF/2,3,7,8-TCDD is 1.7 compared to 1.4 for the ratio of the average RRF values for OCDF/OCDD. This indicates that the actual RRF values for the penta- through heptachlorodibenzofuran congeners might be expected to be somewhat greater than measured for the dibenzo-p-dioxin congeners with the same degrees of chlorination. Thus, the true concentrations of the PCDF congeners may be less than reported in Tables 1-13. This potential difference in the relative response factors reflects the need to develop analytical standards representative of each PCDD and PCDF congener group in future programs.

D. Internal Standard Recovery

The recovery of the internal quantitation standards ($^{13}C_{12}$-2,3,7,8-TCDD and $^{13}C_{12}$-OCDD) were measured using two additional internal recovery standards, ($^{13}C_{12}$-2,3,7,8-TCDF and $^{37}Cl_4$-1,2,3,4,6,7,8-HpCDD). These recovery standards were added just prior to the HRGC/MS analysis. These compounds were also included in the calibration standards to establish the necessary response ratios. The average recovery of the $^{13}C_{12}$-2,3,7,8-TCDD was determined to be 86 ± 26% for extracts taken through the Amoco PX-21/glass fiber column. As noted previously, the $^{13}C_{12}$-OCDD was not detected in these extracts due to its retention on Florisil that was used as part of the broad scan analysis preparation scheme. The $^{13}C_{12}$-TCDD and $^{13}C_{12}$-OCDD recoveries averaged 50 ± 17% and 90 ± 50%, respectively, for the composite extracts eluted through the Carbopak C/Celite column.

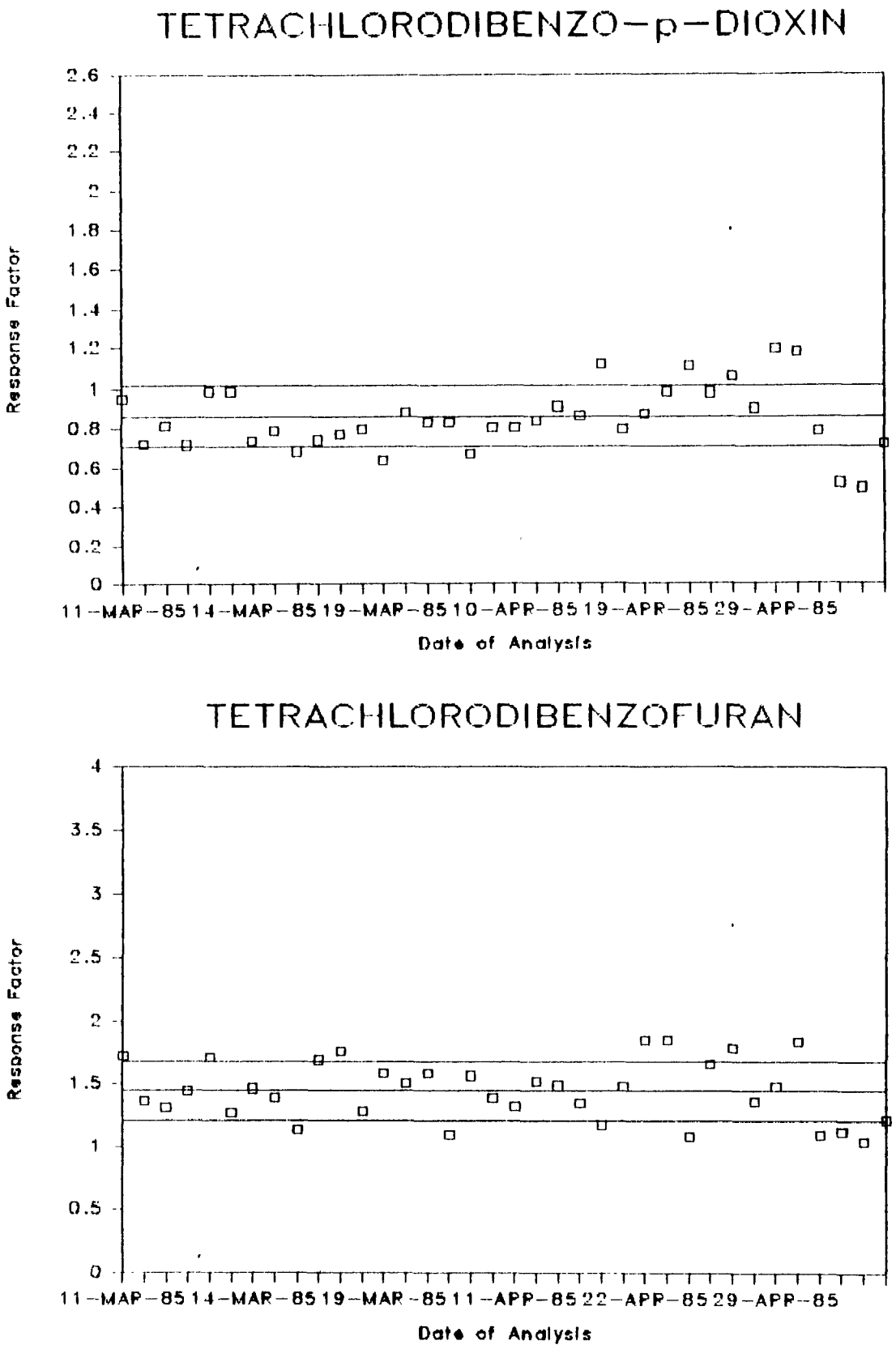


Figure 10. Examples of response factor summary control charts for 2,3,7,8-tetrachlorodibenzo-p-dioxin and 2,3,7,8-tetrachlorodibenzofuran.

Table 17. Relative Response Factor (RRF) Summary for TCDD Versus $^{13}C_{12}$-TCDD

Date	RRF	Concentration (ng/μL)
11-March-1985	0.953	0.100
11-March-1985	0.724	0.010
11-March-1985	0.816	0.001
Average RRF	0.831	
Standard Deviation	0.094	
12-March-1985	0.717	0.010
13-March-1985	0.987	0.010
13-March-1985	0.987	0.010
14-March-1985	0.736	0.010
15-March-1985	0.787	0.010
15-March-1985	0.684	0.001
18-March-1985	0.776	0.010
18-March-1985	0.799	0.001
19-March-1985	0.640	0.001
19-March-1985	0.881	0.010
20-March-1985	0.830	0.010
21-March-1985	0.832	0.010
21-March-1985	0.670	0.001
22-March-1985	0.809	0.010
10-April-1985	0.805	0.010
11-April-1985	0.845	0.010
18-April-1985	0.913	0.001
18-April-1985	0.869	0.010
19-April-1985	1.119	0.001
19-April-1985	0.799	0.001
19-April-1985	0.876	0.010
22-April-1985	0.983	0.100
24-April-1985	1.114	0.010
25-April-1985	0.983	0.100
25-April-1985	1.059	0.010
25-April-1985	0.904	0.100
29-April-1985	1.199	0.001
29-April-1985	1.180	0.010
30-April-1985	0.790	0.001
30-April-1985	0.521	0.001
01-May-1985	0.496	0.001
01-May-1985	0.724	0.010
Average RRF	0.850	
Standard Deviation	0.168	

Table 18. Relative Response Factor (RRF) Summary for TCDF Versus $^{13}C_{12}$-TCDD

Date	RRF	Concentration (ng/μL)
11-March-1985	1.726	0.100
11-March-1985	1.356	0.010
11-March-1985	1.307	0.001
Average RRF	1.463	
Standard Deviation	0.187	
12-March-1985	1.434	0.010
13-March-1985	1.708	0.010
13-March-1985	1.262	0.010
14-March-1985	1.456	0.010
15-March-1985	1.383	0.010
15-March-1985	1.135	0.001
18-March-1985	1.692	0.010
18-March-1985	1.768	0.010
18-March-1985	1.280	0.001
19-March-1985	1.580	0.010
20-March-1985	1.508	0.010
21-March-1985	1.583	0.010
21-March-1985	1.100	0.001
22-March-1985	1.561	0.010
10-April-1985	1.386	0.010
11-April-1985	1.316	0.010
18-April-1985	1.513	0.001
18-April-1985	1.484	0.010
19-April-1985	1.347	0.001
19-April-1985	1.179	0.001
19-April-1985	1.476	0.010
22-April-1985	1.853	0.100
24-April-1985	1.860	0.010
25-April-1985	1.082	0.001
25-April-1985	1.656	0.100
25-April-1985	1.794	0.010
25-April-1985	1.361	0.100
29-April-1985	1.476	0.001
29-April-1985	1.846	0.010
30-April-1985	1.098	0.001
30-April-1985	1.128	0.001
01-May-1985	1.043	0.001
01-May-1985	1.215	0.010
Average RRF	1.442	
Standard Deviation	0.249	

Table 19. Relative Response Factor (RRF) Summary for PCDD and PCDF Calibration Standards[a]

Compound	Calibration range (pg/μL)	RRF[b]	
		Average	Standard deviation
2,3,7,8-TCDD	1-100	0.850	0.168
2,3,7,8-TCDF	1-100	1.442	0.249
1,2,3,7,8-PeCDD	1-100	0.259	0.071
1,2,3,4,7,8-HxCDD	5-500	1.953	0.603
1,2,3,4,6,7,8-HpCDD	5-500	1.410	0.340
OCDD	10-1,000	0.656	0.164
OCDF	10-1,000	0.927	0.225

[a]These data represent a summary of calibration events completed from March 11, 1985 through May 1, 1985.
[b]The RRF values reported are a compilation of all calibration standard analyses rather than a summary of the initial calibration curve data.

E. Confirmation of 2,3,7,8-TCDD

Qualitative confirmation of 2,3,7,8-TCDD in selected extracts was achieved using the following criteria: (1) retention time of the characteristic mass (m/z 320 and 322) corresponding to the 2,3,7,8-TCDD, (2) proper response ratios of m/z 320/322 (0.67 to 0.90), (3) response of fragment ions corresponding to a loss of COCl (m/z 257 and 259), and (4) HRMS (R = 10,000) analysis using the characteristic ions 319.897 and 321.894.

A second chromatographic peak was noted for several of the composite sample analyses that eluted 8 to 10 scans earlier than the 2,3,7,8-TCDD. This peak exhibited response within the acceptable ion ratios for m/z 320 and m/z 322 for the low resolution MS analyses. This peak also exhibited response to these ions for the high resolution MS analyses and the fragment ion corresponding to loss of COCl in a separate analysis. However, this second peak could not be confirmed as a TCDD isomer as a result of a coincidental response noted at m/z 358. Further evaluation of this response is necessary before an identification can be established. This response has not been noted in other studies dealing with PCDD and PCDF in human adipose tissue. This response may be due to some interferent that might be effectively removed by fractionating the sample extract on alumina prior to MS analysis.

VI. REFERENCES

Graham M, Hileman FD, Wendling J, Wilson JD. Chlorocarbons in adipose tissue samples. 5th International Symposium on Chlorinated Dioxins and Related Compounds, Bayreuth, FRG, September 16-19, 1985.

Mack GA, Stanley JS. 1984. Preliminary strategy on the National Human Adipose Tissue Survey. Washington, DC: Office of Toxic Substances. Contracts 68-07-6221 (Task 21) and 68-02-3938 (Work Assignment 8). NHATS-ST-01.

Nygren M, Hansson M, Rappe C, Domellof L, Hardell L. 1985. Analysis of polychlorinated dibenzo-p-dioxins and dibenzofurans in adipose tissue from soft-tissue sarcoma patients and controls. 189th National ACS Meeting Symposium on Chlorinated Dioxins and Dibenzofurans in the Total Environment III, Miami, Florida, 1985. Preprint Division of Environmental Chemistry, ACS 25: 160-163, Paper No. 55.

Patterson DG, Holler JS, Smith SJ, Liddle JA, Sampson EJ, Needham LL. Human tissue data in certain U.S. populations. 5th International Symposium on Chlorinated Dioxins and Related Compounds, Bayreuth, FRG, September 16-19, 1985.

Rappe C, Nygren M, Lindström G, Hanson H. Dioxins and dibenzofurans in human tissues and milk of European origin. 5th International Symposium on Chlorinated Dioxins and Related Compounds, Bayreuth, FRG, September 16-19, 1985.

Ryan JJ. Variation of dioxins and furans in humans with age and organ by country. 5th International Symposium on Chlorinated Dioxins and Related Compounds, Bayreuth, FRG, September 16-19, 1985.

Ryan JJ, Williams DT, Lau BPY, Sakuma T. 1985a. Analysis of human fat tissue for 2,3,7,8-tetrachlorodibenzo-p-dioxin and chlorinated dibenzofuran residues in dioxins and dibenzofurans in the total environment II. Keith LH, Rappe C, Choudhary G, eds. Butterworth Publishers, pp. 205-214.

Ryan JJ, Schecter A, Lizotte R, Sun W-F, Miller L. 1985b. Tissue distribution of dioxins and furans in humans from the general population. Chemosphere 14: 929-932.

Schecter A, Ryan JJ. 1985. Dioxin and furan levels in human adipose tissue from exposed and control populations. 189th National ACS Meeting Symposium on Chlorinated Dioxins and Dibenzofurans in the Total Environment III, Miami, Florida. Preprint Division of Environmental Chemistry, ACS 25:160-163, Paper No. 56.

Schecter A, Tiernan TO, Taylor ML, VanNess GF, Garrett JH, Wagel DJ, Gitlitz G, and Bogdasarian M. 1985. Biological markers after exposure to polychlorinated dibenzo-p-dioxins, dibenzofurans, biphenyls, and biphenylenes. Part I: Findings using fat biopsies to estimate exposure in chlorinated dioxin and dibenzofurans in the total environment II. Keith LH, Rappe C, Choudhary, G, eds. Butterworth Publishers, pp. 215-246.

Smith LM, Stalling DL, Johnson JJ. 1984. Determination of part per trillion levels of polychlorinated dibenzofurans and dioxins in environmental samples. Anal Chem 56:1830-1842.

Stalling DL, Smith LM, Pethy JD. 1979. An expanded approach to the study and measurement of PCBs and selected planar halogenated aromatic environmental pollutants. Ann. N.Y. Acad. Sci. 320, 48-59.

Stanley JS. 1986a. Broad scan analysis of human adipose tissue: Volume I Executive summary. EPA 560/5-86-035.

Stanley JS. 1986b. Broad scan analysis of human adipose tissue: Volume II Volatile organic compounds. EPA 506/5-86-036.

Stanley JS. 1986c. Broad scan analysis of human adipose tissue: Volume III Semivolatile organic compounds. EPA 560/5-86-037.

Stanley JS. 1986d. Broad scan analysis of human adipose tissue: Volume IV Polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF). EPA 560/5-86-038.

Stanley JS, Stockton RA. 1986. Broad scan analysis of human adipose tissue: Volume V Trace elements. EPA 560/5-86-039.

USEPA. 1983. 2,3,7,8-Tetrachlorodibenzo-p-dioxin in soil and sediment by high resolution gas chromatography/low resolution mass spectrometry. Contract laboratory protocol. Solicitation WA-84-A002.

APPENDIX A

GLOSSARY OF TERMS

FY82	Fiscal year 1982
HpCDD	Heptachlorodibenzo-p-dioxin
HpCDF	Heptachlorodibenzofuran
HRGC	High resolution gas chromatography
HxCDD	Hexachlorodibenzo-p-dioxin
HxCDF	Hexachlorodibenzofuran
MS	Mass spectrometry
NHATS	National Human Adipose Tissue Survey
NHMP	National Human Monitoring Program
OCDD	Octachlorodibenzo-p-dioxin
OCDF	Octachlorodibenzofuran
OTS	Office of Toxic Substances
PCDD	Polychlorinated dibenzo-p-dioxin
PCDF	Polychlorinated dibenzofuran
PeCDD	Pentachlorodibenzo-p-dioxin
PeCDF	Pentachlorodibenzofuran
SIM	Selected ion monitoring
TCDD	Tetrachlorodibenzo-p-dioxin
TCDF	Tetrachlorodibenzofuran

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-560/5-86-038		

4. TITLE AND SUBTITLE	5. REPORT DATE
Broad Scan Analysis of Human Adipose Tissue Volume 4 - Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF)	December 1986
	6. PERFORMING ORGANIZATION CODE Midwest Research Institute
7. AUTHOR(S) John S. Stanley	8. PERFORMING ORGANIZATION REPORT NO. 8821-A(01)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Midwest Research Institute 425 Volker Boulevard Kansas City, Missouri, 64110	10. PROGRAM ELEMENT NO.
	11. CONTRACT/GRANT NO. 68-02-3938 68-02-4252
12. SPONSORING AGENCY NAME AND ADDRESS U.S. Environmental Protection Agency Office of Toxic Substances Field Studies Branch (TS-798) Design Development Branch/Exposure Evaluation Division 401 M Street, SW Washington, DC 20460	13. TYPE OF REPORT AND PERIOD COVERED Final
	14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES
J. Remmers and P. Robinson, Work Assignment Managers
J. Breen and C. Stroup, Program Managers

16. ABSTRACT

The U.S. EPA's Office of Toxic Substances (OTS) maintains a unique capability for monitoring human exposure to potential toxic substances through the National Human Adipose Tissue Survey (NHATS). The primary focus for NHATS has been to document trends in human exposure to environmentally persistent contaminants, specifically, organochlorine pesticides and polychlorinated biphenyls (PCBs).

EPA/OTS has recognized a need to expand the use of the NHATS program to provide a more comprehensive assessment of toxic substances that are accumulated in adipose tissues. This report deals specifically with the measurement of polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) in composited adipose tissue samples from the FY82 NHATS repository.

The results of this study demonstrate that the EPA NHATS program is an effective vehicle for documenting the exposure of the general U.S. population to PCDDs and PCDFs. The analysis of the 46 composite samples prepared from the fiscal year 1982 NHATS repository establishes the prevalence of the 2,3,7,8-substituted tetra- through octachloro-PCDD and PCDF congeners in the U.S. population. The PCDD and PCDF levels are comparable to data presented from other studies that focus on samples collected in upstate New York, Canada, and Sweden.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Human Adipose Tissue Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) HRGC/MS Selected ion monitoring Parts per trillion	Analysis Determination	

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS (This Report)	21. NO. OF PAGES
Release unlimited	Unclassified	65
	20. SECURITY CLASS (This page)	22. PRICE
	Unclassified	

EPA Form 2220-1 (9-73)