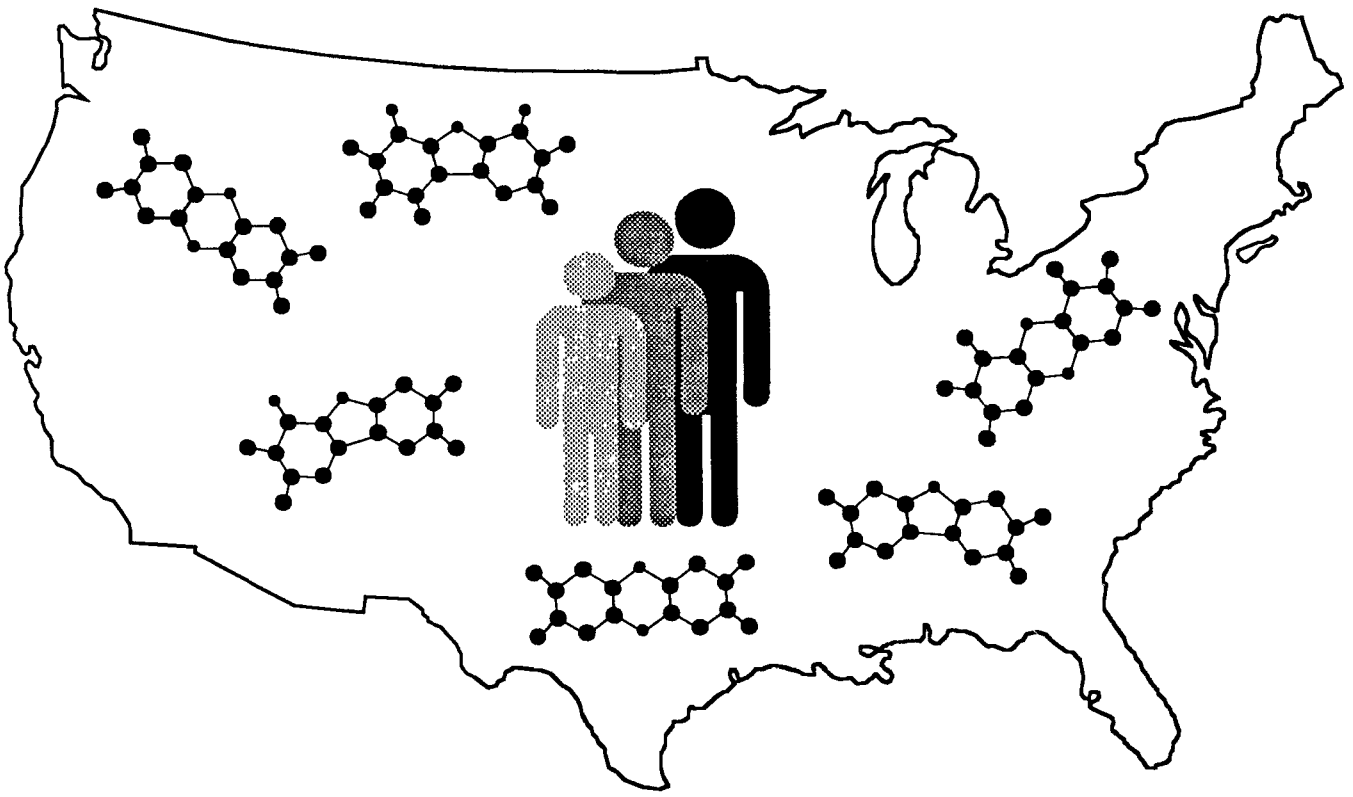

Chlorinated Dioxins and Furans in the General U.S. Population: NHATS FY87 Results

Final Report

Prepared by:

John S. Stanley
Midwest Research Institute
425 Volker Boulevard
Kansas City, MO 64110
EPA Contract No. 68-DO-0137, Work Assignment 27

and

John Orban
Battelle
Columbus Division
505 King Ave.
Columbus, OH 43201
EPA Contract 68-02-4292

for the:

Field Studies Branch
and
Design and Development Branch
Exposure Evaluation Division
Office of Toxic Substances
U.S. Environmental Protection Agency
401 M Street, S.W.
Washington, D.C. 20460

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AUTHORS AND CONTRIBUTORS

The determination of the levels of the polychlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs) in the general population of the United States described in this report was achieved through cooperative efforts of many EPA and contract support staff. EPA staff participating in the program included principal investigators from the Field Studies Branch (FSB) and Design and Development Branch (DDB) of the Exposure Evaluation Division (EED) of the Office of Toxic Substances (OTS). Contract support to OTS was provided by Midwest Research Institute (MRI) under EPA Contract Nos. 68-02-4252 and 68-DO-0137, and Battelle Columbus Division under Contract No. 68-02-4292. The roles and responsibilities of each of these organizations and contributing authors to this report are presented below.

Midwest Research Institute (MRI)

MRI was responsible for the coordination of the collection of the FY87 NHATS specimens, preparation of the NHATS composites and quality control (QC) samples, conducting the HRGC/HRMS analysis of the composites, reporting the results and coordination of the preparation of this final report. Individuals contributing to this final report included: Dr. John Stanley, Ms. Karin Bauer, Mr. Paul Cramer, and Dr. Jerry Flora.

Battelle Columbus Division

Battelle was responsible for developing the FY87 NHATS specimen collection design, creating and maintaining the data bases on the Patient Summary Reports (PSRs), designing the specimen compositing plan and the statistical methodology for data analysis, and conducting the statistical analysis to develop estimates of the PCDD and PCDF residue levels in the general U.S. population based on demographic factors. Individuals contributing to this final report included: Dr. John Orban, Dr. Robert Lordo, Dr. Al Unger, Dr. Ron Menton, Ms. Barbara Leczynski, Ms. Tamara Collins, Ms. Pam Harford, and Ms. Claire Matthews.

Westat

Mr. John Rogers of Westat provided technical support in processing all analysis reports and conducting outlier analyses.

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EPA was responsible for oversight in the development of the study plan, managing and coordinating the conduct of the overall study, and reviewing, editing and finalizing this report. Key staff included: Ms. Janet Remmers, Mr. John Schwemberger as Work Assignment Managers and Dr. Joseph Breen and Ms. Edith Sterrett as Project Officers.

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GLOSSARY

DDB	Design and Development Branch
EED	Exposure Evaluation Division
EPA	Environmental Protection Agency
FSB	Field Studies Branch
FY	Fiscal Year
HpCDD	Heptachlorodibenzo-*p*-dioxin
HpCDF	Heptachlorodibenzofuran
HpCDPE	Heptachlorodiphenylether
HxCDD	Hexachlorodibenzo-*p*-dioxin
HxCDF	Hexachlorodibenzofuran
HxCDPE	Hexachlorodiphenylether
IQS	Internal Quantitation Standard
MRI	Midwest Research Institute
MSA	Metropolitan Statistical Area
NHATS	National Human Adipose Tissue Survey
OCDD	Octachlorodibenzo-*p*-dioxin
OCDF	Octachlorodibenzofuran
OTS	Office of Toxic Substances
PCBs	Polychlorinated Biphenyls
PCDD	Polychlorodibenzo-*p*-dioxin
PCDF	Polychlorodibenzofuran
PCDF	Polychlorinated dibenzofuran
PeCDD	Pentachlorodibenzo-*p*-dioxin
PeCDF	Pentachlorodibenzofuran
RS	Recovery Standard
TCDD	Tetrachlorodibenzo-*p*-dioxin
TCDF	Tetrachlorodibenzofuran
TSCA	Toxic Substances Control Act
VA	Veterans Administration/U.S. Department of Veteran Affairs

EXECUTIVE SUMMARY

BACKGROUND

The National Human Monitoring Program (NHMP), operated by the United States Environmental Protection Agency's Office of Toxic Substances (USEPA/OTS), is an ongoing national program to monitor the human body burden of selected chemicals. The main operative program of the NHMP is the National Human Adipose Tissue Survey (NHATS). The NHATS is an annual survey to collect and analyze a nationwide sample of adipose tissue specimens from autopsied cadavers and surgical patients. The purpose of the NHATS is to identify and quantify the prevalence and levels of selected compounds in human adipose tissue. The analysis results are used to establish an exposure-based chemicals list and to estimate baseline levels and trends of the selected chemicals.

In the past, NHATS data have been used to monitor levels of selected organochlorine pesticides and polychlorinated biphenyls (PCBs) in the United States population. However, in Fiscal Year 1982 (FY82) the specimens collected under NHATS were used in the Broad Scan Analysis Study, in which composited NHATS specimens were analyzed using high resolution gas chromatography/mass spectrometry (HRGC/MS) in order to expand the list of target analytes. The Broad Scan Analysis Study identified 17 volatile organic compounds, 30 semi-volatile organic compounds, 5 polychlorinated dibenzo-*p*-dioxins (dioxins or PCDDs), and 5 polychlorinated dibenzofurans (furans or PCDFs). The Broad Scan Analysis Study demonstrated that the PCDDs and PCDFs could be detected in the general U.S. population across all geographic regions and age groups. Using a similar study design, EPA analyzed the FY87 NHATS samples for PCDDs and PCDFs.

This report presents the objectives, methodology, and results of the FY87 NHATS samples and compares the FY87 NHATS results with results from the analysis of the FY82 NHATS samples and from a related study of PCDDs and PCDFs conducted jointly by the U.S. Veterans Administration and EPA's Office of Toxic Substances (VA/EPA).

OBJECTIVES

The specific objectives of the FY87 NHATS study were to:

- Identify the PCDD and PCDF isomers (specifically those with chlorine substitution in the 2,3,7,8-position) that are present in human adipose tissue,
- Estimate the average concentrations of PCDDs and PCDFs in the adipose tissue of humans in the U.S. population and various demographic subpopulations,

- Determine if any of the demographic factors (geographic region, age, race, and sex) are associated with the average concentrations of PCDDs and PCDFs in human adipose tissue, and

- Compare the estimated average concentration levels of PCDDs and PCDFs found in the FY87 NHATS with estimates from the FY82 NHATS and the VA/EPA studies.

APPROACH

Population estimates were based on data obtained from the chemical analyses of 48 composite samples. The composite samples were prepared from 865 individual specimens collected in a statistically designed survey of autopsied cadavers and surgical patients. A statistical design was also used in creating the composite samples. HRGC/HRMS was used to measure the concentrations of seven PCDDs and 10 PCDFs in the composite samples. The resulting data were statistically analyzed using a model developed specifically for the composite samples to estimate average concentration levels in the U.S. population and to determine if any of the four demographic factors (geographic region, age, race, and sex) are associated with the average concentration levels.

Traditionally, one of the objectives of NHATS has been to estimate the prevalence of chemicals in the U.S. population as well as in various demographic subpopulations. More generally this involves characterizing the distribution (i.e., variability of body burden levels among individual donors. While these are still important objectives for NHATS, it was decided that the FY87 survey would focus available resources on estimating average concentration levels in the U.S. population and characterizing differences among various demographic subpopulations. With the more focused objectives it became more efficient to combine individual specimens into composite samples prior to chemical analysis.

The principal advantage of compositing is that it reduces the number of samples that need to be chemically analyzed. However, this does not necessarily result in a corresponding reduction of precision for the population estimates. With composite samples, the effects of sampling error on the precision of average concentration estimates are substantially reduced because the concentration in a composite sample is the average of individual specimen concentrations.

The main disadvantage of using data from composite samples is that the prevalence and distribution of chemical concentrations among individuals in the population cannot be accurately estimated. However, it was possible to achieve all of the study objectives for the FY87 NHATS as outlined above by using appropriate design criteria for combining specimens into composites.

The overall quality of results was ensured by using statistical sampling and compositing designs, implementing quality control procedures, validating analytical and statistical methodologies, and adhering to data quality objectives. Data quality objectives (DQOs) were established for target ion ratios, percent recovery of target analytes, and the presence of chemical interferences. Only data that met all DQOs were included in the statistical summaries and analyses.

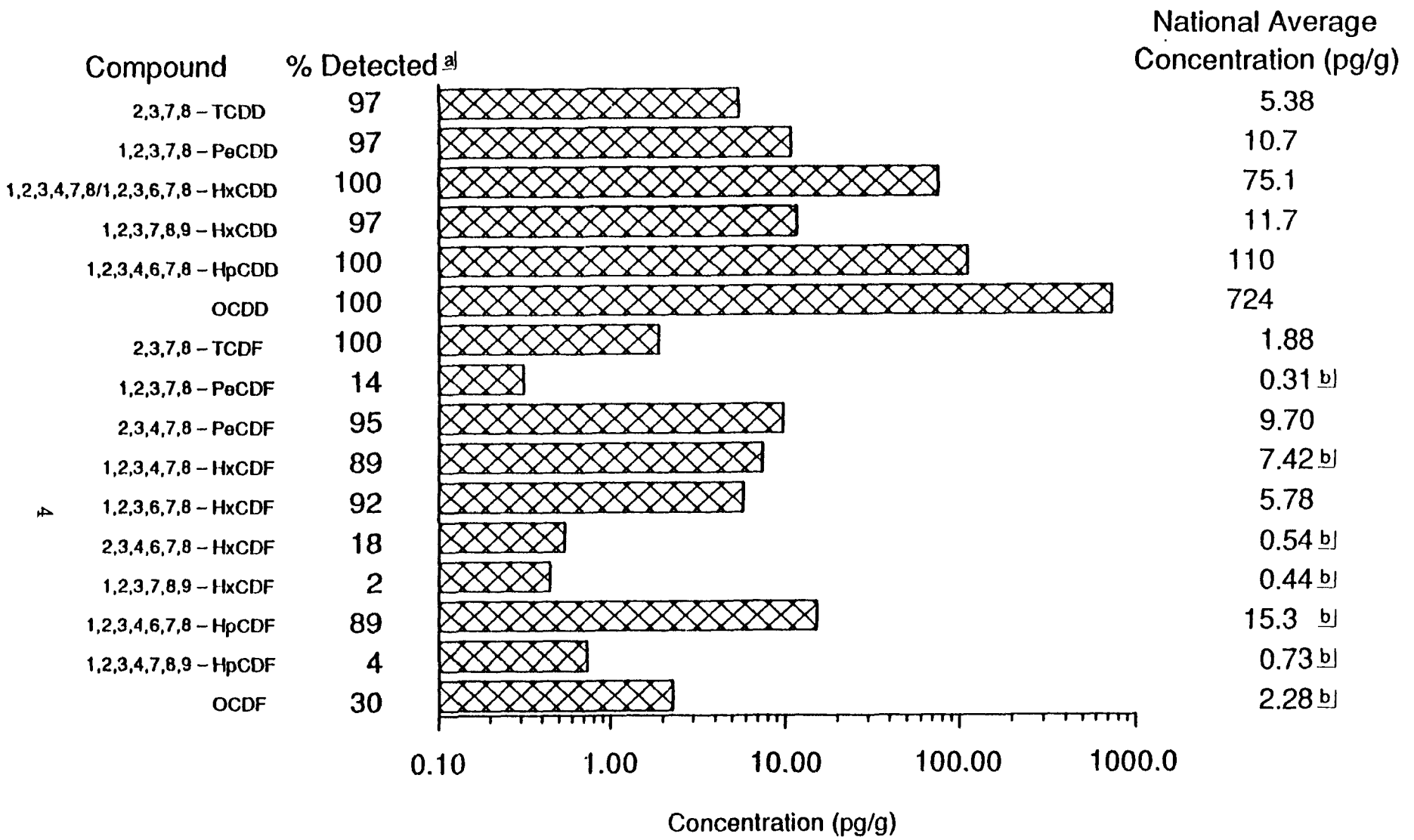
SUMMARY OF THE FY87 NHATS RESULTS

National Weighted Average Concentrations

The estimated national average concentrations of the target analytes are presented graphically in Figure ES-1. The 1,2,3,4,7,8- and 1,2,3,6,7,8-HxCDD isomers have been reported together due to incomplete chromatographic separation. Also, the percent of composite samples in which the compound was detected is listed for each target compound. Data from all of the target PCDDs and three of the 10 PCDFs were analyzed using a statistical modeling approach. Population estimates were calculated using weights determined from the 1980 census counts. Based on the 95% confidence intervals the maximum uncertainty of the estimated average concentrations is 12% for the six PCDDs and 26% for the three PCDFs. The estimates for the remaining PCDFs were calculated using weighted averages of the measured composite concentrations. Weights were determined from the 1980 U.S. Census counts. The seven remaining chemicals (PCDFs) did not meet minimum criteria to perform a more detailed statistical analysis. Several of these compounds were not detected in a sufficient number of composite samples to allow meaningful statistical analysis.

Age Effects

Each composite was prepared with specimens from one of the three age groups (0-14, 15-44 and 45+ years). The average concentrations of the nine PCDDs and PCDFs that were statistically analyzed were found to increase significantly with the age of the donor. For example, the average concentration of 2,3,7,8-TCDD increased from 1.98 pg/g in the youngest age group (0-14 years) to 9.40 pg/g in the oldest age group (45+ years) -- a 375% increase. Increases attributed to age effects in the other chemicals ranged from 24% for 2,3,7,8-TCDF to 863% for 2,3,4,7,8-PECDF.



a Percent of Composite Samples b Estimates Calculated from Simple Weighted Average of Composite Concentrations

Figure ES-1. National average concentration for PCDDs and PCDFs from FY87 NHATS.

Geographic Effects

There were significant differences in the estimated average concentrations of 2,3,4,7,8-PeCDF among the four geographic regions (north central, northeast, west, and south). The estimated average concentration in the west region was 4.49 pg/g compared to an average of 13.7 pg/g in the northeast and a national average of 9.70 pg/g. The data combined with the FY82 NHATS data also suggest the possibility of regional effects on the average concentrations of 2,3,7,8-TCDD and 1,2,3,4,6,7,8-HPCDD, but the estimated regional differences were not statistically significant.

Race and Sex Effects

The differences in estimated average concentrations between caucasian and non-caucasian donors and between male and female donors were not statistically significant for any of the modeled compounds.

COMPARISON WITH FY82 NHATS

The FY87 NHATS estimated national average concentrations of 2,3,7,8-TCDD, 1,2,3,4,6,7,8-HPCDD, and OCDD were consistent with the estimates established in the FY82 NHATS. However, the FY87 estimates for many of the other PCDDs and PCDFs were significantly lower than the estimates obtained in the FY82 NHATS. It is likely that these differences are due in part to advances in the analytical method between FY82 and FY87.

COMPARISON WITH THE VA/EPA STUDY

The VA/EPA study was a retrospective study which used surplus specimens from the NHATS repository to compare PCDD and PCDF concentrations in the adipose tissue of Vietnam veterans with non-Vietnam veterans and civilians. A total of 197 specimens collected between 1971 and 1982 were analyzed individually using analytical methodology equivalent to the FY87 NHATS study. All specimens in the VA/EPA study are within the middle age group (15-44 years) and hence allowed comparison to the same age group evaluated in the FY82 and FY87 NHATS studies. The estimates obtained in the VA/EPA study were two to three times higher than the FY87 NHATS estimates. The average values of the compounds across collection years (1971 to 1987), particularly 2,3,7,8-TCDD, indicate a decrease in adipose tissue residue levels. These differences may be due to a decrease in exposure levels through environmental pathways or consumer products over time or as a result of the differences in storage times between the VA/EPA (up to 16 years) and FY87 NHATS (up to 2 years) studies. Further studies or analyses are needed to resolve these differences.

1.0 INTRODUCTION

1.1 BACKGROUND

The National Human Adipose Tissue Survey (NHATS) is the main operating program of EPA's National Human Monitoring Program (NHMP). Under the NHATS program, human adipose tissues are analyzed to monitor human exposure to potentially toxic compounds. The adipose tissue specimens were collected according to statistically designed sampling plans. The tissues are collected by cooperating pathologists and medical examiners during routine examination of tissues that have been excised during post mortem examinations or for therapeutic reasons during elective surgeries. The tissues are collected based on a statistically developed design of Metropolitan Statistical Areas (MSAs) to provide chemical residue information that can be correlated to demographic data (geographic region, age, sex, and race).

The NHATS program was established in the late 1960's. The chemical residues of interest during that time frame were organochlorine pesticides and PCBs. Recognizing the need to extend the capabilities of the NHATS program, the Office of Toxic Substances (OTS) initiated a series of programs in 1984 to expand the utility of the tissue repository. Foremost in these analysis efforts was the conduct of a study termed the "Broad Scan Analysis Study." The broad scan analysis included the determination of volatile and semivolatile organic compounds based on methods requiring mass spectrometry as a detection device. Included in the semivolatile organic compounds were the polychlorinated dibenzo-*p*-dioxins (PCDDs) and the dibenzofurans (PCDFs). The data from the broad scan analysis effort demonstrated that the PCDDs and PCDFs were prevalent in the adipose tissues of the general U.S. population from all census divisions and within each age group.

1.2 EMPHASIS ON PCDDs AND PCDFs

The ubiquity of the PCDDs and PCDFs in adipose tissues provides evidence of widespread exposure to a class of potentially toxic compounds. On the basis of animal studies, the U.S. EPA considers 2,3,7,8-TCDD to be one of the most potent known carcinogens studied (U.S. EPA 1987, U.S. EPA 1989). Studies on the toxicity of other PCDDs and PCDFs have demonstrated the compounds with chlorine substitutions in the 2,3,7,8-ring positions results in the greatest toxicity. In addition, it has been demonstrated that toxicity varies with the degree of chlorination.

Potential for exposure to PCDDs and PCDFs exists from multiple sources including:

- Diet, particularly fish, poultry, meats, and dairy products that are impacted by environmental releases of PCDDs and PDCFs.

- Incinerator emissions (municipal, hospital and hazardous waste incinerators, automobiles, etc.),

- Halogenated commercial products such as specific herbicides (2,4-D, Agent Orange) or wood preservatives (pentachlorophenol), and

- Bleached paper products and the effluents from pulp and paper bleaching operations.

- Combustion of halogenated aromatics, e.g., PCB transformer fires.

- Metal smelting and reclamation processes.

The PCDDs and PCDFs are environmentally persistent, and the most toxic isomers, the 2,3,7,8-substituted compounds, bioaccumulate in the food chain. EPA has studied the prevalence of these compounds extensively in the general environment through the various programs conducted as part of the National Dioxin Study, and in support of investigations at potential hazardous waste sites. The current studies involving the pulp and paper industry resulted from data generated from fish residue data from the National Dioxin Study. In addition, several states (e.g., California, Vermont, Connecticut, and Mississippi) have conducted monitoring programs to determine background levels of PCDDs and PCDFs in ambient air, soil, sediment, and fish in areas that are potentially impacted by emissions from incineration sources or effluents from the pulp and paper industry (Stanley et al. 1989a, Heil and Fitzgerald 1987, State of Connecticut 1986, personal contacts).

EPA has promulgated regulations to specifically address the releases of PCDDs and PCDFs to the environment. Under Sections 4 and 8 of the Toxic Substances Control Act (TSCA), the manufacturers of specific halogenated products are required to determine and report the levels of halogenated dioxins and furans in those products (U.S. EPA 1987). EPA has also recently developed guidelines for establishing test procedures for release of halogenated dioxins and furans in industrial effluents (USEPA 1991), particularly from pulp and paper bleaching facilities.

The NHATS program provides EPA with a unique capability to monitor the impact of changes in environmentally persistent chemicals. As an example, the NHATS program has demonstrated a decline in the adipose tissue levels of PCBs in the general U.S. population since the restriction and regulation of use and disposal of PCBs in the mid 1970s (Robinson et al. 1990). The NHATS samples should also provide data on the result of efforts to reduce human exposure to PCDDs and PCDFs.

1.3 RELATED STUDIES

Over the past 10 years, considerable attention has been given to the determination of PCDDs and PCDF residues in human tissues. Many of these studies are identified and summarized in Table 1-1. Initial studies focused on body burden levels of individuals who were potentially exposed through occupational exposures (for example, Vietnam veterans to the herbicide Agent Orange) (VA/EPA 1991, Kang et al. 1991, CDC 1987, CDC 1988, CDC 1988a, Pirkle et al. 1989, Schecter 1987, Kahn 1988, Kahn et al. 1988, Kahn et al. 1990, Gross et al. 1986), improper handling of contaminated wastes (Times Beach, Missouri) (Patterson et al. 1986, Andrews et al. 1989, Patterson et al. 1987a, Patterson et al. 1989), contamination of food products (Yusho and Yucheng) (Ryan et al. 1987), and accidental releases from chemical production facilities (Seveso, Italy) (Fachetti et al. 1981, CDC 1988c).

Several studies have been conducted to determine the levels of PCDDs and PCDFs in the tissues of the general population both within the United States and internationally. Table 1-1 provides a summary of the studies that have been reported to date on the residue levels of these compounds. The studies conducted in the United States include a limited number of samples from Binghamton, New York (Schecter et al. 1986); Atlanta, Georgia (Patterson et al. 1986); Salt Lake City, Utah (Patterson et al. 1986); St. Louis, Missouri (Graham et al. 1986a, 1986b); the State of Missouri (Patterson et al.); and the State of California (Stanley et al. 1989). The studies conducted in Binghamton, St. Louis, and the State of California focused on the determination of total PCDDs and PCDFs. The other studies focused on 2,3,7,8-TCDD only.

1.4 OBJECTIVES OF THE FY87 STUDY

The specific objectives of the FY87 NHATS study were to:

- Identify the PCDD and PCDF isomers (specifically those with chlorine substitution in the 2,3,7,8 position) that are present in human adipose tissue,
- Estimate the average concentrations of PCDDs and PCDFs in the adipose tissue of humans in the U.S. population and various demographic subpopulations,
- Determine if any of the demographic factors (geographic region, age, race, and sex) are associated with the average concentrations of PCDDs and PCDFs in human adipose tissue, and
- Compare the estimated average concentration levels of PCDDs and PCDFs found in the FY87 NHATS with estimates from the FY82 NHATS and the VA/EPA studies.

Table 1-1. Summary of Studies Conducted to Determine Levels of PCDDs and PCDFs in Human Tissues

Geographical area	Date of sample collection	No. of samples	Exposure classification	Analytes measured	Tissue matrix	Reference
United States NHATS	1982	46	General population composites from 763 individual specimens	PCDDs/PCDFs	Adipose	Stanley et al., 1986 EPA, 1990
United States NHATS	1972-1982	80 79 36	Civilian Non-Vietnam veteran Vietnam veteran	PCDDs/PCDFs	Adipose	VA/EPA 1990 Kang et al. (1991)
Missouri (Kansas City, St. Louis, Springfield)	1985	128 51	General population. Chemical production work handling recreational residential exposure	TCDD	Adipose Serum	Patterson et al., 1986 Andrews et al., 1989 Patterson et al., 1989 Patterson 1987a
Missouri (St. Louis)	1985-1986	35	General population	PCDDs/PCDFs	Adipose	Graham et al., 1986
Georgia	1984	27	General population	TCDD	Adipose	Patterson et al., 1986c
Utah	1984	8	General population	TCDD	Adipose	Patterson et al., 1986c
New York Binghamton	1983	8	General population. Binghamton office building workers.	PCDDs/PCDFs	Adipose Adipose	Schecter, 1986
New Jersey	1987	19 25	General population Chemical workers	TCDD	Serum Serum	Fingerhut 1989
California (San Francisco, Los Angeles)	1988	57	General population	PCDDs/PCDFs	Adipose	Stanley et al., 1989
General U.S.	1987 1987	97 646	NonVietnam veterans (Army) Vietnam veterans	TCDD TCDD	Serum Serum	CDC 1987, 1988 CDC 1988
General U.S.	1987 1987 1982 1987	49 35 147 10	Vietnam veterans (Air Force) Agent Orange handling (Air Force) Agent Orange handling (Air Force) Agent Orange handling (Air Force), and ground troops	TCDD TCDD TCDD PCDD/PCDF	Serum Serum Serum Adipose	CDC 1988a Pirkle et al., 1989 CDC 1988a Schecter 1987
		20	Army Air Force	PCDD/PCDF	Adipose	Kahn 1988

Table 1-1 (Continued)

Geographical area	Date of sample collection	No. of samples	Exposure classification	Analytes measured	Tissue matrix	Reference
General	a	7	Non-Vietnam veterans (Army)	PCDDs/PCDFs	Adipose	Kahn et al., 1988
		55	Vietnam veterans	PCDDs/PCDFs	Serum	Kahn et al., 1990
General	a	4	Veteran controls	TCDD	Adipose	Gross et al., 1986
	1978	3	Air Force/Army	TCDD	Adipose	
	a	26	U.S. Army	PCDD/PCDF	Adipose	Schecter 1987
Canada	1976	25	General population	PCDD/PCDF	Adipose	Schecter et al., 1987
Canada	1988-1989	59	Firefighters and controls	PCDD/PCDF	Serum	De Wailley et al., 1990
Sweden	a	31	Hospital patients	PCDD/PCDF	Adipose	Rappe et al., 1986
Federal Republic of German (FRG)	a	1	Factory workers	PCDD/PCDF	Adipose	Nygren et al., 1986
	1985	6	Factory workers	PCDD/PCDF	Adipose	Schecter et al., 1988
	1986	45	Factory workers	PCDD/PCDF	Adipose	Beck et al., 1987
	a	4	Factory workers	PCDD/PCDF	Adipose	Rappe et al., 1987
Italy (Seveso)	a	1	Exposed resident	TCDD	Adipose	Fachetti et al., 1981
	1976	9	Exposed resident	TCDD	Serum	CDC 1988c
Japan	1985	12	Cancer patients	PCDD/PCDF	Adipose	Ono et al., 1986
	1984	6	General population	PCDD/PCDF	Adipose	Ryan et al., 1987
	a	17	General population	PCDD/PCDF	Adipose	Ogaki et al., 1987
China	1984	7	General surgical patient	PCDD/PCDF	Adipose	Ryan et al., 1987
Vietnam	1984	24	General	PCDD/PCDF	Adipose	Schecter et al., 1986
				PCDD/PCDF	Human milk	Schecter et al., 1990
Vietnam	a	27	General	PCDD/PCDF	Adipose	Huteau et al., 1990
New Zealand	a	37	General	PCDD/PCDF	Mothers milk	Buckland et al., 1990
France	a	12	General	PCDD/PCDF	Adipose	Huteau et al., 1990

[a] date(s) of sample collection were not specified.

1.5 REPORT ORGANIZATION

This report presents all pertinent information with respect to the assessment of data quality, the statistical design, the conduct and results of the analytical procedures, the statistical analysis procedures and extrapolations to population estimates, and the comparability of the FY87 data set to the previous PCDD and PCDF analysis programs conducted using the NHATS adipose tissue samples.

Section 2.0 (Summary and Recommendations) presents a synopsis of the significant findings from the statistical analysis of the FY87 data set in comparison to the previous studies conducted through the NHATS program. Overall data quality issues with respect to analytical and statistical criteria are discussed in Section 3.0. The study design for collection of individual specimens in the FY87 collection program is described in Section 4.0, and the compositing design and procedures for preparing the composite samples are discussed in Section 5.0. Section 6.0 describe the analytical procedures used to determine of PCDDs and PCDFs and summarizes the supporting analytical quality control (QC) data. The statistical methodologies are described in Section 7.0, and the results of the statistical analysis are presented in Section 8.0. Section 9.0 focuses on the comparisons of the FY87 study with the FY82 and the VA/EPA collaborative study. Study designs, analytical and statistical methodologies, and graphical comparisons of the FY82 and FY87 data are covered.

Supporting information on the individual sample data, the detailed analytical protocol, measures of uncertainty, and plots of QC data are presented in Appendices A through E.

2.0 SUMMARY AND RECOMMENDATIONS

The human adipose tissue specimens analyzed in the FY87 NHATS were collected from October 1986 through September 1987, following a statistically based survey design. A statistical design was also used to composite the specimens prior to chemical analysis. Section 2.1 presents a summary of the FY87 NHATS results. The results include estimated national average concentrations of PCDDs and PCDFs in human adipose tissue and the effects of the geographic region, age, race, and sex on the average concentrations. The statistical comparison of the FY82 and FY87 NHATS results and a descriptive comparison with the EPA/VA results are summarized in Section 2.2. Recommendations for future studies are presented in Section 2.3.

2.1 SUMMARY OF FY87 NHATS RESULTS

Forty eight (48) adipose tissue samples analyzed in the FY87 NHATS were prepared using 865 individual specimens collected in a statistically designed survey of autopsied cadavers and surgical patients. The composite samples were analyzed for PCDDs and PCDFs using high resolution gas chromatography/mass spectrometry (HRGC/HRMS). The resulting data were analyzed using a statistical model developed for composite samples to estimate average concentration levels and to determine if any of the four demographic factors (geographic region, age, race, and sex) are associated with the average concentration levels.

Traditionally, one of the objectives of NHATS has been to estimate the prevalence of chemicals in the U.S. population as well as in various demographic subpopulations. More generally this involves characterizing the distribution (i.e., variability) of body burden levels among individual donors. While these are still important objectives for NHATS, it was decided that the FY87 survey would focus available resources on estimating average concentration levels in the U.S. population and characterizing differences among various demographic subpopulations. With the more focused objectives it became more efficient to combine individual specimens into composite samples prior to chemical analysis.

The principal advantage of compositing is that it reduces the number of samples that need to be chemically analyzed. However, this does not necessarily result in a corresponding reduction of precision for the population estimates. With composite samples the effects of sampling error on the precision of average concentration estimates are substantially reduced because the concentration in a composite sample is the average of individual specimen concentrations.

The main disadvantage of using data from composite samples, however, is that the prevalence and distribution of chemical concentrations among individuals in the population cannot be accurately estimated. But, it was possible to achieve all of the study objectives for the

FY87 NHATS as outlined above by using appropriate design criteria for combining specimens into composites.

Because of criteria based on pre-determined data quality objectives (DQOs) the number of composite samples used in the data analysis varied among the target compounds. Several of the compounds, particularly the PCDFs, were not detected in a sufficient number of composite samples to perform a reliable statistical analysis. This generally occurred for the compounds that were detected at levels near or below the method detection limits. Interferences which precluded accurate measurement, were encountered for two of the HxCDF isomers. Thus, only nine of the target analytes met minimum criteria for applying the statistical model. However, summary statistics are provided for all target chemicals. The criteria for applying the statistical model were:

(1) At least 30 composites must meet all DQOs for chemical analysis, and

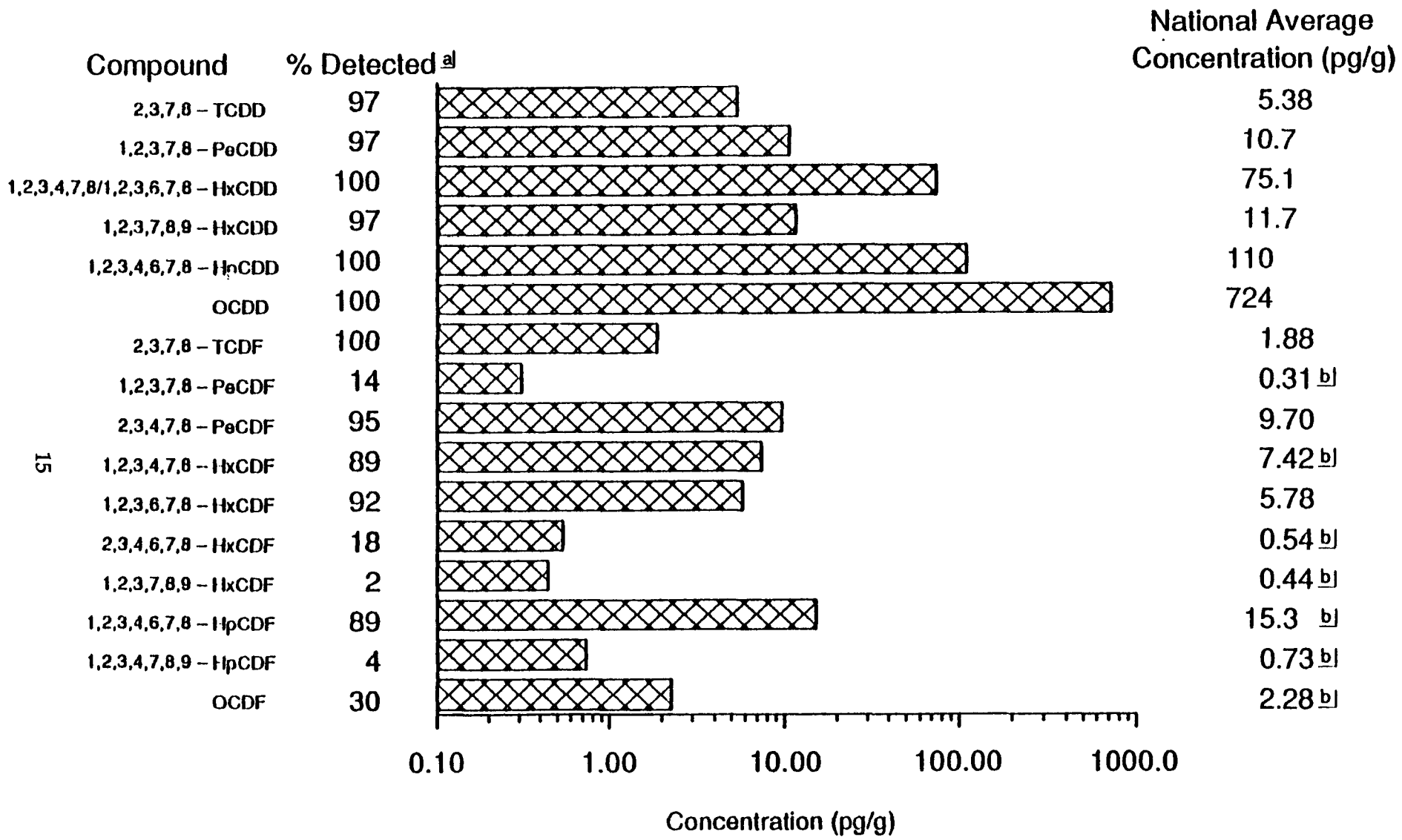
(2) The chemical must be detected in at least 50% of the composites meeting the DQOs.

2.1.1 Population Estimates

Figure 2-1 presents the estimated national average concentrations for the target chemicals. Because of wide range of concentration levels among the target chemicals, the estimates are displayed on a log scale. The estimates were calculated using either a statistical modeling approach or by taking weighted averages of the composite sample concentrations. The weights used in the weighted average estimates are based on 1980 U.S. Census population counts. Further discussion of this approach is presented in Section 9.4.

For the nine chemicals that were statistically analyzed, the error in estimating the average concentration levels, based on 95% confidence limits, was less than 12% for the PCDDs and less than 26% for the PCDFs. The quality of these estimates is supported by validation of both the chemical analysis method and the statistical approach to handling the data and the performance of the method as demonstrated from the results of the Quality Control Samples.

The estimates for the chemicals, particularly PCDFs, that were detected in less than 50% of the samples may be significantly affected by the detection limits of the analytical method. Whenever the chemical was not detected in the sample, it was assumed that the concentration was below the limit of detection (LOD) of the analytical method. Thus, the data value was "censored" and no measured concentration was reported. To calculate an average concentration using censored data, the value of LOD/2 was used in place of a measured concentration whenever the chemical was not detected. This approach minimizes the potential error in the estimated average concentration. But when more than 50% of the data are censored there is



[a] Percent of Composite Samples [b] Estimates Calculated from Simple Weighted Average of Composite Concentrations

Figure 2-1. Estimated national average concentrations for the target PCDDs and PCDFs from the FY87 NHATS.

much more uncertainty in the estimated average. The maximum systematic error due to censuring is the average LOD divided by two.

2.1.2 Demographic Comparisons

Statistical hypothesis tests were performed for each of the nine modeled compounds to determine if there were statistically significant differences in average concentration levels among individuals from different census regions (north central, northeast, west, and south), age groups (0-14 years, 15-44 years, and 45+ years), race groups (caucasian, non-caucasian), and sexes (male, female). Statistical conclusions derived from these analyses are summarized below.

Age Effects. Concentration of PCDDs and PCDFs increase with age. The differences in the average concentrations among age groups was statistically significant at the 0.05 level for all nine of the modeled chemicals. Figure 2-2 displays the average concentrations by age group for each of the modeled chemicals. The highest average concentration was always found in the oldest age group (45+ years) and, except for 2,3,7,8-TCDF, the lowest was found in the youngest age group (0-14 years). The increases from the lower to the upper age groups range from 24% for 2,3,7,8-TCDF to 863% for 2,3,4,7,8-PeCDF.

Geographic Effects. One of the nine modeled chemicals was found to have statistically significant differences among the estimated average concentrations from different census regions. The estimated average concentration of 2,3,4,7,8-PeCDF in the Western census region was 4.49 pg/g compared to an average of 13.7 pg/g in the North East region. The national average was 9.7 pg/g. No other chemical for the FY87 composites was found to have significant geographic effects at the 0.05 level of significance. This geographic effect for the 2,3,4,7,8-PeCDF was also detected in the analysis of the data for the FY82 NHATS samples.

Race Effects. The difference in estimated average concentrations between caucasian and non-caucasian donors was not found to be statistically significant for any of the modeled compounds.

Sex Effects. The difference in estimated average concentrations between male and female donors was not found to be statistically significant for any of the modeled compounds.

2.1.3 Quality Control Results

Twenty (20) quality control (QC) samples were analyzed along with the 48 study samples. Each of the five analysis batches contained eight to ten study samples, a method blank, an unspiked control sample, and two spiked samples—one at a low spike level and one at a high

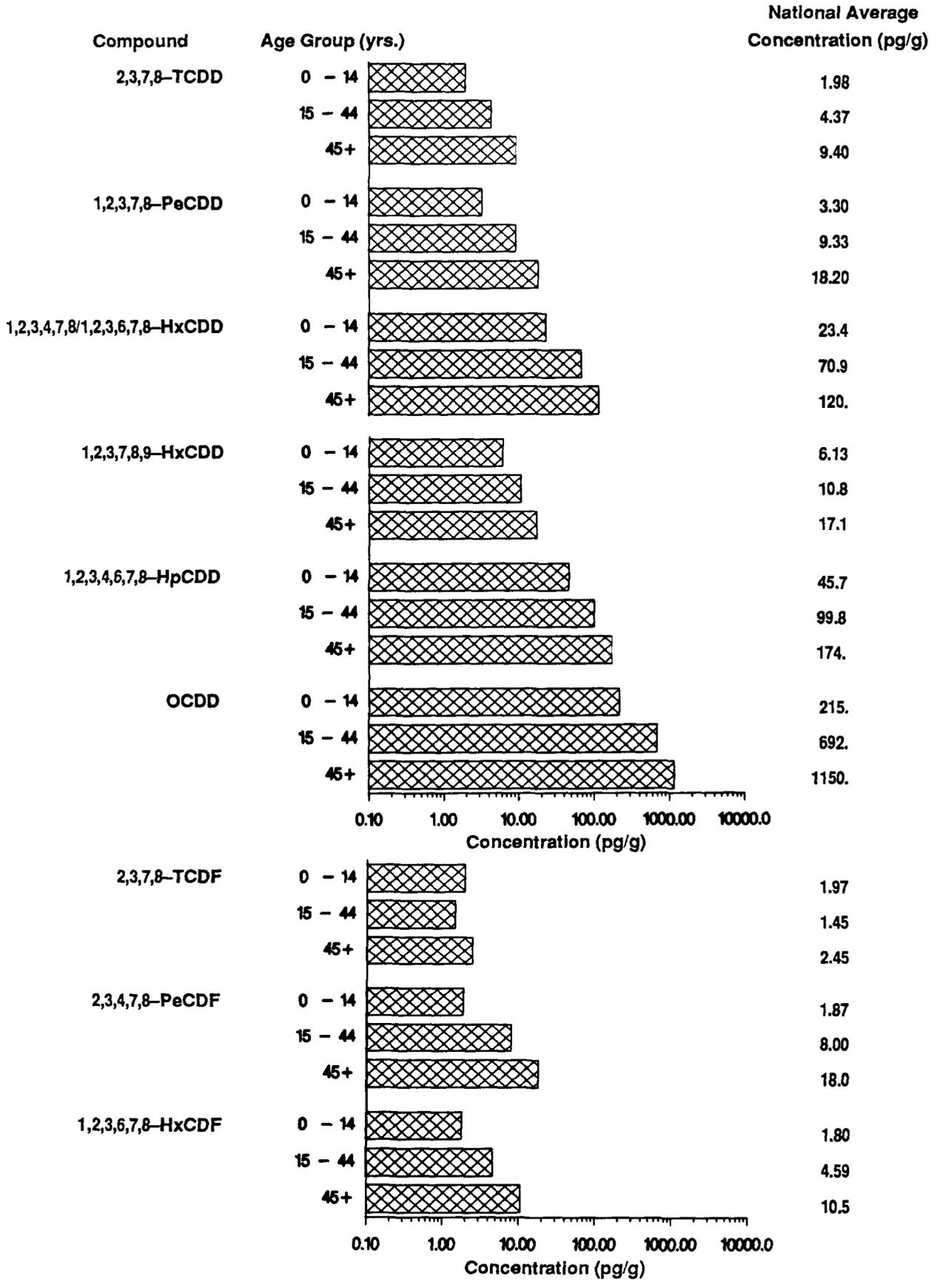


Figure 2-2. Estimated averaged concentrations by age group for selected PCDDs and PCDFs from the FY87 NHATS.

spike level. The QC samples were chemically analyzed to characterize the accuracy and precision of the analytical method and to determine if there were significant batch effects. The statistical analysis of the QC data demonstrated that batch effects were relatively small and that the method recovery criteria were met for 97.5% of the target analytes.

2.2 COMPARISON OF FY87 NHATS WITH OTHER STUDIES

The results from the FY87 NHATS were compared with results from two previous studies involving specimens from the NHATS repository. Section 2.2.1 discusses the comparison with the FY82 NHATS Broad Scan study and Section 2.2.2 discusses the comparison with a collaborative study conducted between the Veterans Administration and the U.S. EPA (VA/EPA).

2.2.1 Comparison with FY82 NHATS

The study designs for FY82 and FY87 NHATS were very similar. Although there are minor differences in the number of specimens collected and their distribution among demographic populations, these differences do not significantly affect the comparison of results. On the other hand, there were substantial improvements in the analytical method between the FY82 and FY87 NHATS. Additional internal quantitation standards were added at the sample extraction step and carried throughout the analysis procedure. These additional internal quantitation standards provide a better characterization of method performance and more accurate determination of the PCDDs and PCDFs via the isotope dilution principle.

Statistical comparisons were performed to determine significant differences for the specific compounds between the FY82 and FY87 NHATS studies. Differences noted in the measured concentrations may be due to either actual changes in body burden levels or changes in the analytical method. In addition to comparing national average concentrations, a more detailed profile analysis was performed to determine if the demographic effects are consistent between FY82 and FY87. The profile analysis provides meaningful results even if there are systematic differences in measured concentration levels.

Table 2-1 compares the estimated national average concentration levels for selected chemicals analyzed in FY82 and FY87. Also presented are the relative changes from FY82 to FY87. For example, the estimated concentration of 2,3,7,8-TCDD is 9% lower for FY87 than determined for FY82. Statistical comparisons were possible for the PCDDs and only one PCDF, the 2,3,4,7,8-PeCDF. The other PCDFs could not be compared statistically because one or both of the estimates are based on data which did not meet the minimum criteria for statistical modeling.

Table 2-1. Estimated National Average Concentrations of PCDDs and PCDFs from the FY82 and FY87 NHATS

	National average			
	FY82 (pg/g)		FY87 (pg/g)	Percent change[a]
PCDDs				
2,3,7,8-TCDD	5.88		5.38	-9%
1,2,3,7,8-PeCDD	73.6	>[b]	10.7	-85%
HxCDD	122	>	86.8	-29%
1,2,3,4,6,7,8-HpCDD	142		110	-23%
OCDD	768		724	-6%
PCDFs				
2,3,7,8-TCDF	39.7	[c]	1.86	-95%
2,3,4,7,8-PeCDF	35.4	>	9.70	-73%
HxCDF	20.9	[c]	14.2	-32%
1,2,3,4,6,7,8-HpCDF	20.6	[c]	15.3	-26%
OCDF	56.0	[c]	2.28	-96%

[a] Percentage calculated as $\frac{(FY87-FY82)}{FY82} \times 100\%$

[b] The FY82 estimate is significantly higher (denoted by >) than the FY87 estimate at the 0.05 level of significance.

[c] No statistical comparisons were possible.

All of the chemicals had higher estimated concentrations in FY82. However, among the modeled chemicals, the differences in estimated concentrations between FY82 and FY87 were not statistically significant for 2,3,7,8-TCDD, 1,2,3,4,6,7,8-HpCDD, and OCDD. These chemicals had estimated relative changes of less than 23%. Some of the differences between FY82 and FY87 can be explained by changes in the analytical method. This is evident in the fact that the detection limits in FY82 were often higher than the estimated average concentration in FY87. For example, the estimated detection limit for OCDF in FY82 was 19.0 pg/g. The average concentration measured for the FY87 samples was 2.28 and the detection limit was 1.67 pg/g. In addition to achieveing lower detection levels, the analysis of the FY87 NHATS composites was enhanced through the use of additional internal quantitation standards that provided more accurate measurements of the PCDD and PCDF residue levels. The effect of using fewer internal standards in the FY82 anaysis may have resulted in over or under estimates of the concentrations of the penta-, hexa-, and hepta-chlorinated PCDDs and PCDFs.

The profile analysis provided additional insight into the demographic effects on PCDDs and PCDFs. As previously discussed, the FY87 data demonstrated that there were geographic effects on the concentrations of 2,3,4,7,8-PeCDF. Although there was a significant difference in the national average concentrations between FY82 and FY87, the FY82 data confirmed the existence of geographic effects. In both years, the highest estimated concentrations of 2,3,4,7,8-PeCDF were found in the northeast. The profile analysis also suggested that there are possible geographic effects on the concentrations of 2,3,7,8-TCDD and 1,2,3,4,6,7,8-HpCDD. The profiles for FY82 and FY87 were very similar with the lowest estimated concentrations in the west and south census regions.

2.2.2 Comparison with the VA/EPA Study

The VA/EPA study was a retrospective study which used surplus specimens from the NHATS repository to compare PCDD and PCDF concentrations in the adipose tissue of Vietnam veterans with non-Vietnam veterans and civilians. The study was conducted using approximately 200 adipose tissue specimens collected between 1971 and 1982 from male donors between the ages of 17 and 46. Each specimen was analyzed for the same PCDDs and PCDFs that were measured in the FY87 composites.

Although there are differences between the VA/EPA study and the FY87 effort in terms of study objectives, sampling plans, and size and composition of samples analyzed, it was expected that the results for the middle age group (15-44) from the studies should allow comparison since all analyses were conducted with equivalent methodology. The estimated concentrations in the VA/EPA were two to three times higher than the corresponding FY87 NHATS estimates. Although no formal statistical tests were performed on these data, the differences are clearly

significant. The average values for the compounds across collection year, particularly 2,3,7,8-TCDD, indicate a decrease in adipose tissue residue levels. These differences may be due to a decrease in exposure levels through environmental pathways or commercial/consumer products over time. Another possible explanation may be the result of differences in specimen storage time between the VA/EPA (up to 16 years) and the FY87 NHATS (up to 2 years) studies.

2.3 RECOMMENDATIONS FOR FUTURE STUDIES

Several recommendations, along with rationale, are presented for consideration for future studies on PCDDs and PCDFs in human adipose tissue. Although the recommendations are presented with respect to human adipose tissue, they also apply to considerations for use of blood (whole, serum, or plasma) as a means to study the body burdens of these compounds in the general U.S. population.

Recommendation: Increase the number of composite samples and individual specimens that are analyzed in future NHATS studies.

Rationale: The FY87 NHATS produced estimates of national average concentration levels of nine PCDDs and PCDFs and demonstrated that the concentrations of these chemicals increase significantly with the age of the donor. These conclusions were based on the chemical analysis of 48 composite samples containing an average of 18 specimens each. Although there were sufficient data to achieve these important objectives, the small number of composites analyzed presents significant challenges to achieving other NHATS objectives. For example, it is very difficult to estimate prevalence with composite sample data. Prevalence is established by estimating the percent of the population with chemical concentrations above specified levels. Estimating prevalence requires that individual specimens be analyzed to characterize the statistical distribution of concentrations among individuals in the population.

Another reason for increasing the number of specimens is to detect smaller but important differences in average concentration levels among geographic regions, race groups, and sexes that cannot be identified statistically because of sampling and measurement errors. Statistical power calculations will be needed to establish the optimal allocation of individual versus composite samples to be analyzed.

Recommendation: Conduct an analysis of PCDDs and PCDFs in human adipose tissue using a similar study design at intervals of one to five years.

Rationale: One of the objectives of NHATS is to estimate time trends in the levels of toxic chemicals. Significant trends require data from at least three to five time periods before they can

be demonstrated. Considering the promulgation of specific regulations to limit releases of these compounds from a variety of sources (incineration emissions, pulp and paper effluents, halogenated commercial products, etc) it is expected that further changes in residue levels may be observed. Correlation of declining residue levels with the promulgation of the residue levels over time may provide the Agency with a good measure of the efficacy of environmental regulation.

Recommendation: Establish and characterize QC samples that are representative of human adipose tissue levels.

Rationale: In order to ensure that an accurate assessment of data comparability can be achieved from one analysis event to the next, it is essential that appropriate reference materials are available for incorporation into the study design. Adipose tissue pools representative of the three age categories could be prepared in sufficient quantity to allow multiple determinations in subsequent NHATS analysis efforts. The QC pools could be stored under the same conditions as the NHATS repository and could be used to address storage stability issues.

3.0 OVERALL DATA QUALITY

The overall quality of results in the FY87 NHATS was achieved by performing several critical activities: planning a well-designed study, validating methods in advance, controlling implementation, verifying assumptions, and documenting procedures. At least one of these activities was involved in every step of the study. Figure 3-1 shows the steps of the study and illustrates how they are related.

The steps are divided into the following five stages:

- Sampling,
- Compositing,
- Chemical analysis,
- Data management and processing, and
- Statistical Analysis.

Each stage involved a design or a plan, a set of controls applied on implementation, and verification and documentation procedures that were followed. Subsections 3.1 to 3.5 describe, for each stage, the specific activities that were carried out.

3.1 SAMPLING

A complete discussion of the FY87 NHATS study sampling design is contained in Section 4.0 of this report and has been fully documented (Panebianco DL, 1986a). The design followed a multistage process in which (1) the conterminous 48 states were divided into geographic strata, (2) Metropolitan Statistical Areas (MSAs) were randomly sampled within each stratum, and (3) cooperators were chosen in each MSA to provide the specimens. Finally, each cooperator was given quotas, derived from the 1980 U.S. Census, on the number of specimens to collect from each age group, sex, and race group.

The cooperators were given guidelines on the types of patients and cadavers which can contribute specimens to the program. The relevant donor information was recorded on Patient Summary Reports (PSRs). Then, matching bar code labels containing sample IDs were affixed to each PSR and the corresponding sample vial. Each PSR was then reviewed by the laboratory responsible for sample collection to determine whether the cooperators followed the guidelines in selecting appropriate donors.

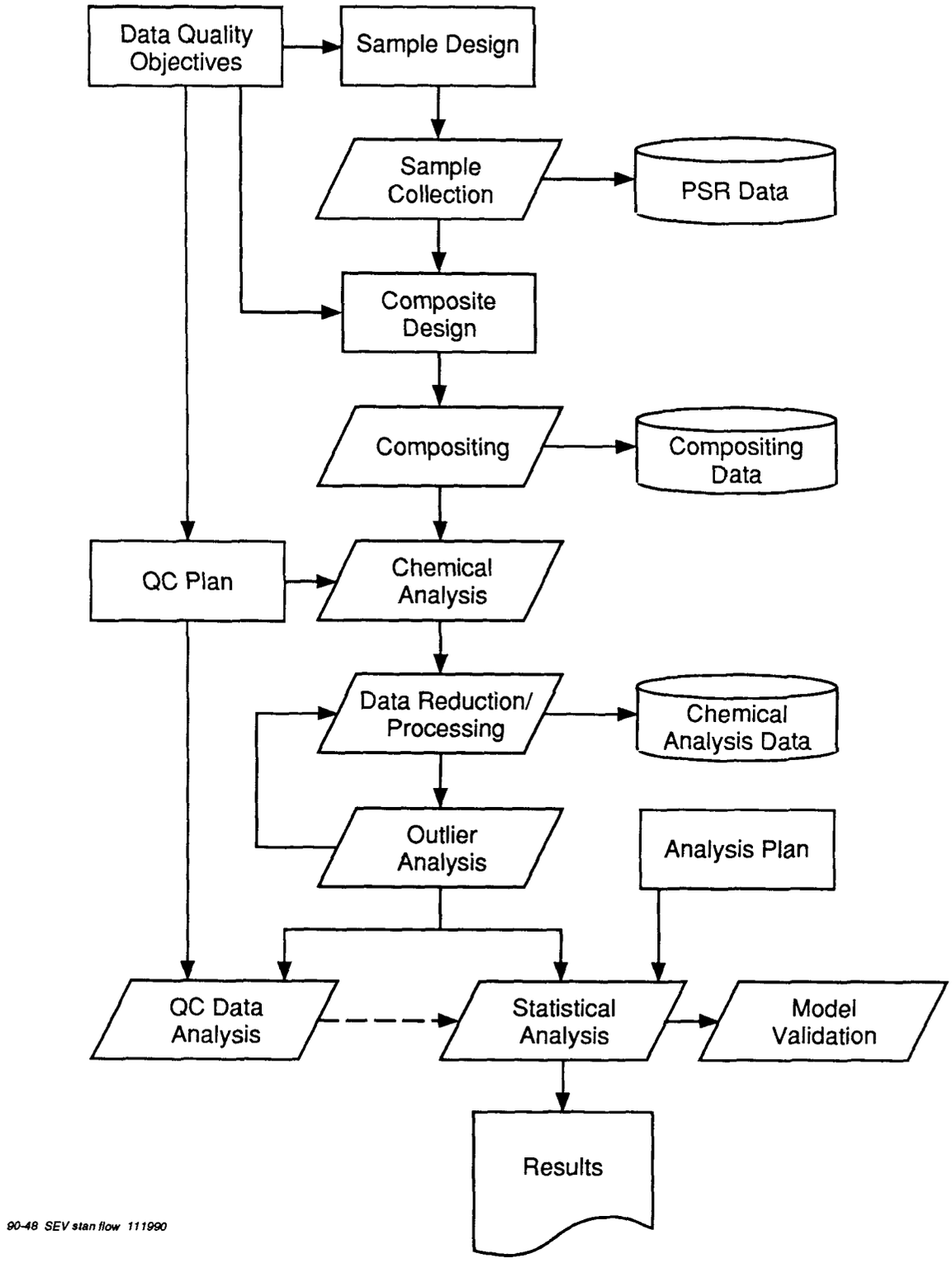


Figure 3-1. Program steps designed to ensure overall program quality.

The sampling design called for collecting 1377 individual specimens with specific quotas for each combination of geographic region and donor age group, race, and sex. Forty one (41) of the 47 MSAs specified in the sample design contributed a total of 956 specimens. Specimens collected within the overall design quota for each MSA were designated as design specimens, and the remaining specimens were designated as surplus. There were 865 design specimens in the FY87 NHATS. As discussed in Section 4.0, the marginal percentages of design specimens from donors in each geographic and demographic category were found to be consistent with the sampling design.

3.2 COMPOSITING

Composite design criteria were developed to ensure that the data obtained from the composites could be interpreted for the populations of interest and that the results could be compared with results from the FY82 NHATS study. Section 5.0 discusses these design criteria.

The composite design criteria specified that only specimens from the same Census division and age group were to be composited together. Also, in FY87 special efforts were made to create more composites containing specimens from donors of the same sex than was achieved in FY82. For example, 24% of the FY82 composites were pure sex (either all males or all females) composites compared with 64% of the composites prepared for the FY87 samples. It was not always possible to form composites containing specimens from the same race group.

The procedures for preparing the composite samples are presented in Section 5.2. Quality assurance/quality control practices that were implemented during compositing were focused on procedures to assure that the study design was accurately followed and reported and that the individual specimens were composited in a manner that preserved sample and specimen integrity and avoided contribution from laboratory background.

Battelle provided MRI the study design, which identified each individual specimen that was to be added to form a single composite. A form was provided for each composite sample and was used to document the specific compositing activities. This form included the identification of each individual specimen and demographic information that was used to confirm the identity of each specimen. All specimens were retrieved from the repository and grouped according to the composite design. The specimens were examined to determine if sufficient tissue was present to meet the composite design. The EPA work assignment managers were notified of potential problems, which were resolved prior to proceeding with the physical compositing.

As the composite samples were prepared, the mass of individual specimens were recorded on the composite forms. All composite forms were compiled as a report and submitted to EPA and Battelle. Data from the compositing reports were keyed to a computer file using double data entry. The data were stored in the FOCUS database system which was also used to perform range and logic checks.

3.3 DEMONSTRATED LABORATORY QUALITY CONTROL

At the outset of the analysis effort for the FY87 NHATS composite samples for PCDDs and PCDFs, specific data quality objectives were developed and included in the Quality Assurance Project Plan. These data quality objectives were identified for calibration criteria (relative response factors [RRFs]) for each analyte and internal standard, HRGC column performance, HRMS resolution, and method performance based on the recoveries of internal quantitation standards (IQS) and compounds spiked into a spiked internal QC sample.

Table 3-1 summarizes the performance achieved versus the specific criteria and data quality objectives for the analysis of the FY87 NHATS composites.

Table 3-1. Summary of DQOs for PCDD/PCDF Analysis Effort

Criteria	Objective	Actual
RRF calibration	±20% TCDD/TCDF ±30% all other	91% (378 of 416) of all RRF factors within DQOs
TCDD isomer separation	≤ 25% valley/-valley	Achieved for all analyses days
Mass resolution	HRMS ≥ 10,000	Maintained and documented all analysis days
Internal quantitation standards (IQS)	40%-150%	96% for all IQS standards (585 of 612); 2.5% (17) of deviation due to ^{13}C-OCDD; 1.5% (9) due to double-spiked sample
Spiked internal QC samples	40%-150%	97.5% of all measurements within criteria (4 of 160 measurements outside DQOs)

A QC plan was proposed by Heath (1988) to monitor and characterize the performance of the analytical method. The plan called for the analysis of a method blank, control sample, and two spiked samples in each of the five analysis batches. The control and spiked samples were formed using a composite sample of a large number of surplus adipose tissue specimens. The method blank was analyzed first to confirm that the sample analytes would not be compromised by laboratory background or calibration standard carryover, then the control and spiked samples were analyzed in random order along with the eight to 10 study samples. The objective of the QC plan was to provide data that could be used to characterize the method's precision and accuracy and to identify any significant batch effects.

The statistical analysis of the QC data involved modeling the measured concentrations versus spike level. Estimates of background levels, batch effects, and method recoveries were established for all of the target chemicals. Also, statistical hypothesis tests were conducted to determine if there were significant batch effects or if the estimated recoveries were significantly less than 100%.

Batch effects were accounted for by including their effect in the estimated measurement precision. Precision, as determined by the coefficient of variation at the control sample concentration level, was estimated to be 5% to 20% for the dioxin compounds and 13% to 48% for the furans.

3.4 STATISTICAL ANALYSIS

Prior to conducting the statistical analysis, a formal analysis plan was prepared to define the statistical objectives and to specify the methods and assumptions that would be used to achieve these objectives. The primary objectives were to estimate average concentrations in the U.S. population and in specified marginal populations, and to determine if there are significant effects associated with geographic and demographic factors. Section 7.0 discusses these objectives and describes the statistical analysis approach.

The statistical model and analysis approach used for the FY87 NHATS were developed specifically for the unique problem of analyzing composite sample data. They were developed and evaluated in a separate study by Orban and Lordo (1989) in which composite data were simulated from actual specimen data obtained in the FY83 NHATS. Their study demonstrated the validity of the analysis approach. They also developed and documented special computer programs to implement the analysis.

Before the statistical analysis was conducted, the data were classified according to restrictions imposed by the predetermined data quality objectives (DQOs). Only data which

met all of the DQOs were included in the analysis. This ensured that the reported results are not affected by biases attributed to the analytical method. Furthermore, the formal statistical analysis to estimate the population averages was only performed when there were a minimum of 30 unrestricted composite measurements, and the chemical was detected in at least 50% of the samples. Nine of the target compounds met these minimum criteria for performing the statistical analyses. Summary statistics are provided for the chemicals which did not meet these criteria.

After fitting the statistical model to the data, the model assumptions were checked through analysis of residuals (i.e., the difference between predicted and measured concentrations), probability plots, and correlations. These analyses demonstrated that the model assumptions were reasonable. Furthermore, as discussed in Section 8.0, eight of the nine chemicals statistically analyzed had R-squared correlations exceeding 68%. That is, at least 68% of the variability in the data could be explained by the assumed model.

4.0 NHATS SAMPLE DESIGN

4.1 SAMPLING DESIGN

The human adipose tissue specimens analyzed in the FY87 NHATS were collected from October 1986 through September 1987, following the NHATS sampling design. The NHATS program uses a statistically based survey design to obtain adipose tissue specimens from autopsied cadavers and surgical patients. Although the NHATS target population is the general, noninstitutionalized U.S. population, the sampling population is limited to cadavers and surgical patients due to the invasive nature of the process required to collect the adipose specimens from living persons.

Each year approximately 800-1200 adipose tissue specimens are collected using a multi-stage sampling plan. The 48 conterminous states are first stratified into geographical areas. From the set of strata, a sample of Metropolitan Statistical Areas (MSAs) is selected with probabilities proportional to MSA population. One or more cooperators (hospital pathologists or medical examiners) are chosen from each MSA and asked to supply a specified quota of tissue specimens. Each year an effort is made to retain the same MSAs and cooperators used in previous years.

Cooperators are given overall quotas, as well as subquotas for supplying specimens in categories defined by the donor's age group, race, and sex. The categories are

Age Group:	0-14 Years, 15-44 Years, and 45+ Years;
Race:	Caucasian and non-Caucasian; and
Sex:	Male and Female.

The subquotas are proportional to the 1980 U.S. census population counts for each sampling stratum. The donors and tissue specimens are selected in a nonprobabilistic manner by the cooperators.

Because the survey requires some divergence from strict probabilistic sampling, the validity of the statistical estimates derived from the data depends on several assumptions. First, the concentrations of toxic substances in the adipose tissue of cadavers and surgical patients are assumed to be comparable to those in the general population. Second, it is assumed that the levels of toxic substances in urban residents are approximately the same as in rural residents, and therefore the selection of only urban hospitals and medical examiners (i.e., those located in MSAs) does not introduce any significant bias into the estimates of average concentration levels. Finally, it is assumed that no systematic bias is introduced by

the fact that the cooperators are not randomly selected, and that the donors and specimens are nonprobabilistically sampled according to pre-specified quotas.

4.1.1 Selection of MSAs

Prior to 1985, the sampling strata from which MSAs were randomly selected were the nine U.S. Census divisions. But in 1985 EPA wanted the ability to obtain estimates of average concentration levels in each of the ten EPA regions. Therefore, beginning with the FY85 NHATS, sampling strata were redefined to be the seventeen geographic areas of the country which resulted from the intersection of the nine Census divisions and the ten EPA regions (Panebianco DL, 1986a). Selecting the sample in this manner made it possible to make comparisons with previous NHATS results and also obtain estimates for the EPA regions. The states, Census divisions, and EPA regions that define the seventeen strata are shown in Table 4-1.

Beginning with the FY85 NHATS, a new sample of MSAs was selected from the set of seventeen strata using a controlled selection technique, known as the Keyfitz technique, which maximized the probability of retaining the MSAs used in the FY84 design (Mack et al. 1984). The MSA sample selected in FY85 has served as the base NHATS sample for FY86 and FY87. The FY87 NHATS sampling design contains forty-seven MSAs. Thirty-eight of the FY87 MSAs were selected in the FY85 sample. The remaining nine FY87 MSAs are replacements. Some of the FY85 sample MSAs were replaced because satisfactory cooperators could not be found.

The forty-seven MSAs included in the FY87 NHATS design are listed by stratum in Table 4-2. Four MSAs (Los Angeles, Chicago, Detroit, and New York) are listed as double collection sites because their populations are much larger than the populations of the other MSAs. While given positive probability of selection, no MSAs were selected from strata 13, 15, and 17 because of the small population size of these strata.

4.1.2 Collection Quotas

Sampling within MSAs is done by quota. A quota of specimens is assigned to each sample MSA. In addition, demographic subquotas are assigned to each MSA so that the specimens collected will be representative of the strata with respect to the three demographic factors: age group, race, and sex. The subquota for each MSA is determined by the demographic makeup of the stratum to which the MSA belongs and is based on the 1980 U.S. Census population counts. Each combination of age group and sex is proportionally represented in the subquota. The race categories are also proportionally represented, but the

Table 4-1. Summary of Sampling Strategy

Stratum	Census division	EPA region	State
1	New England	1	Maine New Hampshire Vermont Massachusetts Rhode Island Connecticut
2	Middle Atlantic	2	New York New Jersey
3	Middle Atlantic	3	Pennsylvania
4	South Atlantic	3	Delaware Maryland District of Columbia Virginia West Virginia
5	South Atlantic	4	North Carolina South Carolina Georgia Florida
6	East South Central	4	Kentucky Tennessee Alabama Mississippi
7	East North Central	5	Ohio Indiana Illinois Michigan Wisconsin
8	West North Central	5	Minnesota
9	West South Central	6	Arkansas Louisiana Oklahoma Texas
10	West North Central	7	Iowa Missouri Nebraska Kansas

Table 4-1 (Continued)

Stratum	Census division	EPA region	State
11	West North Central	8	North Dakota South Dakota
12	Mountain	8	Montana Wyoming Colorado Utah
13	Mountain	9	Arizona Nevada
14	Pacific	9	California
15	Mountain	10	Idaho
16	Pacific	10	Washington Oregon
17	Mountain	6	New Mexico

Table 4-2. Sample MSAs for Fiscal Year 1987
National Human Adipose Tissue Survey

Stratum	Census division	EPA region	MSA
1	New England	1	Springfield, MA Boston, MA
2	Middle Atlantic	2	Syracuse, NY[b] New York, NY[a] Binghamton/Elmira, NY Patterson-Clifton-Passaic, NJ[b]
3	Middle Atlantic	3	Philadelphia, PA Pittsburgh, PA Erie, PA
4	South Atlantic	3	Washington, DC Norfolk, VA
5	South Atlantic	4	Tampa, FL Greenville, SC Jacksonville, FL[b] West Palm Beach-Boca Raton, FL Miami, FL Atlanta, GA
6	East South Central	4	Memphis, TN Birmingham, AL Lexington, KY
7	East North Central	5	Dayton,OH Detroit, MI[a] Columbus, OH Cleveland, OH Akron, OH Chicago, IL[a] Madison, WI Moline, IL
8	West North Central	5	Rochester, MN

Table 4-2 (Continued)

Stratum	Census division	EPA region	MSA
9	West South Central	6	New Orleans, LA[b] Brownsville-Harlingen-San Benito, TX[b] Houston, TX San Antonio, TX Dallas, TX
10	West North Central	7	Omaha, NE St. Louis, MO Wichita, KS [b]
11	West North Central	8	Grand Forks, ND[b]
12	Mountain	8	Salt Lake City, UT Denver, CO
14	Pacific	9	San Francisco, CA Sacramento, CA Los Angeles, CA[a]
16	Pacific	10	Portland, OR Spokane, WA Olympia, WA[b] Tacoma, WA[b]

[a] Indicates a double collection site. A double collection site is an MSA whose population relative to its stratum is so large that its proper representation in the sample requires it to be selected twice.

[b] Indicates replacement MSA. A replacement MSA is an MSA that was not selected in the FY85 probability sample, but was chosen to replace an FY85 sample MSA for which a satisfactory cooperator could not be found.

subquota does not specify that Caucasians and non-Caucasians are to be proportionally represented within each combination of age group and sex. The subquotas only specify the total number of Caucasian and non-Caucasian specimens to be collected from each MSA.

The subquotas for the seventeen sampling strata are presented in Table 4-3. A total quota of twenty-seven specimens was specified for each MSA, except those that were designated as double collection sites. In those MSAs the quotas and subquotas were doubled. Cooperators within an MSA were assigned quotas and subquotas appropriate to that MSA.

The total number of samples specified for the FY87 NHATS was 1377. This is based on the quota of 27 specimens for each of the 47 MSAs plus 27 additional specimens for each of the four MSAs designated as double collection MSAs.

4.2 SAMPLE COLLECTION PROCEDURES

NHATS specimens are adipose tissue samples excised by pathologists and medical examiners during therapeutic or elective surgery or during postmortem examinations. If the specimen was collected postmortem, the tissue was obtained from an unembalmed cadaver which had been dead for less than twenty-four hours and had been kept under refrigeration during that time. The death should have been caused by sudden traumatic injury, such as cardiac arrest, car accident, or gunshot wound.

The following groups were excluded from specimen collection:

- Institutionalized individuals;
- Persons known to be occupationally exposed to toxic chemicals;
- Persons who died of pesticide poisoning; and
- Persons suffering from cachexia.

These guidelines were stipulated so that the levels of substances detected in the specimens were a result of environmental exposure.

All NHATS cooperators in the selected MSAs were provided with target quotas for specimen collections from age, sex and race groups. The cooperators were asked to obtain at least five grams of tissue from each donor. The cooperators were instructed to avoid contamination through contact with disinfectants, parafins, plastics, preservatives and solvents. After collection the specimens were placed in glass jars with Teflon® lids and frozen

Table 4-3. Age, Race, and Sex Subquotas for each NHATS Collection Site within a Stratum--FY87 Design

Stratum	Census division	EPA region	No. of non-Caucasians	0-14 Yr		15-44 Yr		45+ Yr	
				Male	Female	Male	Female	Male	Female
1	New England	1	2	3	3	6	6	4	5
2	Middle Atlantic	2	5	3	3	6	6	4	5
3	Middle Atlantic	3	3	3	3	6	6	4	5
4	South Atlantic	3	6	3	3	6	7	4	4
5	South Atlantic	4	6	3	3	6	6	4	5
6	East South Central	4	5	3	3	6	6	4	5
7	East North Central	5	4	3	3	6	6	4	5
8	West North Central	5	1	3	3	6	6	4	5
9	West South Central	6	6	4	3	6	6	4	4
10	West North Central	7	2	3	3	6	6	4	5
11	West North Central	8	2	3	3	6	6	4	5
12	Mountain	8	2	3	3	7	7	3	4
13	Mountain	9	4	3	3	6	6	4	5
14	Pacific	9	7	3	3	6	7	4	4
15	Mountain	10	1	4	4	6	6	3	4
16	Pacific	10	2	3	3	7	7	3	4
17	Mountain	6	7	4	3	6	7	3	4

at -10° to -20°C. These jars were packed on dry ice for overnight shipment to MRI. Upon receipt at MRI the specimens were checked to determine that they were intact and frozen. The specimens were checked versus the cooperators quota, an approximate specimen weight was determined and the specimens were transferred to storage at -20°C. As part of the check-in process, the patient summary reports (PSRs) were cross checked for consistency with the specimen labels and identification data. The PSRs were forwarded to Battelle for processing.

4.3 SAMPLE COLLECTION SUMMARY

In FY87, 956 specimens were collected from the cooperators. Of these, 771 were collected in accordance with the quotas and subquotas. The rest of the specimens were collected by cooperators, but went beyond what the quotas and subquotas requested.

In designating which collected specimens to chemically analyze, EPA decided to include some specimens that were collected outside of the subquotas. This was done in the anticipation of the use of the linear model, with parameters for the effects of Census region, age group, sex, and race group, to statistically analyze the results. The maximum number of specimens from a MSA remained at the original quota of twenty-seven or fifty-four. In effect, the MSA quotas were maintained, but the MSA subquotas were allowed to vary from what was specified in the design. This approach, while not ideal, was the best means available to deal with MSA non-response. Of the 956 collected specimens, 865 were designated for chemical analysis. These 865 specimens were labeled "Design" specimens.

Table 4-4 is a summary of the collection effort for the FY87 NHATS. Table 4-4 shows collection information for the nine Census divisions. In FY87, EPA chose not to make estimates for EPA regions. Instead, EPA maintained similarity to the FY82 geographic classifications in order to compare FY87 results to FY82 results.

Table 4-5 shows the number of quota specimens, collected specimens, and Design specimens in each category defined by the four analysis factors in the linear model. Because the number of samples in the chemical analysis was not large enough to obtain reliable estimates for all nine Census divisions, the divisions were combined into four Census regions for both the FY82 and FY87 model analyses.

Table 4-4. FY87 NHATS Collection Summary

Census division	Number of sample MSAs	Number of quota specimens	Number of cooperating MSAs	Number of collected specimens	Number of design specimens[a]
New England	2	54	2	33	33
Middle Atlantic	7[b]	216	7[b]	162	142
South Atlantic	8	216	7	228	175
East South Central	3	81	3	48	44
West South Central	5	135	3	73	70
East North Central	8[c]	270	7[c]	199	199
West North Central	5	135	4	108	97
Mountain	2	54	2	29	29
Pacific	7[b]	216	6[b]	76	76
Total	47	1,377	41	956	865

[a] Resulting after the Design/Surplus Indicator was assigned to each specimen.

[b] Includes one double collection MSA.

[c] Includes two double collection MSAs.

Table 4-5. FY87 NHATS Sample Sizes by Categories

Analysis factor	Category	Number of quota specimens	Number of collected specimens	Number of design specimens
Census Region	Northeast	270	195	175
	North Central	405	307	296
	South	432	349	289
	West	270	105	105
	Total	1,377	956	865
Age	0-14 years	311	163	146
	15-44 years	630	353	318
	45+ years	436	440	401
	Total	1,377	956	865
Sex	Male	668	499	436
	Female	709	457	429
	Total	1,377	956	865
Race	Caucasian	1,157	776	707
	Non-Caucasian	220	180	158
	Total	1,377	956	865

5.0 COMPOSITE DESIGN

The 865 design specimens in the FY87 NHATS were assigned to composite samples using specific composite design criteria (Leczynski et al. 1988). The reasons for compositing samples prior to chemical analysis were: (1) at least 10 grams of tissue were needed to meet the limit of detection goals for the target compounds, and (2) the budget for chemical analysis of samples could only support the analysis of 48 samples.

5.1 DESIGN GOALS AND COMPOSITING CRITERIA

The seven goals of the FY87 composite design, listed in order of importance, were

- Create no more than 48 composite samples.
- Maintain similarity to the FY82 composite design.
- Maintain equal tissue mass of individual specimens within the composite samples.
- Specify more pure sex composite samples than in FY82.
- Control the MSA effect.
- Provide the best range of race group percentages across the composite samples.
- Maintain a constant number of specimens across all composite samples.

Because of the constraints imposed by the sampling and compositing protocols and the frequency of collection nonresponse, it was not possible to meet all of the design goals. Each of the last six goals required a different mix of individual specimens within the composite samples. Thus, attempts were made to achieve all goals across the design to the extent possible. The criteria used to design composite samples are discussed below.

(1) Create no more than 48 composite samples.

This criterion was based entirely on the funds available for the chemical analysis.

(2) Maintain similarity to FY82 composite design.

This specification was included to ensure that comparisons between the analysis results from the two years could be made. The design criterion imposed by this objective is that each composite sample had to be constructed from individual specimens collected from exactly one Census division and exactly one age group. Thus, there were 27 distinct categories defined by the intersection of the nine Census divisions and three age groups.

(3) Maintain equal weighing of specimens within the composite samples.

The interpretation of the observed concentrations of the composite samples is far easier when these concentrations can be interpreted as the arithmetic average of the concentrations of the individual specimens. Therefore, this design goal specified that each individual specimen within a composite sample contribute an equal amount of tissue to that composite sample. This specification allows the lipid adjusted concentration of the composite sample to be interpreted as approximately the arithmetic average of the lipid adjusted individual specimen concentrations, with equality whenever all the specimens in the composite sample have the same percentage of lipid material.

(4) Construct more pure sex composites than in FY82.

Pure male or pure female composite samples were constructed when sufficient numbers of specimens were available within a particular Census division/age group category. Pure sex composite samples are samples in which all of the individual specimens were collected from donors of the same sex. Such composites are needed to achieve more precise estimates of sex effects in the population. It was anticipated that including more pure sex composite samples in the FY87 design will lead to smaller standard errors for the sex group estimates (Draper and Smith 1981, pp. 52-55) than was the case in FY82.

(5) Control the effect of the MSAs contributing specimens to each composite.

Controlling the number of MSAs contributing specimens to composite samples is intended to reduce the effect of the MSA on the estimated average concentrations. This is done because MSAs are regarded as being major sources of differences in observed concentrations across the nation due to their varied exposure scenarios (Panebianco 1986b). To avoid confounding the MSA effect with any of the geographic or demographic effects, two design criteria were identified. They were

(5-a) Keep the number of MSAs represented in each composite sample consistent across the design (2-3 MSAs was the target number), and

(5-b) Maintain approximately the same number of pure male and pure female composite samples within a group of MSAs.

The first criterion helps to ensure a constant variance of measured concentrations across the sample whenever the composite sample concentrations are averages over an equal number of MSAs. The second criterion is intended to prevent confounding a large MSA effect with the sex effect.

(6) Vary the percentage of Caucasian and non-Caucasian specimens in the composites as much as possible.

For the same reasons it is important to construct pure sex composite samples, it is important to construct pure race group composite samples. However, this goal is dependent on the number of non-Caucasian specimens collected in the twenty-seven Census division/age group categories and the number of composite samples in the design. Since the number of non-Caucasian specimens collected in the FY87 sample was relatively small, it was decided to provide the best possible range of race group percentages (i.e., mixes of the Caucasian and non-Caucasian specimens within the composite samples) across the design rather than focus on designing pure race group composite samples.

(7) Maintain a constant number of specimens across all composite samples.

This goal, similar to goal 5-a above, could not be fully achieved for the FY87 composite samples.

5.2 LABORATORY COMPOSITING PROCEDURES

The FY87 NHATS specimens from nine census divisions and three age groups were divided into 48 composites, as identified in the composite design provided by Battelle-Columbus Laboratories (Battelle). Battelle provided MRI with the data sheets that identified the individual specimens and their required weights to be included in each composite. Each composite consisted of from 3 to 32 specimens. The composite sample data sheets provided sufficient information (EPA ID number, package number, sample weight, hospital code, etc.) such that the individual specimens could be cross-checked with the study design. The data sheets provided by Battelle were used as work sheets to record the actual laboratory compositing procedures.

Initially, the samples were grouped into composites, and any samples of questionable weights were noted. Three samples identified below did not appear to have the weight required by the composite design. These results were relayed to the EPA Work Assignment Manager (WAM). After consultation with DDB, the EPA WAM forwarded to MRI the following responses regarding the problem samples on June 24, 1988.

Composite number	Sample number	Problem	Response
ACD8700023	8706954	Low weight, ~ 0.1 g need 0.5 g	Include as is
ACD8700032	8701765	No sample	Omit
ACD8700201	8703464	Low weight, ~ 1.3 g need 2.0 g	Include as is

The specimens in a composite were placed on dry ice during the compositing procedure. An electronic four-place balance was used to weigh the samples. The calibration of the balance was checked before any weighing was begun and once during the sample weighings with a Class P set of weights (laboratory grade, tolerance 1/25,000).

To weigh the samples, a clean culture tube was labeled with the composite number and placed on the balance and the weight tared. A specimen jar was opened, and a portion of the frozen adipose tissue removed with a clean stainless steel spatula. The adipose tissue was placed in the culture tube and the weight recorded to three decimal places on the compositing sheets. Additional adipose tissue was added if necessary. A goal of ±10% of the desired weight was attempted where possible. The specimen jar was capped and returned to storage. The weights of the individual specimens were recorded on the data sheets provided by Battelle.

The weight of the culture, beaker, and adipose tissue was rezeroed, and the next specimen in the composite was weighed. A new spatula was used for each sample. This procedure was repeated for each sample in the composite. When the composite was completed, it was capped and stored in a sample freezer at -10° to -20°C. All data on the actual compositing procedures were recorded on the data sheets provided by Battelle. All data sheets were submitted in a separate report to document the compositing activity (Cramer and Stanley 1988).

5.3 SUMMARY OF COMPOSITE SAMPLES

The FY87 NHATS Composite Design resulted in the construction of 48 composite samples, using the 865 design specimens collected from 41 MSAs. Table 5-1 shows the number of composite samples for the 27 distinct combinations of Census division and age group. The sex and race group percentages of the composite samples vary across the design depending on the availability of specimens within specific demographic subpopulations. Table 5-2 shows the demographic makeup of the FY87 NHATS composite samples.

The 48 composite samples were randomly assigned to five batches. Within batches, the composite samples were placed in random order for the chemical analysis.

Table 5-1. Distribution of FY87 NHATS Composite Samples by Census Division and Age Group

	Age group			
Census division	0-14 Years	15-44 Years	45+ Years	Total
New England	1	1	1	3
Middle Atlantic	1	3	2	6
South Atlantic	2	4	4	10
East South Central	1	1	1	3
West South Central	1	1	1	3
East North Central	2	3	5	10
West North Central	1	2	2	5
Mountain	1	1	1	3
Pacific	1	1	3	5
Total	11	17	20	48

Table 5-2. Demographic Makeup of FY87 NHATS Composite Samples

Census Division	Composite ID	Age Group[a]	Number of Individual Specimens	Number of MSAs	Percent Male	Percent Caucasian
New England	ACD8700238	1	3	2	100	100
	ACD8700247	2	12	2	25	100
	ACD8700256	3	18	2	44.4	100
Middle Atlantic	ACD8700149	1	20	6	70	65.0
	ACD8700158	2	18	5	0	83.3
	ACD8700167	2	20	3	100	85.0
	ACD8700176	2	20	3	100	90.0
	ACD8700185	3	32	6	0	90.6
	ACD8700194	3	32	6	100	96.9
South Atlantic	ACD8700318	1	14	5	0	42.9
	ACD8700327	1	17	6	100	41.2
	ACD8700336	2	17	3	100	47.1
	ACD8700345	2	19	4	100	73.7
	ACD8700354	2	19	4	0	84.2
	ACD8700363	2	14	3	0	64.3
	ACD8700372	3	20	4	0	85.0
	ACD8700381	3	17	3	0	82.4
	ACD8700390	3	23	4	100	69.6
	ACD8700407	3	15	3	100	80.0
East South Central	ACD8700112	1	14	2	50	92.9
	ACD8700121	2	13	2	46.2	61.5
	ACD8700130	3	17	2	41.2	88.2
West South Central	ACD8700461	1	13	3	61.5	53.8
	ACD8700470	2	32	3	62.5	71.9
	ACD8700489	3	25	3	56.0	88.0

Table 5-2 (Continued)

Census Division	Composite ID	Age Group[a]	Number of Individual Specimens	Number of MSAs	Percent Male	Percent Caucasian
East North Central	ACD8700014	1	18	6	100	44.4
	ACD8700023	1	20	5	0	85.0
	ACD8700032	2	18	3	44.4	77.8
	ACD8700041	2	16	3	100	81.3
	ACD8700050	2	27	4	0	85.2
	ACD8700069	3	20	2	100	85.0
	ACD8700078	3	16	2	0	87.5
	ACD8700087	3	17	2	100	76.5
	ACD8700096	3	23	2	0	82.6
	ACD8700103	3	24	3	45.8	100
West North Central	ACD8700416	1	17	3	41.2	76.5
	ACD8700425	2	19	4	0	94.7
	ACD8700434	2	24	4	100	91.7
	ACD8700443	3	19	4	0	100
	ACD8700452	3	18	4	100	88.9
Mountain	ACD8700201	1	5	1	80	100
	ACD8700210	2	14	2	57.1	85.7
	ACD8700229	3	10	2	40.0	100
Pacific	ACD8700265	1	5	2	20	100
	ACD8700274	2	16	4	18.8	87.5
	ACD8700283	3	24	6	100	79.2
	ACD8700292	3	16	3	0	93.8
	ACD8700309	3	15	3	0	93.3

[a] Age groups 1 = 0-14 years; 2 = 15-44 years; 3 = 45+ years.

6.0 CHEMICAL ANALYSIS PROCEDURES AND QUALITY CONTROL DATA

The 48 composite samples were prepared in the analysis laboratory for determination of low pg/g (ppt) levels of PCDDs and PCDFs using high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS). The performance of the analysis effort was demonstrated through the determinations of PCDDs and PCDFs in 20 quality control samples (method blanks, controls, and spiked tissues). Chemical analysis performance was also documented through participation in an interlaboratory effort with two other laboratories recognized for their expertise in the determination of PCDDs and PCDFs in human tissues. This section describes the analytical methodology and presents the results (analytical and statistical) for the quality control samples. A detailed presentation of the analytical results for all PCDDs and PCDFs in the FY87 NHATS design samples and the quality control samples is given in Appendix A.

6.1 SAMPLE PREPARATION

The preparation of the composited adipose tissue specimens for determination of PCDDs and PCDFs required a multistep procedure, which included quantitative extraction and cleanup through several chromatographic columns. The procedures described below were carried out for each of the five sample batches for the FY87 NHATS study.

6.1.1 Extraction

After compositing, the adipose tissue samples were stored at -20°C in 50-mL culture tubes sealed with aluminum foil. The extraction procedure was initiated by allowing the samples to come to room temperature and then fortifying them with 100 μL of an internal quantitation standard (IQS) spiking solution (Table 6-1) containing nine carbon-13 labeled PCDDs and PCDFs. Ten milliliters of methylene chloride was added and the sample was homogenized for 1 min with a Tekmar Tissuemizer®. The mixture was allowed to separate, and the methylene chloride was decanted through a funnel of sodium sulfate into a 100-mL volumetric flask. The homogenization was repeated two to three times with fresh 10-mL portions of methylene chloride. The culture tube was rinsed with additional methylene chloride and the remaining contents of the tube transferred to the funnel. Finally, the funnel was rinsed with additional methylene chloride and the final volume was brought to 100 mL.

At this point the flask was stoppered and inverted several times to mix the extract. Next, a 1-mL aliquot was removed with a disposable graduated pipet and placed into a preweighed (measured to 0.0001 g) 1-dram glass vial. The methylene chloride in the vial was reduced under

Table 6-1. Internal Standard Spiking Solution for chlorinated Species[a]

Analyte	Concentration (pg/μL)
Chlorinated Internal Quantitation Standards[b]	
$^{13}C_{12}$-2,3,7,8-TCDD	5
$^{13}C_{12}$-2,3,7,8-TCDF	5
$^{13}C_{12}$-1,2,3,7,8-PeCDD	5
$^{13}C_{12}$-1,2,3,7,8-PeCDF	5
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	12.5
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	12.5
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	12.5
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	12.5
$^{13}C_{12}$--OCDD	25
Internal Recovery Standard[c]	
$^{13}C_{12}$-1,2,3,4-TCDD	50
$^{13}C_{12}$-1,2,3,7,8-HxCDD	125

[a] All internal quantitation and recovery standards were obtained as solutions from Cambridge Isotope Laboratories (Woburn, Massachusetts).
[b] Prepared in isooctane. One hundred microliters spiked. Separate solutions were used for chlorinated and brominated species.
[c] Prepared in tridecane. Used for both chloro and bromo analyses.

flowing nitrogen until a constant weight of lipid was obtained. The weight of the lipid was obtained by difference, and the percent lipid for the composite was calculated and recorded.

The remaining portion of the extract (99 mL) was quantitatively transferred, followed by a 20- to 30-mL rinse, to a 500-mL round-bottomed flask. The extract was concentrated under vacuum to an oily residue (extractable lipids) using rotary evaporation.

6.1.2 Bulk Lipid Removal

Separation and concentration of the PCDDs and PCDFs from the lipids to achieve a final volume of 10 µL is necessary to detect pg/g concentrations. The extractable lipids from some of the composites was as high as 9 g of oily materials. The bulk lipid was removed following an acidic silica gel slurry cleanup procedure . This was accomplished by adding 200 mL of hexane and a Teflon-coated stirring bar to the lipid in the round-bottomed flask. Then, while stirring the extract on a magnetic stir plate, 100 g of 40% w/w sulfuric acid-impregnated silica gel was slowly added to the extract. The mixture was stirred for 2-hours. During the 2-hour slurry period, acid/neutral silica gel columns (4 g 40% H_2SO_4/silica gel, 1 g silica gel) were prepared. After the 2-hour period, the slurry mixture was allowed to settle, and the hexane was decanted off the acid impregnated silica gel through a funnel of sodium sulfate into the acid/neutral silica gel column. The slurry mixture was rinsed for 15 minutes with two additional aliquots (50 mL) of hexane. The rinses were added to the silica gel column through the sodium sulfate funnel. The eluate of the column was collected in a 500-mL Kuderna-Danish evaporation flask. An additional 50 mL of hexane was placed onto the column when the solvent level had reached the level of the chromatographic packing. The extract was then reduced in volume over a steam bath and the final volume adjusted to approximately 1 mL using nitrogen blowdown.

6.1.3 Separation of Chemical Interferences

Separation of chemical interferences, such as pesticides, PCBs, and other chlorinated planar aromatics is essential to avoid false positive measurements. Removal of chemical interference was achieved using two different chromatographic cleanup systems. The first was prepared as a layered column containing 1 g sodium sulfate, 4 g neutral alumina, and 1 g sodium sulfate. The 1 mL extract from the acid/neutral silica gel column was transferred to the alumina column, followed by two 1-mL portions of hexane and 10 mL of 8% (volume/volume, v/v) methylene chloride in hexane. These eluents were collected and archived. The PCDDs and PCDFs were eluted from the column with 15 mL of 60% (v/v) methylene chloride in hexane and the eluent concentrated under a stream of nitrogen to approximately 2 mL.

A disposable column of AX-21 on silica gel was prepared and preeluted with 4 mL of toluene, 2 mL of 75:20:5 methylene chloride/methanol/benzene, and 2 mL of 1:1 cyclohexane/ methylene chloride. The concentrated eluate from the alumina column was added to the AX-21/silica gel column followed by two 1-mL hexane rinses. The column was eluted sequentially with two 0.5-mL aliquots of hexane, 10 mL of 1:1 cyclohexane/methylene chloride, and 5 mL of 75:20:5 methylene chloride/methanol/benzene. These eluents were combined and archived. The columns were turned upside down and the PCDDs and PCDFs eluted with 20 mL of toluene. The extract was then reduced in volume to approximately 100 µL, then 10 µL of recovery standard in tridecane was added (Table 6-1) and the volume was further reduced to 10 µL under nitrogen. The extract was stored in a freezer pending HRGC/HRMS analysis.

6.2 HRGC/HRMS ANALYSIS

Initial calibration of the GC/MS system was conducted by making single 1-µL injections of the standards listed in Table 6-2. Relative response factors calculated from this calibration effort are shown in Table 6-3. A CS7 (2.5 to 12.5 pg/µL) standard was analyzed on a daily basis to ensure adherence to the initial calibration curve. The traceability and comparability of the analytical standards has been demonstrated in a previous NHATS effort (USEPA 1986) and through participation in an interlaboratory comparison study (Bradley, et al. 1990).

HRGS/HRMS analysis of the samples was conducted after initial and routine calibration criteria were met. Prior to the injection of the first sample, an injection of tridecane was analyzed to document system cleanliness. If any evidence of system contamination was found, corrective action was taken by analyzing another tridecane blank or cleaning the injection system. A typical daily sequence of injections is shown in Table 6-4. A 1-µL aliquot of the extracts was injected into the GC/MS system, which was operated under the conditions that previously produced acceptable results with the daily calibration standard.

Selected ion monitoring (SIM) data were acquired according to the acquisition and MS operating conditions previously used to determine the relative response factors (Tables 6-5 and 6-6). Instrument performance was monitored by examining and recording the peak areas for the recovery standard, $^{13}C_{12}$1,2,3,4-TCDD. If this area decreased to less than 50% of the calibration standard, sample analyses were stopped until the problem was identified and corrected.

Table 6-2. Concentration Calibration Solutions for PCDD/PCDF

Analyte	Concentration in calibration solutions in pg/μL							
	CS0.5	CS1	CS2	CS3	CS4	CS5	CS6	CS7
2,3,7,8-TCDD	400	200	100	50	25	120	5	2.5
2,3,7,8-TCDF	400	200	100	50	25	10	5	2.5
1,2,3,7,8-PeCDD	400	200	100	50	25	10	5	2.5
1,2,3,7,8-PeCDF	400	200	100	50	25	10	5	2.5
2,3,4,7,8-PeCDF	400	200	100	50	25	10	5	2.5
1,2,3,4,7,8-HxCDD	1,000	500	250	125	62.5	25	12.5	6.25
1,2,3,6,7,8-HxCDD	1,000	500	250	125	62.5	25	12.5	6.25
1,2,3,7,8,9-HxCDD	1,000	500	250	125	62.5	25	12.5	6.25
1,2,3,4,7,8-HxCDF	1,000	500	250	125	62.5	25	12.5	6.25
1,2,3,6,7,8-HxCDF	1,000	500	250	125	62.5	25	12.5	6.25
2,3,4,6,7,8-HxCDF	1,000	500	250	125	62.5	25	12.5	6.25
1,2,3,7,8,9-HxCDF	1,000	500	250	125	62.5	25	12.5	6.25
1,2,3,4,6,7,8-HpCDD	1,000	500	250	125	62.5	25	12.5	6.25
1,2,3,4,6,7,8-HpCDF	1,000	500	250	125	62.5	25	12.5	6.25
1,2,3,4,7,8,9-HpCDF	1,000	500	250	125	62.5	25	12.5	6.25
OCDD	2,000	1,000	500	250	125	50	25	12.5
OCDF	2,000	1,000	500	250	125	50	25	12.5
Internal Quantitation Standards								
$^{13}C_{12}$-2,3,7,8-TCDD	50	50	50	50	50	50	50	50
$^{13}C_{12}$-2,3,7,8-TCDF	50	50	50	50	50	50	50	50
$^{13}C_{12}$-1,2,3,7,8-PeCDD	50	50	50	50	50	50	50	50
$^{13}C_{12}$-1,2,3,7,8-PeCDF	50	50	50	50	50	50	50	50
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	125	125	125	125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	125	125	125	125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	125	125	125	125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	125	125	125	125	125	125	125	125
$^{13}C_{12}$-OCDD	250	250	250	250	250	250	250	250
Internal Recovery Standard								
$^{13}C_{12}$-1,2,3,4-TCDD	50	50	50	50	50	50	50	50
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	125	125	125	125	125	125	125	125

Table 6-3. Initial Calibration Data--Relative Response Factors

Analyte	CS0.5	CS1	CS2	CS3	CS4	CS5	CS6	CS7	MEAN	% RSD
^{13}C-2,3,7,8-TCDF	2.292	2.116	2.128	2.170	2.134	2.080	2.171	2.161	2.157	2.9
^{13}C-2,3,7,8-TCDD	1.658	1.407	1.440	1.473	1.466	1.499	1.413	1.506	1.483	5.4
^{13}C-1,2,3,7,8-PeCDF	1.100	0.992	1.000	1.001	0.886	1.034	0.971	0.965	0.994	6.1
^{13}C-1,2,3,7,8-PeCDD	0.511	0.454	0.465	0.443	0.379	0.436	0.452	0.434	0.447	8.2
^{13}C-1,2,3,4,7,8-HxCDF	1.522	1.511	1.540	1.620	1.879	1.446	1.624	1.641	1.598	8.3
^{13}C-1,2,3,6,7,8-HxCDD	0.629	0.665	0.626	0.676	0.758	0.574	0.667	0.696	0.661	8.2
^{13}C-1,2,3,4,6,7,8-HpCDF	0.654	0.733	0.720	0.660	0.689	0.651	0.746	0.731	0.698	5.6
^{13}C-1,2,3,4,6,7,8-HpCDD	0.360	0.401	0.385	0.320	0.351	0.338	0.377	0.389	0.365	7.6
^{13}C-OCDD	0.218	0.257	0.218	0.184	0.190	0.216	0.239	0.233	0.219	11.1
2,3,7,8-TCDF	1.137	1.274	1.306	1.357	1.230	1.354	1.240	1.255	1.269	5.7
2,3,7,8-TCDD	0.995	1.037	1.136	1.149	1.023	1.129	1.034	1.308	1.101	9.3
1,2,3,7,8-PeCDF	1.230	1.203	1.131	1.180	1.181	1.218	1.278	1.068	1.186	5.4
2,3,4,7,8-PeCDF	1.294	1.307	1.300	1.282	1.336	1.201	1.192	1.476	1.299	6.8
1,2,3,7,8-PeCDD	1.660	1.587	1.578	1.623	1.682	1.488	1.520	1.490	1.579	4.7
1,2,3,4,7,8-HxCDF	1.172	1.167	1.186	1.221	1.200	1.315	1.338	1.524	1.265	9.7
1,2,3,6,7,8-HxCDF	1.286	1.291	1.278	1.253	1.234	1.302	1.482	1.177	1.288	6.8
2,3,4,6,7,8-HxCDF	1.120	1.182	1.089	1.043	0.960	0.968	1.061	1.244	1.083	9.1
1,2,3,7,8,9-HxCDF	1.048	1.067	1.068	0.995	0.885	0.915	1.042	0.918	0.992	7.6
1,2,3,4,7,8-HxCDD	1.575	1.562	1.577	1.498	1.404	1.284	1.247	1.287	1.429	9.9
1,2,3,6,7,8-HxCDD	1.823	1.624	1.733	1.537	1.720	1.925	1.392	2.281	1.754	15.4
1,2,3,7,8,9-HxCDD	1.791	1.795	1.790	1.626	1.457	2.047	1.680	1.429	1.702	11.8
1,2,3,4,6,7,8-HpCDF	1.600	1.475	1.468	1.523	1.536	1.498	1.418	1.156	1.459	9.2
1,2,3,4,7,8,9-HpCDF	1.172	1.148	1.039	1.012	0.993	0.955	0.970	0.881	1.021	9.6
1,2,3,4,6,7,8-HpCDD	1.291	1.327	1.281	1.337	1.403	1.411	1.334	1.210	1.324	4.9
OCDF	1.393	1.164	1.272	1.149	1.202	1.218	1.066	1.039	1.188	9.5
OCDD	1.128	0.996	1.125	0.909	1.192	0.979	1.049	0.968	1.043	9.3

Table 6-4. Typical Daily Sequence for PCDD/PCDF Analysis

1.	Tune and calibrate mass scale versus perfluorokerosene (PFK).
2.	Inject column performance/window-defining mixture.
3.	Inject concentration calibration solution 2.5 to 12.5 pg/μL (CS-7) solution.
4.	Inject blank (Tridecane).
5.	Inject samples 1 through "N."
6.	Inject concentration calibration solution 2.5 to 12.5 pg/μL (CS-7) solution or other concentration calibration solutions CS1 to CS8 to bracket observed sample concentration.

Table 6-5. Ions Monitored for HRGC/HRMS of PCDD/PCDF

Descriptor	ID	Mass	Nominal dwell time (s)
A1	TCDF	303.902	0.090
		305.899	0.090
	13C12-TCDF	315.942	0.090
		317.939	0.090
	TCDD	319.897	0.090
		321.894	0.090
	$^{13}C_{12}$-TCDD	331.937	0.090
		333.934	0.090
	HxCDPE	377.886	0.090
	PFK (lock mass)	380.976	0.090
A2	TCDF	303.902	0.045
		305.899	0.045
	TCDD	319.897	0.045
		321.894	0.045
	PeCDF	339.863	0.045
		341.860	0.045
	$^{13}C_{12}$-PeCDF	351.900	0.045
		353.894	0.045
	PeCDD	355.858	0.045
		357.855	0.045
	$^{13}C_{12}$-PeCDD	367.895	0.045
		369.889	0.045
	PFK (lock mass)	380.976	0.035
	HpCDPE	409.877	0.035
A3	HxCDF	373.821	0.080
		375.818	0.080
	PFK (lock mass)	380.976	0.080
	$^{13}C_{12}$-HxCDF	383.861	0.080
		385.858	0.080
	HxCDD	389.816	0.080
		391.813	0.080
	$^{13}C_{12}$-HxCDD	401.856	0.080
		403.853	0.080
	OCDPE	445.866	0.080

Table 6-5 (continued)

Descriptor	ID	Mass	Nominal dwell time (s)
A4	PFK (lock mass)	380.976	0.040
	HxCDD	389.816	0.040
		391.813	0.040
	HpCDF	407.782	0.040
		409.779	0.040
	$^{13}C_{12}$-HpCDF	417.822	0.040
		419.819	0.040
	HpCDD	423.777	0.040
		425.774	0.040
	$^{13}C_{12}$-HpCDD	435.817	0.040
		437.814	0.040
	$^{13}C_{12}$-HpCDD	429.768	0.040
		431.765	0.040
	NCDPE	479.856	0.040
A5	PFK (lock mass)	380.976	0.06
	OCDF	441.743	0.07
		443.740	0.07
	$^{13}C_{12}$-OCDF	453.783	0.07
		455.780	0.07
	OCDD	457.738	0.07
		459.735	0.07
	$^{13}C_{12}$-OCDD	469.779	0.07
		471.776	0.07
	DCDPE	513.846	0.06

Table 6-6. HRGC/HRMS Operating Conditions for PCDD/PCDF Analysis

Mass spectrometer (Kratos MS50-TC)	
Accelerating voltage:	8,000 V
Trap current:	500 μA
Electron energy:	70 eV
Electron multiplier voltage:	-1,800 V
Source temperature:	280°C
Resolution:	≥ 10,000 (10% valley definition)
Overall SIM cycle time:	1 s
Gas chromatograph (Carlo Erba MFC-500)	
Column coating:	DB 5
Film thickness:	0.25 μm
Column dimensions:	60 m x 0.25 mm ID
He linear velocity:	~ 25 cm/s
He head pressure:	1.75 kg/cm^2 (25 psi)
Injection type:	Splitless, 45 s
Split flow:	30 mL/min
Purge flow:	6 mL/min
Injector temperature:	270°C
Interface temperature:	300°C
Injection size:	1-2 μL
Initial temperature:	200°C
Initial time:	2 min
Temperature program:	200°C to 270°C at 5°C/min
Second hold time:	10 min
Second temperature ramp:	270° to 330°C at 5°C/min
Final hold time:	5 min

6.3 QA/QC FOR CHEMICAL ANALYSIS

The QA/QC program for this analysis effort included: demonstration of instrumental performance, routine analysis of quality control samples (method blank, controls and spiked tissues), analysis of performance audit samples and participation in an interlaboratory effort for comparison of results on specific samples. Each of these QA/QC efforts and results are discussed.

6.3.1 Instrument Performance

Instrument performance was characterized primarily by three criteria: (1) mass resolution (≥ 10,000) and calibration; (2) relative response factors (RRF), i.e., adherence to the initial RRFs; and (3) column performance as indicated by peak separation between 2,3,7,8-TCDD and other TCDD isomers.

6.3.1.1 Mass Resolution and Calibration

The mass spectrometer was tuned on a daily basis to yield optimum sensitivity and peak shape using an ion *m/z* 380.9760) from PFK. The resolution was visually monitored and maintained at ≥ 10,000 (10% valley definition) to provide adequate noise rejection while maintaining good ion transmission. Static-resolving power checks were performed at the beginning and at the end of each 12-hour operation period. A visual check (i.e., documentation was not required) of the static resolution was made by using the peak matching unit before and after each analysis. Corrective action was implemented whenever the resolving power did not meet the criteria of ≥ 10,000.

Mass calibration of the mass spectrometer for the HRGC/MS analysis of PCDD/PCDF was conducted on a daily basis. The magnetic field was adjusted to pass *m/z* 300 at full accelerating voltage. PFK was admitted to the MS, and an accelerating voltage scan from 8,000 to 4,000 v was acquired by the data system. This corresponded to an effective mass range of 301 to 593 amu. Upon completion of a successful calibration step, the five ion descriptors shown in Table 6-5 were updated to reflect the new mass calibration.

6.3.1.2 Relative Response Factor

As part of the initial and routine instrument performance checks, calibration standards were analyzed and the responses of the respective analytes were compared to specific internal standards to establish the RRF values. The initial and routine calibration criteria required that the precision of the RRF measurements be within ±20% for the tetrachloro congeners and within ±30% for the other compounds.

Sensitivity of the HRMS was documented through the responses noted for the first calibration standard of each analysis day. The method required the analysis of a low level standard (CS7) to document sufficient instrumental response to support instrumental detection limits of 1 pg/μL for TCDD.

Routine checks on the instrument sensitivity, which were documented in the MS log book, was achieved by monitoring the response for the internal recovery standard ($^{13}C_{12}$-1,2,3,4-TCDD) from injection to injection. If the response for this standard dropped by greater than 50% of the response noted in the previous calibration standard, the analyst verified instrumental performance by analyzing an additional calibration standard. Additional details on the initial and routine calibration events are presented in the data reports provided to EPA (Cramer et al. 1989a, 1989b).

6.3.1.3 TCDD Peak Separation

The HRGC column performance was demonstrated at the start of each 12-h analysis period. This was accomplished by injecting 1 μL of the column performance/window-defining check solution and acquiring SIM data for all PCDD and PCDF compounds. The HRGC column performance was determined based on the ability to resolve 2,3,7,8-TCDD from possible coeluting TCDDs.

The chromatographic peak separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers was resolved with a valley of ≤ 25%, where

$$\textbf{valley \%} = (x/y)(100)$$

x = measured height of the valley between the chromatographic peak corresponding to 2,3,7,8-TCDD and the peak of the nearest TCDD isomer

y = peak height of 2,3,7,8-TCDD

Figure 6-1 is an example of the separation of a TCDD isomer mixture and the calculation of isomer resolution. The TCDD isomer resolution was documented to range from 18% to 25% during the analysis effort (Cramer et al. 1989a, 1989b)

6.3.2 QC Samples

Samples included for QC purposes are summarized in Table 6-7. Each of these quality control samples are described in further detail below. These quality control samples were included with the analysis of the FY87 samples. The order of preparation and analysis with respect to the FY87 NHATS composites was specified in the study design.

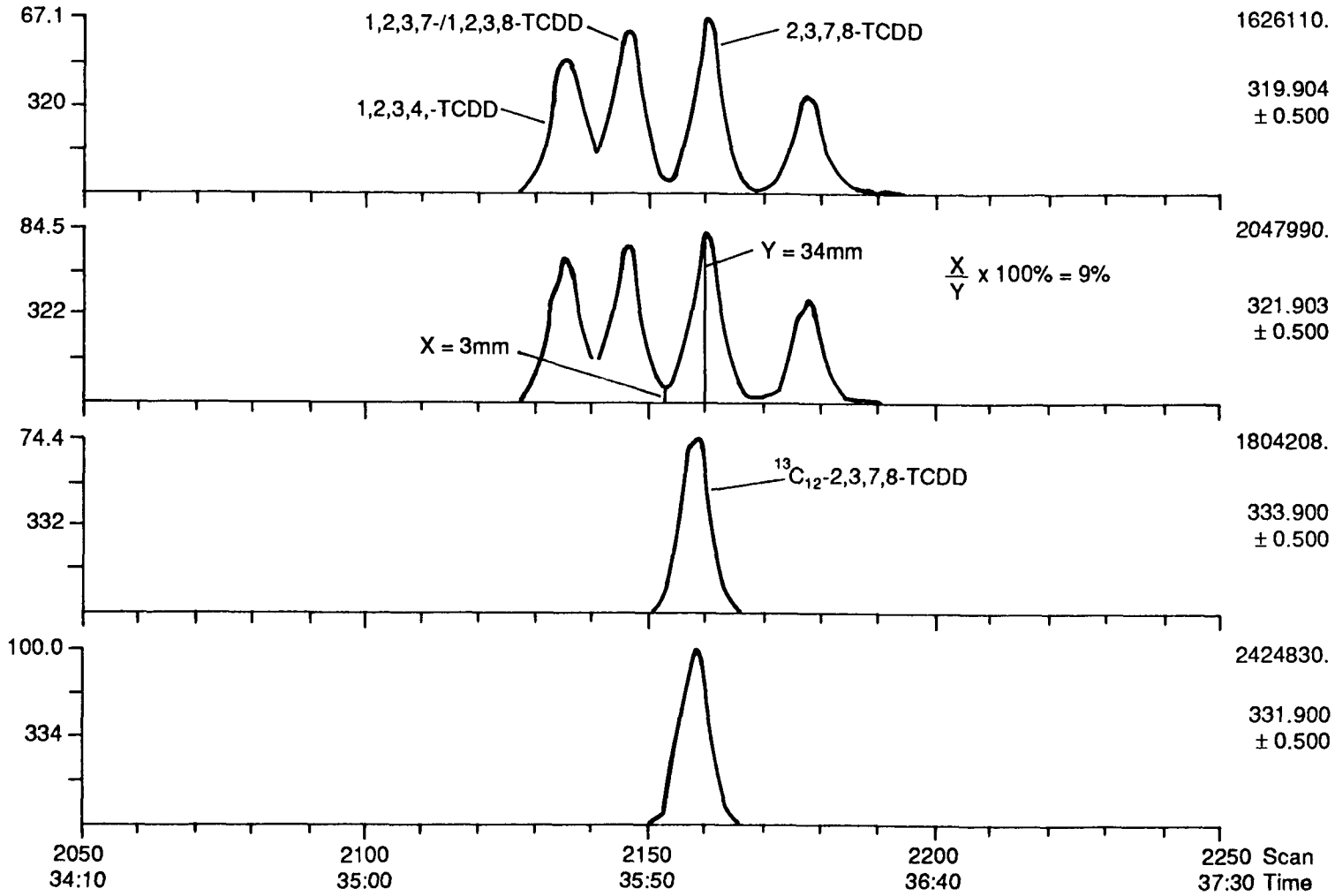


Figure 6-1. Example of the separation of 2,3,7,8-TCDD from other TCDD isomers on a 60-m DB5 column.

Table 6-7. Quality Control Samples

Type	Frequency	Application
Method blank	One per batch	Assess laboratory background contribution
Spiked control adipose tissue sample	Two per batch (two different spike levels)	Evaluate method performance (accuracy and precision)
Unspiked control adipose tissue sample	One per batch	Evaluate method performance (accuracy and precision)

6.3.2.1 Method Blanks

One method blank was generated with each batch of samples. A method blank was generated by performing all steps detailed in the analytical procedure using all reagents, standards, equipment, apparatus, glassware, and solvents that were used for a sample analysis, but omitting the addition of the adipose tissue. The method blank contained the same amounts of carbon-13 labeled internal quantitation standards that were added to samples before bulk lipid cleanup. The five method blanks analyzed with the samples did not contain PCDDs or PCDFs with the exception of the method blank generated for Batch 1 samples. This method blank contained a trace of 2,3,7,8-TCDF which was determined to be equivalent to 0.46 pg/g of tissue. The detailed analysis results for the method blanks are presented in Appendix A.

6.3.2.2 Control Samples

Control samples were prepared from a bulk sample of human adipose tissue. This material was prepared by blending the tissue with methylene chloride, drying the extract by eluting through anhydrous sodium sulfate, and removing the methylene chloride using rotoevaporation at elevated temperatures (80°C). The evaporation process was extended to ensure that all traces of the extraction solvent had been removed. The resulting oily matrix (lipid) was subdivided into 10-g aliquots which were analyzed with each sample batch. A summary of the QC sample results is presented in Section 6.5. A detailed presentation of this data by analyte is given in Appendix A.

6.3.2.3 Internal Spiked Control Samples

Spiked lipid samples were prepared using a portion of the homogenized control lipid. Sufficient spiked lipid matrix was prepared to provide a minimum of two spiked samples, one low and one high, per sample batch. The native spiking solution concentrations are shown in

Table 6-8. Additional OCDD was added to each sample with a 75 pg/µL spiking solution. Low and high spike levels in the control samples are shown in Table 6-9.

The spiking solutions were checked for accuracy prior to spiking the adipose composite with the native isomers. The results of this spike check are shown in Table 6-10. The spike check results for the separate OCDD spiking solution (needed to reach the higher level for OCDD in the adipose tissue) are given in Table 6-11. A summary of the results from the analysis of these spiked materials is presented in Section 6.5. The results are presented for each spiked sample in Appendix A.

6.3.3 Performance Audit Samples (PAS)

Performance audit samples (PAS) were submitted for analysis before the first sample was analyzed with batch 1 and batch 5. These samples consisted of unlabeled PCDDs and PCDFs in a solvent matrix. The samples were prepared by the project quality control coordinator (QCC) and turned over to project personnel who fortified the samples with IQS and RS solutions and submitted the prepared sample for HRGC/HRMS analysis. The performance audit samples were prepared from a mixture of standards used for an interlaboratory effort to establish consensus values for concentrations (Bradley et al 1989). Results from the analysis were given directly to the QCC. Acceptability criteria were 70% to 130% accuracy for each of the isomers in the sample.

Table 6-12 provides a summary of the results from the analysis of the two performance audit samples. As presented the measurements were within the criteria specified for each analyte.

6.3.4 Interlaboratory Comparisons

External QC samples and solutions were submitted to two outside laboratories. The contacts and laboratories were Dr. Donald Patterson with the Centers for Disease Control in Atlanta and Dr. John J. Ryan with the Health Protection Branch in Canada. Each laboratory received one spike check solution sealed in an amber ampule and three blind control lipid samples. The lipid samples were spiked identically to those used with the analysis of the FY87 NHATS samples and included an unspiked sample, a low level spike sample, and a high level spike sample. Data from the interlaboratory comparison are presented in Tables 6-13 through 6-16. These data demonstrate that although some differences were apparent in the analytical standards and hence the results for the analysis of the quality control samples, the data for the respective compounds from the laboratories are generally within 30% relative percent difference.

Table 6-8. PCDD and PCDF Native Spiking Solution[a]

Analyte	Concentration (pg/μL)
2,3,7,8-TCDD	5
2,3,7,8-TCDF	5
1,2,3,7,8-PeCDD	5
1,2,3,7,8-PeCDF	5
2,3,4,7,8-PeCDF	5
1,2,3,4,7,8-HxCDD	12.5
1,2,3,6,7,8-HxCDD	12.5
1,2,3,7,8,9-HxCDD	12.5
1,2,3,4,7,8-HxCDF	12.5
1,2,3,6,7,8-HxCDF	12.5
1,2,3,7,8,9-HxCDF	12.5
2,3,4,6,7,8-HxCDF	12.5
1,2,3,4,6,7,8-HpCDD	12.5
1,2,3,4,6,7,8-HpCDF	12.5
1,2,3,4,7,8,9-HpCDF	12.5
OCDD	25[b]
OCDF	25

[a] Prepared in isooctane. This solution also contained similar concentrations of the available brominated dioxin and furan congeners.

[b] The level of OCDD was adjusted based on the endogenous level of OCDD in adipose tissue. An additional, separate solution of OCDD at 75 pg/μL was used to achieve the higher spiking level needed.

Table 6-9. Control Sample Spike Levels

Analyte	Low spike level (pg/g)	High spike level (pg/g)
2,3,7,8-TCDF	10	50
2,3,7,8-TCDD	10	50
1,2,3,7,8-PeCDF	10	50
2,3,4,7,8-PeCDF	10	50
1,2,3,7,8-PeCDD	10	50
1,2,3,4,7,8-HxCDF	25	125
1,2,3,6,7,8-HxCDF	25	125
2,3,4,6,7,8-HxCDF	25	125
1,2,3,7,8,9-HxCDF	25	125
1,2,3,4,7,8/1,2,3,6,7,8-HxCDD	50	250
1,2,3,7,8,9-HxCDD	25	125
1,2,3,4,6,7,8-HpCDF	25	125
1,2,3,4,7,8,9-HpCDF	25	125
1,2,3,4,6,7,8-HpCDD	25	125
1,2,3,4,6,7,8,9-OCDF	50	250
1,2,3,4,6,7,8,9-OCDD	350	700

Table 6-10. PCDD and PCDF Spike Check Results

Analyte	Spike level (pg/µL)	Spike check 1		Spike check 2		Average recovery (%)
		(pg/µL)	Recovery	(pg/µL)	Recovery	
2,3,7,8-TCDF	50	44.3	89	41.1	82	85
2,3,7,8-TCDD	50	51.0	102	49.5	99	101
1,2,3,7,8-PCDF	50	47.3	95	56.8	114	104
2,3,4,7,8-PCDF	50	48.6	97	54.7	109	103
1,2,3,7,8-PCDD	50	46.5	93	46.1	92	93
1,2,3,4,7,8-HxCDF	125	124.0	99	121.3	97	98
1,2,3,6,7,8-HxCDF	125	119.4	96	116.2	93	94
2,3,4,6,7,8-HxCDF	125	104.0	83	105.3	84	84
1,2,3,7,8,9-HxCDF	125	93.0	74	107.0	86	80
1,2,3,4,7,8-HxCDD	125	115.9	93	115.1	92	92
1,2,3,6,7,8-HxCDD	125	118.4	95	122.6	98	96
1,2,3,7,8,9-HxCDD	125	107.3	86	114.5	92	89
1,2,3,4,6,7,8-HpCDF	125	114.3	91	114.4	92	91
1,2,3,4,7,8,9-HpCDF	125	114.9	92	99.7	80	86
1,2,3,4,6,7,8-HpCDD	125	115.4	92	118.0	94	93
OCDF	250	207.8	83	210.6	84	84
OCDD	250	226.7	91	224.6	90	90

Table 6-11. OCDD Spike Check Results

Replicate no.	Spike level (pg/µL)	Amount found (pg/µL)	Recovery %
1	150	142.6	95
2	150	154.6	103
3	150	138.1	92
Average recovery (%)			97

Table 6-12. Results of Performance Audit Samples Analyzed With the FY87 NHATS Composites

Actual	PAS 06091			PAS 06092		
	Actual concentration (pg/μL)	Found concentration (pg/μL)	% Accuracy	Actual concentration (pg/μL)	Found concentration (pg/μL)	% Accuracy
2,3,7,8-TCDF	100	90.8	91	20	17.8	89
2,3,7,8-TCDD	100	92.7	93	200	173	86
1,2,3,7,8-PeCDF	100	115	115	20	21.6	108
2,3,4,7,8-PeCDF	100	114	114	198	177	89
1,2,3,7,8-PeCDD	100	113	113	200	195	98
1,2,3,4,7,8-HxCDF	250	211	85	49.6	41.0	83
1,2,3,6,7,8-HxCDF	250	234	94	49.6	43.3	87
2,3,4,6,7,8-HxCDF	250	239	96	496	522	105
1,2,3,7,8,9-HxCDF	250	228	91	496	432	87
1,2,3,4,7,8-HxCDD	250	280	112	a	a	a
1,2,3,6,7,8-HxCDD	250	239	96	432	358	83
1,2,3,7,8,9-HxCDD	250	230	92	32	37.4	117
1,2,3,4,6,7,8-HpCDF	250	293	117	49.6	51.2	103
1,2,3,4,7,8,9-HpCDF	250	266	106	496	605	122
1,2,3,4,6,7,8-HpCDD	250	229	92	400	322	80
OCDF	500	632	126	49.6	59.1	119
OCDD	500	569	114	400	392	98

[a] The results for 1,2,3,4,7,8-HxCDD were summed and reported with the 1,2,3,6,7,8-HxCDD isomer. The results for 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD isomers were reported as a combined value in the analysis report forms because of the overlap of these peaks on the DB-5 column. However, where chromatographic resolution was sufficient to separate these isomers, each compound was quantitated separately and was reported as such in the remarks section of the respective analysis report form.

Table 6-13. Interlaboratory Comparison—Spike Check Solution

Analyte	Theoretical level (pg/μL)	Lab 1		Lab 2	
		Amt. found (pg/μl)	% Accuracy	Amt. found (pg/μL)	% Accuracy
2,3,7,8-TCDF	8.33	8.6	103	8.30	100
2,3,7,8-TCDD	8.33	8.0	96	6.95	83
1,2,3,7,8-PeCDF	8.33	9.1	109	6.47	78
2,3,4,7,8-PeCDF	8.33	11	132	6.83	82
1,2,3,7,8-PeCDD	8.33	9.5	114	7.28	87
1,2,3,4,7,8,-HxCDF	20.83	24	115	20.44	98
1,2,3,6,7,8-HxCDF	20.83	27	130	17.93	86
2,3,4,6,7,8-HxCDF	20.83	32	154	21.77	105
1,2,3,7,8,9-HxCDF	20.83	34	163	14.98	72
1,2,3,4,7,8-HxCDD	20.83	20	96	17.62	85
1,2,3,6,7,8-HxCDD	20.83	25	120	17.49	84
1,2,3,7,8,9-HxCDD	20.83	28	134	15.74	76
1,2,3,4,6,7,8-HpCDF	20.83	41	197	16.26	78
1,2,3,4,7,8,9-HpCDF	20.83	31	149	13.74	66
1,2,3,4,6,7,8-HpCDD	20.83	26	125	17.04	82
1,2,3,4,6,7,8,9-OCDF	41.70	47	113	NR[a]	-
1,2,3,4,6,7,8,9-OCDD	291.7	550	189	NR	-

[a] Not reported.

Table 6-14. Interlaboratory Comparison—Control Lipid Results (pg/g)

Analyte	Lab 1 n=1	Lab 2 n=4		Lab 3 n=5	
		Average	RSD (%)	Average	RSD (%)
2,3,7,8-TCDF	1.2	1.3	23.1	1.12[a]	-
2,3,7,8-TCDD	8	10.6	9.4	9.29	9.7
1,2,3,7,8-PeCDF	ND(1)[b]	ND(0.7)	-	0.48[c]	-
2,3,4,7,8-PeCDF	16	27.1	7.4	25.1	7.5
1,2,3,7,8-PeCDD	15	26.6	9.4	20.8	4.6
1,2,3,4,7,8-HxCDF	19	31.7	8.8	18.7[c]	-
1,2,3,6,7,8-HxCDF	9	14.6	13.0	10.8	21.4
2,3,4,6,7,8-HxCDF	1.6	3.0	20.2	ND(9.84)	-
1,2,3,7,8,9-HxCDF	ND(1)	ND(0.3)	-	ND(0.66)	-
1,2,3,4,7,8-HxCDD	11	20.2	11.9	-	-
1,2,3,6,7,8-HxCDD	99	131	5.3	136.8[d]	6.8
1,2,3,7,8,9-HxCDD	17	18.5	16.8	21.1[e]	8.4
1,2,3,4,6,7,8-HpCDF	36	31.5	18.1	29.8	6.3
1,2,3,4,7,8,9-HpCDF	ND(1)	ND(0.4)	-	1.42	-
1,2,3,4,6,7,8-HpCDD	132	212	7.5	140	6.9
1,2,3,4,6,7,8,9-OCDF	ND(1)	NR[f]	-	4.02[g]	70.6
1,2,3,4,6,7,8,9-OCDD	986	1030	6.9	1,184	3.7

[a] Avenge of two positive quantifiable or trace values (n=2).
[b] Not detected. Detection limit in parenthesis.
[c] One trace value (n=1).
[d] 1,2,3,4,7,8- and 1,2,3,6,7,8-HxCDD reported as total amount in Lab 3 results.
[e] Average of four positive quantifiable or trace values (n=4).
[f] Not reported.
[g] Average of three positive quantifiable or trace values (n=3).

Table 6-15. Interlaboratory Comparison—Low Level Spiked Lipid (% Recovery)

	Lab 1 n=1	Lab 2 n=4		Lab 3 n=5	
Analyte		Average	RSD (%)	Average	RSD (%)
2,3,7,8-TCDF	70	119	3.8	95	7.1
2,3,7,8-TCDD	70	98	4.4	83	23.3
1,2,3,7,8-PeCDF	56	91	6.6	115[a]	8.8
2,3,4,7,8-PeCDF	50	100	5.7	99[a]	17.9
1,2,3,7,8-PeCDD	70	110	8.8	94[b]	39.1
1,2,3,4,7,8-HxCDF	56	120	13.5	-[c]	-
1,2,3,6,7,8-HxCDF	56	109	12.7	95[a]	13.6
2,3,4,6,7,8-HxCDF	62	107	11.1	99[d]	35.2
1,2,3,7,8,9-HxCDF	68	95	5.1	79	15.1
1,2,3,4,7,8-HxCDD	60	88	8.5	-[e]	-
1,2,3,6,7,8-HxCDD	120	128	3.1	88[a]	32.3
1,2,3,7,8,9-HxCDD	72	93	3.1	95	20.0
1,2,3,4,6,7,8-HpCDF	100	103	5.6	114	14.6
1,2,3,4,7,8,9-HpCDF	76	133	9.6	115	4.2
1,2,3,4,6,7,8-HpCDD	56	260	7.6	89[a]	11.5
1,2,3,4,6,7,8,9-OCDF	56	NR[c]	-	99[a]	14.9
1,2,3,4,6,7,8,9-OCDD	64	NR	-	87[a]	13.3

[a] Average of four positive quantifiable or trace values (n=4).
[b] Average of three positive quantifiable or trace values (n=3).
[c] Diphenylether interference observed.
[d] Average of two positive quantifiable or trace values (n=2).
[e] 1,2,3,4,7,8- and 1,2,3,6,7,8-HxCDD isomers were reported as a sum.
[f] Not reported.

Table 6-16. Interlaboratory Comparison—High Level Spiked Lipid (% Recovery)

	Lab 1 N=1	Lab 2 n=4		Lab 3 n=5	
Analyte		Average	RSD (%)	Average	RSD (%)
2,3,7,8-TCDF	74	111	2.1	92	4.7
2,3,7,8-TCDD	86	91	10.0	92	3.4
1,2,3,7,8-PeCDF	60	93	6.9	100	6.2
2,3,4,7,8-PeCDF	62	103	6.2	89	9.2
1,2,3,7,8-PeCDD	78	120	9.7	88	11.5
1,2,3,4,7,8-HxCDF	62	112	15.1	-[a]	-
1,2,3,6,7,8-HxCDF	72	104	13.9	68	13.2
2,3,4,6,7,8-HxCDF	70	105	17.2	78[b]	0.9
1,2,3,7,8,9-HxCDF	62	86	5.6	74	13.3
1,2,3,4,7,8-HxCDD	58	89	9.2	-[c]	-
1,2,3,6,7,8-HxCDD	70	93	7.7	97	12.7
1,2,3,7,8,9-HxCDD	78	88	8.6	91	8.8
1,2,3,4,6,7,8-HpCDF	100	95	6.7	108	15.2
1,2,3,4,7,8,9-HpCDF	82	120	21.3	104	10.0
1,2,3,4,6,7,8-HpCD	80	121	13.8	90	19.3
1,2,3,4,6,7,8-OCDF	81	NR[d]	-	93	9.0
1,2,3,4,7,8,9-OCDD	89	NR	-	93[e]	29.2

[a] Diphenylether interference observed
[b] Average of two positive quantifiable or trace values (n=2).
[c] 1,2,3,4,7,8- and 1,2,3,6,7,8-HxCDD isomers were reported as a sum.
[d] Not reported.
[e] Average of four positive quantifiable or trace values (n=4)

6.4 SYNOPSIS OF ANALYTICAL RESULTS

The detailed results from the analysis of all samples have been submitted as separate reports to EPA (Cramer et al. 1989a, 1989b). Appendix A of this report provides detailed documentation on the results of each FY87 NHATS sample and QC sample. The data in Appendix A identifies: batch number, laboratory identification; NHATS sample identification; number of specimens in the composite; census region and age group; composition of composite by sex and race; identification of data quality as positive quantifiable (PQ), trace (TR) or not detected (ND); measured concentrations and limits of detection; data restrictions; and IQS recoveries.

Successful analyses were achieved for all samples except one, ACD8700425. This sample appeared to have been fortified with twice the specified amount of IQS. In the remaining samples, the range of 2,3,7,8-TCDD detected ranged from a nondetect value of 0.0689 pg/g to a maximum of 15.1 pg/g. Positive quantifiable OCDD ranged from 136 to 1,660 pg/g.

It should also be noted that in many of the analyses, responses to octachlorodiphenyl ethers (OCDPE) overlapped with the responses of the 1,2,3,4,7,8-HxCDF and the 2,3,4,6,7,8-HxCDF isomers. In these cases the observed responses were quantitated but were reported as a nondetected (ND) value.

The recoveries of the internal quantitation standards (IQS) were within the QA data quality objective of 40% to 150%, with the exceptions that are identified below. As previously noted, composite ACD8700425 appeared to have been fortified with twice the specified amount of IQS. Data for this composite are considered suspect. In composites ACD8700023 and ACD8700256, the recoveries of $^{13}C_{12}$-PeCDD (28%) and $^{13}C_{12}$-PeCDF (17%), respectively, were outside the data quality objectives. Since only one out of nine IQS recoveries per sample were not in control, the samples were not reanalyzed. The PeCDD and PeCDF data for these two composites were flagged as not meeting the DQOs. In batches 3 through 5, the recoveries of the carbon-13 labeled HpCDD in three composites and the labeled octachlorodioxin in 17 composites and/or QC samples were outside the DQOs.

The analysis results for the spiked control QC samples (Table 6-17) indicated that the accuracy of the method met the QA objectives of 40% to 150% for all compounds with the following exceptions: the 1,2,3,4,7,8- and 1,2,3,6,7,8-HxCDD pair in the low level batch 2 spike (13% recovery); OCDD in the high level batch 3 spike (39%);, 1,2,3,4,6,7,8-HpCDD in the low level batch 5 spike (32%); and 2,3,4,7,8-PeCDF in the low level batch 4 spike (not recovered). The PeCDF recovery was affected by a momentary reduction in sensitivity resulting from the apparent coelution of a high level compound interference. The remaining compounds showed recoveries ranging from 44% to 137%.

Table 6-17. Summary of QC Data for the FY87 NHATS PCDDs and PCDFs

Analyte	Spike level (pg/g)	N[a]	Number detected[b]	Average measured concentration (pg/g)	Background adjusted recovery[c] (%)	Standard deviation (pg/g)	Coefficient of variation (%)
2,3,7,8-TCDF	0	2	2	1.12	-	0.170	15.2
	10	5	5	10.5	93.3	0.856	8.19
	50	5	5	46.8	91.3	2.27	4.86
2,3,7,8-TCDD	0	5	5	9.29	-	0.899	9.68
	10	5	5	17.6	83.5	2.56	14.5
	50	5	5	55.2	91.8	1.82	3.30
1,2,3,7,8-PeCDF	0	5	1	0.210	-	0.180	85.7
	10	4	4	11.6	114	0.956	8.24
	50	5	5	50.1	99.8	3.09	6.16
2,3,4,7,8-PeCDF	0	5	5	25.1	-	1.89	7.53
	10	5	5	32.6	75.2	6.33	19.4
	50	5	5	69.6	89.1	4.21	6.05
1,2,3,7,8-PeCDD	0	5	5	20.8	-	0.970	4.66
	10	3	3	30.8	100	3.73	12.1
	50	5	5	64.8	88.1	5.77	8.90
1,2,3,4,7,8-HxCDF	0	1	1	18.7	-	-	-
1,2,3,6,7,8-HxCDF	0	5	5	10.8	-	2.32	21.4
	25	4	4	34.4	94.1	2.48	7.23
	125	5	5	95.6	67.8	12.9	13.5

Table 6-17 (continued)

Analyte	Spike level (pg/g)	N[a]	Number detected[b]	Average measured concentration (pg/g)	Background adjusted recovery[c] (%)	Standard deviation (pg/g)	Coefficient of variation (%)
2,3,4,6,7,8-HxCDF	0	1	1	1.93	-	-	-
	25	2	2	25.7	94.9	7.28	28.4
	125	2	2	97.9	76.8	2.40	2.46
1,2,3,7,8,9-HxCDF	0	5	0	0.334	-	0.0824	24.7
	25	5	5	19.9	78.1	3.00	15.1
	125	5	5	92.2	73.5	12.3	13.4
1,2,3,4,7,8/	0	5	5	137	-	9.36	6.85
1,2,3,6,7,8-HxCDD	50	5	5	173	73.2	17.3	10.0
	250	5	5	379	96.9	36.3	9.57
							8.41
1,2,3,7,8,9-HxCDD	0	4	4	21.1	-	1.77	
	25	5	5	44.9	95.2	3.68	8.20
	125	5	5	134	90.5	9.88	7.37
1,2,3,4,6,7,8-HpCDF	0	5	5	29.8	-	1.88	6.30
	25	5	5	58.3	114	3.01	5.16
	125	5	5	165	108	19.6	11.9
1,2,3,4,7,8,9-HpCDF	0	5	2	0.895	-	0.514	57.5
	25	5	5	29.4	114	1.32	4.49
	125	5	5	130	103	12.8	9.85

Table 6-17 (continued)

Analyte	Spike level (pg/g)	N[a]	Number detected[b]	Average measured concentration (pg/g)	Background adjusted recovery[c] (%)	Standard deviation (pg/g)	Coefficient of variation (%)
1,2,3,4,6,7,8-HpCDD	0	5	5	140	-	9.56	6.84
	25	5	5	159	78.4	11.0	6.93
	125	5	5	253	90.6	22.0	8.72
1,2,3,4,6,7,8,9-OCDF	0	4	3	3.35	-	2.68	80.2
	50	3	3	56.1	105	8.30	14.8
	250	2	2	239	94.3	4.24	1.78
1,2,3,4,6,7,8,9-OCDD	0	4	4	1190	-	49.2	4.15
	350	3	3	1520	96.0	25.2	1.65
	700	2	2	1980	113	91.9	4.65
1,2,3,4,7,8-HxCDD	0	2	2	8.76	-	0.567	6.46
	25	4	4	29.0	80.8	10.6	36.5
	125	4	4	158	120	32.1	20.3
1,2,3,6,7,8-HxCDD	0	2	2	128	-	4.24	3.31
	25	4	4	145	68.0	9.31	6.42
	125	4	4	235	85.2	11.6	4.93

[a] N = number of samples meeting DQO criteria.
[b] The number of samples with PQ or TR qualifiers.

[c] Background adjusted recovery (%) = $100 \frac{\text{Average} - \text{Background}}{\text{Spike Level}}$

Background is average measured amount for control samples (0 spike level).

6.5 STATISTICAL ANALYSIS OF THE QUALITY CONTROL DATA

The statistical analysis of the FY87 NHATS QC samples for the PCDD and PCDF analytical results are summarized in Table 6-17. The objectives of the analysis were to

- Estimate the percent recovery of the analytical method,
- Determine if there are significant differences in the analytical performance among the batches,
- Characterize the precision of the analytical method,
- Establish the relationship between the precision of the analytical method and the level of the spiked concentration, and
- Identify anomalous results that suggest potential problems in the analytical measurements.

Of the 68 samples analyzed for PCDDs and PCDFs in the FY87 study, 20 were QC samples. Each of the five analysis batches contained one method blank, one unspiked control sample, and two spiked samples. The sampling plan for the allocation of these QC samples has been described by Heath (1988). Leczynski et al. (1988) determined the assignment of the QC samples to the analysis batches.

Because it was agreed that population estimates would be calculated using only the data that met the data quality objectives (DQOs), the same criteria were applied before evaluating the QC data. The DQO criteria are:

(1) Internal quantitation standard (IQS) recovery must be between 40% and 150%,
(2) Ion ratio must be within 20% of the theoretical ratio, and
(3) There must be no problems with coelution or fragmented peaks.

If an analyte was not detected (ND), the measured concentration of the QC sample was computed as one half of the detection limit (LOD/2). This same approach was used in the statistical analysis of the field samples.

A descriptive summary of the QC data is presented in Section 6.5.1. In Section 6.5.2 the statistical approach to analyzing the QC data is discussed and the results of these analyses are presented in Section 6.5.3. Conclusions are presented in Section 6.6.4.

6.5.1 Summary of the QC

Table 6-17 presents the data for each chemical in the quality control samples. Table 6-17 presents the data by chemical and spike level (pg/g) and provides information on the number of QC samples for which the DQOs were met, the number of positive quantifiable (PQ) and trace (TR) measurements, the average measured concentration, the background adjusted recovery (BAR) for spiked samples, the standard deviation (SD) of the measured concentration, and the coefficient of variation (CV). The background adjusted recoveries at the low (L) and high (H) spike levels were computed as

$$\mathbf{BAR(j) = 100\%*[Avg(j) - Avg(0)] / spike\ level,}$$

where Avg(j) is the average measured concentration at spike level j (j=L or H) and Avg(0) is the average measured concentration of the unspiked control sample. The coefficient of variation was computed as

$$\mathbf{CV(j) = 100\%*[SD(j)/Avg(j)],\ for\ j= 0,\ L,\ and\ H.}$$

All background adjusted recoveries were between 67 and 120% and the coefficients of variation were generally between 2 and 20% at both the low and high spike levels. In the control samples analytes at levels below the detection limit of the analytical method generally had the higher coefficients of variation.

Only one analyte was detected in the method blank. The measured concentration of 2,3,7,8-TCDF in the batch 1 method blank was 0.460 pg/g, or 41% of the average measured 2,3,7,8-TCDF concentration (1.12 pg/g) in the two control samples that met the DQO criteria. Because this indicated a potential for a bias affecting all batch 1 samples, the measured 2,3,7,8-TCDF concentrations of the 48 study samples were compared across the five batches. This comparison did not reveal any statistical evidence of a batch effect on the study samples. Therefore, no adjustments were made to the measured 2,3,7,8-TCDF concentrations of the study or the QC samples in batch 1. In particular, all 2,3,7,8-TCDF QC data meeting the original DQOs were included in the statistical analysis.

Appendix D contains the plots of the measured concentrations against the spike levels for all study compounds. It is evident from these plots that the relationship between measured concentration and spiked concentration is nearly linear for all the compounds. Because only one measurement met the DQO criteria for 1,2,3,4,7,8-HxCDF, a figure is not given for this chemical. Also presented in these figures are the predicted concentrations with tolerance bounds for individual measured concentrations. The statistical methods for calculating the predicted values and tolerance bounds are discussed in the following section.

6.5.2 Statistical Approach to Analyzing the QC Data

The QC data were statistically analyzed using linear regression models fitted to the measured concentrations for each compound. Three models, as described in Table 6-18, were fitted to the data to determine the best fit and to test for significant batch effects.

Initially the full batch effects (FB) model was fitted for each compound. The FB model was used to test for two types of batch effects: fixed effects and proportional effects. A fixed batch effect is the constant amount by which the measured concentrations in the batch differ from the average for all batches. It is calculated using the intercepts ($\propto_i$, i=1,...,5) of the FB model. For example, the fixed effect of batch 1 is represented by $\propto_1-\propto$, where $\propto$ is the average intercept. Proportional batch effects are characterized by differences in the batch recoveries. The proportional batch effect for batch 1, for example, is the difference between the recovery (slope) for batch 1 and the average recovery for all batches. Using the notation in Table 6-18, the proportional effect (also called the recovery effect) for batch 1 is denoted by $\beta_1-\beta$.

For those analytes providing sufficient data, F-tests were performed to determine the significance of the fixed and proportional batch effects. In virtually all cases where batch effects were detected, they were due to variations in the slopes from batch to batch. Therefore, a second statistical model containing a constant intercept (i.e., $\alpha_i = \propto$ in the full batch effects model) was fitted to the data for each analyte. This model is called the batch slopes (BS) model because it can be used to test for significant differences in the recoveries (slopes) between batches.

The BS model was used to estimate recoveries for each batch, overall average recovery, and predicted concentrations at each spike level. Statistical F-tests were performed to test for significant background levels and batch effects. Background levels are indicated when the estimated intercept is found to be significantly different from zero and batch effects are indicated when at least one of the estimated batch slopes is found to be significantly different from the others. Predicted concentrations at each spike level were calculated from the estimated intercept and average recovery.

For some analytes it was not possible to fit either the FB or BS models because there were insufficient data after applying the DQO restriction criteria. In these cases a simple linear regression (SLR) model was used to estimate average recovery and test for significant background levels. However, using the SLR model, it is not possible to test for significant batch effects.

Table 6-18. Regression Models Used to Analyze FY87 NHATS QC Data

Model	Equation[a] (number of parameters)	Remarks
Full Batch Effects (FB)	$EC_{ij} = \alpha_i + \beta_i SC_j$ (10 parameters)	Intercepts (α_i) are the batch background levels. Slopes (β_i) are the batch recoveries. When the data are balanced,[b] the average of the estimated batch recoveries from this model equals the estimated recovery from the SLR model, and the average of the estimated intercepts equals the estimated intercept from the SLR model.
Batch Slopes Model (BS)	$EC_{ij} = \alpha + \beta_i SC_j$ (6 parameters)	Intercept (α) is the background level. Slopes (β_i) are the batch recoveries. When the data are balanced,[a] the average of the estimated batch recoveries from this model equals the estimated recovery from the SLR model, and the estimated intercept equals the estimated intercept from the SLR model.
Simple Linear Regression (SLR)	$EC_{ij} = \alpha + \beta SC_j$ (2 parameters)	Intercept (α) is the background level. Slope (β) is the recovery.

[a] i = 1, 2, 3, 4, and 5 (batches)
j = Control, low spike, and high spike levels
EC_{ij} = expected value of the concentration in the jth QC sample of batch i
SC_j = spike levels of the jth QC sample

[b] In this application, the data are balanced whenever there are equal numbers of control, low spike, and high spike samples that meet the DQO criteria.

Although the analysis established that there were statistically significant batch effects for most of the analytes it was decided that there would be no adjustments made to the measured concentrations of the study samples. Thus, a model was developed in which the differences in recoveries from batch to batch were treated as random effects; thus affecting the precision of the analytical method. The model assumes that the standard deviation of the measured concentration has two components: (1) a component associated with the within-batch measurement error, estimated by the mean squared error (MSE) from the BS model; and (2) a random component associated with the random-batch effects. The standard deviation of the ith measured concentration ($\hat{C}_{ij}$) at spiked concentration SC_j was computed as:

$$SD(\hat{C}_{ij}) = \sqrt{MSE + (SC_j * SD(\beta_j))^2}$$

where MSE is the mean squared error from the BS model, and $SD(\beta_j)$ is the sample standard deviation of the estimated batch recoveries. According to this model, the standard deviation increases with the concentration of the sample; however, it is not necessarily proportional to the concentration. When there was insufficient data to fit the BS model, the square root of the MSE from the SLR model was used to estimate the standard deviation of the predicted concentration.

Approximate prediction bounds on the individual measured concentrations were calculated by adding plus or minus three times the estimated standard deviation to the estimated predicted concentration. The probability that an individual measured concentration will fall within the prediction interval is approximately 99% according to asymptotic distribution theory.

6.5.3 Results

The results of the statistical analyses are summarized in Tables 6-19 and 6-20. Table 6-19 contains an estimate of the average recovery, its standard error (SE), and the estimated batch recoveries for each analyte. When there was sufficient data, the individual batch recoveries were estimated from the BS model, otherwise the SLR model was used to calculate the average recovery.

For each compound, a hypothesis test was performed to determine if the average recovery was significantly different (at the 5% significance level) from 100%. The result of this hypothesis test is denoted by an " * " next to the estimated average recovery. The average recoveries were determined to be significantly less than 100% for nine compounds, and the lowest average recovery was estimated to be 66.1% for 1,2,3,6,7,8-HxCDF. There was only one compound, 1,2,3,4,6,7,8-HpCDF, for which the average recovery is statistically greater than 100%.

Table 6-19. Estimated Recoveries for Each Batch and Estimated Average Recovery Over All Five Batches

Analyte	Estimated average recoveries (%) and standard error: Recovery (%)[a]	SE	Estimated batch recoveries (%): 1	2	3	4	5	Significant batch effect?
2,3,7,8-TCDF	91.0*	0.83	96.1	94.2	86.1	92.2	86.4	Yes[f]
2,3,7,8-TCDD	92.4*	2.1	97.9	95.3	88.9	90.5	89.4	No
1,2,3,7,8-PeCDF	99.0	1.0	108	98.2	99.1	98.3	90.7	Yes
2,3,4,7,8-PeCDF	90.1	5.3	96.7	100	78.4	89.1	86.4	No
1,2,3,7,8-PeCDD	87.7*	2.2	76.0	107	82.2	88.2	84.5	Yes
1,2,3,4,7,8-HxCDF[b]								
1,2,3,6,7,8-HxCDF	66.1*	2.2	69.7	67.8	48.6	71.5	73.2	Yes
2,3,4,6,7,8-HxCDF[c,d]	74.6*	4.2	[d]					
1,2,3,7,8,9-HxCDF	73.2*	1.1	82.2	77.4	57.2	76.8	72.2	Yes
1,2,3,4,7,8/ 1,2,3,6,7,8-HXCDD	98.6	3.8	98.7	105	115	97.2	77.2	Yes
1,2,3,7,8,9-HxCDD	90.1*	1.4	89.9	91.5	102	84.7	82.2	Yes
1,2,3,4,6,7,8-HpCDF	107*	1.9	85.3	108	119	122	102	Yes
1,2,3,4,7,8,9-HpCDF	103	1.3	97.4	108	105	114	88.7	Yes
1,2,3,4,6,7,8-HpCDD	91.4	4.6	116	93.7	98.5	70.6	78.8	Yes
1,2,3,4,6,7,8,9-OCDF[c,d]	93.8*	2.0						
1,2,3,4,6,7,8,9-OCDD[c,d]	111	7.0						
1,2,3,4,7,8-HxCDD[c]	124	12.4	114	126	158	100	[e]	Yes
1,2,3,6,7,8-HxCDD[c]	87.3*	5.5	87.5	87.2	76.6	97.8	[e]	No

[a] The asterisk (*) indicates that the average estimated recovery was determined to be significantly different from 100% at the 5% significance level.
[b] Insufficient data to fit a linear model to the QC samples for 123478-HxCDF.
[c] Average recovery was estimated using the SLR model.
[d] Insufficient data to model the batch recoveries.
[e] Insufficient data to estimate the recovery for this batch.
[f] Indicates statistically significant differences among the estimated batch recoveries at the 5% significance level.

Table 6-20. Predicted Concentration ($\hat{C}$, pg/g) and Coefficient of Variation at Each Spike Level

	Spike level					
	Control		Low		High	
	$\hat{C}$	CV($\hat{C}$) (%)	$\hat{C}$	CV($\hat{C}$) (%)	$\hat{C}$	CV($\hat{C}$) (%)
2,3,7,8-TCDF	1.27*[a]	48.3	10.4	7.1	46.8	4.5
2,3,7,8-TCDD	8.90*	19.9	18.1	10.0	55.1	4.8
1,2,3,7,8-PeCDF	0.786	104	10.7	9.6	50.3	6.4
2,3,4,7,8-PeCDF	24.4*	18.1	33.4	13.5	69.5	8.8
1,2,3,7,8-PeCDD	21.1*	8.7	29.8	7.3	64.9	9.6
1,2,3,4,7,8-HxCDF[b]						
1,2,3,6,7,8-HxCDF	13.6*	33.0	30.1	17.0	96.3	13.8
2,3,4,6,7,8-HxCDF	5.05	100	23.7	21.3	98.3	5.1
1,2,3,7,8,9-HxCDF	0.881	265	19.2	17.5	92.4	13.2
1,2,3,4,7,8/ 1,2,3,6,7,8-HxCDD	131*	12.0	180	9.5	378	10.1
1,2,3,7,8,9-HxCDD	21.7*	12.8	44.2	7.7	134	7.6
1,2,3,4,6,7,8-HpCDF	30.5*	13.2	57.4	9.5	165	11.6
1,2,3,4,7,8,9-HpCDF	2.17	127	27.8	13.3	130	9.6
1,2,3,4,6,7,8-HpCDD	138*	6.9	161	6.5	252	9.5
1,2,3,4,6,7,8,9-OCDF	5.52	106	52.4	11.1	240	2.4
1,2,3,4,6,7,8,9-OCDD	1170*	4.9	1,560	3.7	1,950	3.0
1,2,3,4,7,8-HxCDD	2.06	561	33.1	39.5	157	20.8
1,2,3,6,7,8-HxCDD	125*	6.6	147	5.8	234	5.8

[a] The asterisk (*) indicates that the predicted background concentration for the control sample was determined to be significantly greater than zero at the 5% significance level.

[b] Insufficient data to fit a linear model to the QC samples for this compound.

The individual estimated batch recoveries are shown in the remaining columns of Table 6-19. Also presented are the results of the hypothesis tests for differences among the batch recoveries. Notice that there are significant batch effects for virtually all analytes. However, because there were no apparent patterns to the batch effects, it was decided to treat the batch effects as random. This means that no "batch" corrections were made to the measured concentrations of the study samples. Instead, as discussed in the previous section, the batch effects were treated as random and included in the estimated precision of the analytical method.

Table 6-20 contains the model-derived predicted average concentration and estimated coefficient of variation (CV) for each analyte and spike level. For each analyte, a hypothesis test was performed to determine if the predicted concentration in the control sample was greater than zero at the 5% significance level. For example, the predicted concentration of 2,3,7,8-TCDD in the control samples was estimated to be 8.90 pg/g and this estimate is significantly greater than zero at the 5% significance level. This is consistent with the fact that 2,3,7,8-TCDD was detected in all five control samples. The background concentrations were determined to be significantly greater than zero for six of the seven PCDDs (not including 1,2,3,4,7,8/1,2,3,6,7,8-HxCDD because the individual isomers are already represented) and for four of the nine PCDFs (1,2,3,4,7,8-HxCDF is not included because of insufficient data).

In general, the relative precision of measured concentrations is much better for PCDDs than PCDFs. At the control level, the CVs of the measured concentrations for the four PCDFs which had significant background levels were between 13% and 48%. All of the CVs for the PCDFs were less than 21% in the spiked samples. The CVs at the control level for the six PCDDs with significant background levels were between 5% and 20%. Although this might be explained by the higher concentration levels of the PCDDs, the relative precision for measuring PCDDs is also much better in the spiked samples. With the exception of 1,2,3,4,7,8-HxCDD, the CVs for PCDD measurements are between 3% and 10% in spiked samples while the CVs for the PCDF measurements in spiked samples were between 2% and 21%.

In general, the linear model provided a good fit to the measured concentrations for most analytes. This is evident in the figures presented in Appendix D. There does appear to be some lack of fit for compounds 1,2,3,6,7,8-HxCDF and 1,2,3,4,7,8-HxCDD. The lack of fit for 1,2,3,6,7,8-HxCDF is most probably due to the rather low measured concentration of 73 pg/g in batch 3 at the high spike level. The measured concentrations for 1,2,3,4,7,8-HxCDD were reported as a combined value with the 1,2,3,6,7,8-HxCDD and therefore could not be effectively evaluated using the linear model.

6.5.4 Summary of QC Data

The results from the statistical analysis of the QC data are summarized as follows:

1. Nine of the analytes had estimated average recoveries that were found to be less than 100% at the 5% significance level and one analyte had an estimated average recovery that was significantly greater than 100%. The estimated average recoveries were between 66.1% and 124% for all analytes.

2. There are statistically significant differences in the recoveries from batch to batch for most of the analytes. However, it is recommended that no batch adjustment be made to the study samples. Instead, the estimated measurement precision will account for the batch effects.

3. Measurement precision, determined by the estimated coefficients of variation in the control samples, is generally between 5% and 20% for the PCDDs and between 13% and 48% for the PCDFs. For the spiked samples, the PCDDs had CVs between 3% and 10%, and the PCDFs had CVs between 2% and 21%. These estimated CVs include random batch effects.

4. Statistically significant background levels of four PCDFs and six PCDDs were identified in the control samples.

5. The relationship between measured and spiked concentrations is generally linear over the range of spiked concentrations.

7.0 STATISTICAL METHODOLOGY

There were three objectives for the statistical analysis of FY87 NHATS data:

1. Estimate average concentration levels in the adipose tissue of individuals in the U.S. population and in various demographic subpopulations,

2. Construct confidence intervals for these averages, and

3. Determine if average concentration levels of chlorinated dioxins and furans in the U.S. population differ significantly by any of the four demographic factors (geographic region, age, race, and sex).

The statistical analysis methods used in this report are based on an additive model for the demographic effects. Previous studies of the effect of using composite samples demonstrated the validity of the additive model. A technique of iterative weighted generalized least squares was used to estimate model parameters. The resulting estimates are approximately normal for large samples. This approximate normality was used in constructing confidence intervals and hypothesis tests. The remainder of this section provides details of the statistical model and process as well as references to background work.

7.1 STATISTICAL MODEL

The use of composite samples for determination of the levels of PCDDs and PCDFs created a need to reevaluate the approach to estimate general population levels of these compounds. The statistical models previously used to assess NHATS data for OCl pesticides and PCBs using individual sample data were not adequate for extrapolating the composite sample data. Section 7.1.1 discusses the background on the development of a new statistical model, the additive model, which is presented in Section 7.1.2.

7.1.1 Background

Mack and Panebianco (1986) developed and used a "multiplicative" model to analyze the composite sample data from the NHATS FY82 Broad Scan Analysis Study. In their model the analyte concentrations in a composite sample are represented as a product of fixed and random effects associated with geographic and demographic characteristics of individuals who contributed specimens to the samples. Orban et al. (1987) studied this problem further and recommended the "additive" model which assumes additive effects of the donors' geographic and demographic characteristics.

The multiplicative and additive models were later evaluated by Orban and Lordo (1989). They showed that under certain distributional assumptions, both models produce asymptotically unbiased estimates of average concentration levels in the target populations. However, the multiplicative model requires that the sampling and measurement errors be distributed according to lognormal distributions. No specific distributional forms are required to achieve asymptotically unbiased estimates using the additive model. Orban and Lordo (1989) also compared the two models using simulated composite sample data which were generated from actual specimen data obtained in the FY83 NHATS. Their analysis demonstrated, using actual NHATS data, that the standard errors of the estimates from these models are nearly equal.

Following the study, the additive model was chosen to be used in the FY87 NHATS and all future NHATS for the following reasons: (1) under very general assumptions, the additive model produces asymptotically unbiased estimates of average concentration levels in the population, and (2) the additive model establishes a more tractable relationship between the distribution of analyte concentrations in individuals and the distribution of measured concentrations from the composite samples. The second reason is particularly important because individual specimens are collected but composites are chemically analyzed.

7.1.2 The Additive Model

Table 7-1 lists the categories of the four analysis factors of interest to NHATS. The additive model assumes that the four analysis factors have fixed additive effects on the average concentrations in specimens. This assumption creates 48 subpopulations defined by the 4 x 3 x 2 x 2 unique combinations of categories.

Table 7-1. NHATS Analysis Factors and Categories

Analysis factor	Category
Census region	Northeast North Central South West
Age	0-14 years 15-44 years 45+ years
Sex	Male Female
Race group	Caucasian Noncaucasian

In addition to the four analysis factors, there are three ancillary factors that are assumed to have random effects on NHATS data. They are (1) sampling of MSAs, (2) sampling of individuals within MSAs, and (3) measurement of analyte concentrations in the composite samples. The second factor, sampling individuals from MSAs, also includes the effects of selecting specimens from individual donors. The first two ancillary factors have random effects on the actual concentrations in individual specimens, and the third has a random effect on the measured concentrations of composites.

From the assumptions above, the actual concentration in a specimen from the i-th donor in MSA j, census region k, age group ℓ, sex m, and race group n, is represented by

$$C_{ijk\ell mn} = \mu + CR_k + A_\ell + S_m + R_n + MSA_j + \epsilon_{ij}$$

where

μ is a constant
CR_k is the fixed effect of census region k; k = 1,...,4
A_ℓ is the fixed effect of age group ℓ; ℓ = 1,2,3
S_m is the fixed effect of sex m; m = 1,2
R_n is the fixed effect of race group n; n = 1,2
MSA_j is the random effect of selecting MSA j; j = 1,2,...
ϵ_{ij} is the random effect of selecting individual i in MSA j.

Furthermore, to uniquely define the parameters, we let

$$\sum_{k=1}^{4} CR_k = \sum_{\ell=1}^{3} A_\ell = \sum_{m=1}^{2} S_m = \sum_{n=1}^{2} R_n = 0$$

The random effects MSA_{jk} and ϵ_{ijk} are assumed to have independent error distributions with mean zero and variances σ_m^2 and σ_ϵ^2, respectively. Also, because of evidence from previous NHATS and other environmental studies that the variation in specimen concentrations increases with average concentration levels, it is assumed that $\sigma_\epsilon^2 = b^2\mu_s^2$, where μ_s is the average concentration level is subpopulation s. For notational simplicity we let

$$\mu_s = \mu + CR_k + A_\ell + S_m + R_n$$

for some particular combination of indices k, ℓ, m, and n.

This defines the model for the actual concentration in a specimen collected in the survey. However, the specimens are composited prior to chemical analysis. Thus, data are obtained from the chemical analyses of composite samples. Letting Y_h represent the measured concentration of composite h (h = 1, . . . , C), the natural additive effects of compositing imply that

$$Y_h = \frac{1}{M_h} \sum_s \sum_j \sum_i C_h(i,j,s) C_{ijs} + \gamma_h,$$

where C_{ijs} is the actual concentration in specimen i from MSA j and subpopulation s; γ_h is a random measurement error; M_h is the number of specimens in composite h; and $C_h(i,j,s)$ is equal to 1 if specimen (i,j,s) is in composite h, and is equal to zero, otherwise. The error distribution of γ_h has mean zero and variance σ_γ^2.

At this point the notation for representing the model is rather complex. However, the main points can be illustrated using matrix notation. Let

$$\underset{\sim}{\beta} = (\mu, CR_1, CR_2, CR_3, A_1, A_2, S_1, R_1)'$$

be the 8x1 vector of fixed effects and $\underset{\sim}{\mu} = (\mu_1, \ldots, \mu_{48})'$ be a 48x1 vector representing the 48 subpopulation average concentrations. Then

$$\underset{\sim}{\mu} = X\underset{\sim}{\beta} ,$$

where X is a 48x8 design matrix. Letting $\underset{\sim}{Y} = (Y_1, \ldots, Y_C)'$ be the Cx1 vector of measured composite concentrations, Orban and Lordo (1989) show that the expected value of $\underset{\sim}{Y}$ is

$$\underset{\sim}{E(Y)} = Z\underset{\sim}{X\beta} = D\underset{\sim}{\beta} ,$$

where Z is a Cx48 composite design matrix. Thus, according to the additive model, both the actual concentrations of the individual specimens and the measured concentrations of the composite samples have expected values that are linear combinations of the additive effects of the fixed analysis factors.

Orban and Lordo (1989) also show that the variance-covariance matrix of $\underset{\sim}{Y}$ is a block diagonal matrix that depends on σ_m^2 , σ_e^2 , and σ_Y^2.

7.2 STATISTICAL ANALYSIS

This section describes the specific methods used to achieve the statistical objectives. The estimation methods are discussed in Section 7.2.1 and the hypothesis testing procedures are presented in Section 7.2.2.

7.2.1 Estimation

The specific quantities estimated for the FY87 NHATS are the average analyte concentrations in the adipose tissue of the U.S. population and the averages for each of the marginal populations defined by the categories listed in Table 7-1. The estimates were calculated in three steps:

1. The additive model parameters associated with the four analysis factors were estimated using a method called iterative weighted generalized least squares (IWGLS).

2. Estimates of average concentration levels in all 48 subpopulations defined by the analysis factors were calculated from the parameter estimates.

3. National and marginal population estimates were obtained by taking weighted averages of the appropriate subpopulation estimates. Weights were proportional to the population counts of the 48 subpopulations from the 1980 U.S. census.

According to the model described in Section 7.1.2, the vector of measured composite sample concentrations, denoted by

$$\underset{\sim}{Y} = (Y_1, \ldots, Y_c)' ,$$

has a multivariate distribution with mean

$$E(\underset{\sim}{Y}) = D\underset{\sim}{\beta}$$

and a variance-covariance matrix V. The vector

$$\underset{\sim}{\beta} = (\mu, CR_1, CR_2, CR_3, A_1, A_2, S_1, R_1)'$$

is the vector of fixed effects to be estimated.

To obtain asymptotically unbiased estimates of the fixed effects it is not necessary to make any assumptions about the form of the distributions of the random effects. If the variance-covariance matrix V were known, the method of generalized least squares (GLS) produces estimates of $\underset{\sim}{\beta}$ that are unbiased and have minimum variance among all unbiased estimates. Furthermore, if the errors are normally distributed, the GLS estimates are equivalent to the maximum likelihood estimates. The GLS estimate of $\underset{\sim}{\beta}$ is

$$\hat{\underset{\sim}{\beta}} = (D'V^{-1}D)^{-1}D'V^{-1}\underset{\sim}{Y}$$

Unfortunately, V depends on three unknown variance components (σ_m^2, σ_e^2, and σ_γ^2) as well as the vector $\underset{\sim}{\beta}$. Therefore, Orban and Lordo (1989) proposed a method involving iterative weighting. Thus, the method is called iterative weighted generalized least squares (IWGLS).

Starting values for the fixed effect parameters and estimates of the variance components were calculated using a maximum likelihood procedure. This step was performed using the P3V program in the BMDP program package. The resulting estimate of V was then used in the GLS formula to produce a revised estimate of $\underset{\sim}{\beta}$. Each time the GLS formula was applied, the estimate of V was updated. This process was continued until certain convergence criteria were met. Orban and Lordo (1989) discuss this method in more detail and describe special computer programs for implementing IWGLS. They also provide formulas for calculating the standard errors of the estimates.

An estimate of the average concentration level in each of the 48 subpopulations was then calculated by

$$\underset{\sim}{\hat{\mu}} = X\underset{\sim}{\hat{\beta}}$$

Weighted averages of the appropriate subpopulation predicted concentrations were then calculated to estimate marginal averages for the categories of each analysis factor. For example, the average concentration in the Northeast census region was estimated by the weighted average of predicted concentrations in all subpopulations in the Northeast region. Marginal estimates were calculated for four census regions, three age groups, two sexes, and two race groups. The U.S. population estimate was calculated in a similar manner. An approximate 95% confidence interval for each estimate was calculated by adding and subtracting two times the standard error of the estimate.

7.2.2 Hypothesis Testing

Hypothesis tests were performed to determine if average concentration levels differ significantly by any of the geographic or demographic factors. The specific hypotheses tested were:

H_1: $CR_1 = CR_2 = CR_3 = CR_4 = 0$,

H_2: $A_1 = A_2 = A_3 = 0$,

H_3: $R_1 = R_2 = 0$, and

H_4: $S_1 = S_2 = 0$.

The hypothesis, H_1, for example, states that there are no differences in average concentration levels among the four census regions. The alternative hypothesis is that there is at least one pair of regions for which the average concentrations are different. The results from these hypothesis tests are somewhat related to the confidence intervals for averages in individual subpopulations. Generally, if the confidence intervals for any pair of subpopulation averages do not intersect, then the hypothesis of no differences among subpopulations is likely to be rejected. However, the contrapositive is not always true.

In order to test these hypotheses, it was necessary to make specific distribution assumptions for the random effects. It was assumed that the errors associated with sampling MSAs, sampling individuals within MSAs, and measuring concentrations were independent and normally distributed. The additive effect of compositing specimens suggests that the normality assumption for sampling error is reasonable because concentrations of individuals are averaged in the composite sample. Statistical theory states that averages and sums are approximately normally distributed. Distributional assumptions were tested for all analytes using probability plots and residual analysis. The model validation results are discussed later in Section 8.5.

The likelihood ratio method was used to test hypotheses H_1 through H_4. According to asymptotic theory, the log of the ratio of the likelihood functions (calculated under the full and null hypothesis models) has approximately a chi squared distribution. The number of degrees of freedom is equal to the number of independent parameters constrained under the null hypothesis. Orban and Lordo (1989) wrote computer programs to perform these tests.

8.0 RESULTS

This section contains the results of the statistical analysis of the FY87 NHATS for PCDDs and PCDFs in human adipose tissue. The analysis was performed on data obtained from 48 composite samples, each containing an average of 18 adipose tissue specimens from sampled cadavers and surgical patients.

A descriptive summary of the data is provided in Section 8.1 and the results of the formal statistical analyses are presented in Sections 8.2 and 8.3. Section 8.4 provides estimated rates of change of selected PCDD and PCDF concentrations. Section 8.5 describes the outlier detection procedures that identified potential data errors to be corrected prior to conducting the statistical analysis. Finally, Section 8.6 discusses the steps that were taken to demonstrate the validity of the statistical methodology applied to the FY87 NHATS data.

8.1 DATA RESTRICTIONS AND DESCRIPTIVE STATISTICS

Prior to conducting the statistical analysis, the data were classified according to the specified data restrictions and data qualifiers. For each of the target analytes, Table 8-1 shows the number of composite samples for which the measured concentrations were restricted or qualified.

Data restrictions indicate whether specific data quality objectives (DQOs) were met during chemical analysis. In the data listings of Appendix A, the data restrictions are indicated by C, coelution; F, fragmented peaks; I, ion ratio criterion not met; P, peak separation; and R, IQS recovery criterion not met. If any of the data restrictions were noted for a particular sample and analyte, the measured concentration was not included in the data summaries or statistical analyses. For example, 15 of the 48 composites failed at least one of the DQOs for 2,3,7,8-TCDF. Thus, there are only 33 unrestricted measurements. Preliminary analyses demonstrated that significant biases can occur if restricted measurements are included in the population estimates.

Data qualifiers are defined in terms of the analytical method's limit of detection (LOD) for each analyte. The analyte is reported as not detected (ND) if the measured concentration is below the LOD, trace (TR) if it is between the LOD and five times the LOD, and positive quantifiable (PQ) if it is greater than five times the LOD. Measured concentrations are reported only for detected (i.e., TR and PQ) analytes. Table 8-1 shows the number of PQ, TR, and ND measurements for each analyte among the unrestricted composites. For example, of the 33 unrestricted measurements of 2,3,7,8-TCDF, 32 were positive quantifiable and one was a trace value.

Table 8-1. Frequency Counts of Data Restrictions and Data Qualifiers in the 48 Samples for FY87 NHATS Composite Samples

	No composites with data restrictions[a]					No. unrestricted composites[c]				
Analyte	Coelution present	Peak separation/ frag. peak present	Ion ratio criteria failed	IQS recovery not in proper range	Total number restricted[b]	PQ	TR	ND	Total number unrestricted[d]	Percent detected[e]
2,3,7,8-TCDF	0	1	14	1	15	32	1	0	33	100
2,3,7,8-TCDD	0	0	11	1	12	35	0	1	36	97
1,2,3,7,8-PeCDF	0	0	3	2	5	3	3	37	43	14
2,3,4,7,8-PeCDF	0	0	7	2	9	38	1	2	41	95
1,2,3,7,8-PeCDD	0	0	13	2	13	31	3	1	35	97
1,2,3,4,7,8-HxCDF	34	0	5	1	39	8	0	1	9	89
1,2,3,6,7,8-HxCDF	0	0	10	1	11	29	5	3	37	92
2,3,4,6,7,8-HxCDF	29	0	2	1	31	1	2	14	17	18
1,2,3,7,8,9-HxCDF	0	0	2	1	3	0	1	44	45	2
1,2,3,4,7,8/ 1,2,3,6,7,8-HxCDD	0	0	6	1	7	41	0	0	41	100
1,2,3,7,8,9-HxCDD	0	2	6	1	9	38	0	1	39	97
1,2,3,4,6,7,8-HpCDF	0	0	21	1	21	24	0	3	27	89
1,2,3,4,7,8,9-HpCDF	0	0	1	1	2	0	2	44	46	4
1,2,3,4,6,7,8-HpCDD	0	0	2	4	6	42	0	0	42	100
OCDF	8	4	13	12	25	3	4	16	23	30
OCDD	0	0	5	12	16	32	0	0	32	100
1,2,3,4,7,8-HxCDD[f]	0	0	1	1	2	-	-	-	16	100
1,2,3,6,7,8-HxCDD[f]	0	0	2	1	3	-	-	-	16	100

[a] Restricted data did not meet data quality objectives.
[b] May be restricted for more than one reason.
[c] PQ = positive quantifiable, TR = trace, ND = not detected.
[d] Number of samples used in the statistical analyses (PQs, TRs, and NDs).
[e] Percent of unrestricted samples.
[f] Results reflect composites where separation of these two isomers was possible. Data qualifiers were not reported.

Table 8-2 gives a summary of the unrestricted data for FY87 NHATS PCDDs and PCDFs. The average, standard deviation, minimum, median, and maximum concentrations were calculated from the unrestricted measurements. However, in calculating these statistics, the value of LOD/2 was used in place of the measured concentration whenever the analyte was not detected. In some cases, the minimum is reported as < LOD, where LOD is the smallest detection limit reported for samples in which the analyte was not detected. The average LOD for all samples and the percent detected are also presented. Detailed data summaries are provided in Appendix E.

The results presented in Table 8-2 and Appendix E are based on simple unweighted averages of the measured concentrations from the composite samples. As such they only summarize the data. Statistical conclusions and estimates of population average concentration levels should only be based on the statistical analysis presented in Sections 8.3 and 8.4.

8.2 POPULATION ESTIMATES

Not all of the analytes provided sufficient data to warrant a meaningful statistical analysis. Two criteria were used to determine which analytes would be statistically analyzed. First, the analyte must be detected (TR or PQ) in at least 50% of the unrestricted composites. This ensures that there will be minimal bias caused by substituting LOD/2 for the measured concentration whenever the analyte was not detected by the analytical method. Also, because sufficient data are needed to estimate model parameters associated with the four analysis factors and three variance analytes, it was decided that a minimum of 30 unrestricted measurements was needed to achieve sufficient precision for the population estimates. Thus, of the original 16 analytes (the pair 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD is counted as one analyte because they could not be separated in most samples) there were nine that met both criteria for performing statistical analyses.

For each of the nine analytes analyzed statistically, Table 8-3 lists the estimated average concentration in the entire U.S. population and in each of the categories defined by the four analysis factors. Also presented is the relative standard error (percent) for each estimate. The estimates and standard errors are based on the additive model analysis described in Section 7.2. The estimates are asymptotically unbiased and were adjusted for population percentages based on the 1980 U.S. Census.

Table 8-2. Summary of Unrestricted Data for FY87 NHATS Composite Samples

Analyte	Sample size[a]	Average LOD (pg/g)	Percent detected[b]	Average concentration[c] (pg/g)	Standard deviation (pg/g)	Concentration range (pg/g)		
						Minimum	Median	Maximum
2,3,7,8-TCDF	33	0.205	100	2.05	0.81	0.893	1.89	3.88
2,3,7,8-TCDD	36	0.291	97	6.60	3.54	< 0.980*	6.54	15.1
1,2,3,7,8-PeCDF	43	0.434	14	0.323	0.292	< 0.066*	0.249	1.42
2,3,4,7,8-PeCDF	39	0.385	95	11.8	7.8	< 0.264*	9.21	29.2
1,2,3,7,8-PeCDD	35	1.34	97	11.8	6.5	< 2.44*	10.2	24.4
1,2,3,4,7,8-HxCDF	9	0.706	89	8.20	4.45	< 3.11*	7.30	17.0
1,2,3,6,7,8-HxCDF	37	0.741	92	5.73	3.72	< 0.556*	5.03	14.3
2,3,4,6,7,8-HxCDF	17	1.00	18	0.707	0.641	< 0.337*	0.479	2.49
1,2,3,7,8,9-HxCDF	45	0.877	2	0.454	0.348	< 0.290*	0.341	1.98
1,2,3,4,7,8/ 1,2,3,6,7,8-HxCDD	41	1.25	100	82.2	42.1	13.3	76.1	174.
1,2,3,7,8,9-HxCDD	39	1.23	97	12.8	5.0	< 3.86*	11.5	22.0
1,2,3,4,6,7,8-HpCDF	27	1.00	89	15.9	7.6	< 1.15*	17.7	32.5
1,2,3,4,7,8,9-HpCDF	46	1.41	4	0.741	0.293	< 0.731*	0.715	1.74
1,2,3,4,6,7,8-HpCDD	42	1.07	100	119.	57.	20.9	110.	230.
OCDF	23	1.67	30	2.05	2.90	< 0.680*	1.19	13.2
OCDD	32	3.75	100	806.	419.	152.	838.	1630.

[a] Number of unrestricted composite samples meeting all DQOs.
[b] Percent of unrestricted samples
[c] Not detected (ND) concentrations are replaced by LOD/2.
* The minimum concentration is less than the minimum reported LOD.

Table 8-3. Estimated Average Concentrations (pg/g) with Relative Standard Errors (%) for Selected PCDDs and PCDFs from FY87 NHATS Composite Samples

Analyte	Entire Nation	Census region				Age group (years)			Race group		Sex group	
		NE	NC	S	W	0-14	15-44	45 +	Caucasian	Non-Caucasian	Male	Female
Population percentages[a]	100	22	26	33	19	23	46	31	83	17	49	51
PCDDs												
2,3,7,8-TCDD	5.38[b]	6.02	5.72	5.19	4.54	1.98	4.37	9.40	4.62	9.15	4.22	6.48
	(6)[c]	(10)	(11)	(11)	(16)	(41)	(12)	(4)	(14)	(29)	(11)	(8)
1,2,3,7,8-PeCDD	10.7	11.3	10.8	10.4	10.3	3.30	9.33	18.2	10.6	11.4	10.8	10.6
	(4)	(8)	(7)	(8)	(12)	(22)	(7)	(4)	(7)	(23)	(6)	(7)
1,2,3,4,7,8/ 1,2,3,6,7,8-HxCDD	75.1	75.5	82.8	66.9	78.7	23.4	70.9	120	71.7	92.0	73.7	76.5
	(4)	(8)	(6)	(7)	(8)	(23)	(6)	(3)	(7)	(19)	(6)	(5)
1,2,3,7,8,9-HxCDD	11.7	10.7	12.7	11.5	11.7	6.13	10.8	17.1	11.5	12.3	11.0	12.3
	(4)	(9)	(7)	(7)	(10)	(18)	(7)	(4)	(8)	(28)	(7)	(6)
1,2,3,4,6,7,8-HpCDD	110	120	115	105	103	45.7	99.8	174	108	123	104	117
	(3)	(7)	(6)	(6)	(9)	(11)	(5)	(3)	(5)	(14)	(5)	(4)
1,2,3,4,6,7,8,9-OCDD	724	765	759	738	608	215	692	1150	718	755	676	771
	(4)	(7)	(6)	(6)	(15)	(17)	(7)	(5)	(5)	(15)	(7)	(5)
PCDFs												
2,3,7,8-TCDF	1.88	1.85	2.00	1.53	2.33	1.97	1.45	2.45	1.81	2.21	1.84	1.91
	(7)	(14)	(14)	(15)	(14)	(11)	(15)	(7)	(12)	(33)	(11)	(11)
2,3,4,7,8-PeCDF	9.70	13.7	10.5	9.51	4.49	1.87	8.00	18.0	9.25	11.9	8.70	10.7
	(8)	(13)	(13)	(15)	(47)	(100)	(15)	(6)	(16)	(44)	(13)	(12)
1,2,3,6,7,8-HxCDF	5.78	7.15	5.57	5.80	4.49	1.80	4.59	10.5	5.60	6.68	5.00	6.52
	(13)	(27)	(24)	(25)	(43)	(83)	(26)	(13)	(25)	(73)	(24)	(19)

[a] population percentage based on 1980 U.S. Census
[b] average concentration (pg/g)
[c] relative standard error (%)

The standard errors are used to characterize the statistical uncertainty in the individual estimates. Uncertainty of an estimate is best expressed by calculating a confidence interval. Approximate 95% confidence intervals are calculated by adding plus or minus two times the standard error to the estimate. For example, the national average concentration of 2,3,7,8-TCDD was estimated to be 5.38 pg/g with a relative standard error of 6%. Thus the approximate 95% confidence interval for the national average is 5.38 ± 0.65 pg/g (4.73 to 6.03 pg/g calculated as 5.38 ± 2x0.06x5.38).

Estimates of the average concentrations in the population categories defined by the four analysis factors are presented even if the effects of those factors were not found to be statistically significant. For example, the regional estimates of 2,3,7,8-TCDD average concentrations range from 4.54 pg/g in the West to 6.02 pg/g in the North East. However, as will be discussed in Section 8.3, the differences among regions for 2,3,7,8-TCDD were not found to be statistically significant.

The age group estimates in Table 8-3 suggest that the concentrations of nearly all analytes increase with the age of the donor. All of the analytes occur at higher concentrations in the oldest age group (45+ years). However, conclusions about the age group effects are based on the statistical tests discussed in the next section.

8.3 HYPOTHESIS TESTING

Statistical hypothesis tests were conducted for each of the target analytes to determine if there are statistically significant differences in average concentrations between individuals from different geographic regions, age groups, sex groups, and race groups. The tests were based on likelihood ratio tests using the additive model analysis as described in Section 7.2.

Table 8-4 lists the attained significance levels for the tests associated with the four analysis factors. The attained significance level is the smallest significance level that will result in rejection of the hypothesis that there are no differences between population averages. For example, the differences among estimated averages of 2,3,7,8-TCDD in the four census regions could only be considered significant at the 0.15 level of significance. On the other hand, the differences in age group averages are significant at the 0.002 level. A 5% (0.05) level of significance is generally the smallest level used to declare statistical significance.

It is clear from Table 8-4 that there are statistically significant differences among the average concentrations in the three age groups. The differences in concentrations between each age group for each compound, except TCDF, were found to be significant. For each of the nine analytes (Table 8-3), the highest average concentration is found in the oldest age

Table 8-4. Significance Levels from Hypothesis Tests for Differences Between Demographic Groups for NHATS FY87 PCDDs and PCDFs

Analyte	Effects due to . . .			
	Census Region[a]	Age Group[b]	Sex Group[c]	Race Group[c]
2,3,7,8-TCDF	0.150	0.002[e]	0.918	0.672
2,3,7,8-TCDD	0.247	< 0.001[e]	0.232	0.341
2,3,4,7,8-PeCDF	0.011[d]	< 0.001[e]	0.603	0.605
1,2,3,7,8-PeCDD	0.960	< 0.001[e]	0.627	0.593
1,2,3,6,7,8-HxCDF	0.847	< 0.001[e]	0.648	0.795
1,2,3,4,7,8/ 1,2,3,6,7,8-HxCDD	0.087[f]	< 0.001[e]	0.934	0.395
1,2,3,7,8,9-HxCDD	0.289	< 0.001[e]	0.597	0.725
1,2,3,4,6,7,8-HpCDD	0.299	< 0.001[e]	0.424	0.393
OCDD	0.655	< 0.001[e]	0.495	0.520

[a] Likelihood ratio tests based on the $\chi^2_{(3)}$ distribution.

[b] Likelihood ratio tests based on the $\chi^2_{(2)}$ distribution.

[c] Likelihood ratio test based on $\chi^2_{(1)}$ distribution.

[d] Indicates significance at the 0.05 level.
[e] Indicates significance at the 0.01 level.
[f] Indicates significance at the 0.10 level.

group (45+ years) and, except for TCDF, the lowest is found in the youngest age group (0-14 years).

The only other statistically significant finding, at the 0.05 level of significance, was that there are possible regional differences in the average concentrations of 2,3,4,7,8-PeCDF. The average concentration in the Western census region was estimated to be 4.49 pg/g compared to the national average of 9.70 pg/g. The highest concentrations of 2,3,4,7,8-PeCDF were found in the North East census region (13.7 pg/g). This regional effect is discussed further in Section 9.0 in consideration of the comparison of the FY87 and FY82 broad scan effort.

Some potential difference in the estimated average concentration between caucasian and non-caucasian and males and females are noted for each of the PCDDs and PCDFs presented in Table 8-3. However, these differences were not statistically significant for any of the modeled compounds based on the statistical hypotesis tests which are summarized in Table 8-4.

8.4 ESTIMATED RATES OF CHANGE OF SELECTED PCDD AND PCDF CONCENTRATIONS

The additive model analysis compared mean concentrations across three age groups, lower (0-14 years), middle (15-44 years) and upper (45+ years). The analysis showed that significant differences existed among mean age group concentrations for the nine modeled analytes. However, the analysis did not quantify rates of change between ages. Therefore, a second set of analyses using linear regression was performed to address this issue.

For each analyte, the measured concentration in each composite sample was regressed against the mean age of all individuals whose specimens had been pooled into that composite. Two linear regressions were performed, the first to estimate the average rate of change in concentration levels from the lower to the middle age groups and the second to estimate the average rate from the middle to the upper age group. These rates were taken as the regression slopes times ten to convert them into rates of pg/g per decade. The plot of the measured concentration of 2,3,7,8-TCDD versus average age with estimated regression lines is presented in Figure 8-1.

The average ages taken over all composites from the lower, middle, and upper age groups were 3.0, 30.8, and 65.0 years, respectively. Therefore, the first rate was the estimated average rate of change per decade from ages 0-31 years, and the second rate was the estimated average rate of change per decade from ages 32+ years. These rates were further standardized by dividing them by the arithmetic average concentration in the first age group

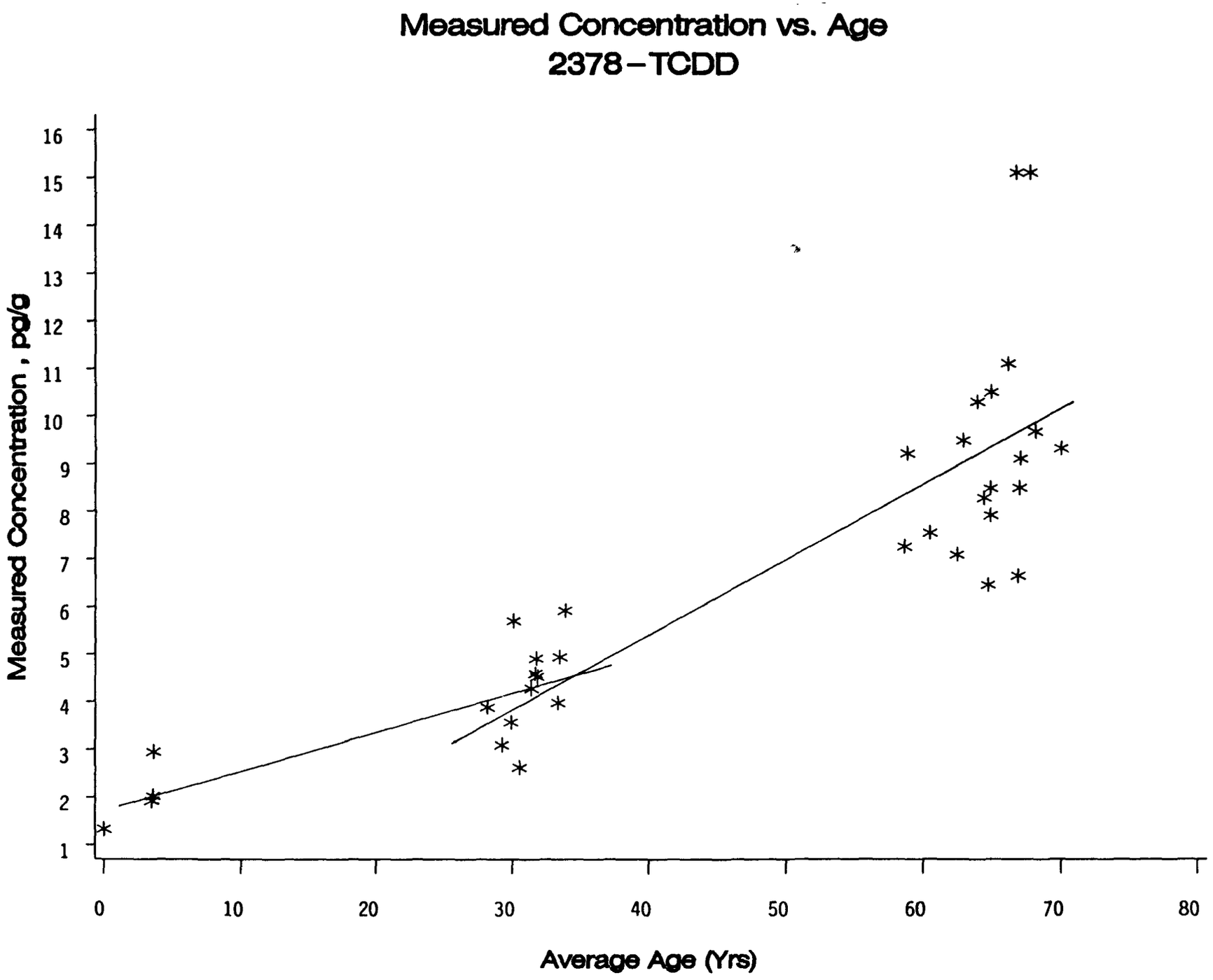


Figure 8-1. Measured concentrations of 2,3,7,8-TCDD versus average age with estimated regression lines.

within each regression set. That is, the first rate was standardized by dividing by the mean concentration for the lower age group, and the second rate was standardized by dividing by the mean concentration for the middle age group. The standardized rates of change were then reported as rates of change per decade, relative to the mean concentration at the beginning of the time interval analyzed. Because the average ages tend to cluster around three points, it is not possible to characterize how the rates change over time. The analysis only provide estimates of the average rates in the two age intervals.

Table 8-5 displays national average concentrations for selected PCDDs and PCDFs, with the two rates of changes expressed as pg/g per decade and percentage of initial mean concentrations per decade, respectively. For example, the national average concentration of 2,3,7,8-TCDD is 5.38 pg/g (as reported in Table 8-3). The rate of change between the lower and middle age groups was estimated by the regression slope as 0.83 pg/g per decade, with a standard error of 0.17. The average concentration of TCDD in the lower age group was 2.06 pg/g (not shown), so the rate of 0.83 divided by 2.06 resulted in the standardized rate of 40% per decade. Similarly, the rate of change between the middle and upper age groups was 1.52 pg/g per decade, with a standard error of 0.20. The average concentration of 2,3,7,8-TCDD in the middle age group was 4.33 pg/g (not shown), so the rate of 1.52 divided by 4.33 resulted in a standardized rate of 35% per decade.

The rate of change of 2,3,7,8-TCDD of 1.52 pg/g decade from the middle to the upper age groups is similar to the value of 2 ppt/decade reported by Patterson et al (1985). Their study investigated adipose tissue samples from individuals of age 35-85, whereas the present study analyzed composite samples from individuals representing all age groups.

All of the rates of change shown in Table 8-5, except for the first rate for 2,3,7,8-TCDF, were positive and highly significant ($p < 0.0001$). For seven of the nine analytes shown, the rates of change per decade increased after age 31. However, when the rates were converted to a percent of initial average concentration, eight of the nine analytes showed a decreased in standardized rates per decade after age 31.

8.5 OUTLIER DETECTION

Prior to conducting the statistical analyses of the FY87 NHATS data, outlier detection procedures were performed to identify possible data entry errors and errors associated with the analytical method (Rogers, 1989). Outlier detection was performed on four types of data: (1) measured concentrations of native analytes, (2) internal quantitation standard recoveries, (3) LODs, and (4) percent lipid values for composite and QC samples.

Table 8-5. Estimated Rates of Change of Selected Dioxin and Furan Concentrations in Human Adipose Tissue

Analyte	National Average Concentration (pg/g)	Donor Age 0-31 Years			Donor Age 32+ Years		
		Avg. Rate pg/g/dec	(SE)	Rate %[a] % per dec	Avg. Rate pg/g/dec	(SE)	Rate %[b] % per dec
Dioxins							
2,3,7,8-TCDD	5.38	0.83	(0.17)	40	1.52	(0.20)	35
1,2,3,7,8-PeCDD	10.7	1.96	(0.23)	52	2.62	(0.33)	28
1,2,3,4,7,8/ 1,2,3,6,7,8-HxCDD	75.1	16.1	(1.11)	61	15.2	(1.72)	21
1,2,3,7,8,9-HxCDD	11.7	1.40	(0.25)	20	1.91	(0.26)	18
1,2,3,4,6,7,8-HpCDD	110	19.2	(2.55)	41	21.8	(2.44)	22
OCDD	724	182	(20.8)	74	130	(24.0)	18
Furans							
2,3,7,8-TCDF	1.88	-0.23	(0.09)	-12	0.37	(0.08)	27
2,3,4,7,8-PeCDF	9.70	1.82	(0.34)	51	2.64	(0.58)	30
1,2,3,6,7,8-HxCDF	5.78	1.03	(0.12)	49	1.49	(0.18)	30

[a] Rate of change expressed as a percent of average concentration per decade (dec) for young age group.
[b] Rate of change expressed as a percent of average concentration per decade (dec) for middle age group.

Logic checks were performed to identify obvious inconsistencies in the data. For example, logic checks would reveal records having recorded concentrations but a data qualifier of "not detected." The extreme studentized deviate (ESD) test statistic was applied to the residuals of a simple linear regression model fit to the measured concentrations and recoveries as another means of detecting outliers. Finally, the secondary outlier procedures were performed using tests for normality, multivariate techniques, and graphical techniques (boxplots).

No data problems were detected in the logic checks phase. However, a total of 62 of 1620 data items were identified as potential outliers. Of these, total 45 were found to be correct readings by the analytical laboratory. The other 17 data items were incorrect readings which were recalculated. The laboratory also reported additional data changes resulting from its own review of the outlier analysis. All data corrections were made to the master dataset before proceeding with the statistical analysis.

8.6 MODEL VALIDATION

Three types of analyses were performed to evaluate the adequacy of the additive model for use on the FY87 NHATS data. All three analyses were based on comparisons of the observed (i.e., measured) and predicted concentrations for the composite samples. Predicted concentrations were calculated using the statistical analysis approaches discussed in Section 7.2. Residuals, which were also used in the model validation analysis, were calculated by taking the differences between the observed and predicted concentrations.

Model validation analyses included (1) residual analysis, (2) normal probability plots, and (3) R-squared analysis. The use of the Shapiro-Wilk tests for normality was also considered. However, in this application, the Shapiro-Wilk test is not appropriate because the data are correlated and variances increase with increasing concentrations.

The residual plots confirmed the model assumption that the variance of the measured concentrations will increase with the average concentration. The plots also show that the distribution of residuals is symmetric. This supports the use of normal models for the sampling and measurement errors. As discussed in Section 7.2.2 the normality assumption is important for ensuring the validity of the hypothesis tests. Nearly all of the probability plots were linear, thus supporting the normality assumption for the errors. Those that were not linear could be explained by the larger variances at the high concentration levels.

Finally, Table 8-6 lists the R-squared correlations between the observed and predicted composite concentrations calculated for each analyte. R-squared can be interpreted as the percent of the total variability in the data (observed concentrations) that can be explained by the model. For example, 81% of the variation in measured composite concentrations of TCDD can be explained by the fixed effects of the additive model. Overall, these correlations demonstrate excellent agreement between the data and the model. Eight of the nine analytes produced R-squared correlations of at least 68%. The lowest value of R-squared (46%) occurs with 2,3,7,8-TCDF. This can be explained by the relatively small age effect on 2,3,7,8-TCDF concentrations. Although there were statistically significant age effects for all nine analytes, the ratio of average concentrations in the lower and upper age groups was only 1.2 (2.45/1.97) for 2,3,7,8-TCDF. The ratios exceeded 2.5 for all the other analytes. Thus, for each of these analytes, the additive model accounts for a large percentage of the total variability in the data.

Table 8-6. R-Squared Correlation[a] Between Predicted and Observed Concentrations for FY87 Dioxins and Furans

Chemical	R^2 (%)
2,3,7,8-TCDF	46
2,3,7,8-TCDD	81
2,3,4,7,8-PeCDF	68
1,2,3,7,8-PeCDD	85
1,2,3,6,7,8-HxCDF	87
1,2,3,4,7,8/1,2,3,6,7,8-HxCDD	88
1,2,3,7,8,9-HxCDD	79
1,2,3,4,6,7,8-HpCDD	88
OCDD	82

[a] R-squared is the square of the Pearson correlation coefficient. It represents the percent of variability in the data that is explained by the predictive model.

9.0 COMPARISON OF FY87 DATA WITH FY82 AND VA/EPA DATA BASES

The analysis of the FY87 NHATS specimens as composites provides a reference point for body burden levels of PCDDs and PCDFs in the general U.S. population. The data generated from the analysis of the FY87 NHATS specimens can be compared with other data bases that have been developed from the analyses of samples collected in North America and Europe. The documentation on the total effort (the compositing design, chemical analysis and statistical treatment of data) offers a means of comparing the significance of the FY87 NHATS data set with two other analysis programs conducted using the NHATS specimen repository. These two studies are the FY82 NHATS broad scan analysis effort and a collaborative study conducted between the U.S. Department of Veterans Affairs (VA) and EPA's Office of Toxic Substances (VA/EPA). The comparisons of these studies extends the utility of the data bases in establishing trends in body burdens of PCDDs and PCDFs and identifies limitations in comparing the results to other data sets. This section gives an overview of these programs and presents comparisons of the compositing designs (FY82 and FY87 NHATS), analytical procedures, results, and statistical methodologies.

The objectives of the FY82 and FY87 NHATS were quite different from those of the VA/EPA study. The FY82 and FY87 NHATS studies were conducted to develop baseline estimates of tissue concentrations. The FY82 and FY87 NHATS tissue specimens were obtained from cadavers and surgical patients. The target population was all noninstitutionalized U.S. citizens in the conterminous 48 states. On the other hand, the primary objective of the VA/EPA study was to compare PCDD and PCDF levels in Vietnam veterans with those found in similar groups of non-Vietnam veterans and civilians. The VA/EPA study was a retrospective study based on archived NHATS specimens. Only specimens from male donors born between 1936 and 1954 were included in the VA/EPA study.

Despite the differences in study objectives, it is possible to compare the average concentrations found in the VA/EPA study and those found in the 15-44 year age group from the two NHATS surveys. The donors in the VA/EPA study were all between 17 and 46 years old at the time of their death or surgery. In Section 8.0 it was concluded that the only factor consistently affecting concentrations of dioxins and furans was the age of the donor. Additional information on the programs are presented in Section 9.1. Comparisons of the study designs, chemical analysis procedures, and the significant results are presented in Sections 9.2, 9.3, and 9.4, respectively.

9.1 OVERVIEW OF THE ANALYTICAL PROGRAMS

The FY82 NHATS specimens were analyzed as composites as part of a broad scan analysis program conducted to expand the utility of the NHATS program beyond the monitoring of organochlorine pesticides and PCBs. Forty-six composite samples were analyzed for tetra- through octachloro PCDDs and PCDFs. The FY82 effort was designed to provide body burden estimates for the general U.S. population based on age, sex, and geographic region.

The VA/EPA study used approximately 200 individual specimens collected from 1971 through 1982. The specimens were from adult males with birthdates between 1936 to 1954 who potentially might have served in the Vietnam War and who possibly had been exposed to the herbicide Agent Orange. These specimens were categorized into three groups: Vietnam veterans, veterans with no military records indicating service in Vietnam, and civilians. The design of the study was intended to determine whether there was any possible difference in the levels of 2,3,7,8-TCDD between groups. The analysis program, however, provided data on all of the 2,3,7,8-substituted chlorinated dioxin and furan congeners. Hence, this data base has generated a considerable amount of information that can be compared with other data bases.

9.2 COMPARISON OF STUDY DESIGNS

Similar sampling designs were used for collecting tissue specimens in the FY82 and FY87 NHATS. Both studies used a multi-staged sampling plan. The conterminous 48 states were divided into strata; MSAs were selected with probabilities proportional to size; and cooperators were solicited and assigned quotas for collecting specimens. There was a minor difference only in the method of stratification. Prior to the FY85 NHATS, MSAs were selected from strata defined by U.S. Census divisions. Beginning with the FY85 NHATS, sampling strata were redefined to be the 17 geographic areas that resulted from the intersection of the nine Census divisions and the ten EPA Regions (Panebianco DL, 1986a). A controlled selection technique, known as the Keyfitz technique (Mack et al, 1984), was used to maximize the probability of retaining MSAs used in previous years. As a result, there were 47 MSAs in the FY87 design compared to 35 in FY82. Otherwise, the sampling designs for the FY82 and FY87 were essentially the same.

A total of 763 specimens were used to generate the composites for the FY82 study, and 865 specimens were included in the composites for the FY87 study. As shown in Table 9-1, the distributions of specimens among the various geographic and demographic subpopulations were similar.

Table 9-1. Marginal Comparisons of FY82 and FY87 NHATS Individual Specimens Used for PCDD and PCDF Analysis

Category	No. of specimens (%) FY82	No. of specimens (%) FY87	1980 Census population (%)
Census Region			
Northeast	166(22)	175(20)	26
North Central	206(27)	296(34)	22
South	331(43)	289(33)	33
West	60(8)	105(12)	19
Total	763	865	
Age Group			
0-14 years	178(23)	146(17)	23
15-44 years	312(41)	318(37)	46
45+ years	273(36)	401(46)	31
Total	763	865	
Sex			
Male	412(54)	436(50)	49
Female	351(46)	429(50)	51
Total	763	865	
Race Group			
Caucasian	632(83)	707(82)	83
Non-Caucasian	131(17)	158(18)	17
Total	763	865	

The FY82 and FY87 NHATS had comparable compositing designs. One of the design criteria for compositing FY87 specimens was to maintain similarity to the FY82 design. Table 9-2 gives a comparison of the marginal percentages of composites in each of the categories defined by the four analysis factors. Population percentages from the 1980 Census are also provided.

Overall, the marginal percentages from the FY82 and FY87 NHATS agree reasonably well with each other and with the census figures. The only differences are the FY87 NHATS had more "pure sex" composites (31 versus 11) than FY82, while the FY82 NHATS had more "pure race" composites (17 versus 8) than FY87.

Because the VA/EPA investigation was a retrospective study using surplus specimens from the NHATS archives, the method used to select specimens was different from the NHATS sampling strategy. Of the approximately 8,000 unused NHATS specimens collected between 1971 and 1982, 528 were collected from males born between 1936 and 1954. However, there was sufficient background information on only 494 donors. Specimens from 40 donors who served in Vietnam were selected, along with randomly selected specimens from 80 non-Vietnam military veterans. Finally, specimens from 80 civilian men were included in the study by matching the birth year (±2 years) and sample collection year (±2 years) of two civilians with each Vietnam veteran. Thus, a total of about 200 specimens from male donors between the ages of 17 and 46 formed the basis for the VA/EPA study. Of the specimens identified, successful analysis was achieved for 197 individuals.

Another major difference between the two NHATS and the VA/EPA study is that specimens in the NHATS studies were composited prior to chemical analysis, while specimens selected for the VA/EPA study were analyzed individually. This difference affects the way in which the data are statistically analyzed, but, as discussed in Section 9.4.2, it does not affect the comparison of average concentration levels.

9.3 COMPARISON OF ANALYTICAL PROCEDURES

To compare the data for the three studies, it is necessary to review the analytical procedures (see Figure 9-1). While the analytical procedures used for the VA/EPA and FY87 efforts were fairly comparable, the figure illustrates that the FY82 approach was considerably different. The changes in the analytical procedures were incorporated in VA/EPA and FY87 studies as an effort to improve the state-of-the-art of the analytical technology between the time frames that each study was conducted.

Table 9-2. Marginal Comparisons of FY82 AND FY87 NHATS Composite Designs

Category	No. of Composites (%)[a] FY82	FY87	1980 Census population %
Census Region			
North Central	12 (26)	15 (31)	26
Northeast	9 (20)	9 (19)	22
South	19 (41)	16 (33)	33
West	6 (13)	8 (17)	19
Total	46	48	
Age Group			
0-14 years	12 (26)	11 (23)	23
15-44 years	17 (37)	17 (35)	46
45+ years	17 (37)	20 (42)	31
Total	46	48	
Sex			
Pure male	6 (55)	16 (52)	49
Mixed	35	17	
Pure female	5 (45)	15 (48)	51
Total	46	48	
Race Group			
Pure Caucasian	11 (65)	8 (100)	83
Mixed	29	40	
Pure Non-Caucasian	6 (35)	0 (0)	17
Total	46	48	

[a] The percent estimates for sex and race groups are calculated as the total number of pure composites within each study design. For example, 6 of the 11 (55%) pure sex composites in the FY82 study design were composed of males only.

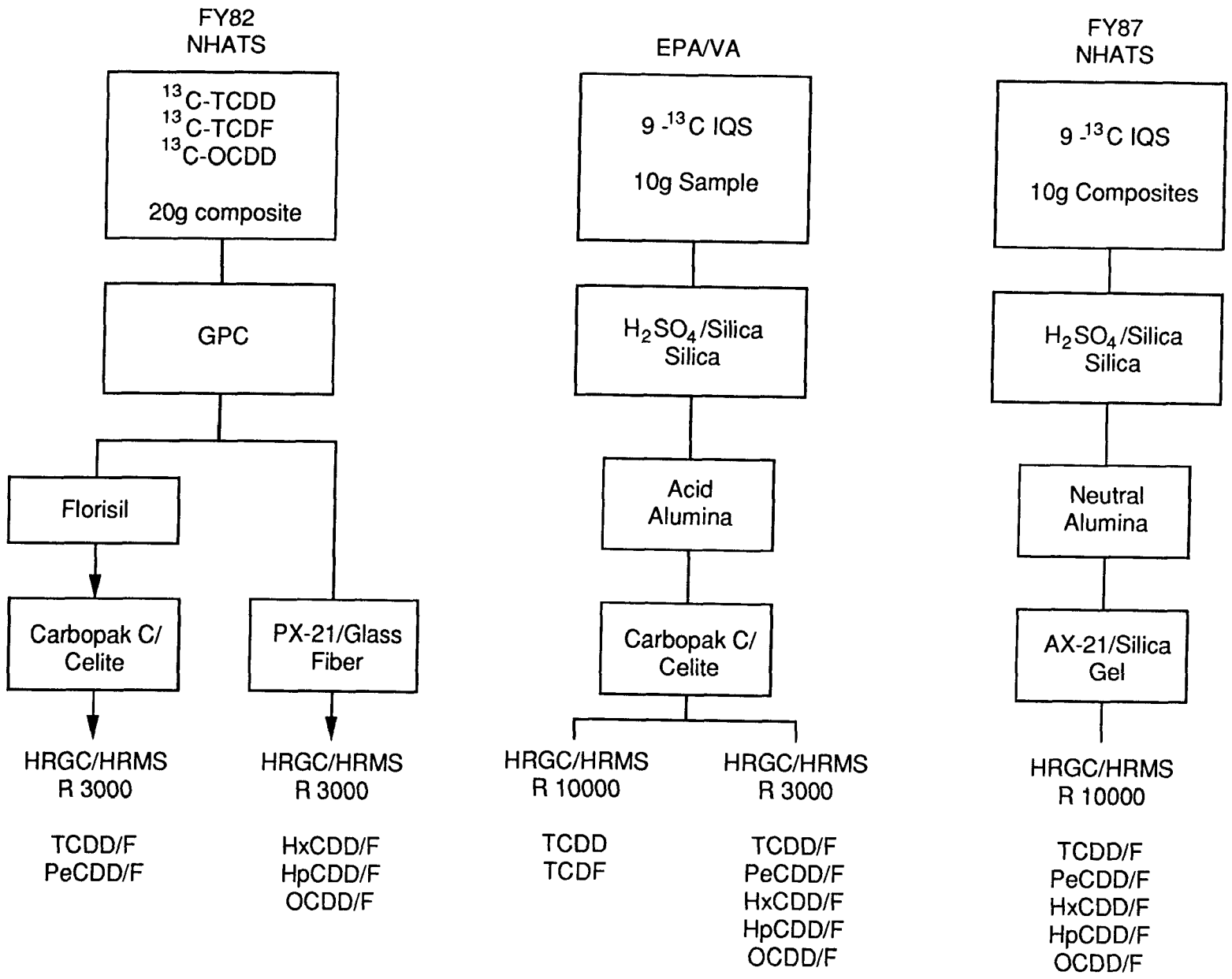


Figure 9-1. Comparison of analytical methods for FY82, VA/EPA, and FY87 studies.

Each procedure required fortification with internal quantitation standards (IQS), extraction, removal of bulk lipid, and separation of interferences from the PCDDs and PCDFs. The techniques for all three studies were essentially equivalent. Extraction was achieved with methylene chloride using a Tekmar Tissuemizer to promote thorough extraction of lipids. Bulk lipid removal for the FY87 and the VA/EPA studies was conducted using identical techniques, consisting of treatment with sulfuric acid-modified silica gel slurries and further cleanup via a chromatographic column of the same material. Gel permeation chromatography was used to remove the bulk lipids in the FY82 composites.

The separation of chemical interferences was achieved using Florisil (FY82 NHATS), acidic alumina (VA/EPA), or neutral alumina (FY87 NHATS). Neutral alumina was used for the FY87 samples rather than acidic alumina to improve method recovery and reduce possible background contributions due to hepta- and octachloro-PCDDs. Previous efforts using Florisil on the FY82 composites had demonstrated poor recovery of the hexa- through octachloro-congeners (USEPA 1986a).

For the final cleanup of sample extracts, a carbon-based column was used. However, as noted in Figure 9-1, three different carbon adsorbents were used between the studies. Two separate extracts were cleaned for the FY82 NHATS composites. Because recovery of the higher chlorinated compounds was poor, an aliquot of the extract taken through the GPC cleanup, but not through Florisil chromatography, was taken through a PX-21/glass fiber column to determine the hexa- through octachloro-PCDDs and PCDFs.

The AX-21/silica gel column used for the FY87 NHATS composites did not provide the degree of cleanup demonstrated with the Carbopak C/Celite used with VA/EPA. This was primarily noted through the detection of octachlorodiphenylethers that interfered with the determination of 1,2,3,4,7,8-HxCDF and 2,3,4,6,7,8-HxCDF in the FY87 composites. The HRGC/HRMS conditions varied across studies (Figure 9-1), depending on whether analyses were conducted using a mass resolution of R = 3,000 or R = 10,000. The higher the R value, the more specific the analyses and the higher the confidence in compound identification. The pattern or fingerprint of the major PCDDs and PCDFs observed in the HRGC/HRMS chromatograms for human adipose tissue was consistent across all these studies.

Two factors have the largest potential effect on data comparability among the three studies: the type and number of IQS and the consistent use of analytical standards (Figure 9-2). Only three IQS compounds were available for the FY82 composites. Since the calculation for PCDDs and PCDFs is based on an isotope dilution principle, the limitation on the FY82 composites is the assumption that all compounds will recover the same as the IQS. Recovery data for the additional IQS compounds in the FY87 and VA/EPA studies demonstrate

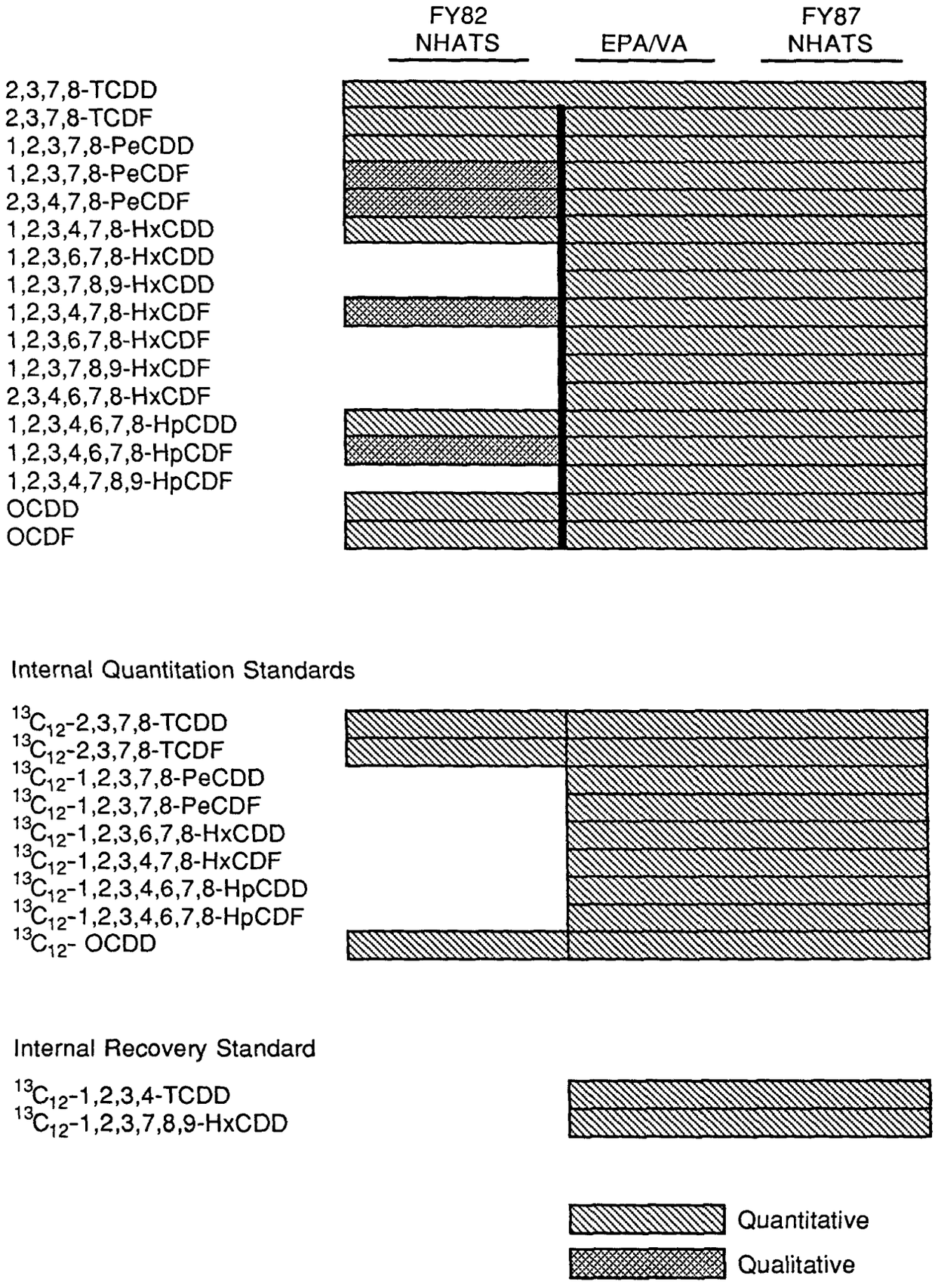


Figure 9-2. Comparison of analytical standards from the FY82, VA/EPA, and FY87 studies.

that PCDDs and PCDFs recoveries differ depending on the degree of chlorination. For these reasons, only the data for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and OCDD are directly comparable between the other two studies. Since the same sets of IQS standards were used between the studies, the FY87 and VA/EPA studies are comparable.

The second factor, standard traceability, is another serious consideration. Figure 9-2 shows that all standards are directly comparable between the FY87 and the VA/EPA studies. When considering the standards for the FY82 composites, only the 2,3,7,8-TCDD standard is directly comparable across all three studies. The standards analyzed with the VA/EPA and FY87 studies (including the 2,3,7,8-TCDD for FY82) were verified through participation in interlaboratory comparisons and the analysis of an NBS standard reference material. The results of these interlaboratory studies support the quantitation of results reported in these studies and also promote the comparability between human tissue data sets generated by the other laboratories participating in these studies.

9.4 COMPARISON OF RESULTS

The results from the FY87 NHATS, the FY82 NHATS, and the VA/EPA studies are compared in this section. Because the same study design was used for the two NHATS surveys, it is possible to make a more detailed comparison of those two sets of results. A statistical comparison of the FY82 and FY87 results is presented in Section 9.4.1. The VA/EPA results are compared with the FY82 and FY87 NHATS in Section 9.4.2.

9.4.1 Statistical Comparison of FY82 and FY87 NHATS Results

The results from the FY82 and FY87 NHATS were statistically compared to determine if there were significant changes in average PCDD and PCDF concentration levels over the five-year period. However, as discussed in Section 9.3, advancements were made in the analytical method for the FY87 survey. The most significant change was that additional internal quantitative standards (IQSs) were available for the penta-, hexa-, and heptachloro-compounds. Therefore, only results for the tetra- and octa- compounds are expected to be directly comparable. Statistical comparisons were performed on all compounds for which data were generated in both years. Comparisons were made, not only between the predicted national averages, but also between the demographic profiles from the two surveys. The profile analysis, discussed in detail later (9.4.1.3), examines possible changes in the differences across demographic subpopulations. This type of comparison is valuable even if systematic differences in concentration levels exist that can be attributed to the analytical methodologies.

Statistical comparisons were possible only when sufficient data were available from both surveys. The criteria for performing a model-based comparison were: (1) the chemical must be detected in at least 50% of the composite samples each year, and (2) there must be at least 30 analyzed composite samples in each year. The additive model, described in Section 7.0, was used for the model-based comparisons between the FY82 and FY87 NHATS. Since the FY82 data were originally analyzed using a multiplicative model (EPA 1989), the FY82 results presented in this comparison are different from those previously published (USEPA 1990a).

Table 9-3 shows the type of comparison used for each of the target chemicals. Six analytes met the criteria for a model-based (M) comparison. The other four analytes, analyzed in both years, were compared in a descriptive manner using weighted averages (WA). This approach is discussed later. (See 9.4.1.4.)

As discussed in Section 8.0, data restrictions derived from the data quality objectives were imposed on the FY87 data but were not applied in FY82. Thus, in Table 9-3, the number of composites listed for FY87 represents the number with unrestricted measurements, while the number for FY82 is the total number of available composites.

The comparison of FY82 and FY87 results is divided into four parts. Section 9.4.1.1 compares the FY82 and FY87 results in terms of limits of detection (LODs) and the percent of composite samples for which each analyte was detected. Estimates of the national averages for the six analytes statistically analyzed using the additive model are compared in Section 9.4.1.2. The results of the profile analyses are presented in Section 9.4.1.3. Finally, in Section 9.4.1.4, a descriptive comparison of weighted national averages is presented for the four analytes that were not statistically modeled.

9.4.1.1 <u>**Comparison of LODs and Prevalence Detected**</u>. Table 9-4 compares the percent of composite samples in which the analytes were detected and the average detection limit (LOD) for each year. In FY82, LODs were only calculated when the concentration was either not detected (ND) or qualified as a trace (TR) value. Thus, the sample size for calculating the average LOD in FY82 was often much less than the number of samples analyzed. For example, TCDD was either not detected or found at a trace level in 13 of the 43 composite samples for the FY82 study. The average LOD reported for the 13 samples was 7.28 pg/g. In FY87, LODs were calculated for all composite samples.

Table 9-3. Number of Composite Samples and Types of Statistical Comparisons Made Between FY82 and FY87 NHATS Results

Analyte	Number of composite samples		Type of comparison[c]
	FY82[a]	FY87[b]	
2,3,7,8-TCDF	43	33	WA
2,3,7,8-TCDD	43	36	M
1,2,3,7,8-PeCDF	-	43	-
2,3,4,7,8-PeCDF	43	39	M
1,2,3,7,8-PeCDD	41	35	M
HxCDF[d]	45	9-45	WA
HxCDD[d]	45	39-41	M
1,2,3,4,6,7,8-HpCDF	45	27	WA
1,2,3,4,7,8,9-HpCDF	-	46	-
1,2,3,4,6,7,8-HpCDD	45	42	M
OCDF	45	23	WA
OCDD	45	32	M

[a] Number of available measurements. Two outliers were removed for 1,2,3,7,8-PeCDD.
[b] Number of unrestricted composite sample measurements.
[c] WA = weighted averages
M = model results and profile analysis
- = no data available for FY82
[d] Analyte analysis results for specific isomers of HxCDF and HxCDD were combined (summed) for comparisons between FY82 and FY87.

Table 9-4. Comparisons of LODs and Percent Detected for PCDDs and PCDFs Between NHATS FY82 and FY87

Analyte	FY82 N[a]	FY82 Percent detected	FY82 N[b]	FY82 Average LOD (pg/g)	FY87 N[c]	FY87 Percent detected	FY87 Average LOD (pg/g)	Change in percent detected (87-82)
2,3,7,8-TCDF	43	26	34	9.94	33	100	0.205	74*[d]
2,3,7,8-TCDD	43	74	13	7.28	36	97	0.291	23*
1,2,3,7,8-PeCDF	-	-	-	-	43	14	0.434	-
2,3,4,7,8-PeCDF	43	88	9	17.7	39	95	0.385	7
1,2,3,7,8-PeCDD	43	93	15	39.3	35	97	1.34	4
HxCDF[e]	45	71	19	17.0	47	79[f]	0.696[g]	8
HxCDD[e]	45	98	4	29.3	47	100[f]	1.19[g]	2
1,2,3,4,6,7,8-HpCDF	45	93	7	9.01	27	89	1.00	-4
1,2,3,4,7,8,9-HpCDF	-	-	-	-	46	4	1.41	-
1,2,3,4,6,7,8-HpCDD	45	98	1	26.0	42	100	1.07	2
OCDF	45	40	33	19.0	23	30	1.67	-10
OCDD	45	100	0	-	32	100	3.75	0

[a] Number of composites analyzed.
[b] Number of composites with calculated LODs.
[c] Number of unrestricted composites.
[d] Indicates difference is greater than zero at 0.05 level of significance according to Fisher's exact test.
[e] Chemical analysis results for specific isomers of HxCDF and HxCDD were combined (summed) for comparison between FY82 and FY87.
[f] Analyte is detected if any of the four (HXCDF) or three (HXCDD) isomers were detected.
[g] The smallest of the LODs among the isomers is used to calculate the average LOD.

There clearly was a significant improvement in the sensitivity of the analytical method in FY87. The average detection limits in FY82 were in the range of 9 to 50 times higher than those in FY87. This, most likely, explains the statistically significant increase in the percent of samples in which TCDF and TCDD were detected between FY82 and FY87. For example, 2,3,7,8-TCDD was detected in only 74% of the samples in FY82 but was detected in 97% of the samples in FY87. This difference (23%) is statistically significant; however, the average detection limit was decreased from 7.28 pg/g to 0.291 pg/g. The average 2,3,7,8-TCDD concentration in both years was estimated to be around 5.5 pg/g.

9.4.1.2 Comparison of National Average Estimates for Modeled Analytes. Table 9-5 shows the estimated national average concentrations for FY82 and FY87 and the estimated difference (FY87-FY82) in concentrations for the six analytes statistically analyzed. The standard error of each estimate and the significance level for testing that the difference is different from zero also are provided. The test was based on the approximate t-statistic of the form

$$t = \frac{NA_{87} - NA_{82}}{\sqrt{SE_{87}^2 + SE_{82}^2}}$$

where NA_{82} and NA_{87} are the FY82 and FY87 national average estimates and SE_{82} and SE_{87} their standard errors, respectively. Approximate significance levels were calculated using the standard normal distribution.

Generally, levels less than 0.05 are used to indicate statistical significance. Using this criterion, the average predicted concentrations of 2,3,4,7,8-PeCDF, 1,2,3,7,8-PeCDD, and HxCDD are significantly lower in FY87 than in FY82. However, as mentioned earlier, additional internal quantitation standards were used in the FY87 analytical procedures. Thus, we cannot conclude that the average concentration in the U.S. population changed between FY82 and FY87. The differences may be due only to the changes in the analytical method. Data from the VA/EPA study also demonstrated significantly different concentrations for 2,3,4,7,8-PeCDF and 1,2,3,7,8-PeCDD comparable with the FY87 levels. No significant differences in the average or predicted levels of 2,3,7,8-TCDD and OCDD were noted between FY82 and FY87. The same internal quantitation standards were used in both years for these specific analytes.

Table 9-5. Comparisons of NHATS FY82 and FY87 Predicted National Average Concentrations of PCDDs and PCDFs in Human Adipose Tissue

Analyte	Estimated national average pg/g FY82 Avg.	FY82 (SE)[a]	FY87 Avg.	FY87 (SE)[a]	Difference FY87-FY82 Diff.	(SE)[b]	Statistical significance level
2,3,7,8-TCDD	5.88	(0.534)	5.38	(0.330)	-0.499	(0.628)	0.213
2,3,4,7,8-PeCDF	35.4	(3.57)	9.70	(0.800)	-25.7	(3.66)	< 0.001
1,2,3,7,8-PeCDD	73.6	(20.0)	10.7	(0.426)	-62.9	(20.0)	0.001
HxCDD[c]	122	(20.1)	86.8	(3.16)	-34.8	(20.3)	0.043
1,2,3,4,6,7,8-HpCDD	142	(24.8)	110	(3.73)	-31.6	(25.0)	0.104
OCDD	768	(79.7)	724	(28.6)	-43.9	(84.7)	0.302

[a] Standard error (pg/g) of estimated average.
[b] Standard error (pg/g) of difference.
[c] Results for specific isomers of HxCDD were combined (summed) for comparison between FY82 and FY87.

9.4.1.3 Results of Profile Analysis. Profile analysis is a multivariate statistical technique that is used to compare profiles of two or more populations. In this context, a profile is a vector of estimates of subpopulation averages. An example of a profile is the set of estimated average TCDD concentrations for the four geographic regions (NC, NE, S, and W) in the FY87 NHATS.

Profile plots were used to make a visual assessment of the differences in the FY82 and FY87 results. In order to determine what differences may exist and whether these difference are statistically significant, a formal profile analysis was conducted.

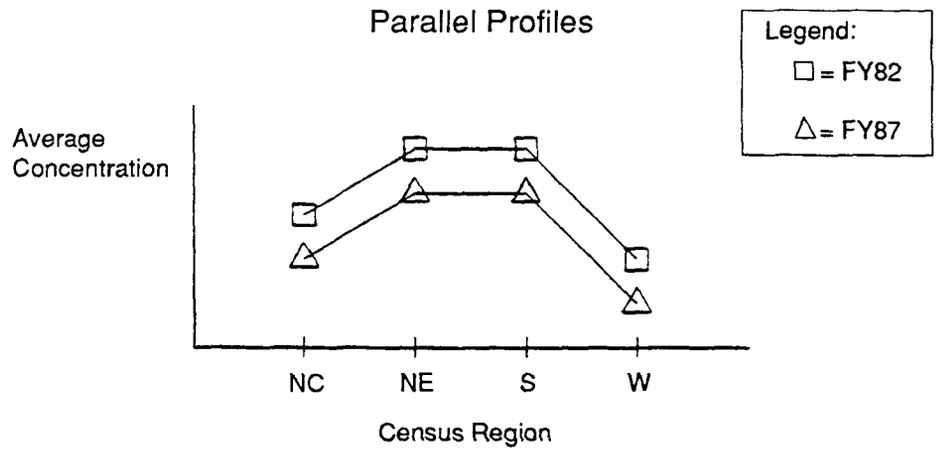
Profile analysis tests a sequence of statistical hypotheses to determine how two or more multivariate populations may differ. The hypotheses address the following questions in order:

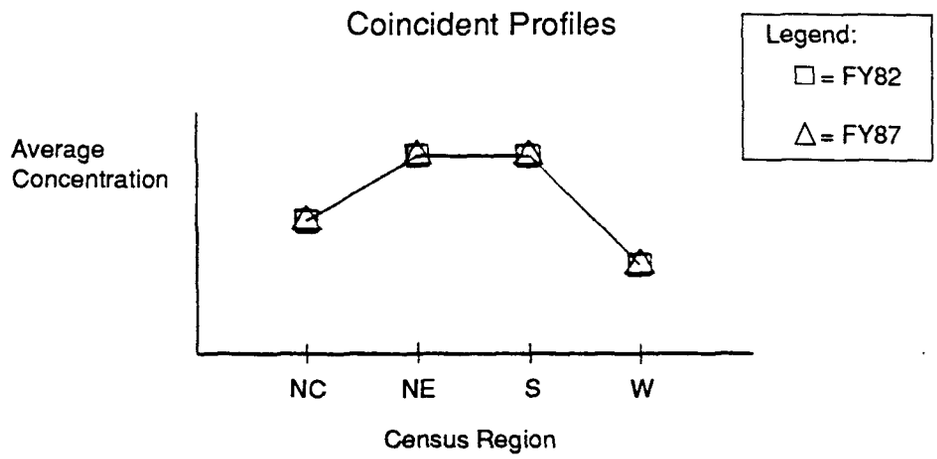
1. Are the profiles from the two fiscal years parallel?
2. Assuming the profiles are parallel, are they coincident?
3. Assuming the profiles are coincident, do they have equal levels?

Two profiles are said to be parallel if all pairwise differences between the average concentrations across the levels of a demographic factor are equal. For coincident profiles, all pairwise differences are equal to zero. Finally, coincident profiles are said to have equal levels if there are no differences among the average concentrations at different level of a demographic factor. The different types of profiles are illustrated in Figure 9-3.

Profile tests are performed sequentially. The hypothesis of coincident profiles is only tested if the hypothesis of parallelism is not rejected. Similarly, the hypothesis of equal levels is only tested if the hypotheses of parallelism and coincidence are not rejected. The third test combines the FY82 and FY87 data to test for significant effects of the demographic factors. This approach described by Johnson and Wichern (1982) was used to conduct the profile analysis.

To provide the background for the profile analysis, Table 9-6 compares the FY82 and FY87 significance levels from testing for differences among demographic groups. The additive model was used to perform the tests, with the assumption of normally distributed errors for both years. Although these assumptions were found to be reasonable for the FY87 data, the large measurement errors from the chemical analysis of FY82 composites indicate that the assumption of normality may not be true for the FY82 data. However, because of these data in FY82, it is difficult to verify any distributional assumptions.





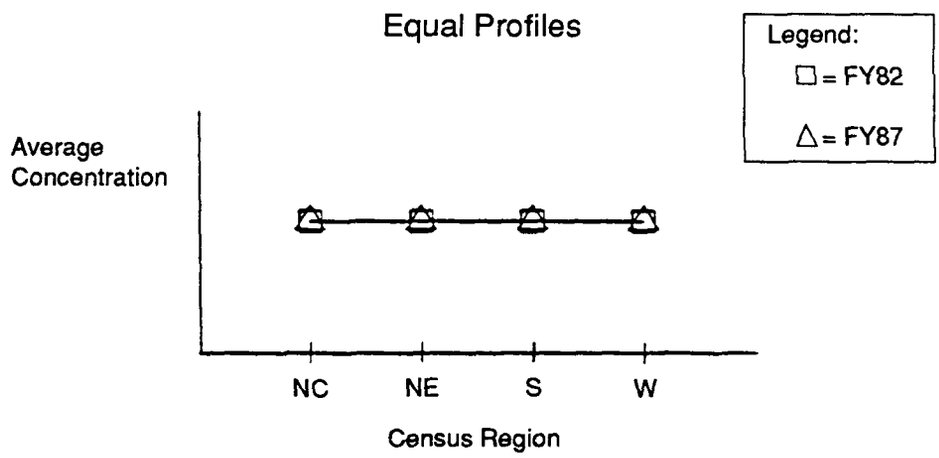


Figure 9-3. Example of profile plots.

Table 9-6. Comparisons of NHATS FY82 and FY87 Significance Levels[a] from Hypothesis Tests for Differences Between Demographic Groups for Selected PCDD and PCDF Levels

Analyte	Effects due to ...							
	Census region		Age group		Sex group		Race group	
	FY82	FY87	FY82	FY87	FY82	FY87	FY82	FY87
2,3,7,8-TCDD	0.484	0.247	0.031*	< 0.001*	0.962	0.232	0.861	0.341
2,3,4,7,8-PeCDF	0.047*[b]	0.011*	0.054	< 0.001*	0.613	0.603	0.657	0.605
1,2,3,7,8-PeCDD	0.006*	0.960	0.018*	< 0.001*	0.922	0.627	0.985	0.593
HxCDD (Total)	< 0.001*	--	0.885	--	0.413	--	0.967	--
(1,2,3,4,7,8/1,2,3,6,7,8)	--	0.087	--	< 0.001*	--	0.934	--	0.395
(1,2,3,7,8,9)	--	0.289	--	< 0.001*	--	0.597	--	0.725
1,2,3,4,6,7,8-HPCDD	0.003*	0.299	0.913	< 0.001*	0.451	0.424	0.735	0.393
OCDD	0.260	0.655	0.054	< 0.001*	0.709	0.495	0.953	0.520

[a] Significance levels for both years were calculated using the additive model analysis.
[b] The asterisk (*) indicates significance at the 0.05 level.

In FY82, measured concentrations for four of the six analytes were found to be significantly different among the four census regions. In FY87, 2,3,4,7,8-PeCDF was the only analyte with significant regional differences. On the other hand, in FY87, age effects were much more evident. In FY82, only 2,3,7,8-TCDD and 1,2,3,7,8-PeCDD showed significant age effects at the 0.05 significance level, and 2,3,4,7,8-PeCDF and OCDD were significant at the 0.054 level. None of the analytes in either year showed significant differences in measured concentrations among the different sex or race groups.

Profiles of the six modeled analytes in FY82 and FY87 are plotted for each of the four analysis factors (region, age, race, and sex) in Figures 9-4 through 9-9. The estimated average concentrations within each analysis factor and fiscal year are connected with straight lines. The vertical lines define approximate 95% confidence limits of the estimates. The confidence limits were calculated by adding ±2 times the standard error of the estimated average. The estimated national averages with 95% confidence limits are also plotted.

The numerical results—estimated average concentrations and their standard errors—of the profile analyses are presented in Tables 9-7 through 9-10. The estimated difference (FY87-FY82) and their standard error for each region are also presented in each table. Finally, the significance levels of sequential tests for parallel profiles, coincident profiles, and equal levels are provided.

Profile analyses were performed for five of the six modeled analytes. Total HxCDD was not included, because in FY82 only the total HxCDD was measured, while in FY87 each individual isomer was measured. To perform the profile analysis on HxCDD, it would have been necessary to combine the individual isomer data for FY87. This would have required making certain assumptions that would produce extremely conservative results.

2,3,7,8-TCDD

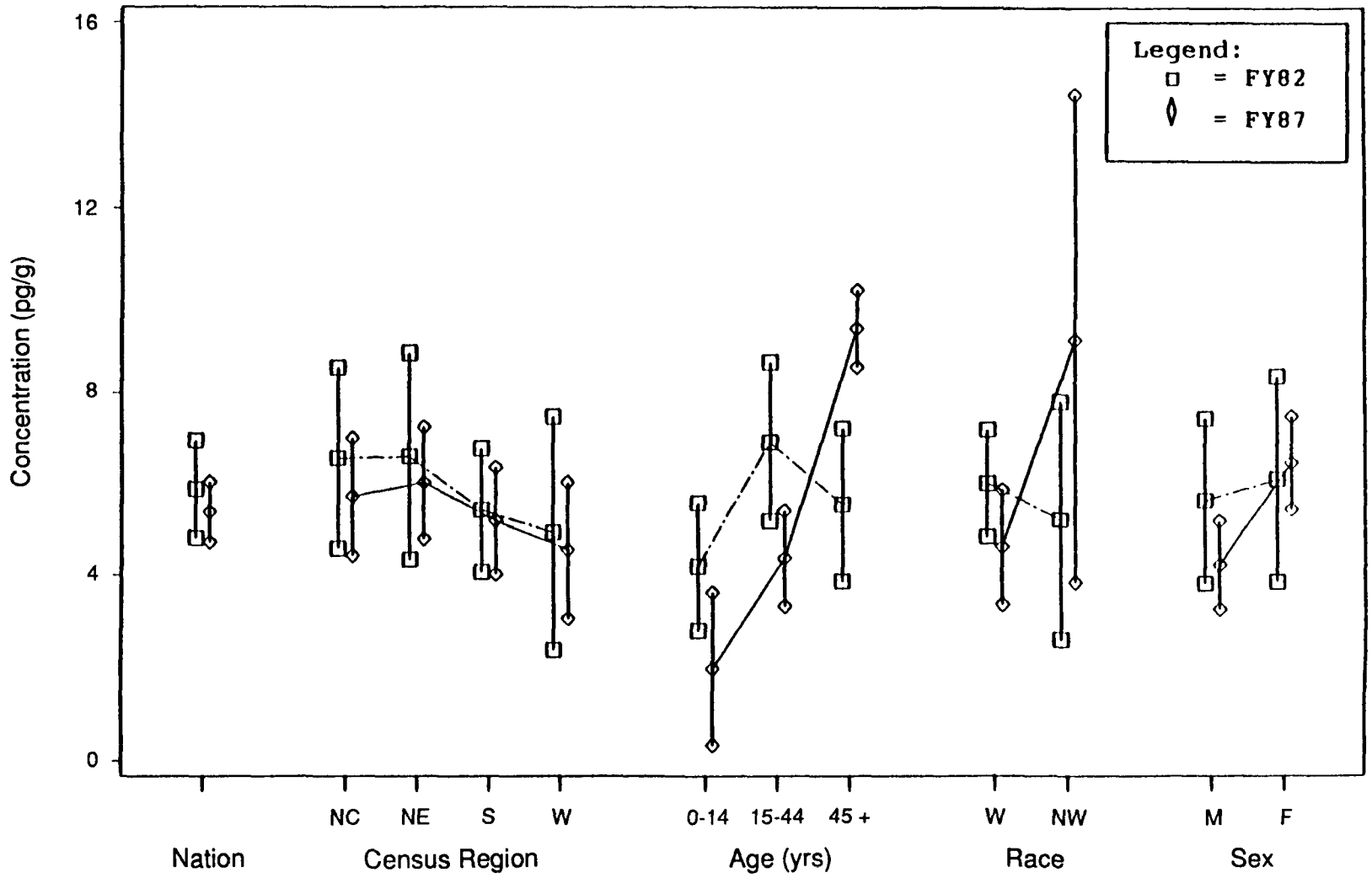


Figure 9-4. Profile plots of average 2,3,7,8-TCDD concentrations with 95% confidence limits for FY82 and FY87 adipose tissue samples.

2,3,4,7,8-PeCDF

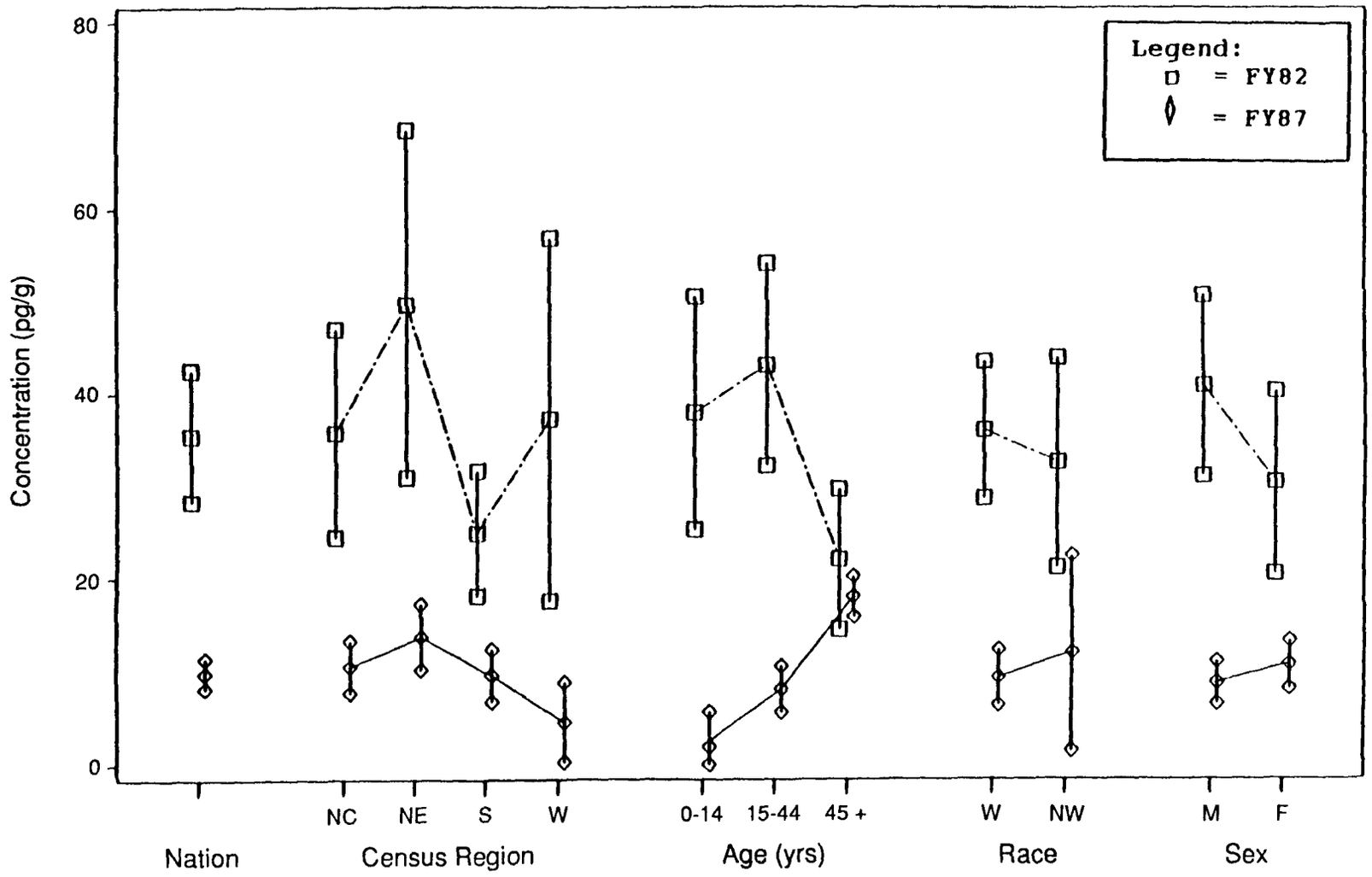


Figure 9-5. Profile plots of average 2,3,4,7,8-PeCDF concentrations with 95% confidence limits for FY82 and FY87 adipose tissue samples.

1,2,3,7,8-PeCDD

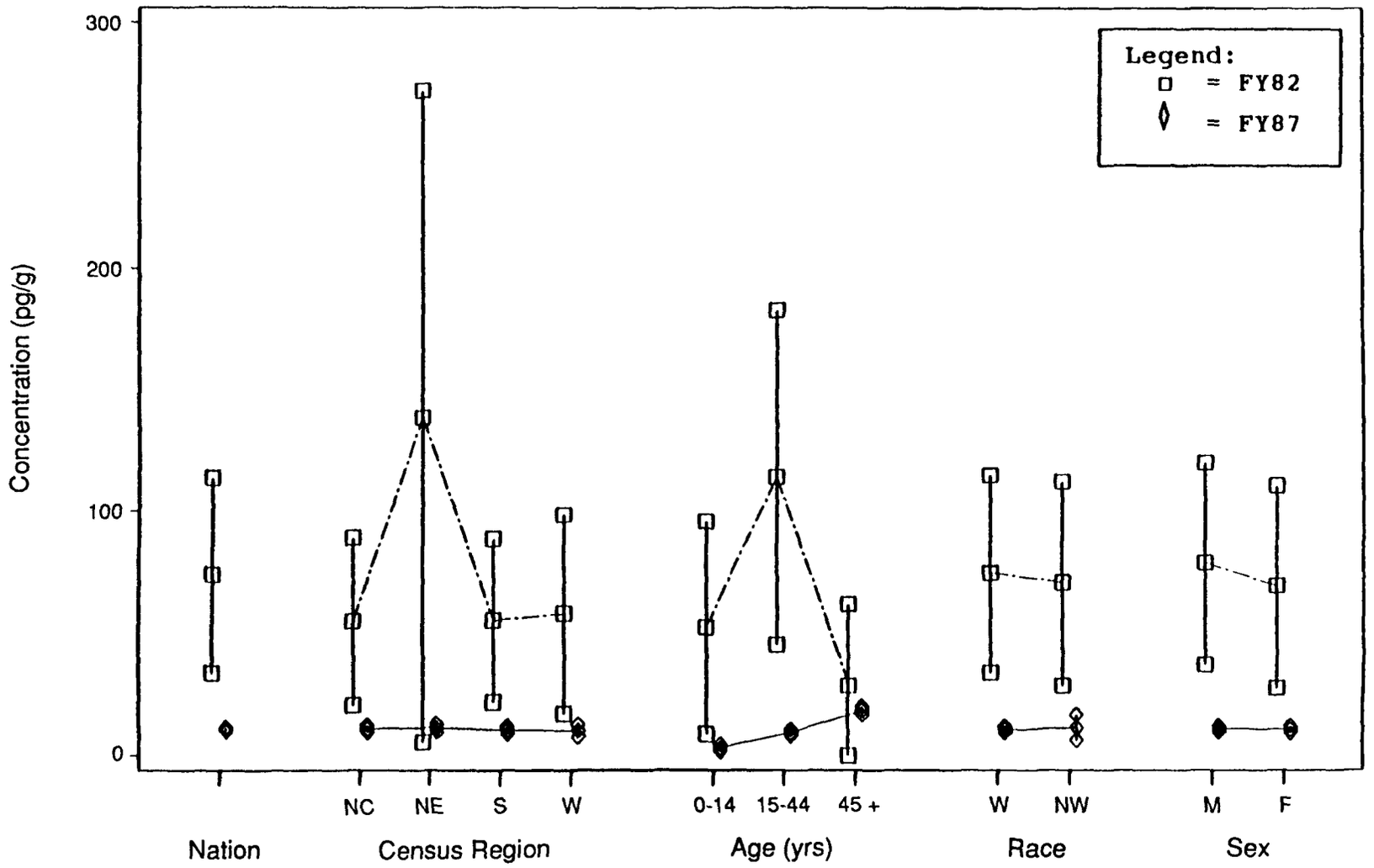


Figure 9-6. Profile plots of average 1,2,3,7,8-PeCDD concentrations with 95% confidence limits for FY82 and FY87 adipose tissue samples.

HxCDD

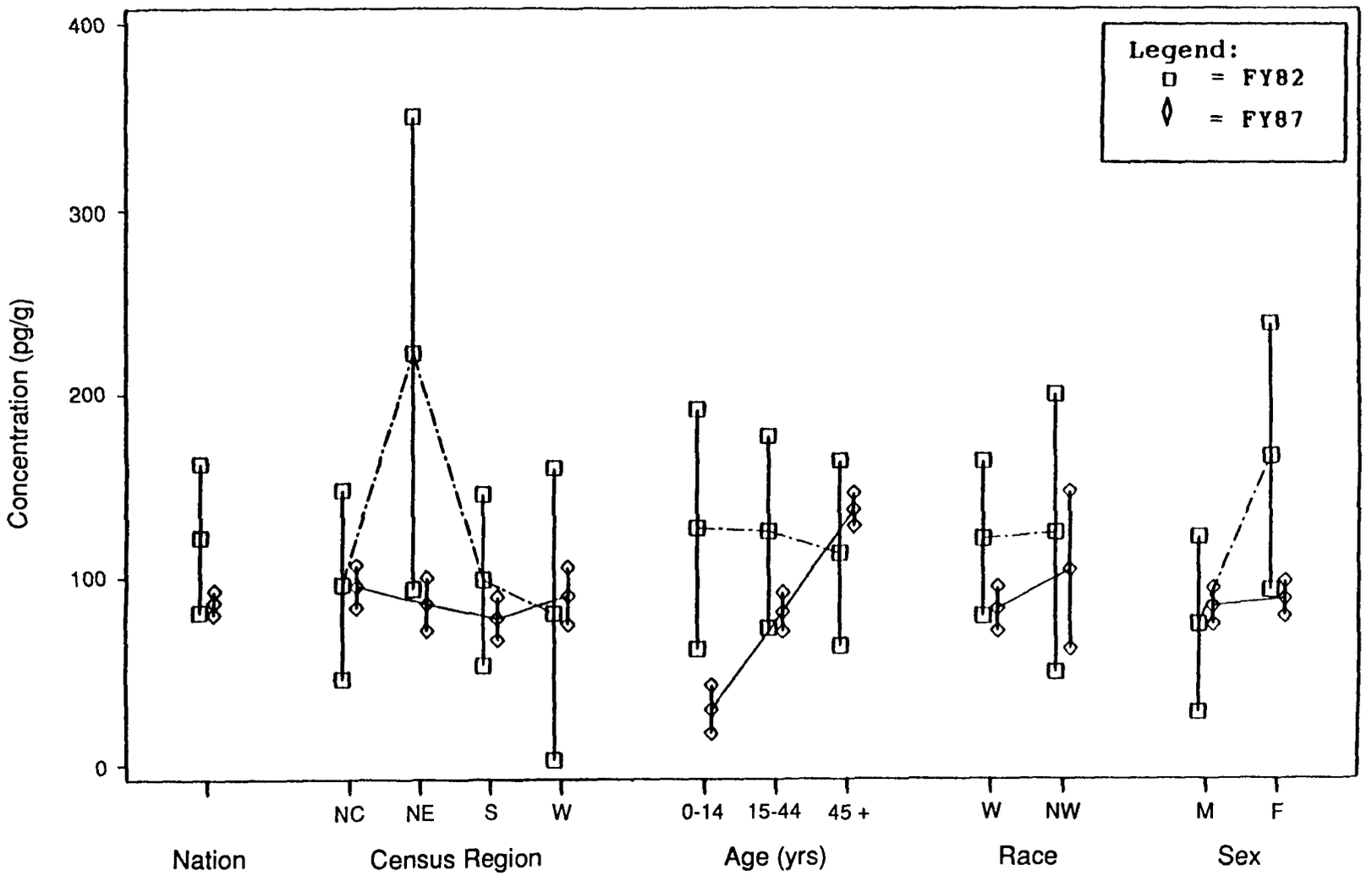


Figure 9-7. Profile plots of average HxCDD concentrations with 95% confidence limits for FY82 and FY87 adipose tissue samples.

1,2,3,4,6,7,8-HpCDD

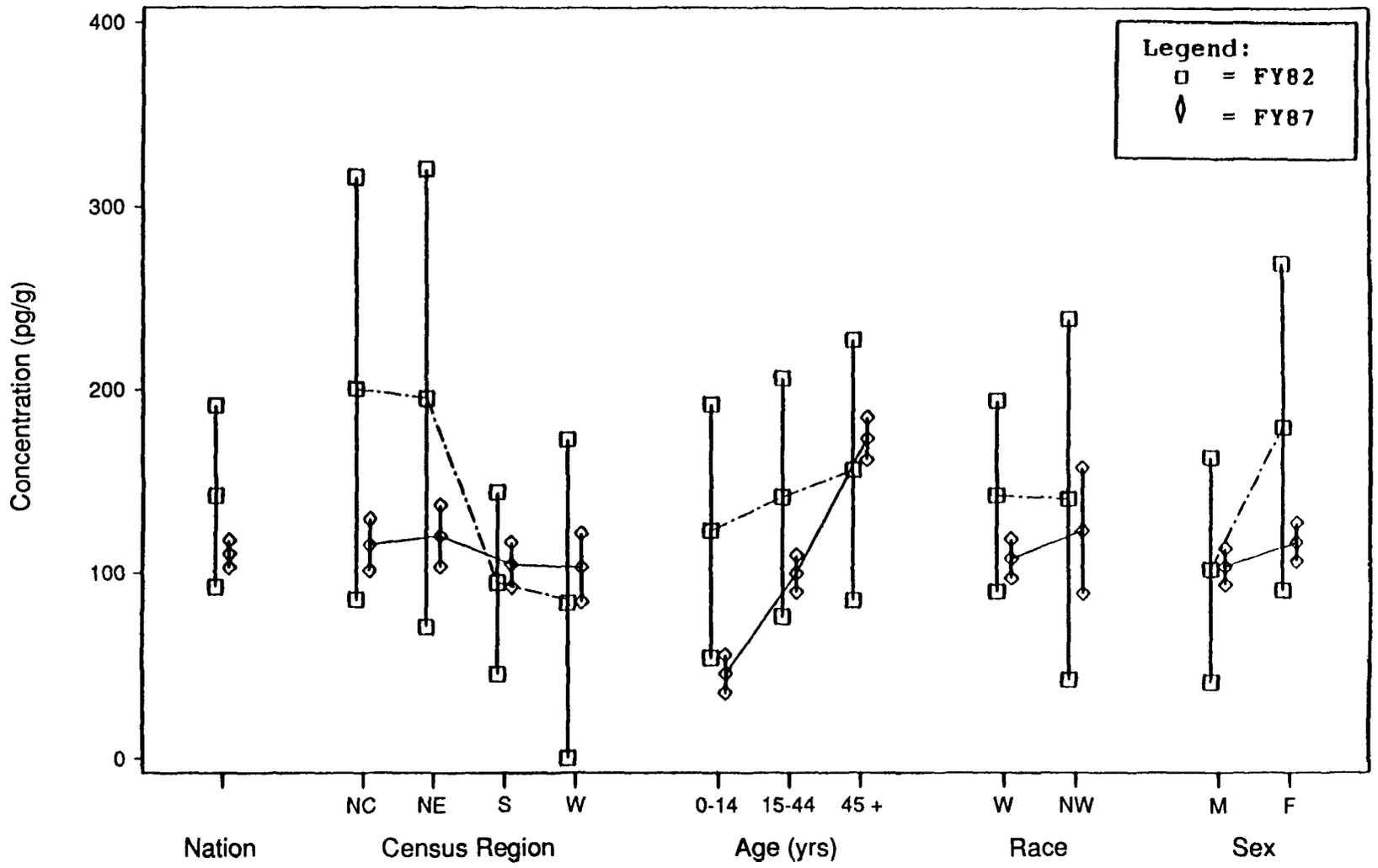


Figure 9-8. Profile plots of average 1,2,3,4,6,7,8-HpCDD concentrations with 95% confidence limits for FY82 and FY87 adipose tissue samples.

OCDD

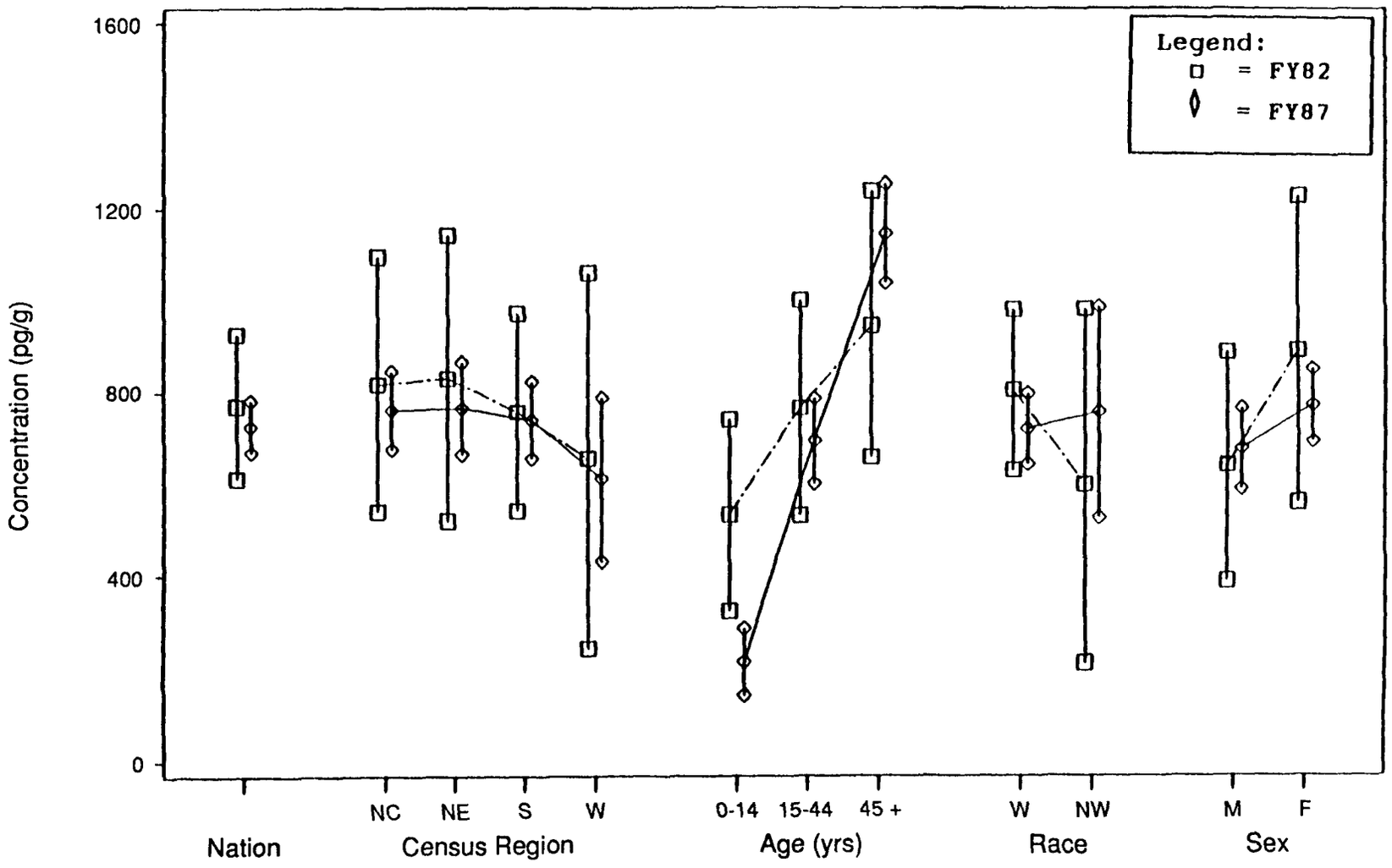


Figure 9-9. Profile plots of average OCDD concentrations with 95% confidence limits for FY82 and FY87 adipose tissue samples.

Table 9-7. Results of Profile Analysis Comparing FY82 and FY87 Predicted Concentrations of Selected PCDDs and PCDFs in Human Adipose Tissue - By Census Region

Analyte	Census region	Estimated average (pg/g) FY82 Avg.	FY82 (SE)	FY87 Avg.	FY87 (SE)	Difference (pg/g) FY87-FY82 Diff.	(SE)	Significant levels for sequential test of. . . Parallel profiles	Coincident profiles	Equal levels
2,3,7,8-TCDD	North Central	6.57	(0.992)	5.72	(0.645)	-0.852	1.18	0.977	0.435	0.235
	North East	6.60	(1.13)	6.02	(0.615)	-0.583	1.29			
	South	5.42	(0.677)	5.18	(0.584)	-0.235	0.894			
	West	4.92	(1.27)	4.54	(0.743)	-0.381	1.48			
2,3,4,7,8-PeCDF	North Central	35.7	(5.47)	10.5	(1.39)	-25.2	5.81	0.060	< 0.001*	--
	North East	49.6	(9.40)	13.7	(1.77)	-36.0	9.56			
	South	24.8	(3.36)	9.51	(1.38)	-15.3	3.63			
	West	37.2	(9.80)	4.49	(2.12)	-32.7	10.0			
1,2,3,7,8-PeCDD	North Central	54.6	(17.1)	10.8	(0.762)	-43.8	17.1	0.666	0.002*	--
	North East	139	(66.7)	11.3	(0.854)	-127	66.7			
	South	55.1	(16.6)	10.4	(0.801)	-44.7	16.6			
	West	57.8	(20.3)	10.3	(1.26)	-47.5	20.3			
1,2,3,4,6,7,8-HpCDD	North Central	201	(57.6)	115	(6.98)	-85.4	58.0	0.268	0.212	0.088
	North East	196	(62.3)	120	(8.32)	-75.3	62.9			
	South	94.6	(24.6)	105	(6.07)	9.9	25.3			
	West	83.6	(44.7)	103	(9.32)	19.0	45.6			
OCDD	North Central	817	(140)	759	(43.3)	-58.0	146	0.994	0.605	0.603
	North East	831	(156)	765	(51.0)	-66.2	164			
	South	756	(108)	738	(42.6)	-18.1	116			
	West	652	(204)	608	(89.1)	-44.4	223			

* Indicates statistical significance at 0.05 level.

Table 9-8. Results of Profile Analysis Comparing FY82 and FY87 Predicted Concentrations of Selected PCDDs and PCDFs in Human Adipose Tissue - By Age Group

Analyte	Age group	Estimated average (pg/g) FY82 Avg.	FY82 (SE)	FY87 Avg.	FY87 (SE)	Difference (pg/g) FY87-FY82 Diff.	(SE)	Significant levels for sequential test of. . . Parallel profiles	Coincident profiles	Equal levels
2,3,7,8-TCDD	0 - 14 yr	4.19	(0.694)	1.98	(0.819)	-2.21	1.07	< 0.001*	--	--
	15 - 44 yr	6.93	(0.871)	4.37	(0.520)	-2.56	1.01			
	45 + yr	5.54	(0.842)	9.40	(0.413)	3.90	0.938			
2,3,4,7,8-PeCDF	0 - 14 yr	37.9	(6.32)	1.87	(1.86)	-36.0	6.59	< 0.001*	--	--
	15 - 44 yr	43.1	(5.49)	8.00	(1.23)	-35.1	5.62			
	44 + yr	22.0	(3.78)	18.0	(1.08)	-3.98	3.93			
1,2,3,7,8-PeCDD	0 - 14 yr	52.2	(21.7)	3.29	(0.726)	-48.9	21.7	0.004*	--	--
	15 - 44 yr	114	(34.4)	9.33	(0.667)	-105	34.4			
	45 + yr	28.5	(16.5)	18.2	(0.793)	-10.3	16.5			
1,2,3,4,6,7,8-HpCDD	0 - 14 yr	123	(34.6)	45.7	(5.11)	-77.7	35.0	0.097	0.166	0.002*
	15 - 44 yr	142	(32.6)	99.8	(5.02)	-41.7	33.0			
	45 + yr	156	(35.6)	174	(5.83)	17.0	36.0			
OCDD	0 - 14 yr	531	(104)	215	(35.7)	-316	110	0.018*	--	--
	15 - 44 yr	765	(118)	692	(47.0)	-73.0	127			
	45 - yr	947	(145)	1150	(53.0)	199	155			

* Indicates statistical significance at 0.05 level.

Table 9-9. Results of Profile Analysis Comparing FY82 and FY87 Predicted Concentrations of Selected PCDDs and PCDFs in Human Adipose Tissue - By Race Group

Analyte	Race group	Estimated average (pg/g) FY82 Avg.	FY82 (SE)	FY87 Avg.	FY87 (SE)	Difference (pg/g) FY87-FY82 Diff.	(SE)	Significant levels for sequential test of. . . Parallel profiles	Coincident profiles	Equal levels
2,3,7,8-TCDD	Caucasian	6.02	(0.591)	4.62	(0.629)	-1.40	0.863	0.125	0.332	0.286
	Non-Caucasian	5.21	(1.30)	9.15	(2.65)	3.90	2.96			
2,3,4,7,8-PeCDF	Caucasian	36.0	(3.71)	9.25	(1.47)	-26.7	4.00	0.462	< 0.001*	--
	Non-Caucasian	32.4	(5.69)	11.9	(5.24)	-20.5	7.73			
1,2,3,7,8-PeCDD	Caucasian	74.3	(20.3)	10.6	(0.703)	-63.7	20.3	0.664	0.002*	--
	Non-Caucasian	70.3	(20.9)	11.4	(2.61)	-58.9	21.1			
1,2,3,4,6,7,8-HpCDD	Caucasian	142	(26.2)	108	(5.37)	-34.4	26.7	0.759	0.402	0.808
	Non-Caucasian	141	(49.2)	123	(17.2)	-17.4	52.1			
OCDD	Caucasian	803	(88.4)	718	(39.0)	-85.1	96.6	0.338	0.751	0.503
	Non-Caucasian	596	(193)	755	(115)	159	225			

* Indicates statistical significance at 0.05 level.

Table 9-10. Results of Profile Analysis Comparing FY82 and FY87 Predicted Concentrations of Selected PCDDs and PCDFs in Human Adipose Tissue - By Sex

Analyte	Sex	Estimated average (pg/g) FY82 Avg.	FY82 (SE)	FY87 Avg.	FY87 (SE)	Difference (pg/g) FY87-FY82 Diff.	(SE)	Significant levels for sequential test of... Parallel profiles	Coincident profiles	Equal levels
2,3,7,8-TCDD	Male	5.63	(0.909)	4.22	(0.480)	-1.41	1.03	0.352	0.399	0.151
	Female	6.21	(1.13)	6.48	(0.511)	0.36	1.24			
2,3,4,7,8-PeCDF	Male	40.8	(4.90)	8.70	(1.12)	-32.1	5.03	0.077	< 0.001*	--
	Female	30.3	(4.95)	10.7	(1.27)	-19.7	5.11			
1,2,3,7,8-PeCDD	Male	78.5	(20.7)	10.8	(0.638)	-67.7	20.7	0.405	0.002*	--
	Female	69.0	(20.8)	10.6	(0.733)	-58.4	20.8			
1,2,3,4,6,7,8-HpCDD	Male	102	(30.6)	104	(4.93)	1.60	31.0	0.278	0.215	0.123
	Female	180	(44.7)	117	(5.17)	-62.9	45.0			
OCDD	Male	638	(125)	676	(44.2)	38.0	133	0.538	0.618	0.176
	Female	892	(167)	771	(39.9)	-121	172			

* Indicates statistical significance at 0.05 level.

Census Region Profiles

Table 9-7 shows that between FY82 and FY87 there are no differences at the 5% significance level in the profiles of 2,3,7,8-TCDD, 1,2,3,4,6,7,8-HpCDD, and OCDD. Furthermore, based on the combined FY82 and FY87 data, there are no significant differences in the concentrations of these analytes among the geographic regions (parallel profiles). The profile comparisons for 2,3,7,8-TCDD and OCDD are consistent with the individual test results (see Table 9-6), which indicate no significant geographic effects for these analytes. There was a significant geographic effect for 1,2,3,4,6,7,8-HpCDD in FY82, but the effect was not statistically confirmed in FY87. However, the marginal significance (0.088 significance level) of the combined tests for geographic effects and the fact that the South and West Census Regions had the lowest estimated average concentrations in both FY82 and FY87 suggest the possibility of geographic effects for 1,2,3,4,6,7,8-HpCDD.

The profile analysis suggests that the differences in 2,3,4,7,8-PeCDF and 1,2,3,7,8-PeCDD concentrations between FY82 and FY87 are constant across all geographic regions (parallel profiles). This could be due to changes in the body burden levels or, as suspected, differences in the analytical methods. The hypothesis of coincident profiles was rejected at the 5% level for these two analytes. This could be explained by systematic differences in measured concentrations. As mentioned earlier, because the profiles for 2,3,4,7,8-PeCDF and 1,2,3,7,8-PeCDD were not coincident, the combined test for geographic effects was not performed. However, in both FY82 and FY87, the highest estimated average concentrations were found in the Northeast Census Region. The geographic effect was found to be statistically significant for 2,3,4,7,8-PeCDF concentrations in both FY82 and FY87, but for 1,2,3,7,8-PeCDD, it was only significant in FY82.

Age Group Profiles

The comparison of profiles by age is presented in Table 9-8. The profile analysis rejects the hypothesis of parallel profiles at the 0.05 level for four of the five analytes, the only exception being 1,2,3,4,6,7,8-HpCDD. Even in this case, the test was marginally significant at the 0.10 level. In FY87, the test for age effects (Table 9-6) was significant at the 0.05 level for each of the modeled analytes. They were also significant or nearly significant at the 0.05 level for the same analytes in FY82, but the estimated average concentrations of 2,3,7,8-TCDD, 2,3,4,7,8-PeCDF, and 1,2,3,7,8-PeCDD in FY82 were not increasing with age. Also, the estimated concentrations of OCDD were increasing in FY82, the rate of increase is lower than the rate observed in FY87. Thus, even though there were significant age effects for these four analytes in both FY82 and FY87, the hypothesis of parallel profiles was rejected in each case.

The hypothesis of parallel profiles for 1,2,3,4,6,7,8-HpCDD was only marginally rejected at the significance level of 0.10. In both FY82 and FY87, the estimated average concentrations increased with age group, but the differences among age groups in FY82 were not statistically significant, possibly because of large measurement errors. Also, the rate of increase of estimated concentrations of 1,2,3,4,6,7,8-HpCDD was lower in FY82 than in FY87. Assuming that the profiles of 1,2,3,4,6,7,8-HpCDD are parallel, the hypothesis of coincident profiles is not rejected, but that of equal concentrations among the three age groups based on the combined data is rejected at the 0.002 significance level.

Race and Sex Profiles

The profile analysis comparing race groups (Table 9-9) and sexes (Table 9-10) produced results similar to those for age groups. The findings are: (1) there are no significant differences in the profiles of 2,3,7,8-TCDD, 1,2,3,4,6,7,8-HpCDD, and OCDD between FY82 and FY87; (2) the estimated differences in the concentrations of 2,3,4,7,8-PeCDF and 1,2,3,7,8-PeCDD between FY82 and FY87 are statistically significant and consistent across different age groups and sexes; and (3) there are no significant differences in the concentrations of any of these analytes among different age groups and sexes.

9.4.1.4 **Weighted Average Comparison of Analytes Not Statistically Modeled.** Four analytes, measured in FY82 and FY87, but which did not meet the criteria for statistical modeling in both years, were compared using weighted averages. First, the average concentration of composites in each of the three age groups was computed for each analyte. For example, the 2,3,7,8-TCDF averages in FY87 were 2.03, 1.34, and 2.50 pg/g in the youngest to oldest age groups, respectively. Next, these averages were weighted by the population percentages from the 1980 census. For 2,3,7,8-TCDF, the weighted average concentration in FY87 was

$$1.86 \text{ (pg/g)} = 0.23(2.03 \text{ pg/g}) + 0.46(1.34 \text{ pg/g}) + 0.31(2.50 \text{ pg/g}).$$

This type of national average estimate is likely to be more accurate than the simple average of the composite concentrations, because there was strong evidence from the statistical analysis of FY87 modeled compounds that age has a significant effect on concentrations of PCDDs and PCDFs in human adipose tissue. Additional calculations were needed to compare HxCDF concentrations, since each of the HxCDF isomers were individually analyzed in FY87, while only the total HxCDF was measured in FY82. Thus, the weighted averages of the individually measured isomers were summed to estimate the total HxCDF concentration in FY87.

Table 9-11, which gives the weighted average estimates and standard errors for each of these compounds, shows large decreases in the average measured concentrations of 2,3,7,8-TCDF and OCDF from FY82 to FY87. The average measured concentration of OCDF was 56.0 pg/g in FY82 and only 2.28 pg/g in FY87. In addition, there were significant advances in the HRMS methodology for analyzing the FY87 composites. For example, as presented in Table 9-4, the average LOD for OCDF was 19.0 pg/g in FY82 and 1.67 pg/g in FY87. The standard errors of the estimated average concentrations are also considerably larger for the FY82 data. These facts suggest the differences may be due to changes in the analytical method.

There also are differences in the weighted averages of HxCDF and 1,2,3,4,6,7,8-HpCDF. The averages are 47% and 35% higher in FY82 than in FY87 for the two analytes, respectively.

9.4.2 Comparison of FY82, FY87 NHATS and VA/EPA Study Results

The results of the VA/EPA study, as reported by Kang et al. (1990) and Bauer et al. (1990), are presented in Table 9-12 with data from the 15 to 44 age group from the FY82 and FY87 NHATS. No statistical comparisons are made, because the NHATS and VA/EPA studies had different objectives and data collection strategies.

Table 9-12 shows the average concentrations of selected analytes from specimens collected in three-year periods beginning in 1971 from the VA/EPA study. The combined averages for all specimens collected between 1971 and 1982 are also presented. Since the specimens were taken from male donors born between 1936 and 1954, the ages of the donors were between 17 and 46 years. These averages are compared with the average concentrations from NHATS composites containing specimens from donors in the 15-44 year age group. These composites did contain specimens from female donors; however, there has not been any statistical evidence linking concentrations of these analytes with the sex of the donor. Therefore, comparisons of the average concentrations from the NHATS and VA/EPA studies is possible. The standard errors of the average concentrations and the number of specimens or composite samples used to calculate the averages are also presented in Table 9-12.

There are obvious differences between the NHATS and VA/EPA results. Except for concentrations of 1,2,3,7,8-PeCDD in FY82, the NHATS averages are considerably lower than the corresponding averages from the VA/EPA study. For example, the average concentrations of 2,3,7,8-TCDD from FY82 and FY87 NHATS are 6.87 and 4.33 pg/g, respectively, while the average concentration in the VA/EPA specimens is 14.1 pg/g.

Table 9-11. Weighted Average Concentrations and Standard Errors (SE) of Selected PCDFs from FY82 and FY87 NHATS

Analyte	FY82 N[a]	FY82 Weighted[b] average Conc. (pg/g)	FY82 (SE)	FY87 N[c]	FY87 Weighted[b] average Conc. (pg/g)	FY87 (SE)
2,3,7,8-TCDF	43	39.7	(19.4)	33	1.86	(0.0949)
HxCDF	45	20.9	(2.15)	45	14.2[d]	(1.42)
1,2,3,4,6,7,8-HpCDF	45	20.6	(2.41)	27	15.3	(1.38)
OCDF	45	56.0	(21.8)	23	2.28	(0.980)

[a] Number of composite samples.
[b] Age group averages are weighted by population counts.
[c] Number of unrestricted composite samples.
[d] Sum of weighted averages from the four isomers of HXCDF.

Table 9-12. Arithmetic Averages (pg/g), Standard Errors, and Number of Samples for Selected PCDDs Obtained from the VA/EPA, FY82 NHATS (15-44 Age Group), and FY87 NHATS (15-44 Age group) Studies by Collection Year Category

Analyte	VA Study[a] 1971-73	VA Study[a] 1974-76	VA Study[a] 1977-79	VA Study[a] 1980-82	VA Study[b] 1971-82	NHATS[c] FY82	NHATS[c] FY87
2,3,7,8-TCDD	19.9[d]	17.3	11.6	12.6	14.1	6.87	4.33
	1.52[e]	1.92	0.80	1.50	0.78	0.68	0.28
	27[f]	29	57	82	197	17	12
1,2,3,7,8-PeCDD	21.7	23.7	17.9	17.3	18.9	125	9.48
	1.64	2.92	1.09	1.42	0.86	47.5	0.31
	27	29	57	82	197	17	12
1,2,3,4,7,8/ 1,2,3,6,7,8-HxCDD	173	190	159	158	164	g	70.8
	12.5	18.0	11.5	14.5	7.6	g	2.28
	27	29	57	82	197	g	13
1,2,3,4,6,7,8-HpCDD	364	291	267	244	273	114	99.8
	38.7	36.3	26.7	33.2	17.7	23.7	5.13
	27	29	57	82	197	16	14
OCDD	1,530	1,400	1,230	1,130	1,270	760	726
	143	186	114	130	71.8	66	59.5
	27	29	57	82	197	16	9

[a] Concentrations for not detected were replaced with LOD/2. The specimens for the four groups added to 195 (age information was not available for two specimens).
[b] Statistics based on all study specimens; concentrations not detected (ND) were replaced with LOD/2.
[c] Statistics based on composites in 15-44 yr. age group; concentrations not detected (ND) were replaced with LOD/2.
[d] Arithmetic average (pg/g).
[e] Standard error.
[f] Number of individual specimens or composites analyzed.
[g] Data reported for the FY82 composites included the measured levels of the 1,2,3,7,8,9-HxCDD.

Average concentrations, with standard errors for additional analytes, are provided in Table 9-13. The VA/EPA results (Bauer et al. 1990) are compared with the average concentrations for NHATS composite samples containing specimens from donors in the 15-44 year age group. As indicated, many of the compounds were detected in fewer than 50% of the composites. In those cases, the reported average concentrations may be significantly affected by the estimated LODs. For compounds detected in more than 50% of the composites, the average concentrations from the NHATS studies are considerably lower than those obtained in the VA/EPA study. The only exception is that the measured concentrations of 2,3,4,7,8-PeCDD are much higher in the FY82 NHATS.

Two explanations for the differences in the levels found in these studies are possible. First, the apparent decline in PCDD and PCDF concentrations reflects a decline in PCDD and PCDF residues in the general environment over the same time frame. This is a logical possibility resulting from regulations promulgated and enforced since 1970 and environmental awareness that has focused attention on releases of toxic chemicals via industrial effluents and handling of hazardous wastes.

The second possibility for the differences observed between the VA/EPA and the FY87 NHATS results may be attributed to storage stability. Since PCDDs and PCDFs are very persistent, stability is affected by the integrity of the tissue rather than the chemicals. Some of the specimens in the VA/EPA study had been stored since 1971 before being analyzed in 1986 (16 years later). The NHATS composite samples from FY82 and FY87 were analyzed within two to three years of the specimen collections.

Although further evidence is required, the data in Table 9-12 suggest a correlation between time in storage and measured concentrations. However, the effect on storage time is completely confounded with the effect of collection year. Thus, it is not possible to determine which factor is causing the observed effect on concentration. To address the issue of storage time versus collection year, further studies will be necessary to either directly study the storage effect or identify a surrogate measure of tissue stability. Storage stability can be studied through development of quality control pools that can be stored with the NHATS archives and pulled for analysis with each analysis program.

Table 9-13. Arithmetic Averages (pg/g), Standard Errors, and Sample Sizes for Selected Analytes Obtained from the VA/EPA, FY82 NHATS (15-44 Age Group), and FY87 NHATS (15-44 Age Group) Studies

Analyte	VA Study[a] 1971-82	NHATS[b] FY82	NHATS[b] FY87
2,3,7,8-TCDF	2.1[c]		1.34
	0.177[d]		0.098
	197[e]		9
1,2,3,7,8-PeCDF	0.6		0.244*[f]
	0.062		0.042
	197		16
2,3,4,7,8-PeCDF	23.0	39.4	8.71
	1.071	4.62	0.543
	197	17	14
1,2,3,4,7,8-HxCDF	21.4		7.13
	1.005		0.817
	197		5
1,2,3,6,7,8-HxCDF	10.9		4.63
	0.581		0.376
	197		15
2,3,4,6,7,8-HxCDF	3.3		0.281*
	0.192		0.001
	197		2
1,2,3,7,8,9-HxCDF	0.33		0.370*
	0.021		0.043
	197		14
1,2,3,7,8,9-HxCDD	18.0		10.7
	0.837		0.298
	197		15
1,2,3,4,6,7,8-HpCDF	36.5	21.1	15.9
	1.822	3.39	1.88
	197	16	10
1,2,3,4,7,8,9-HpCDF			
	1.4		0.726*
	0.082		0.048
	197		15
OCDF	3.1		2.89*
	0.290		2.07
	197		6

[a] Includes all study specimens; concentrations not detected (ND) were replaced with LOD/2.
[b] Statistics based on composites in 15-44 yr. age group; concentrations not detected (ND) were replaced with LOD/2.
[c] Arithmetic average (pg/g).
[d] Standard error.
[e] Number of individual specimens or composites analyzed.
[f] * Indicates detection in fewer than 50% of the FY87 composites.

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APPENDIX A

FY87 NHATS COMPOSITE DATA LISTED BY ANALYTE FOR EACH COMPOSITE

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 2,3,7,8-TCDF
Internal Quantitation Standard = 13C12-2,3,7,8-TCDF
Target Ion Ratio = 0.76

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	PQ	0.1	1.76			76.7
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.049	0.953	R		162.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	IR10		1.42	I	0.17	56.0
1	16258	ACD8700318	14	S	1	0.0	42.9	PQ	0.109	1.83			66.8
1	16259	ACD8700112	14	S	1	50.0	92.9	PQ	0.088	1.35			76.6
1	16260	ACD8700069	20	NC	3	100.0	85.0	PQ	0.055	2.01			86.7
1	16261	ACD8700461	13	S	1	61.5	53.8	IR10		2.56	I	1.06	73.4
1	16262	ACD8700194	32	NE	3	100.0	96.9	PQ	0.114	2.11			77.6
1	16263	ACD8700390	23	S	3	100.0	69.6	IR10		1.96	I	1.10	76.7
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	0.16	1.26			76.9
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	0.163	3.66			81.4
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	0.126	0.893			79.3
2	16269	ACD8700283	24	W	3	100.0	79.2	PQ	0.179	3.32			75.6
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	0.182	3.37			83.1
2	16272	ACD8700489	25	S	3	56.0	88.0	PQ	0.18	1.69			74.3
2	16274	ACD8700096	23	NC	3	0.0	82.6	IR10		3.74	I	0.92	79.7
2	16275	ACD8700416	17	NC	1	41.2	76.5	PQ	0.147	1.49			73.2
2	16276	ACD8700014	18	NC	1	100.0	44.4	PQ	0.216	1.99			75.9
2	16277	ACD8700274	16	W	2	18.8	87.5	IR25		1.49	I	0.96	70.4
3	16279	ACD8700185	32	NE	3	0.0	90.6	PQ	0.314	1.54			62.0
3	16280	ACD8700292	16	W	3	0.0	93.8	IR10		2.69	I	0.42	63.5
3	16281	ACD8700354	19	S	2	0.0	84.2	PQ	0.312	1.44			54.2
3	16282	ACD8700434	24	NC	2	100.0	91.7	IR25		0.817	IF	0.55	62.8

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 2,3,7,8-TCDF
Internal Quantitation Standard = 13C12-2,3,7,8-TCDF
Target Ion Ratio = 0.76

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	0.225	2.53			68.2
3	16284	ACD8700238	3	NE	1	100.0	100.0	PQ	0.47	1.95			63.3
3	16286	ACD8700381	17	S	3	0.0	82.4	IR10		2.45	I	1.03	65.0
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.139	1.74			65.0
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	0.156	1.11			60.3
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.211	3.88			61.5
4	16293	ACD8700201	5	W	1	80.0	100.0	PQ	0.216	1.54			63.2
4	16294	ACD8700130	17	S	3	41.2	88.2	PQ	0.103	1.89			68.8
4	16295	ACD8700103	24	NC	3	45.8	100.0	IR25		1.31	I	1.30	73.1
4	16297	ACD8700158	18	NE	2	0.0	83.3	IR10		2.68	I	0.47	69.5
4	16299	ACD8700345	19	S	2	100.0	73.7	IR25		0.954	I	1.22	69.5
4	16300	ACD8700149	20	NE	1	70.0	65.0	PQ	0.397	1.9			62.5
4	16301	ACD8700032	18	NC	2	44.4	77.8	IR25		0.965	I	1.06	62.5
4	16302	ACD8700041	16	NC	2	100.0	81.3	IR25		1.12	I	1.07	74.0
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	0.34	2.23			63.8
5	16306	ACD8700363	14	S	2	0.0	64.3	IR10		1.15	I	0.95	70.3
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	0.194	1.29			68.0
5	16309	ACD8700265	5	W	1	20.0	100.0	PQ	0.191	3.73			53.4
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	0.126	1.37			65.6
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	0.099	1.49			64.3
5	16313	ACD8700452	18	NC	3	100.0	88.9	PQ	0.153	2.4			68.6
5	16314	ACD8700327	17	S	1	100.0	41.2	PQ	0.186	2.46			59.0
5	16315	ACD8700443	19	NC	3	0.0	100.0	PQ	0.294	2.49			58.7

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 2,3,7,8-TCDF
Internal Quantitation Standard = 13C12-2,3,7,8-TCDF
Target Ion Ratio = 0.76

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	TR	0.428	1.09			56.4
5	16317	ACD8700407	15	S	3	100.0	80.0	PQ	0.389	2.97			71.1

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 2,3,7,8-TCDD
Internal Quantitation Standard = 13C12-2,3,7,8-TCDD
Target Ion Ratio = 0.76

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	PQ	0.066	4.9			78.7
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.05	1.91	R		153.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	PQ	0.239	2.02			68.4
1	16258	ACD8700318	14	S	1	0.0	42.9	IR25		1.51	I	0.48	69.8
1	16259	ACD8700112	14	S	1	50.0	92.9	IR25		1.73	I	1.73	72.8
1	16260	ACD8700069	20	NC	3	100.0	85.0	PQ	0.121	11.1			81.8
1	16261	ACD8700461	13	S	1	61.5	53.8	IR10		2.6	I	0.56	66.5
1	16262	ACD8700194	32	NE	3	100.0	96.9	PQ	0.122	8.48			68.9
1	16263	ACD8700390	23	S	3	100.0	69.6	PQ	0.286	7.08			69.7
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	0.136	3.56			76.0
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	0.12	9.1			98.6
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	0.139	5.69			71.0
2	16269	ACD8700283	24	W	3	100.0	79.2	PQ	0.291	9.31			75.3
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	0.255	10.5			75.5
2	16272	ACD8700489	25	S	3	56.0	88.0	PQ	0.394	9.21			64.9
2	16274	ACD8700096	23	NC	3	0.0	82.6	PQ	0.285	10.3			69.2
2	16275	ACD8700416	17	NC	1	41.2	76.5	IR25		1.5	I	0.93	67.8
2	16276	ACD8700014	18	NC	1	100.0	44.4	IR25		2.25	I	1.35	71.2
2	16277	ACD8700274	16	W	2	18.8	87.5	PQ	0.236	3.09			64.0
3	16279	ACD8700185	32	NE	3	0.0	90.6	PQ	0.329	9.67			70.9
3	16280	ACD8700292	16	W	3	0.0	93.8	PQ	0.67	8.49			69.8
3	16281	ACD8700354	19	S	2	0.0	84.2	PQ	0.349	4.27			65.3
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	0.221	3.87			77.1

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 2,3,7,8-TCDD
Internal Quantitation Standard = 13C12-2,3,7,8-TCDD
Target Ion Ratio = 0.76

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	0.199	7.91			76.1
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	0.98		LOD/2		73.9
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	0.15	15.1			72.3
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.129	3.97			68.5
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	0.254	4.58			63.8
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.151	15.1			69.6
4	16293	ACD8700201	5	W	1	80.0	100.0	PQ	0.169	1.33			69.0
4	16294	ACD8700130	17	S	3	41.2	88.2	PQ	0.142	7.25			72.3
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	0.444	7.54			74.4
4	16297	ACD8700158	18	NE	2	0.0	83.3	PQ	0.565	5.91			78.4
4	16299	ACD8700345	19	S	2	100.0	73.7	PQ	0.381	2.62			89.4
4	16300	ACD8700149	20	NE	1	70.0	65.0	PQ	0.626	2.95			68.2
4	16301	ACD8700032	18	NC	2	44.4	77.8	IR10		3.16	I	0.97	53.6
4	16302	ACD8700041	16	NC	2	100.0	81.3	IR10		3.62	I	0.97	79.7
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	0.536	6.63			75.1
5	16306	ACD8700363	14	S	2	0.0	64.3	IR10		4.41	I	0.94	81.1
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	0.219	4.52			81.2
5	16309	ACD8700265	5	W	1	20.0	100.0	IR10		1.72	I	0.96	73.3
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	0.171	9.48			82.3
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	0.09	4.93			75.8
5	16313	ACD8700452	18	NC	3	100.0	88.9	PQ	0.174	6.44			79.0
5	16314	ACD8700327	17	S	1	100.0	41.2	PQ	0.233	1.92			75.9
5	16315	ACD8700443	19	NC	3	0.0	100.0	IR10		7.12	I	0.91	66.5

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 2,3,7,8-TCDD
Internal Quantitation Standard = 13C12-2,3,7,8-TCDD
Target Ion Ratio = 0.76

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	IR10		4.04	I	1.00	79.4
5	16317	ACD8700407	15	S	3	100.0	80.0	PQ	0.621	8.28			91.4

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,7,8-PECDF
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDF
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	ND	0.066		LOD/2		90.0
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.072	0.428	R		175.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	ND	0.419		LOD/2		43.1
1	16258	ACD8700318	14	S	1	0.0	42.9	ND	0.103		LOD/2		77.3
1	16259	ACD8700112	14	S	1	50.0	92.9	ND	0.157		LOD/2		87.3
1	16260	ACD8700069	20	NC	3	100.0	85.0	ND	0.73		LOD/2		83.3
1	16261	ACD8700461	13	S	1	61.5	53.8	ND	0.233		LOD/2		82.8
1	16262	ACD8700194	32	NE	3	100.0	96.9	ND	0.361		LOD/2		87.7
1	16263	ACD8700390	23	S	3	100.0	69.6	ND	0.918		LOD/2		84.1
1	16264	ACD8700176	20	NE	2	100.0	90.0	ND	0.383		LOD/2		92.3
2	16267	ACD8700256	18	NE	3	44.4	100.0	ND	1.54		R		26.1
2	16268	ACD8700470	32	S	2	62.5	71.9	ND	0.63		LOD/2		87.9
2	16269	ACD8700283	24	W	3	100.0	79.2	ND	0.323		LOD/2		76.9
2	16270	ACD8700087	17	NC	3	100.0	76.5	ND	0.373		LOD/2		98.3
2	16272	ACD8700489	25	S	3	56.0	88.0	ND	0.407		LOD/2		88.2
2	16274	ACD8700096	23	NC	3	0.0	82.6	TR	0.435	0.865			86.9
2	16275	ACD8700416	17	NC	1	41.2	76.5	ND	0.322		LOD/2		82.9
2	16276	ACD8700014	18	NC	1	100.0	44.4	ND	0.836		LOD/2		86.0
2	16277	ACD8700274	16	W	2	18.8	87.5	ND	0.175		LOD/2		84.6
3	16279	ACD8700185	32	NE	3	0.0	90.6	ND	0.631		LOD/2		63.6
3	16280	ACD8700292	16	W	3	0.0	93.8	ND	0.817		LOD/2		66.7
3	16281	ACD8700354	19	S	2	0.0	84.2	ND	0.631		LOD/2		81.1
3	16282	ACD8700434	24	NC	2	100.0	91.7	ND	0.459		LOD/2		87.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,7,8-PECDF
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDF
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	ND	0.84		LOD/2		92.9
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	0.84		LOD/2		82.6
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	0.353	1.2			87.2
3	16288	ACD8700210	14	W	2	57.1	85.7	TR	0.194	0.302			87.3
3	16289	ACD8700167	20	NE	2	100.0	85.0	ND	0.287		LOD/2		80.7
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.218	1.42			85.3
4	16293	ACD8700201	5	W	1	80.0	100.0	ND	0.351		LOD/2		84.5
4	16294	ACD8700130	17	S	3	41.2	88.2	IR25		0.511	I	2.93	90.7
4	16295	ACD8700103	24	NC	3	45.8	100.0	ND	0.699		LOD/2		94.0
4	16297	ACD8700158	18	NE	2	0.0	83.3	ND	0.716		LOD/2		97.8
4	16299	ACD8700345	19	S	2	100.0	73.7	ND	0.501		LOD/2		92.6
4	16300	ACD8700149	20	NE	1	70.0	65.0	ND	0.529		LOD/2		92.4
4	16301	ACD8700032	18	NC	2	44.4	77.8	ND	0.415		LOD/2		78.8
4	16302	ACD8700041	16	NC	2	100.0	81.3	ND	0.499		LOD/2		95.8
4	16304	ACD8700229	10	W	3	40.0	100.0	IR25		0.638	I	1.19	84.3
5	16306	ACD8700363	14	S	2	0.0	64.3	PQ	0.172	0.733			92.5
5	16308	ACD8700121	13	S	2	46.2	61.5	ND	0.197		LOD/2		96.6
5	16309	ACD8700265	5	W	1	20.0	100.0	ND	0.241		LOD/2		62.2
5	16310	ACD8700372	20	S	3	0.0	85.0	IR25		0.615	I	0.53	86.5
5	16311	ACD8700050	27	NC	2	0.0	85.2	ND	0.104		LOD/2		89.1
5	16313	ACD8700452	18	NC	3	100.0	88.9	ND	0.242		LOD/2		97.2
5	16314	ACD8700327	17	S	1	100.0	41.2	TR	0.289	0.88			80.4
5	16315	ACD8700443	19	NC	3	0.0	100.0	ND	0.289		LOD/2		86.2

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,7,8-PECDF
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDF
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	ND	0.675		LOD/2		92.1
5	16317	ACD8700407	15	S	3	100.0	80.0	ND	0.602		LOD/2		109.0

CODES USED FOR DATA RESTRICTION:

LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 2,3,4,7,8-PECDF
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDF
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	PQ	0.06	9.56			90.0
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.066	3.66	R		175.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	IR10		4.03	I	14.22	43.1
1	16258	ACD8700318	14	S	1	0.0	42.9	PQ	0.094	3.36			77.3
1	16259	ACD8700112	14	S	1	50.0	92.9	IR25		3.39	I	2.67	87.3
1	16260	ACD8700069	20	NC	3	100.0	85.0	PQ	0.156	7.73			83.3
1	16261	ACD8700461	13	S	1	61.5	53.8	IR25		2.62	I	5.24	82.8
1	16262	ACD8700194	32	NE	3	100.0	96.9	PQ	0.271	21.0			87.7
1	16263	ACD8700390	23	S	3	100.0	69.6	PQ	0.194	19.2			84.1
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	0.349	11.3			92.3
2	16267	ACD8700256	18	NE	3	44.4	100.0	ND	0.97		R		26.1
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	0.26	8.23			87.9
2	16269	ACD8700283	24	W	3	100.0	79.2	TR	0.295	7.11			76.9
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	0.341	29.2			98.3
2	16272	ACD8700489	25	S	3	56.0	88.0	PQ	0.372	12.6			88.2
2	16274	ACD8700096	23	NC	3	0.0	82.6	PQ	0.398	22.4			86.9
2	16275	ACD8700416	17	NC	1	41.2	76.5	PQ	0.294	2.63			82.9
2	16276	ACD8700014	18	NC	1	100.0	44.4	PQ	0.323	5.37			86.0
2	16277	ACD8700274	16	W	2	18.8	87.5	IR10		7.77	I	1.22	84.6
3	16279	ACD8700185	32	NE	3	0.0	90.6	PQ	0.576	25.6			63.6
3	16280	ACD8700292	16	W	3	0.0	93.8	IR10		17.5	I	1.93	66.7
3	16281	ACD8700354	19	S	2	0.0	84.2	PQ	0.576	10.3			81.1
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	0.419	7.41			87.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 2,3,4,7,8-PECDF
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDF
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	0.767	4.01			92.9
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	2.47		LOD/2		82.6
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	0.322	28.1			87.2
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.177	7.84			87.3
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	0.262	13.3			80.7
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.199	27.1			85.3
4	16293	ACD8700201	5	W	1	80.0	100.0	PQ	0.321	2.93			84.5
4	16294	ACD8700130	17	S	3	41.2	88.2	PQ	0.219	11.3			90.7
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	0.638	16.9			94.0
4	16297	ACD8700158	18	NE	2	0.0	83.3	IR10		13.5	I	1.86	97.8
4	16299	ACD8700345	19	S	2	100.0	73.7	PQ	0.458	7.76			92.6
4	16300	ACD8700149	20	NE	1	70.0	65.0	PQ	0.483	3.61			92.4
4	16301	ACD8700032	18	NC	2	44.4	77.8	PQ	0.379	8.09			78.8
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	0.455	9.21			95.8
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	0.509	14.2			84.3
5	16306	ACD8700363	14	S	2	0.0	64.3	PQ	0.157	6.75			92.5
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	0.18	8.34			96.6
5	16309	ACD8700265	5	W	1	20.0	100.0	IR10		2.52	I	0.90	62.2
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	0.173	16.9			86.5
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	0.095	4.91			89.1
5	16313	ACD8700452	18	NC	3	100.0	88.9	PQ	0.183	17.0			97.2
5	16314	ACD8700327	17	S	1	100.0	41.2	ND	0.264		LOD/2		80.4
5	16315	ACD8700443	19	NC	3	0.0	100.0	PQ	0.264	16.4			86.2

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 2,3,4,7,8-PECDF
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDF
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	PQ	0.616	8.92			92.1
5	16317	ACD8700407	15	S	3	100.0	80.0	PQ	0.445	21.7			109.0

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,7,8-PECDD
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDD
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	PQ	0.536	8.43			96.8
1	16255	ACD8700425	19	NC	2	0.0	94.7	IR10		3.71	IR		169.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	IR25		10.8	IR	1.92	28.1
1	16258	ACD8700318	14	S	1	0.0	42.9	TR	0.543	1.65			86.8
1	16259	ACD8700112	14	S	1	50.0	92.9	PQ	0.628	3.21			81.2
1	16260	ACD8700069	20	NC	3	100.0	85.0	IR10		9.36	I	0.31	88.1
1	16261	ACD8700461	13	S	1	61.5	53.8	PQ	0.527	5.54			76.3
1	16262	ACD8700194	32	NE	3	100.0	96.9	PQ	2.4	24.4			86.0
1	16263	ACD8700390	23	S	3	100.0	69.6	PQ	0.782	15.3			96.7
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	1.26	9.98			98.4
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	1.69	18.0			76.8
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	1.3	11.3			94.1
2	16269	ACD8700283	24	W	3	100.0	79.2	IR10		10.5	I	1.94	75.6
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	1.58	23.8			96.7
2	16272	ACD8700489	25	S	3	56.0	88.0	PQ	2.28	17.8			87.3
2	16274	ACD8700096	23	NC	3	0.0	82.6	PQ	0.92	20.6			101.0
2	16275	ACD8700416	17	NC	1	41.2	76.5	PQ	0.766	4.6			94.2
2	16276	ACD8700014	18	NC	1	100.0	44.4	IR10		6.13	I	0.86	86.4
2	16277	ACD8700274	16	W	2	18.8	87.5	PQ	1.19	10.2			94.5
3	16279	ACD8700185	32	NE	3	0.0	90.6	IR10		16.5	I	0.92	77.5
3	16280	ACD8700292	16	W	3	0.0	93.8	IR10		16.9	I	0.84	77.1
3	16281	ACD8700354	19	S	2	0.0	84.2	IR10		11.3	I	1.06	98.3
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	1.49	10.7			94.8

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,7,8-PECDD
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDD
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	IR10		11.0	I	1.02	83.0
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	2.44		LOD/2		95.6
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	1.53	22.9			103.0
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.624	7.74			112.0
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	0.799	9.77			97.4
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	1.05	20.3			103.0
4	16293	ACD8700201	5	W	1	80.0	100.0	PQ	0.616	3.85			90.0
4	16294	ACD8700130	17	S	3	41.2	88.2	PQ	1.24	12.4			113.0
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	1.61	12.0			123.0
4	16297	ACD8700158	18	NE	2	0.0	83.3	PQ	1.32	8.96			127.0
4	16299	ACD8700345	19	S	2	100.0	73.7	IR10		8.3	I	1.07	121.0
4	16300	ACD8700149	20	NE	1	70.0	65.0	TR	1.12	3.68			123.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	PQ	1.64	8.07			116.0
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	1.35	9.94			127.0
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	1.05	13.3			114.0
5	16306	ACD8700363	14	S	2	0.0	64.3	IR10		7.68	I	0.81	109.0
5	16308	ACD8700121	13	S	2	46.2	61.5	IR10		10.4	I	1.19	119.0
5	16309	ACD8700265	5	W	1	20.0	100.0	IR10		3.52	I	0.99	69.7
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	1.98	18.5			107.0
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	1.6	9.1			97.0
5	16313	ACD8700452	18	NC	3	100.0	88.9	PQ	0.756	17.4			121.0
5	16314	ACD8700327	17	S	1	100.0	41.2	TR	0.95	3.65			104.0
5	16315	ACD8700443	19	NC	3	0.0	100.0	PQ	2.88	16.8			115.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,7,8-PECDD
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDD
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	PQ	0.91	9.53			132.0
5	16317	ACD8700407	15	S	3	100.0	80.0	PQ	3.59	17.9			143.0

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	010		11.8	C		60.0
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.087	8.56	R		178.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	010		8.62	C		66.0
1	16258	ACD8700318	14	S	1	0.0	42.9	IR25		4.59	I	4.34	64.9
1	16259	ACD8700112	14	S	1	50.0	92.9	010		6.85	C		83.4
1	16260	ACD8700069	20	NC	3	100.0	85.0	010		26.7	C		86.0
1	16261	ACD8700461	13	S	1	61.5	53.8	010		6.35	C		70.8
1	16262	ACD8700194	32	NE	3	100.0	96.9	010		31.8	C		98.4
1	16263	ACD8700390	23	S	3	100.0	69.6	010		31.6	C		82.4
1	16264	ACD8700176	20	NE	2	100.0	90.0	010		16.6	C		85.2
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	0.324	12.3			69.9
2	16268	ACD8700470	32	S	2	62.5	71.9	010		15.6	C		86.2
2	16269	ACD8700283	24	W	3	100.0	79.2	010		39.9	C		73.0
2	16270	ACD8700087	17	NC	3	100.0	76.5	010		36.9	C		80.1
2	16272	ACD8700489	25	S	3	56.0	88.0	010		24.9	C		59.9
2	16274	ACD8700096	23	NC	3	0.0	82.6	010		31.5	C		47.7
2	16275	ACD8700416	17	NC	1	41.2	76.5	IR10		4.92	IC	0.83	80.7
2	16276	ACD8700014	18	NC	1	100.0	44.4	025		10.2	C		86.1
2	16277	ACD8700274	16	W	2	18.8	87.5	010		10.6	C		89.3
3	16279	ACD8700185	32	NE	3	0.0	90.6	010		38.6	C		70.3
3	16280	ACD8700292	16	W	3	0.0	93.8	010		48.5	C		59.5
3	16281	ACD8700354	19	S	2	0.0	84.2	010		16.6	C		71.1
3	16282	ACD8700434	24	NC	2	100.0	91.7	010		15.2	C		71.5

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	0.503	17.0			72.4
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	3.11		LOD/2		75.8
3	16286	ACD8700381	17	S	3	0.0	82.4	O10		26.7	C		74.8
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.458	6.76			60.4
3	16289	ACD8700167	20	NE	2	100.0	85.0	O10		23.3	C		64.2
3	16291	ACD8700078	16	NC	3	0.0	87.5	O10		31.9	C		63.7
4	16293	ACD8700201	5	W	1	80.0	100.0	O10		2.87	C		62.0
4	16294	ACD8700130	17	S	3	41.2	88.2	O10		25.4	C		63.7
4	16295	ACD8700103	24	NC	3	45.8	100.0	O10		30.6	C		68.5
4	16297	ACD8700158	18	NE	2	0.0	83.3	PQ	0.406	7.42			68.1
4	16299	ACD8700345	19	S	2	100.0	73.7	O10		18.9	C		70.4
4	16300	ACD8700149	20	NE	1	70.0	65.0	IR25		3.27	I	1.74	60.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	IR10		8.04	I	0.82	71.6
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	0.228	4.84			68.7
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	0.535	7.3			67.1
5	16306	ACD8700363	14	S	2	0.0	64.3	O10		14.3	C		79.5
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	0.5	6.73			74.1
5	16309	ACD8700265	5	W	1	20.0	100.0	IR25		2.42	I	2.10	60.3
5	16310	ACD8700372	20	S	3	0.0	85.0	O10		27.1	C		74.1
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	0.287	9.91			67.0
5	16313	ACD8700452	18	NC	3	100.0	88.9	O10		22.7	C		71.6
5	16314	ACD8700327	17	S	1	100.0	41.2	O10		4.05	C		58.8
5	16315	ACD8700443	19	NC	3	0.0	100.0	O10		20.7	C		69.6

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	010		23.3	C		66.1
5	16317	ACD8700407	15	S	3	100.0	80.0	010		31.0	C		64.7

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,6,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	PQ	0.218	3.92			60.0
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.085	2.02	R		178.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	PQ	0.607	2.72			66.0
1	16258	ACD8700318	14	S	1	0.0	42.9	PQ	0.352	1.96			64.9
1	16259	ACD8700112	14	S	1	50.0	92.9	ND	2.68		LOD/2		83.4
1	16260	ACD8700069	20	NC	3	100.0	85.0	PQ	0.293	8.99			86.0
1	16261	ACD8700461	13	S	1	61.5	53.8	TR	0.346	1.25			70.8
1	16262	ACD8700194	32	NE	3	100.0	96.9	IR10		6.47	I	1.70	98.4
1	16263	ACD8700390	23	S	3	100.0	69.6	IR10		7.43	I	2.06	82.4
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	0.47	5.03			85.2
2	16267	ACD8700256	18	NE	3	44.4	100.0	IR10		8.79	I	1.59	69.9
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	0.347	4.25			86.2
2	16269	ACD8700283	24	W	3	100.0	79.2	PQ	0.34	8.32			73.0
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	0.283	9.93			80.1
2	16272	ACD8700489	25	S	3	56.0	88.0	IR10		7.78	I	1.53	59.9
2	16274	ACD8700096	23	NC	3	0.0	82.6	PQ	0.305	14.2			47.7
2	16275	ACD8700416	17	NC	1	41.2	76.5	PQ	0.386	2.17			80.7
2	16276	ACD8700014	18	NC	1	100.0	44.4	TR	1.28	1.96			86.1
2	16277	ACD8700274	16	W	2	18.8	87.5	TR	0.885	3.34			89.3
3	16279	ACD8700185	32	NE	3	0.0	90.6	PQ	1.1	14.3			70.3
3	16280	ACD8700292	16	W	3	0.0	93.8	IR10		12.0	I	1.50	59.5
3	16281	ACD8700354	19	S	2	0.0	84.2	PQ	0.753	5.5			71.1
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	0.861	6.12			71.5

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,6,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	0.494	9.94			72.4
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	3.05		LOD/2		75.8
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	0.826	11.9			74.8
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.449	3.9			60.4
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	0.472	6.0			64.2
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.566	11.5			63.7
4	16293	ACD8700201	5	W	1	80.0	100.0	TR	0.371	1.46			62.0
4	16294	ACD8700130	17	S	3	41.2	88.2	IR10		4.78	I	1.63	63.7
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	0.767	8.05			68.5
4	16297	ACD8700158	18	NE	2	0.0	83.3	IR10		7.46	I	0.92	68.1
4	16299	ACD8700345	19	S	2	100.0	73.7	PQ	0.531	5.21			70.4
4	16300	ACD8700149	20	NE	1	70.0	65.0	PQ	1.01	2.39			60.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	ND	0.556		LOD/2		71.6
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	0.224	4.66			68.7
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	0.526	7.41			67.1
5	16306	ACD8700363	14	S	2	0.0	64.3	PQ	1.06	4.85			79.5
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	0.485	5.99			74.1
5	16309	ACD8700265	5	W	1	20.0	100.0	IR25		1.12	I	5.10	60.3
5	16310	ACD8700372	20	S	3	0.0	85.0	IR10		6.45	I	1.52	74.1
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	0.282	5.4			67.0
5	16313	ACD8700452	18	NC	3	100.0	88.9	IR10		8.15	I	1.67	71.6
5	16314	ACD8700327	17	S	1	100.0	41.2	TR	2.07	2.76			58.8
5	16315	ACD8700443	19	NC	3	0.0	100.0	PQ	0.709	9.8			69.6

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,6,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	PQ	0.674	4.96			66.1
5	16317	ACD8700407	15	S	3	100.0	80.0	PQ	0.789	8.85			64.7

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 2,3,4,6,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	IR25		0.917	I	2.31	60.0
1	16255	ACD8700425	19	NC	2	0.0	94.7	ND	0.564		R		178.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	ND	1.62		LOD/2		66.0
1	16258	ACD8700318	14	S	1	0.0	42.9	ND	0.418		LOD/2		64.9
1	16259	ACD8700112	14	S	1	50.0	92.9	ND	0.74		LOD/2		83.4
1	16260	ACD8700069	20	NC	3	100.0	85.0	O10		16.3	C		86.0
1	16261	ACD8700461	13	S	1	61.5	53.8	TR	0.411	0.947			70.8
1	16262	ACD8700194	32	NE	3	100.0	96.9	ND	0.958		LOD/2		98.4
1	16263	ACD8700390	23	S	3	100.0	69.6	TR	0.72	0.793			82.4
1	16264	ACD8700176	20	NE	2	100.0	90.0	ND	0.559		LOD/2		85.2
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	0.378	2.49			69.9
2	16268	ACD8700470	32	S	2	62.5	71.9	ND	0.564		LOD/2		86.2
2	16269	ACD8700283	24	W	3	100.0	79.2	ND	1.47		LOD/2		73.0
2	16270	ACD8700087	17	NC	3	100.0	76.5	ND	0.337		LOD/2		80.1
2	16272	ACD8700489	25	S	3	56.0	88.0	ND	0.44		LOD/2		59.9
2	16274	ACD8700096	23	NC	3	0.0	82.6	ND	0.362		LOD/2		47.7
2	16275	ACD8700416	17	NC	1	41.2	76.5	ND	0.485		LOD/2		80.7
2	16276	ACD8700014	18	NC	1	100.0	44.4	ND	1.52		LOD/2		86.1
2	16277	ACD8700274	16	W	2	18.8	87.5	IR10		5.57	IC	0.74	89.3
3	16279	ACD8700185	32	NE	3	0.0	90.6	O10		21.1	C		70.3
3	16280	ACD8700292	16	W	3	0.0	93.8	O10		20.8	C		59.5
3	16281	ACD8700354	19	S	2	0.0	84.2	O10		11.4	C		71.1
3	16282	ACD8700434	24	NC	2	100.0	91.7	O10		12.3	C		71.5

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 2,3,4,6,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	010		14.0	C		72.4
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	3.64		LOD/2		75.8
3	16286	ACD8700381	17	S	3	0.0	82.4	010		15.3	C		74.8
3	16288	ACD8700210	14	W	2	57.1	85.7	010		12.9	C		60.4
3	16289	ACD8700167	20	NE	2	100.0	85.0	010		13.9	C		64.2
3	16291	ACD8700078	16	NC	3	0.0	87.5	010		21.2	C		63.7
4	16293	ACD8700201	5	W	1	80.0	100.0	010		1.48	C		62.0
4	16294	ACD8700130	17	S	3	41.2	88.2	010		13.3	C		63.7
4	16295	ACD8700103	24	NC	3	45.8	100.0	010		13.6	C		68.5
4	16297	ACD8700158	18	NE	2	0.0	83.3	010		10.7	C		68.1
4	16299	ACD8700345	19	S	2	100.0	73.7	010		13.6	C		70.4
4	16300	ACD8700149	20	NE	1	70.0	65.0	010		2.32	C		60.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	010		7.39	C		71.6
4	16302	ACD8700041	16	NC	2	100.0	81.3	010		13.6	C		68.7
4	16304	ACD8700229	10	W	3	40.0	100.0	010		13.6	C		67.1
5	16306	ACD8700363	14	S	2	0.0	64.3	010		8.5	C		79.5
5	16308	ACD8700121	13	S	2	46.2	61.5	010		10.9	C		74.1
5	16309	ACD8700265	5	W	1	20.0	100.0	025		0.725	C		60.3
5	16310	ACD8700372	20	S	3	0.0	85.0	010		13.3	C		74.1
5	16311	ACD8700050	27	NC	2	0.0	85.2	010		8.18	C		67.0
5	16313	ACD8700452	18	NC	3	100.0	88.9	010		23.0	C		71.6
5	16314	ACD8700327	17	S	1	100.0	41.2	ND	2.46		LOD/2		58.8
5	16315	ACD8700443	19	NC	3	0.0	100.0	010		12.2	C		69.6

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 2,3,4,6,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	010		24.0	C		66.1
5	16317	ACD8700407	15	S	3	100.0	80.0	010		23.1	C		64.7

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,7,8,9-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	IR25		0.469	I	2.06	60.0
1	16255	ACD8700425	19	NC	2	0.0	94.7	ND	0.179		R		178.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	ND	0.789		LOD/2		66.0
1	16258	ACD8700318	14	S	1	0.0	42.9	ND	0.457		LOD/2		64.9
1	16259	ACD8700112	14	S	1	50.0	92.9	ND	0.63		LOD/2		83.4
1	16260	ACD8700069	20	NC	3	100.0	85.0	ND	0.38		LOD/2		86.0
1	16261	ACD8700461	13	S	1	61.5	53.8	ND	0.449		LOD/2		70.8
1	16262	ACD8700194	32	NE	3	100.0	96.9	ND	0.66		LOD/2		98.4
1	16263	ACD8700390	23	S	3	100.0	69.6	ND	0.786		LOD/2		82.4
1	16264	ACD8700176	20	NE	2	100.0	90.0	ND	0.611		LOD/2		85.2
2	16267	ACD8700256	18	NE	3	44.4	100.0	ND	0.413		LOD/2		69.9
2	16268	ACD8700470	32	S	2	62.5	71.9	ND	0.45		LOD/2		86.2
2	16269	ACD8700283	24	W	3	100.0	79.2	ND	0.441		LOD/2		73.0
2	16270	ACD8700087	17	NC	3	100.0	76.5	ND	0.368		LOD/2		80.1
2	16272	ACD8700489	25	S	3	56.0	88.0	ND	0.48		LOD/2		59.9
2	16274	ACD8700096	23	NC	3	0.0	82.6	ND	0.396		LOD/2		47.7
2	16275	ACD8700416	17	NC	1	41.2	76.5	ND	0.501		LOD/2		80.7
2	16276	ACD8700014	18	NC	1	100.0	44.4	ND	1.66		LOD/2		86.1
2	16277	ACD8700274	16	W	2	18.8	87.5	ND	1.15		LOD/2		89.3
3	16279	ACD8700185	32	NE	3	0.0	90.6	ND	1.43		LOD/2		70.3
3	16280	ACD8700292	16	W	3	0.0	93.8	ND	2.16		LOD/2		59.5
3	16281	ACD8700354	19	S	2	0.0	84.2	ND	0.978		LOD/2		71.1
3	16282	ACD8700434	24	NC	2	100.0	91.7	ND	1.12		LOD/2		71.5

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,7,8,9-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	ND	0.642		LOD/2		72.4
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	3.96		LOD/2		75.8
3	16286	ACD8700381	17	S	3	0.0	82.4	ND	1.07		LOD/2		74.8
3	16288	ACD8700210	14	W	2	57.1	85.7	ND	0.584		LOD/2		60.4
3	16289	ACD8700167	20	NE	2	100.0	85.0	IR25		0.932	I	2.94	64.2
3	16291	ACD8700078	16	NC	3	0.0	87.5	TR	0.735	1.06			63.7
4	16293	ACD8700201	5	W	1	80.0	100.0	ND	0.429		LOD/2		62.0
4	16294	ACD8700130	17	S	3	41.2	88.2	ND	0.726		LOD/2		63.7
4	16295	ACD8700103	24	NC	3	45.8	100.0	ND	0.996		LOD/2		68.5
4	16297	ACD8700158	18	NE	2	0.0	83.3	ND	0.517		LOD/2		68.1
4	16299	ACD8700345	19	S	2	100.0	73.7	ND	0.689		LOD/2		70.4
4	16300	ACD8700149	20	NE	1	70.0	65.0	ND	1.31		LOD/2		60.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	ND	0.721		LOD/2		71.6
4	16302	ACD8700041	16	NC	2	100.0	81.3	ND	0.29		LOD/2		68.7
4	16304	ACD8700229	10	W	3	40.0	100.0	ND	0.683		LOD/2		67.1
5	16306	ACD8700363	14	S	2	0.0	64.3	ND	1.38		LOD/2		79.5
5	16308	ACD8700121	13	S	2	46.2	61.5	ND	0.629		LOD/2		74.1
5	16309	ACD8700265	5	W	1	20.0	100.0	ND	0.419		LOD/2		60.3
5	16310	ACD8700372	20	S	3	0.0	85.0	ND	0.519		LOD/2		74.1
5	16311	ACD8700050	27	NC	2	0.0	85.2	ND	0.366		LOD/2		67.0
5	16313	ACD8700452	18	NC	3	100.0	88.9	ND	0.987		LOD/2		71.6
5	16314	ACD8700327	17	S	1	100.0	41.2	ND	2.68		LOD/2		58.8
5	16315	ACD8700443	19	NC	3	0.0	100.0	ND	0.921		LOD/2		69.6

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,7,8,9-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	ND	0.875		LOD/2		66.1
5	16317	ACD8700407	15	S	3	100.0	80.0	ND	1.02		LOD/2		64.7

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,7,8/1,2,3,6,7,8-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	IR10		46.9	I	1.68	68.0
1	16255	ACD8700425	19	NC	2	0.0	94.7	*** DATA MISSING FOR THIS SAMPLE ***					
1	16257	ACD8700023	20	NC	1	0.0	85.0	PQ	1.2	41.7			68.0
1	16258	ACD8700318	14	S	1	0.0	42.9	PQ	0.43	20.2			65.4
1	16259	ACD8700112	14	S	1	50.0	92.9	IR10		25.4	I	1.49	83.4
1	16260	ACD8700069	20	NC	3	100.0	85.0	PQ	0.492	151.			79.6
1	16261	ACD8700461	13	S	1	61.5	53.8	PQ	0.381	25.6			67.5
1	16262	ACD8700194	32	NE	3	100.0	96.9	PQ	1.95	122.			83.8
1	16263	ACD8700390	23	S	3	100.0	69.6	PQ	0.917	90.0			80.1
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	1.09	83.9			87.5
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	0.845	113.			66.9
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	1.41	72.7			74.8
2	16269	ACD8700283	24	W	3	100.0	79.2	PQ	0.934	144.			72.6
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	0.761	128.			71.6
2	16272	ACD8700489	25	S	3	56.0	88.0	IR10		112.	I	1.76	67.5
2	16274	ACD8700096	23	NC	3	0.0	82.6	PQ	0.636	121.			63.4
2	16275	ACD8700416	17	NC	1	41.2	76.5	PQ	0.704	29.3			71.7
2	16276	ACD8700014	18	NC	1	100.0	44.4	PQ	0.978	39.8			83.7
2	16277	ACD8700274	16	W	2	18.8	87.5	PQ	1.12	66.5			76.2
3	16279	ACD8700185	32	NE	3	0.0	90.6	PQ	1.63	95.7			80.3
3	16280	ACD8700292	16	W	3	0.0	93.8	PQ	3.0	113.			68.2
3	16281	ACD8700354	19	S	2	0.0	84.2	PQ	1.44	70.7			77.8
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	0.858	67.7			72.8

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,7,8/1,2,3,6,7,8-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	0.97	118.			72.4
3	16284	ACD8700238	3	NE	1	100.0	100.0	PQ	3.14	13.3			68.4
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	1.32	147.			69.3
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	1.12	73.8			67.7
3	16289	ACD8700167	20	NE	2	100.0	85.0	IR10		82.0	I	5.05	76.2
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.826	174.			76.6
4	16293	ACD8700201	5	W	1	80.0	100.0	PQ	0.639	22.8			73.0
4	16294	ACD8700130	17	S	3	41.2	88.2	PQ	0.631	93.5			72.9
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	1.01	90.5			68.5
4	16297	ACD8700158	18	NE	2	0.0	83.3	PQ	1.17	76.1			72.2
4	16299	ACD8700345	19	S	2	100.0	73.7	PQ	0.919	64.3			74.3
4	16300	ACD8700149	20	NE	1	70.0	65.0	PQ	1.75	24.4			68.3
4	16301	ACD8700032	18	NC	2	44.4	77.8	IR10		54.3	I	1.74	78.9
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	0.917	70.4			77.4
4	16304	ACD8700229	10	W	3	40.0	100.0	IR10		90.6	I	1.49	84.9
5	16306	ACD8700363	14	S	2	0.0	64.3	PQ	1.58	50.2			77.6
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	2.07	80.2			67.7
5	16309	ACD8700265	5	W	1	20.0	100.0	PQ	1.41	20.0			65.9
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	1.16	97.1			76.1
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	0.841	74.4			71.1
5	16313	ACD8700452	18	NC	3	100.0	88.9	PQ	1.56	133.			75.6
5	16314	ACD8700327	17	S	1	100.0	41.2	PQ	1.62	27.6			68.8
5	16315	ACD8700443	19	NC	3	0.0	100.0	PQ	2.64	128.			63.6

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,7,8/1,2,3,6,7,8-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	PQ	1.66	69.5			68.4
5	16317	ACD8700407	15	S	3	100.0	80.0	PQ	1.43	125.			77.6

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,7,8,9-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	PQ	0.113	9.88			68.0
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.279	5.25	R		154.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	PQ	1.24	10.2			68.0
1	16258	ACD8700318	14	S	1	0.0	42.9	IR25		4.74	I	0.92	65.4
1	16259	ACD8700112	14	S	1	50.0	92.9	PQ	1.16	7.13			83.4
1	16260	ACD8700069	20	NC	3	100.0	85.0	IR10		19.6	I	1.54	79.6
1	16261	ACD8700461	13	S	1	61.5	53.8	PQ	0.392	5.45			67.5
1	16262	ACD8700194	32	NE	3	100.0	96.9	PQ	2.01	17.8			83.8
1	16263	ACD8700390	23	S	3	100.0	69.6	PQ	0.945	11.8			80.1
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	1.12	9.14			87.5
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	0.87	16.4			66.9
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	1.45	11.2			74.8
2	16269	ACD8700283	24	W	3	100.0	79.2	PQ	0.963	19.7			72.6
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	0.784	20.7			71.6
2	16272	ACD8700489	25	S	3	56.0	88.0	PQ	0.719	21.6			67.5
2	16274	ACD8700096	23	NC	3	0.0	82.6	PQ	0.656	22.0			63.4
2	16275	ACD8700416	17	NC	1	41.2	76.5	PQ	0.726	6.15			71.7
2	16276	ACD8700014	18	NC	1	100.0	44.4	PQ	1.01	8.3			83.7
2	16277	ACD8700274	16	W	2	18.8	87.5	PQ	1.15	11.7			76.2
3	16279	ACD8700185	32	NE	3	0.0	90.6	PQ	1.68	11.3			80.3
3	16280	ACD8700292	16	W	3	0.0	93.8	PQ	3.09	16.2			68.2
3	16281	ACD8700354	19	S	2	0.0	84.2	PQ	1.48	11.8			77.8
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	0.885	10.9			72.8

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,7,8,9-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	1.0	17.1			72.4
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	3.86		LOD/2		68.4
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	1.36	20.5			69.3
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	1.15	11.5			67.7
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	0.658	9.93			76.2
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.851	19.1			76.6
4	16293	ACD8700201	5	W	1	80.0	100.0	IR10		2.95	I	1.51	73.0
4	16294	ACD8700130	17	S	3	41.2	88.2	PQ	0.65	13.6			72.9
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	1.04	13.6			68.5
4	16297	ACD8700158	18	NE	2	0.0	83.3	PQ	1.2	12.8			72.2
4	16299	ACD8700345	19	S	2	100.0	73.7	PQ	0.947	10.3			74.3
4	16300	ACD8700149	20	NE	1	70.0	65.0	PQ	1.8	4.27			68.3
4	16301	ACD8700032	18	NC	2	44.4	77.8	PQ	1.22	9.15			78.9
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	0.945	11.3			77.4
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	0.815	14.7			84.9
5	16306	ACD8700363	14	S	2	0.0	64.3	PQ	1.63	8.63			77.6
5	16308	ACD8700121	13	S	2	46.2	61.5	PS10		11.7	P		67.7
5	16309	ACD8700265	5	W	1	20.0	100.0	IR25		2.25	I	0.48	65.9
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	1.19	18.1			76.1
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	0.867	11.1			71.1
5	16313	ACD8700452	18	NC	3	100.0	88.9	PS10		18.2	P		75.6
5	16314	ACD8700327	17	S	1	100.0	41.2	IR10		3.2	I	2.36	68.8
5	16315	ACD8700443	19	NC	3	0.0	100.0	PQ	2.72	19.3			63.6

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,7,8,9-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	PQ	1.71	11.0			68.4
5	16317	ACD8700407	15	S	3	100.0	80.0	IR10		11.8	I	2.41	77.6

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,6,7,8-HPCDF
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDF
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	IR10		192.	I	19.87	66.2
1	16255	ACD8700425	19	NC	2	0.0	94.7	IR10		8.8	IR		163.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	IR25		7.6	I	0.49	49.9
1	16258	ACD8700318	14	S	1	0.0	42.9	IR10		95.7	I	17.12	75.1
1	16259	ACD8700112	14	S	1	50.0	92.9	IR10		19.3	I	1.70	88.1
1	16260	ACD8700069	20	NC	3	100.0	85.0	PQ	0.511	27.8			91.9
1	16261	ACD8700461	13	S	1	61.5	53.8	IR10		35.9	I	10.53	73.3
1	16262	ACD8700194	32	NE	3	100.0	96.9	IR10		28.3	I	1.93	79.3
1	16263	ACD8700390	23	S	3	100.0	69.6	PQ	0.812	24.0			67.2
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	0.652	19.1			74.5
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	0.557	17.4			58.1
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	0.913	22.7			74.0
2	16269	ACD8700283	24	W	3	100.0	79.2	IR10		23.5	I	2.06	69.3
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	1.19	32.5			77.7
2	16272	ACD8700489	25	S	3	56.0	88.0	IR10		24.8	I	1.56	87.4
2	16274	ACD8700096	23	NC	3	0.0	82.6	IR10		32.6	I	1.26	86.1
2	16275	ACD8700416	17	NC	1	41.2	76.5	IR10		84.9	I	15.60	69.5
2	16276	ACD8700014	18	NC	1	100.0	44.4	PQ	1.35	18.4			75.0
2	16277	ACD8700274	16	W	2	18.8	87.5	PQ	1 21	15.4			70.4
3	16279	ACD8700185	32	NE	3	0.0	90.6	IR10		17.8	I	0.72	72.1
3	16280	ACD8700292	16	W	3	0.0	93.8	PQ	1.64	15.7			94.4
3	16281	ACD8700354	19	S	2	0.0	84.2	IR10		16.8	I	0.76	76.7
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	1.25	17.8			71.6

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,6,7,8-HPCDF
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDF
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	1.15	14.0			76.6
3	16284	ACD8700238	3	NE	1	100.0	100.0	PQ	0.87	10.5			74.8
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	0.673	17.8			74.7
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.996	14.1			69.5
3	16289	ACD8700167	20	NE	2	100.0	85.0	IR10		26.5	I	2.09	82.3
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.913	20.5			89.8
4	16293	ACD8700201	5	W	1	80.0	100.0	PQ	0.864	3.64			77.1
4	16294	ACD8700130	17	S	3	41.2	88.2	ND	1.79		LOD/2		57.3
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	1.3	18.4			70.3
4	16297	ACD8700158	18	NE	2	0.0	83.3	ND	1.26		LOD/2		71.2
4	16299	ACD8700345	19	S	2	100.0	73.7	IR10		16.1	I	0.65	79.4
4	16300	ACD8700149	20	NE	1	70.0	65.0	IR10		18.3	I	2.46	63.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	PQ	1.17	19.3			105.0
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	0.772	17.7			85.5
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	0.871	13.8			77.6
5	16306	ACD8700363	14	S	2	0.0	64.3	PQ	0.634	14.0			89.8
5	16308	ACD8700121	13	S	2	46.2	61.5	IR10		54.2	I	0.30	73.5
5	16309	ACD8700265	5	W	1	20.0	100.0	ND	1.15		LOD/2		71.4
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	0.819	19.2			80.7
5	16311	ACD8700050	27	NC	2	0.0	85.2	IR10		15.8	I	1.42	69.8
5	16313	ACD8700452	18	NC	3	100.0	88.9	IR10		36.1	I	2.21	76.4
5	16314	ACD8700327	17	S	1	100.0	41.2	PQ	0.85	16.8			62.6
5	16315	ACD8700443	19	NC	3	0.0	100.0	IR10		72.2	I	6.47	78.1

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,6,7,8-HPCDF
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDF
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	PQ	0.892	17.8			69.2
5	16317	ACD8700407	15	S	3	100.0	80.0	IR10		38.9	I	2.97	85.1

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,7,8,9-HPCDF
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDF
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	ND	1.51		LOD/2		66.2
1	16255	ACD8700425	19	NC	2	0.0	94.7	ND	0.607		R		163.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	ND	1.62		LOD/2		49.9
1	16258	ACD8700318	14	S	1	0.0	42.9	ND	0.777		LOD/2		75.1
1	16259	ACD8700112	14	S	1	50.0	92.9	ND	1.09		LOD/2		88.1
1	16260	ACD8700069	20	NC	3	100.0	85.0	ND	0.731		LOD/2		91.9
1	16261	ACD8700461	13	S	1	61.5	53.8	ND	0.755		LOD/2		73.3
1	16262	ACD8700194	32	NE	3	100.0	96.9	ND	1.5		LOD/2		79.3
1	16263	ACD8700390	23	S	3	100.0	69.6	ND	1.55		LOD/2		67.2
1	16264	ACD8700176	20	NE	2	100.0	90.0	ND	0.932		LOD/2		74.5
2	16267	ACD8700256	18	NE	3	44.4	100.0	ND	0.797		LOD/2		58.1
2	16268	ACD8700470	32	S	2	62.5	71.9	ND	1.3		LOD/2		74.0
2	16269	ACD8700283	24	W	3	100.0	79.2	ND	0.837		LOD/2		69.3
2	16270	ACD8700087	17	NC	3	100.0	76.5	ND	1.7		LOD/2		77.7
2	16272	ACD8700489	25	S	3	56.0	88.0	ND	0.879		LOD/2		87.4
2	16274	ACD8700096	23	NC	3	0.0	82.6	TR	0.666	0.887			86.1
2	16275	ACD8700416	17	NC	1	41.2	76.5	ND	1.44		LOD/2		69.5
2	16276	ACD8700014	18	NC	1	100.0	44.4	ND	1.93		LOD/2		75.0
2	16277	ACD8700274	16	W	2	18.8	87.5	ND	1.73		LOD/2		70.4
3	16279	ACD8700185	32	NE	3	0.0	90.6	ND	3.25		LOD/2		72.1
3	16280	ACD8700292	16	W	3	0.0	93.8	ND	2.34		LOD/2		94.4
3	16281	ACD8700354	19	S	2	0.0	84.2	ND	1.87		LOD/2		76.7
3	16282	ACD8700434	24	NC	2	100.0	91.7	ND	1.79		LOD/2		71.6

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,7,8,9-HPCDF
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDF
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	ND	1.64		LOD/2		76.6
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	1.24		LOD/2		74.8
3	16286	ACD8700381	17	S	3	0.0	82.4	ND	0.961		LOD/2		74.7
3	16288	ACD8700210	14	W	2	57.1	85.7	ND	1.42		LOD/2		69.5
3	16289	ACD8700167	20	NE	2	100.0	85.0	IR10		1.83	I	3.56	82.3
3	16291	ACD8700078	16	NC	3	0.0	87.5	TR	1.31	1.74			89.8
4	16293	ACD8700201	5	W	1	80.0	100.0	ND	1.23		LOD/2		77.1
4	16294	ACD8700130	17	S	3	41.2	88.2	ND	2.55		LOD/2		57.3
4	16295	ACD8700103	24	NC	3	45.8	100.0	ND	1.86		LOD/2		70.3
4	16297	ACD8700158	18	NE	2	0.0	83.3	ND	1.8		LOD/2		71.2
4	16299	ACD8700345	19	S	2	100.0	73.7	ND	1.36		LOD/2		79.4
4	16300	ACD8700149	20	NE	1	70.0	65.0	ND	1.0		LOD/2		63.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	ND	1.67		LOD/2		105.0
4	16302	ACD8700041	16	NC	2	100.0	81.3	ND	1.1		LOD/2		85.5
4	16304	ACD8700229	10	W	3	40.0	100.0	ND	1.24		LOD/2		77.6
5	16306	ACD8700363	14	S	2	0.0	64.3	ND	0.907		LOD/2		89.8
5	16308	ACD8700121	13	S	2	46.2	61.5	ND	2.1		LOD/2		73.5
5	16309	ACD8700265	5	W	1	20.0	100.0	ND	1.64		LOD/2		71.4
5	16310	ACD8700372	20	S	3	0.0	85.0	ND	1.17		LOD/2		80.7
5	16311	ACD8700050	27	NC	2	0.0	85.2	ND	1.02		LOD/2		69.8
5	16313	ACD8700452	18	NC	3	100.0	88.9	ND	1.08		LOD/2		76.4
5	16314	ACD8700327	17	S	1	100.0	41.2	ND	1.22		LOD/2		62.6
5	16315	ACD8700443	19	NC	3	0.0	100.0	ND	1.55		LOD/2		78.1

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,7,8,9-HPCDF
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDF
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	ND	1.27		LOD/2		69.2
5	16317	ACD8700407	15	S	3	100.0	80.0	ND	1.55		LOD/2		85.1

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,6,7,8-HPCDD
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDD
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	IR10		86.8	I	1.29	93.5
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.317	57.1	R		208.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	PQ	0.737	56.0			102.0
1	16258	ACD8700318	14	S	1	0.0	42.9	PQ	0.828	48.9			93.9
1	16259	ACD8700112	14	S	1	50.0	92.9	PQ	0.267	59.1			106.0
1	16260	ACD8700069	20	NC	3	100.0	85.0	PQ	1.06	181.			115.0
1	16261	ACD8700461	13	S	1	61.5	53.8	PQ	0.596	36.2			93.1
1	16262	ACD8700194	32	NE	3	100.0	96.9	PQ	0.85	183.			90.6
1	16263	ACD8700390	23	S	3	100.0	69.6	PQ	1.68	122.			89.2
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	0.81	90.0			103.0
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	1.57	184.			72.5
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	1.45	104.			104.0
2	16269	ACD8700283	24	W	3	100.0	79.2	PQ	0.731	208.			90.3
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	1.34	175.			114.0
2	16272	ACD8700489	25	S	3	56.0	88.0	PQ	0.878	141.			96.3
2	16274	ACD8700096	23	NC	3	0.0	82.6	PQ	0.629	207.			99.3
2	16275	ACD8700416	17	NC	1	41.2	76.5	PQ	1.13	50.4			94.0
2	16276	ACD8700014	18	NC	1	100.0	44.4	PQ	2.03	69.4			86.8
2	16277	ACD8700274	16	W	2	18.8	87.5	PQ	3.29	89.8			94.5
3	16279	ACD8700185	32	NE	3	0.0	90.6	PQ	2.04	173.			105.0
3	16280	ACD8700292	16	W	3	0.0	93.8	PQ	1.77	160.			141.0
3	16281	ACD8700354	19	S	2	0.0	84.2	PQ	0.962	96.8			118.0
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	0.766	94.3			102.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,6,7,8-HPCDD
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDD
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	0.937	177.			106.0
3	16284	ACD8700238	3	NE	1	100.0	100.0	IR10		34.5	I	1.31	112.0
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	1.4	201.			107.0
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.662	86.0			123.0
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	0.454	100.			147.0
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	0.602	178.	R		163.0
4	16293	ACD8700201	5	W	1	80.0	100.0	PQ	0.589	20.9			125.0
4	16294	ACD8700130	17	S	3	41.2	88.2	PQ	0.663	230.			88.5
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	0.956	151.			93.6
4	16297	ACD8700158	18	NE	2	0.0	83.3	PQ	1.31	135.			106.0
4	16299	ACD8700345	19	S	2	100.0	73.7	PQ	0.514	76.6			132.0
4	16300	ACD8700149	20	NE	1	70.0	65.0	PQ	0.582	51.0			110.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	PQ	0.722	89.3	R		153.0
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	0.855	140.			115.0
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	0.417	52.6	R		284.0
5	16306	ACD8700363	14	S	2	0.0	64.3	PQ	1.16	88.2			112.0
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	0.925	115.			109.0
5	16309	ACD8700265	5	W	1	20.0	100.0	PQ	1.6	25.1			112.0
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	0.624	176.			109.0
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	0.943	105.			106.0
5	16313	ACD8700452	18	NC	3	100.0	88.9	PQ	0.893	170.			111.0
5	16314	ACD8700327	17	S	1	100.0	41.2	PQ	1.5	50.9			97.9
5	16315	ACD8700443	19	NC	3	0.0	100.0	PQ	1.14	158.			100.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = 1,2,3,4,6,7,8-HPCDD
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDD
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	PQ	1.1	76.1			101.0
5	16317	ACD8700407	15	S	3	100.0	80.0	PQ	0.859	146.			120.0

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = OCDF
Internal Quantitation Standard = 13C12-OCDD
Target Ion Ratio = 0.87

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	ND	0.68		LOD/2		98.7
1	16255	ACD8700425	19	NC	2	0.0	94.7	ND	0.666		R		239.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	ND	1.14		LOD/2		122.0
1	16258	ACD8700318	14	S	1	0.0	42.9	ND	1.58		LOD/2		100.0
1	16259	ACD8700112	14	S	1	50.0	92.9	PQ	0.865	8.13			111.0
1	16260	ACD8700069	20	NC	3	100.0	85.0	ND	1.89		LOD/2		143.0
1	16261	ACD8700461	13	S	1	61.5	53.8	PQ	0.273	1.88			101.0
1	16262	ACD8700194	32	NE	3	100.0	96.9	ND	2.41		LOD/2		90.3
1	16263	ACD8700390	23	S	3	100.0	69.6	ND	1.4		LOD/2		85.6
1	16264	ACD8700176	20	NE	2	100.0	90.0	TR	0.997	1.19			102.0
2	16267	ACD8700256	18	NE	3	44.4	100.0	ND	0.916		LOD/2		76.2
2	16268	ACD8700470	32	S	2	62.5	71.9	IR25		2.32	I	0.62	111.0
2	16269	ACD8700283	24	W	3	100.0	79.2	ND	1.81		LOD/2		92.3
2	16270	ACD8700087	17	NC	3	100.0	76.5	TR	1.03	1.73			102.0
2	16272	ACD8700489	25	S	3	56.0	88.0	TR	0.832	2.99			78.5
2	16274	ACD8700096	23	NC	3	0.0	82.6	TR	0.989	1.78			71.1
2	16275	ACD8700416	17	NC	1	41.2	76.5	ND	1.65		LOD/2		91.9
2	16276	ACD8700014	18	NC	1	100.0	44.4	IR25		5.21	I	0.57	83.4
2	16277	ACD8700274	16	W	2	18.8	87.5	ND	1.82		LOD/2		91.4
3	16279	ACD8700185	32	NE	3	0.0	90.6	DC		11.3	C		109.0
3	16280	ACD8700292	16	W	3	0.0	93.8	IR10		4.27	IF	0.41	135.0
3	16281	ACD8700354	19	S	2	0.0	84.2	IR10		2.47	IF	0.71	132.0
3	16282	ACD8700434	24	NC	2	100.0	91.7	IR10		3.74	IF	1.18	111.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = OCDF
Internal Quantitation Standard = 13C12-OCDD
Target Ion Ratio = 0.87

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	IR10		2.32	IF	1.43	125.0
3	16284	ACD8700238	3	NE	1	100.0	100.0	ND	4.16		LOD/2		115.0
3	16286	ACD8700381	17	S	3	0.0	82.4	ND	2.58		LOD/2		149.0
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	0.662	2.95	R		190.0
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	0.527	3.31	R		213.0
3	16291	ACD8700078	16	NC	3	0.0	87.5	IR10		3.86	IR	0.63	248.0
4	16293	ACD8700201	5	W	1	80.0	100.0	DC		5.73	CR		184.0
4	16294	ACD8700130	17	S	3	41.2	88.2	DC		11.1	CR		164.0
4	16295	ACD8700103	24	NC	3	45.8	100.0	IR25		1.68	I	0.30	107.0
4	16297	ACD8700158	18	NE	2	0.0	83.3	DC		7.81	C		122.0
4	16299	ACD8700345	19	S	2	100.0	73.7	DC		6.58	CR		169.0
4	16300	ACD8700149	20	NE	1	70.0	65.0	DC		8.4	C		139.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	DC		5.28	CR		207.0
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	0.53	5.52	R		153.0
4	16304	ACD8700229	10	W	3	40.0	100.0	IR25		1.63	IR	1.48	151.0
5	16306	ACD8700363	14	S	2	0.0	64.3	ND	1.2		LOD/2		135.0
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	1.04	13.2			118.0
5	16309	ACD8700265	5	W	1	20.0	100.0	IR10		4.92	IR	0.61	170.0
5	16310	ACD8700372	20	S	3	0.0	85.0	DC		8.92	CR		154.0
5	16311	ACD8700050	27	NC	2	0.0	85.2	IR10		3.41	I	0.42	126.0
5	16313	ACD8700452	18	NC	3	100.0	88.9	IR10		4.0	I	1.13	142.0
5	16314	ACD8700327	17	S	1	100.0	41.2	ND	4.3		LOD/2		130.0
5	16315	ACD8700443	19	NC	3	0.0	100.0	ND	2.65		LOD/2		133.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = OCDF
Internal Quantitation Standard = 13C12-OCDD
Target Ion Ratio = 0.87

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	ND	2.19		LOD/2		118.0
5	16317	ACD8700407	15	S	3	100.0	80.0	IR10		2.68	I	0.46	132.0

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = OCDD
Internal Quantitation Standard = 13C12-OCDD
Target Ion Ratio = 0.87

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16254	ACD8700247	12	NE	2	25.0	100.0	IR10		631.	I	1.09	98.7
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	1.4	384.	R		239.0
1	16257	ACD8700023	20	NC	1	0.0	85.0	PQ	1.4	274.			122.0
1	16258	ACD8700318	14	S	1	0.0	42.9	PQ	1.96	221.			100.0
1	16259	ACD8700112	14	S	1	50.0	92.9	PQ	1.22	298.			111.0
1	16260	ACD8700069	20	NC	3	100.0	85.0	PQ	1.25	1200.			143.0
1	16261	ACD8700461	13	S	1	61.5	53.8	PQ	4.12	233.			101.0
1	16262	ACD8700194	32	NE	3	100.0	96.9	PQ	5.71	1190.			90.3
1	16263	ACD8700390	23	S	3	100.0	69.6	PQ	0.477	790.			85.6
1	16264	ACD8700176	20	NE	2	100.0	90.0	PQ	2.64	586.			102.0
2	16267	ACD8700256	18	NE	3	44.4	100.0	PQ	8.95	1230.			76.2
2	16268	ACD8700470	32	S	2	62.5	71.9	PQ	1.34	725.			111.0
2	16269	ACD8700283	24	W	3	100.0	79.2	PQ	3.37	1330.			92.3
2	16270	ACD8700087	17	NC	3	100.0	76.5	PQ	1.69	1270.			102.0
2	16272	ACD8700489	25	S	3	56.0	88.0	PQ	1.74	887.			78.5
2	16274	ACD8700096	23	NC	3	0.0	82.6	PQ	4.27	1510.			71.1
2	16275	ACD8700416	17	NC	1	41.2	76.5	PQ	1.13	234.			91.9
2	16276	ACD8700014	18	NC	1	100.0	44.4	PQ	2.25	328.			83.4
2	16277	ACD8700274	16	W	2	18.8	87.5	PQ	4.16	441.			91.4
3	16279	ACD8700185	32	NE	3	0.0	90.6	PQ	5.78	1040.			109.0
3	16280	ACD8700292	16	W	3	0.0	93.8	PQ	9.33	1160.			135.0
3	16281	ACD8700354	19	S	2	0.0	84.2	PQ	3.37	707.			132.0
3	16282	ACD8700434	24	NC	2	100.0	91.7	PQ	3.02	597.			111.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = OCDD
Internal Quantitation Standard = 13C12-OCDD
Target Ion Ratio = 0.87

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
3	16283	ACD8700309	15	W	3	0.0	93.3	PQ	7.92	874.			125.0
3	16284	ACD8700238	3	NE	1	100.0	100.0	PQ	6.48	152.			115.0
3	16286	ACD8700381	17	S	3	0.0	82.4	PQ	4.04	1630.			149.0
3	16288	ACD8700210	14	W	2	57.1	85.7	PQ	2.03	589.	R		190.0
3	16289	ACD8700167	20	NE	2	100.0	85.0	PQ	1.37	711.	R		213.0
3	16291	ACD8700078	16	NC	3	0.0	87.5	PQ	1.42	1120.	R		248.0
4	16293	ACD8700201	5	W	1	80.0	100.0	PQ	0.973	136.	R		184.0
4	16294	ACD8700130	17	S	3	41.2	88.2	PQ	1.2	1190.	R		164.0
4	16295	ACD8700103	24	NC	3	45.8	100.0	PQ	3.23	839.			107.0
4	16297	ACD8700158	18	NE	2	0.0	83.3	PQ	3.8	981.			122.0
4	16299	ACD8700345	19	S	2	100.0	73.7	PQ	1.94	431.	R		169.0
4	16300	ACD8700149	20	NE	1	70.0	65.0	PQ	1.59	235.			139.0
4	16301	ACD8700032	18	NC	2	44.4	77.8	PQ	1.71	465.	R		207.0
4	16302	ACD8700041	16	NC	2	100.0	81.3	PQ	1.66	710.	R		153.0
4	16304	ACD8700229	10	W	3	40.0	100.0	PQ	1.85	791.	R		151.0
5	16306	ACD8700363	14	S	2	0.0	64.3	PQ	8.16	695.			135.0
5	16308	ACD8700121	13	S	2	46.2	61.5	PQ	4.52	969.			118.0
5	16309	ACD8700265	5	W	1	20.0	100.0	IR10		133.	IR	1.06	170.0
5	16310	ACD8700372	20	S	3	0.0	85.0	PQ	6.45	1660.	R		154.0
5	16311	ACD8700050	27	NC	2	0.0	85.2	PQ	1.11	837.			126.0
5	16313	ACD8700452	18	NC	3	100.0	88.9	PQ	5.21	1160.			142.0
5	16314	ACD8700327	17	S	1	100.0	41.2	IR10		283.	I	1.05	130.0
5	16315	ACD8700443	19	NC	3	0.0	100.0	PQ	4.65	1170.			133.0

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS (CONT.)

Compound = OCDD
Internal Quantitation Standard = 13C12-OCDD
Target Ion Ratio = 0.87

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
5	16316	ACD8700336	17	S	2	100.0	47.1	IR10		523.	I	1.14	118.0
5	16317	ACD8700407	15	S	3	100.0	80.0	IR10		936.	I	1.06	132.0

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,7,8-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16255	ACD8700425	19	NC	2	0.0	94.7	ND	2.58		R		154.0
1	16258	ACD8700318	14	S	1	0.0	42.9			2.04			65.4
1	16260	ACD8700069	20	NC	3	100.0	85.0			13.4			79.6
1	16262	ACD8700194	32	NE	3	100.0	96.9			8.3			83.8
1	16263	ACD8700390	23	S	3	100.0	69.6			5.32			80.1
2	16276	ACD8700014	18	NC	1	100.0	44.4			3.81			83.7
3	16280	ACD8700292	16	W	3	0.0	93.8			19.1			68.2
3	16283	ACD8700309	15	W	3	0.0	93.3			12.2			72.4
3	16289	ACD8700167	20	NE	2	100.0	85.0				I	5.05	76.2
3	16291	ACD8700078	16	NC	3	0.0	87.5			46.6			76.6
4	16293	ACD8700201	5	W	1	80.0	100.0			3.11			73.0
4	16297	ACD8700158	18	NE	2	0.0	83.3			9.32			72.2
4	16299	ACD8700345	19	S	2	100.0	73.7			4.88			74.3
5	16306	ACD8700363	14	S	2	0.0	64.3			2.88			77.6
5	16311	ACD8700050	27	NC	2	0.0	85.2			6.91			71.1
5	16314	ACD8700327	17	S	1	100.0	41.2			2.53			68.8
5	16315	ACD8700443	19	NC	3	0.0	100.0			13.6			63.6
5	16316	ACD8700336	17	S	2	100.0	47.1			6.41			68.4

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET
P = PEAK MAXIMA SEPARATED BY 3 SCANS
R = IQS RECOVERY NOT IN RANGE OF 40 TO 150 PERCENT

LISTING OF FY87 NHATS COMPOSITE SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,6,7,8-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Specimen Count	Census Region	Age Group	Percent Male	Percent White	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16255	ACD8700425	19	NC	2	0.0	94.7	PQ	0.271	31.1	R		154.0
1	16258	ACD8700318	14	S	1	0.0	42.9			18.2			65.4
1	16260	ACD8700069	20	NC	3	100.0	85.0			138.			79.6
1	16262	ACD8700194	32	NE	3	100.0	96.9			114.			83.8
1	16263	ACD8700390	23	S	3	100.0	69.6			84.7			80.1
2	16276	ACD8700014	18	NC	1	100.0	44.4			36.0			83.7
3	16280	ACD8700292	16	W	3	0.0	93.8			93.9			68.2
3	16283	ACD8700309	15	W	3	0.0	93.3			106.			72.4
3	16289	ACD8700167	20	NE	2	100.0	85.0				I	1.63	76.2
3	16291	ACD8700078	16	NC	3	0.0	87.5			127.			76.6
4	16293	ACD8700201	5	W	1	80.0	100.0			19.7			73.0
4	16297	ACD8700158	18	NE	2	0.0	83.3			66.8			72.2
4	16299	ACD8700345	19	S	2	100.0	73.7			59.4			74.3
4	16301	ACD8700032	18	NC	2	44.4	77.8				I	1.74	78.9
5	16306	ACD8700363	14	S	2	0.0	64.3			47.3			77.6
5	16311	ACD8700050	27	NC	2	0.0	85.2			67.5			71.1
5	16314	ACD8700327	17	S	1	100.0	41.2			25.1			68.8
5	16315	ACD8700443	19	NC	3	0.0	100.0			114.			63.6
5	16316	ACD8700336	17	S	2	100.0	47.1			63.1			68.4

CODES USED FOR DATA RESTRICTION:
LOD/2 = CONCENTRATION USED IN STATISTICAL ANALYSIS IS LOD/2
C = COELUTION PRESENT IN CHEMICAL ANALYSIS
F = FRAGMENTED PEAK NOTED IN CHEMICAL ANALYSIS
I = ION RATIO CRITERIA NOT MET

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 2,3,7,8-TCDF
Internal Quantitation Standard = 13C12-2,3,7,8-TCDF
Target Ion Ratio = 0.76

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	PQ	0.06	0.46			76.9
1	16252	LOW SPIKE	PQ	0.096	11.4			80.7
1	16253	HIGH SPIKE	PQ	0.167	49.2			81.4
1	16256	CONTROL	PQ	0.14	1.24			71.7
2	16265	METHOD BLANK	ND	0.084		LOD/2		64.6
2	16266	CONTROL	PQ	0.108	0.999			78.5
2	16271	LOW SPIKE	PQ	0.196	11.1			74.2
2	16273	HIGH SPIKE	PQ	0.136	48.3			81.3
3	16278	METHOD BLANK	ND	0.095		LOD/2		59.0
3	16285	LOW SPIKE	PQ	0.183	10.6			70.0
3	16287	CONTROL	IR10		0.892	I	1.27	70.4
3	16290	HIGH SPIKE	PQ	0.107	44.2			69.8
4	16292	METHOD BLANK	ND	0.08		LOD/2		60.5
4	16296	CONTROL	IR25		0.74	I	1.21	72.3
4	16298	HIGH SPIKE	PQ	0.232	47.6			71.5
4	16303	LOW SPIKE	PQ	0.528	9.44			71.4
5	16305	METHOD BLANK	ND	0.294		LOD/2		59.2
5	16307	CONTROL	IR10		0.812	I	1.38	73.5
5	16312	HIGH SPIKE	PQ	0.135	44.5			71.7
5	16318	LOW SPIKE	PQ	0.303	9.7			68.4

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 2,3,7,8-TCDD
Internal Quantitation Standard = 13C12-2,3,7,8-TCDD
Target Ion Ratio = 0.76

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.196		LOD/2		70.0
1	16252	LOW SPIKE	PQ	0.053	21.3			72.3
1	16253	HIGH SPIKE	PQ	0.072	57.3			77.6
1	16256	CONTROL	PQ	0.188	10.6			66.4
2	16265	METHOD BLANK	ND	0.244		LOD/2		62.4
2	16266	CONTROL	PQ	0.135	9.13			73.1
2	16271	LOW SPIKE	PQ	0.218	17.2			69.0
2	16273	HIGH SPIKE	PQ	0.164	56.8			73.6
3	16278	METHOD BLANK	ND	0.098		LOD/2		69.3
3	16285	LOW SPIKE	PQ	0.201	17.9			80.0
3	16287	CONTROL	PQ	0.154	9.73			71.6
3	16290	HIGH SPIKE	PQ	0.143	53.3			77.3
4	16292	METHOD BLANK	ND	0.168		LOD/2		73.1
4	16296	CONTROL	PQ	0.461	8.66			79.9
4	16298	HIGH SPIKE	PQ	0.55	54.9			79.5
4	16303	LOW SPIKE	PQ	0.519	14.1			105.0
5	16305	METHOD BLANK	ND	0.416		LOD/2		64.2
5	16307	CONTROL	PQ	0.107	8.34			90.1
5	16312	HIGH SPIKE	PQ	0.131	53.6			79.6
5	16318	LOW SPIKE	PQ	0.313	17.7			77.7

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,7,8-PECDF
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDF
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.066		LOD/2		86.9
1	16252	LOW SPIKE	PQ	0.178	12.9			88.1
1	16253	HIGH SPIKE	PQ	0.423	54.7			95.5
1	16256	CONTROL	ND	0.106		LOD/2		85.6
2	16265	METHOD BLANK	ND	0.011		LOD/2		78.8
2	16266	CONTROL	ND	0.084		LOD/2		94.8
2	16271	LOW SPIKE	IR10		9.61	I	1.21	87.5
2	16273	HIGH SPIKE	PQ	0.274	49.9			94.4
3	16278	METHOD BLANK	ND	0.013		LOD/2		71.9
3	16285	LOW SPIKE	PQ	0.363	11.5			90.2
3	16287	CONTROL	TR	0.149	0.478			89.8
3	16290	HIGH SPIKE	PQ	0.281	50.2			92.9
4	16292	METHOD BLANK	ND	0.07		LOD/2		73.2
4	16296	CONTROL	ND	0.543		LOD/2		87.1
4	16298	HIGH SPIKE	PQ	0.612	49.8			103.0
4	16303	LOW SPIKE	PQ	0.617	11.4			149.0
5	16305	METHOD BLANK	ND	0.193		LOD/2		65.0
5	16307	CONTROL	ND	0.408		LOD/2		96.7
5	16312	HIGH SPIKE	PQ	0.265	46.0			100.0
5	16318	LOW SPIKE	PQ	0.402	10.6			103.0

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 2,3,4,7,8-PECDF
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDF
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.256		LOD/2		86.9
1	16252	LOW SPIKE	PQ	0.162	40.7			88.1
1	16253	HIGH SPIKE	PQ	0.386	71.4			95.5
1	16256	CONTROL	PQ	0.096	28.2			85.6
2	16265	METHOD BLANK	ND	0.01		LOD/2		78.8
2	16266	CONTROL	PQ	0.077	24.5			94.8
2	16271	LOW SPIKE	PQ	0.19	33.9			87.5
2	16273	HIGH SPIKE	PQ	0.25	74.5			94.4
3	16278	METHOD BLANK	ND	0.012		LOD/2		71.9
3	16285	LOW SPIKE	PQ	0.331	33.3			90.2
3	16287	CONTROL	PQ	0.136	24.6			89.8
3	16290	HIGH SPIKE	PQ	0.256	63.4			92.9
4	16292	METHOD BLANK	ND	0.07		LOD/2		73.2
4	16296	CONTROL	PQ	0.496	24.9			87.1
4	16298	HIGH SPIKE	PQ	0.559	71.0			103.0
4	16303	LOW SPIKE	PQ	0.563	23.0			149.0
5	16305	METHOD BLANK	ND	0.176		LOD/2		65.0
5	16307	CONTROL	PQ	0.373	23.1			96.7
5	16312	HIGH SPIKE	PQ	0.242	67.8			100.0
5	16318	LOW SPIKE	PQ	0.367	32.0			103.0

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,7,8-PECDD
Internal Quantitation Standard = 13C12-1,2,3,7,8-PECDD
Target Ion Ratio = 1.55

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.146		LOD/2		78.0
1	16252	LOW SPIKE	IR10		23.9	I	4.56	103.0
1	16253	HIGH SPIKE	PQ	0.958	59.1			111.0
1	16256	CONTROL	PQ	1.06	19.5			92.9
2	16265	METHOD BLANK	ND	0.788		LOD/2		91.9
2	16266	CONTROL	PQ	0.635	21.9			101.0
2	16271	LOW SPIKE	PQ	1.39	34.0			92.9
2	16273	HIGH SPIKE	PQ	0.756	74.3			101.0
3	16278	METHOD BLANK	ND	0.82		LOD/2		74.2
3	16285	LOW SPIKE	PQ	2.46	31.7			103.0
3	16287	CONTROL	PQ	1.38	20.9			103.0
3	16290	HIGH SPIKE	PQ	0.687	61.7			110.0
4	16292	METHOD BLANK	ND	0.117		LOD/2		85.8
4	16296	CONTROL	PQ	1.92	20.2			118.0
4	16298	HIGH SPIKE	PQ	1.92	65.2			145.0
4	16303	LOW SPIKE	IR10		25.5	I	1.94	110.0
5	16305	METHOD BLANK	ND	0.12		LOD/2		75.4
5	16307	CONTROL	PQ	1.76	21.5			112.0
5	16312	HIGH SPIKE	PQ	1.14	63.9			130.0
5	16318	LOW SPIKE	PQ	1.76	26.7			134.0

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.115		LOD/2		84.5
1	16252	LOW SPIKE	010		66.7	C		69.9
1	16253	HIGH SPIKE	010		134.	C		75.3
1	16256	CONTROL	010		42.9	C		75.7
2	16265	METHOD BLANK	ND	0.046		LOD/2		77.0
2	16266	CONTROL	010		29.2	C		89.1
2	16271	LOW SPIKE	010		55.6	C		81.1
2	16273	HIGH SPIKE	010		128.	C		91.1
3	16278	METHOD BLANK	ND	0.043		LOD/2		89.3
3	16285	LOW SPIKE	010		54.5	C		82.4
3	16287	CONTROL	PQ	0.503	18.7			75.9
3	16290	HIGH SPIKE	010		105.	C		58.4
4	16292	METHOD BLANK	ND	0.13		LOD/2		50.5
4	16296	CONTROL	010		37.2	C		65.9
4	16298	HIGH SPIKE	010		119.	C		65.5
4	16303	LOW SPIKE	010		51.0	C		76.9
5	16305	METHOD BLANK	ND	0.208		LOD/2		68.9
5	16307	CONTROL	010		33.6	C		67.3
5	16312	HIGH SPIKE	010		117.	C		79.4
5	16318	LOW SPIKE	010		54.1	C		71.5

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,6,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.177		LOD/2		84.5
1	16252	LOW SPIKE	PQ	0.164	36.5			69.9
1	16253	HIGH SPIKE	PQ	0.41	99.6			75.3
1	16256	CONTROL	PQ	0.416	12.5			75.7
2	16265	METHOD BLANK	ND	0.045		LOD/2		77.0
2	16266	CONTROL	PQ	0.394	12.2			89.1
2	16271	LOW SPIKE	IR10		23.9	I	0.86	81.1
2	16273	HIGH SPIKE	PQ	0.393	98.3			91.1
3	16278	METHOD BLANK	ND	0.042		LOD/2		89.3
3	16285	LOW SPIKE	PQ	1.06	32.2			82.4
3	16287	CONTROL	PQ	0.494	7.56			75.9
3	16290	HIGH SPIKE	PQ	0.464	73.0			58.4
4	16292	METHOD BLANK	ND	0.13		LOD/2		50.5
4	16296	CONTROL	PQ	0.711	9.21			65.9
4	16298	HIGH SPIKE	PQ	1.13	102.			65.5
4	16303	LOW SPIKE	PQ	0.564	36.5			76.9
5	16305	METHOD BLANK	ND	0.204		LOD/2		68.9
5	16307	CONTROL	PQ	0.561	12.7			67.3
5	16312	HIGH SPIKE	PQ	0.527	105.			79.4
5	16318	LOW SPIKE	PQ	0.888	32.2			71.5

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 2,3,4,6,7,8-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.278		LOD/2		84.5
1	16252	LOW SPIKE	PQ	0.196	30.8			69.9
1	16253	HIGH SPIKE	PQ	0.488	96.2			75.3
1	16256	CONTROL	IR25		1.96	I	3.23	75.7
2	16265	METHOD BLANK	ND	0.053		LOD/2		77.0
2	16266	CONTROL	PQ	0.469	1.93			89.1
2	16271	LOW SPIKE	PQ	0.576	20.5			81.1
2	16273	HIGH SPIKE	PQ	0.468	99.6			91.1
3	16278	METHOD BLANK	ND	0.05		LOD/2		89.3
3	16285	LOW SPIKE	O10		31.9	C		82.4
3	16287	CONTROL	O10		14.7	C		75.9
3	16290	HIGH SPIKE	O10		99.0	C		58.4
4	16292	METHOD BLANK	ND	0.15		LOD/2		50.5
4	16296	CONTROL	O10		15.8	C		65.9
4	16298	HIGH SPIKE	O10		114.	C		65.5
4	16303	LOW SPIKE	O10		34.2	C		76.9
5	16305	METHOD BLANK	ND	0.243		LOD/2		68.9
5	16307	CONTROL	O10		14.8	C		67.3
5	16312	HIGH SPIKE	O10		119.	C		79.4
5	16318	LOW SPIKE	O10		32.7	C		71.5

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,7,8,9-HXCDF
Internal Quantitation Standard = 13C12-1,2,3,4,7,8-HXCDF
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.038		LOD/2		84.5
1	16252	LOW SPIKE	PQ	0.213	24.9			69.9
1	16253	HIGH SPIKE	PQ	0.533	103.			75.3
1	16256	CONTROL	ND	0.54		LOD/2		75.7
2	16265	METHOD BLANK	ND	0.058		LOD/2		77.0
2	16266	CONTROL	ND	0.512		LOD/2		89.1
2	16271	LOW SPIKE	PQ	0.629	18.7			81.1
2	16273	HIGH SPIKE	PQ	0.511	98.0			91.1
3	16278	METHOD BLANK	ND	0.054		LOD/2		89.3
3	16285	LOW SPIKE	PQ	1.38	20.2			82.4
3	16287	CONTROL	ND	0.641		LOD/2		75.9
3	16290	HIGH SPIKE	PQ	0.602	71.4			58.4
4	16292	METHOD BLANK	ND	0.17		LOD/2		50.5
4	16296	CONTROL	ND	0.923		LOD/2		65.9
4	16298	HIGH SPIKE	PQ	1.47	97.3			65.5
4	16303	LOW SPIKE	PQ	0.732	18.1			76.9
5	16305	METHOD BLANK	ND	0.265		LOD/2		68.9
5	16307	CONTROL	ND	0.728		LOD/2		67.3
5	16312	HIGH SPIKE	PQ	0.685	91.4			79.4
5	16318	LOW SPIKE	PQ	1.15	17.4			71.5

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,7,8/1,2,3,6,7,8-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.280		LOD/2		74.3
1	16252	LOW SPIKE	PQ	0.298	195.			76.9
1	16253	HIGH SPIKE	PQ	0.431	375.			75.4
1	16256	CONTROL	PQ	0.687	134.			78.8
2	16265	METHOD BLANK	ND	0.875		LOD/2		65.6
2	16266	CONTROL	PQ	1.11	140.			77.4
2	16271	LOW SPIKE	PQ	0.964	147.			75.2
2	16273	HIGH SPIKE	PQ	0.809	400.			81.4
3	16278	METHOD BLANK	ND	0.757		LOD/2		78.4
3	16285	LOW SPIKE	PQ	1.93	179.			78.5
3	16287	CONTROL	PQ	1.1	151.			75.9
3	16290	HIGH SPIKE	PQ	0.899	421.			65.3
4	16292	METHOD BLANK	ND	0.8		LOD/2		49.4
4	16296	CONTROL	PQ	1.79	126.			74.6
4	16298	HIGH SPIKE	PQ	1.35	375.			71.2
4	16303	LOW SPIKE	PQ	0.844	175.			77.1
5	16305	METHOD BLANK	ND	0.941		LOD/2		73.0
5	16307	CONTROL	PQ	1.16	133.			69.5
5	16312	HIGH SPIKE	PQ	1.34	324.			84.4
5	16318	LOW SPIKE	PQ	1.59	171.			76.6

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,7,8,9-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.405		LOD/2		74.3
1	16252	LOW SPIKE	PQ	0.307	49.5			76.9
1	16253	HIGH SPIKE	PQ	0.444	133.			75.4
1	16256	CONTROL	PQ	0.708	19.6			78.8
2	16265	METHOD BLANK	ND	0.902		LOD/2		65.6
2	16266	CONTROL	PQ	1.14	23.0			77.4
2	16271	LOW SPIKE	PQ	0.994	44.9			75.2
2	16273	HIGH SPIKE	PQ	0.834	136.			81.4
3	16278	METHOD BLANK	ND	0.78		LOD/2		78.4
3	16285	LOW SPIKE	PQ	1.99	45.2			78.5
3	16287	CONTROL	IR10		20.8	I	1.69	75.9
3	16290	HIGH SPIKE	PQ	0.927	150.			65.3
4	16292	METHOD BLANK	ND	0.82		LOD/2		49.4
4	16296	CONTROL	PQ	1.85	19.5			74.6
4	16298	HIGH SPIKE	PQ	1.39	127.			71.2
4	16303	LOW SPIKE	PQ	0.87	45.5			77.1
5	16305	METHOD BLANK	ND	0.97		LOD/2		73.0
5	16307	CONTROL	PQ	1.19	22.1			69.5
5	16312	HIGH SPIKE	PQ	1.38	125.			84.4
5	16318	LOW SPIKE	PQ	1.64	39.2			76.6

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,6,7,8-HPCDF
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDF
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.197		LOD/2		78.2
1	16252	LOW SPIKE	PQ	0.387	57.6			71.4
1	16253	HIGH SPIKE	PQ	0.797	136.			74.3
1	16256	CONTROL	PQ	0.676	31.8			78.0
2	16265	METHOD BLANK	ND	0.195		LOD/2		63.5
2	16266	CONTROL	PQ	0.595	27.0			71.6
2	16271	LOW SPIKE	PQ	0.967	61.3			84.2
2	16273	HIGH SPIKE	PQ	1.3	165.			81.7
3	16278	METHOD BLANK	ND	0.372		LOD/2		64.7
3	16285	LOW SPIKE	PQ	1.56	59.0			82.3
3	16287	CONTROL	PQ	0.316	29.1			94.0
3	16290	HIGH SPIKE	PQ	0.792	180.			83.0
4	16292	METHOD BLANK	ND	0.13		LOD/2		67.6
4	16296	CONTROL	PQ	0.952	30.1			65.4
4	16298	HIGH SPIKE	PQ	1.03	185.			66.9
4	16303	LOW SPIKE	PQ	0.839	53.5			79.4
5	16305	METHOD BLANK	ND	0.546		LOD/2		75.3
5	16307	CONTROL	PQ	0.682	31.1			72.4
5	16312	HIGH SPIKE	PQ	0.665	157.			83.5
5	16318	LOW SPIKE	PQ	1.26	60.1			72.6

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,7,8,9-HPCDF
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDF
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.175		LOD/2		78.2
1	16252	LOW SPIKE	PQ	0.553	31.4			71.4
1	16253	HIGH SPIKE	PQ	1.14	123.			74.3
1	16256	CONTROL	PQ	0.965	1.64			78.0
2	16265	METHOD BLANK	ND	0.279		LOD/2		63.5
2	16266	CONTROL	ND	0.915		LOD/2		71.6
2	16271	LOW SPIKE	PQ	1.38	29.6			84.2
2	16273	HIGH SPIKE	PQ	1.86	137.			81.7
3	16278	METHOD BLANK	ND	0.532		LOD/2		64.7
3	16285	LOW SPIKE	PQ	2.23	28.2			82.3
3	16287	CONTROL	TR	0.452	1.21			94.0
3	16290	HIGH SPIKE	PQ	1.13	133.			83.0
4	16292	METHOD BLANK	ND	0.18		LOD/2		67.6
4	16296	CONTROL	ND	1.36		LOD/2		65.4
4	16298	HIGH SPIKE	PQ	1.48	145.			66.9
4	16303	LOW SPIKE	PQ	1.2	28.2			79.4
5	16305	METHOD BLANK	ND	0.781		LOD/2		75.3
5	16307	CONTROL	ND	0.974		LOD/2		72.4
5	16312	HIGH SPIKE	PQ	0.951	112.			83.5
5	16318	LOW SPIKE	PQ	1.81	29.6			72.6

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,6,7,8-HPCDD
Internal Quantitation Standard = 13C12-1,2,3,4,6,7,8-HPCDD
Target Ion Ratio = 1.02

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.64		LOD/2		97.2
1	16252	LOW SPIKE	PQ	0.26	170.			89.8
1	16253	HIGH SPIKE	PQ	0.57	282.			91.3
1	16256	CONTROL	PQ	0.628	151.			104.0
2	16265	METHOD BLANK	ND	0.076		LOD/2		77.6
2	16266	CONTROL	PQ	2.1	144.			96.4
2	16271	LOW SPIKE	PQ	1.37	168.			113.0
2	16273	HIGH SPIKE	PQ	1.47	254.			110.0
3	16278	METHOD BLANK	ND	0.208		LOD/2		76.6
3	16285	LOW SPIKE	PQ	1.61	149.			117.0
3	16287	CONTROL	PQ	0.459	125.			134.0
3	16290	HIGH SPIKE	PQ	0.629	264.			133.0
4	16292	METHOD BLANK	ND	0.22		LOD/2		137.0
4	16296	CONTROL	PQ	1.15	140.			99.1
4	16298	HIGH SPIKE	PQ	0.77	225.			118.0
4	16303	LOW SPIKE	PQ	0.862	163.			112.0
5	16305	METHOD BLANK	ND	1.1		LOD/2		113.0
5	16307	CONTROL	PQ	0.879	138.			104.0
5	16312	HIGH SPIKE	PQ	0.797	239.			120.0
5	16318	LOW SPIKE	PQ	0.997	146.			109.0

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = OCDF
Internal Quantitation Standard = 13C12-OCDD
Target Ion Ratio = 0.87

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	0.532		LOD/2		88.0
1	16252	LOW SPIKE	IR10		51.	I	1.06	83.2
1	16253	HIGH SPIKE	PQ	1.39	236.			93.3
1	16256	CONTROL	ND	2.65		LOD/2		116.0
2	16265	METHOD BLANK	ND	1.52		LOD/2		65.7
2	16266	CONTROL	TR	0.795	2.37			89.9
2	16271	LOW SPIKE	PQ	2.13	57.1			106.0
2	16273	HIGH SPIKE	PQ	1.82	242.			106.0
3	16278	METHOD BLANK	ND	1.24		LOD/2		72.8
3	16285	LOW SPIKE	PQ	2.98	47.3			127.0
3	16287	CONTROL	ND	0.825		R		199.0
3	16290	HIGH SPIKE	PQ	0.553	222.	R		204.0
4	16292	METHOD BLANK	ND	0.343		R		159.0
4	16296	CONTROL	PQ	1.08	7.3			117.0
4	16298	HIGH SPIKE	PQ	1.03	268.	R		155.0
4	16303	LOW SPIKE	PQ	1.06	63.8			146.0
5	16305	METHOD BLANK	ND	3.4		LOD/2		128.0
5	16307	CONTROL	TR	0.616	2.39			131.0
5	16312	HIGH SPIKE	PQ	1.51	207.	R		165.0
5	16318	LOW SPIKE	PQ	1.34	42.7	R		162.0

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = OCDD
Internal Quantitation Standard = 13C12-OCDD
Target Ion Ratio = 0.87

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16251	METHOD BLANK	ND	1.45		LOD/2		88.0
1	16252	LOW SPIKE	IR10		1530.	I	1.26	83.2
1	16253	HIGH SPIKE	PQ	0.56	2040.			93.3
1	16256	CONTROL	PQ	3.85	1130.			116.0
2	16265	METHOD BLANK	ND	1.12		LOD/2		65.7
2	16266	CONTROL	PQ	3.44	1250.			89.9
2	16271	LOW SPIKE	PQ	2.74	1550.			106.0
2	16273	HIGH SPIKE	PQ	5.15	1910.			106.0
3	16278	METHOD BLANK	ND	1.03		LOD/2		72.8
3	16285	LOW SPIKE	PQ	3.87	1500.			127.0
3	16287	CONTROL	PQ	1.75	1170.	R		199.0
3	16290	HIGH SPIKE	IR10		1440.	IR	1.34	204.0
4	16292	METHOD BLANK	ND	0.993		R		159.0
4	16296	CONTROL	PQ	5.8	1180.			117.0
4	16298	HIGH SPIKE	PQ	1.88	1760.	R		155.0
4	16303	LOW SPIKE	PQ	6.99	1520.			146.0
5	16305	METHOD BLANK	FP25		3.28	F		128.0
5	16307	CONTROL	PQ	3.8	1190.			131.0
5	16312	HIGH SPIKE	PQ	4.32	1650.	R		165.0
5	16318	LOW SPIKE	PQ	4.62	1440.	R		162.0

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,4,7,8-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16252	LOW SPIKE			40.6			76.9
1	16253	HIGH SPIKE			142.			75.4
2	16266	CONTROL			9.16			77.4
2	16271	LOW SPIKE			15.0			75.2
2	16273	HIGH SPIKE			163.			81.4
3	16285	LOW SPIKE			31.0			78.5
3	16290	HIGH SPIKE			201.			65.3
4	16298	HIGH SPIKE			127.			71.2
4	16303	LOW SPIKE			29.2			77.1
5	16307	CONTROL			8.36			69.5

LISTING OF FY87 NHATS QC SAMPLE DATA
FOR CHLORINATED DIOXINS AND FURANS

Compound = 1,2,3,6,7,8-HXCDD
Internal Quantitation Standard = 13C12-1,2,3,6,7,8-HXCDD
Target Ion Ratio = 1.22

Batch	Laboratory Number	Sample ID	Data Qualifier	LOD (pg/g)	Conc. (pg/g)	Data Restriction	Ion Ratio	IQS Recovery
1	16252	LOW SPIKE			154.			76.9
1	16253	HIGH SPIKE			233.			75.4
2	16266	CONTROL			131.			77.4
2	16271	LOW SPIKE			132.			75.2
2	16273	HIGH SPIKE			237.			81.4
3	16285	LOW SPIKE			148.			78.5
3	16290	HIGH SPIKE			220.			65.3
4	16298	HIGH SPIKE			248.			71.2
4	16303	LOW SPIKE			146.			77.1
5	16307	CONTROL			125.			69.5

APPENDIX B

ANALYTICAL PROTOCOL

1.0 SAMPLE EXTRACTION

1.1 Extraction of Adipose Tissue

Addition of internal quantitation standards -- Allow the adipose tissue composite to reach room temperature and then add the carbon-13 internal quantitation spiking solution such that it delivers 500 to 2,500 pg of each of the ^{13}C-labeled surrogates.

Add 10 mL of methylene chloride and homogenize the mixture for approximately 1 min with a Tekmar Tissuemizer®.

Allow the mixture to separate and decant the methylene chloride extract from the residual solid material using a disposable pipette. The methylene chloride is eluted through a filter funnel containing a plug of clean glass wool and 5 to 10 g of anhydrous sodium sulfate. The dried extract is collected in a 100-mL volumetric flask.

Add to the sample a second 10-mL aliquot of methylene chloride and homogenized for 1 min. The methylene chloride is decanted, dried, and transferred to the 100-mL volumetric flask.

Rinse culture tube with at least two additional aliquots (10 mL each) of methylene chloride, and transfer the entire contents to the filter funnel containing the anhydrous sodium sulfate. The filter funnel and contents are rinsed with additional methylene chloride (20 to 40 mL). The total eluant from the filter funnel is collected in the 100-mL volumetric flask. Discard the sodium sulfate.

Adjust the final volume of the extract for each sample to 100 mL in the volumetric flask using methylene chloride.

1.2 Lipid Determination

Preweigh a clean 1-dram glass vial to the nearest 0.0001 g using an analytical balance tared to zero.

Accurately transfer 1.0 mL of the final extract (100 mL) to the 1-dram vial. Reduce the volume of methylene chloride from the extract using a water bath (50-60°C) gentle stream of purified nitrogen until an oil residue remains.

Accurately weigh the 1-dram vial and residue to the nearest 0.001 g and calculate the weight of lipid present in the vial based on difference. Nitrogen blowdown is continued until a constant weight is achieved.

Calculate the percent lipid content of the original sample to the nearest 0.1% as shown in Equation B-1.

$$\text{Lipid content, LC (\%)} = \frac{W_{LR} \times V_{EXT}}{W_{AT} \times V_{AL}} \times 100\% \qquad \text{Eq. B-1}$$

where: W_{LR} = weight of the lipid residue to the nearest 0.0001 g;

V_{EXT} = total volume of the extract in milliliters;

W_{AT} = weight of the original adipose tissue composite to the nearest 0.01 g; and

V_{AT} = volume of the aliquot of the final extract in mL used for the quantitative measure of the lipid residue (1.0 mL).

1.3 Extract Concentration

Quantitatively transfer the remaining extract volume (99.0 mL) to a 500-mL Erlenmeyer flask. Rinse the volumetric flask with 20 to 30 mL of additional methylene chloride to ensure quantitative transfer.

Concentrate the extract to an oily residue using rotary evaporation.

2.0 CLEANUP PROCEDURES

2.1 Bulk Lipid Removal

Add a total of 200 mL of *n*-hexane to the spiked lipid residue in the 500-mL Erlenmeyer flask. Slowly add, with stirring, 100 g of the 40% w/w sulfuric acid impregnated silica gel. Stir with a magnetic stir-plate for 2 h.

Allow solids to settle and decant liquid through a powder funnel containing 20 g of anhydrous sodium sulfate and collect in a 500-mL sample bottle. Rinse solids with two 50-mL portions of hexane. Stir each rinse for 15 min, decant, and dry by elution through sodium sulfate combining the hexane extracts.

After the rinses have gone through the sodium sulfate, rinse the sodium sulfate with an additional 25 mL of hexane and combine with the hexane extracts.

Prepare an acidic silica and a neutral alumina column as follows: Pack a 1 cm x 10 cm chromatographic column with a glass wool plug, add approximately 25 mL of hexane, add 1.0 g of silica gel, and then add 4.0 g of 40% w/w sulfuric acid impregnated silica gel and allow to settle. Elute the excess hexane from the column until the solvent level reaches the top of the chromatographic packing. Verify that the column does not contain any bubbles or channels. Pack a second chromatographic column (1 cm x 30 cm) with a glass wool plug, add 4 g of sodium sulfate, add 4.0 g of neutral alumina and allow to settle, and then top with a 4-g layer of sodium sulfate. Elute the column with 10 mL of hexane until the solvent level reaches the top of the chromatographic packing. Inspect the column to ensure it is free of channels and air bubbles.

Quantitatively transfer the hexane extract from the Erlenmeyer flask to the silica gel column reservoir. Allow the hexane extract to percolate through the column and collect in a KD concentrator.

Complete the elution of the extract from the silica gel column with 50 mL of hexane in the KD concentrator. Concentrate the eluate to approximately 1.0 mL, using nitrogen blowdown as necessary.

Note: If the 40% sulfuric acid/silica gel in noted to be highly discolored throughout the length of the adsorbent bed, it is necessary to repeat the cleaning procedure beginning with the acidic silica gel slurry procedure.

2.2 Separation of Chemical Interferences

Transfer the concentrate (1.0 mL) to the top of the alumina column. Rinse the K-D concentrator with two 1.0-mL portions of hexane and transfer the rinses to the top of the alumina column. Elute the alumina column with 10 mL of 8% (v/v) methylene chloride in hexane until the solvent level is just below the top of the sodium sulfate. Archive this eluate. Columns must not be allowed to reach dryness (i.e., a solvent "head" must be maintained).

Elute the column with 15 mL of 60% (v/v) methylene chloride in hexane and collect this fraction containing the dioxins and furans in a 50-mL culture tube. Concentrate the fraction to a volume of approximately 2 mL.

Prepare an AX-21/silica gel mixture by thoroughly mixing 1 g of AX-21 (100/325 mesh) and 19 g of silica gel in a 40-mL vial. Activate at 130°C for 6 h. Store in a desiccator. Cut off a clean 5-mL disposable glass pipet (6 to 7 mm ID) at the 4-mL mark. Insert a plug of glass wool and push to the 2-mL mark. Add 1 g of the activated AX-21/silica gel mixture followed by another glass wool plug. Using two glass rods, push both glass wool plugs simultaneously towards the AX-21/silica gel mixture and gently compress the AX-21/silica gel plug to a length of 4 cm. Preelute the column with 4 mL of toluene followed by 2 mL of 75:20:5 methylene chloride/methanol/benzene, and 4 mL of 1:1 cyclohexane in methylene chloride. The flow rate should be less than 0.5 mL/min. While the column is still wet, add the concentrated eluate from the alumina column to the top of the AX-21/silica gel column. Rinse the culture tube which contained the extract twice with 1 mL of hexane and add the rinsates to the top of the column. Elute the column sequentially with two 0.5-mL aliquots of hexane, 10 mL of 1:1 cyclohexane in methylene chloride, and 5 mL of 75:20:5 methylene chloride/methanol/benzene. Combine and archive the first three eluates. Turn the column upside down and elute the PCDD/PCDF fraction with 20 mL of toluene into a 6-dram vial.

Using a stream of nitrogen, reduce the toluene volume to approximately 1 mL. Carefully transfer the concentrate into a 1-mL minivial and reduce the volume to about 200 µL using a stream of nitrogen.

Rinse the concentrator tube with three washings using 500 µL of 1% toluene in methylene chloride. Concentrate to 200-500 µL and add 10 µL of the

tridecane solution containing the internal recovery standard and store the sample in a refrigerator until HRGC/MS analysis.

Prior to analysis, using a gentle stream of nitrogen at room temperature, remove toluene and methylene chloride. Submit sample to HRGC/MS once a stable 10-µL volume of tridecane is attained.

2.3 HRGC/HRMS Analysis for PCDD/PCDF

Once initial and routine calibration criteria are met, the instrument is ready for sample analysis. Prior to the first sample, an injection of tridecane will be analyzed to document system cleanliness. If any evidence of system contamination is found, corrective action must be taken and another tridecane blank analyzed.

Note: Syringe Technique -- Congeners of PCDD/PCDF can carry-over between injections in the syringes used for HRGC/MS analysis unless the syringes are properly cleaned between samples. The following procedure has been found to be very effective for PCDD/PCDF removal from contaminated syringes and will be used throughout these analyses.

- Rinse the syringe 10 times with isooctane.
- Fill the syringe with toluene and sonicate syringe and plunger in toluene for 5 min and repeat at least twice.
- Rinse the syringe 10 times with tridecane and pull up 1 µL of clean tridecane.
- Syringe is ready for use.

At no time should air be introduced into the HRGC column by using an air plug in the syringe. The oxygen present in the air plug will quickly degrade a nonbonded GC phase.

Inject a 1-µL aliquot of the extract into the GC, operated under the conditions previously used to produce acceptable results with the performance check solution.

Acquire SIM data according to the same acquisition and MS operating conditions previously used to determine the relative response factors.

Instrument performance will be monitored by examining and recording the peak areas for the recovery standard, $^{13}C_{12}$-1,2,3,4-TCDD. If this area should decrease to less than 50% of the calibration standard, sample analyses will be stopped until the problem is found and corrected.

2.4 HRGC/MS Analysis for PBDD/PBDF

Procedures for the analysis of the brominated species will be similar to the procedures for the PCDD/PCDF analysis, with the following exceptions:

1. There is no column performance window-defining mix available.

2. Sensitivity and daily RRF check will be established by injecting the CS5 level calibration standard.

SIM data will be acquired using the same chromatographic and MS operating conditions used to determine the relative response factors. Instrument performance will be monitored by examining and recording the peak areas for the recovery standard, $^{13}C_{12}$-1,2,3,4,7,8-HxCDD. If this area should decrease to less than 50% of the daily calibration standard, sample analysis will be stopped until the problem is found and corrected.

3.0 CALCULATIONS

In this section, the procedures for the data reduction are outlined for the analysis of data from the HRGC/HRMS method for PCDD/PCDF and the HRGC/MS method for PBDD/PBDF. Figure B-1 presents a schematic of the qualitative criteria for identifying PCDDs and PCDFs. Identical calculations and qualitative criteria will be applied to the data generated for the brominated analogs of the dioxins and furans.

3.1 Qualitative Identification

The ion current responses for each mass for a particular analyte must be within ±1 s to attain positive identification of that analyte. For example, m/z 338 and m/z 340 must have maximum peak responses that are within ±1 s to be positively identified as a pentachlorodibenzofuran.

The ion current intensities for a particular PCDD/PCDF or PBDD/PBDF must be ≥ 2.5 times the noise level (S/N ≥ 2.5) for positive identification of that isomer. The integrated ion current ratios of the analytical masses for a particular PCDD/PCDF must fall within the ranges shown in Tables B-1 and B-2.

3.2 Quantitative Calculations

Relative response factors for native PCDD and PCDF analytes (RRF) are calculated from the data obtained during the analysis of concentration calibration solutions using the following formula:

$$RRF = \frac{A_{STD} \cdot C_{IS}}{A_{IS} \cdot C_{STD}} \qquad \text{Eq. B-2}$$

where: A_{STD} = the sum of the areas of the integrated ion abundances for the analyte in question. For example, for TCDD, A_{STD} would be the sum of the integrated ion abundances for m/z 320 and 322;

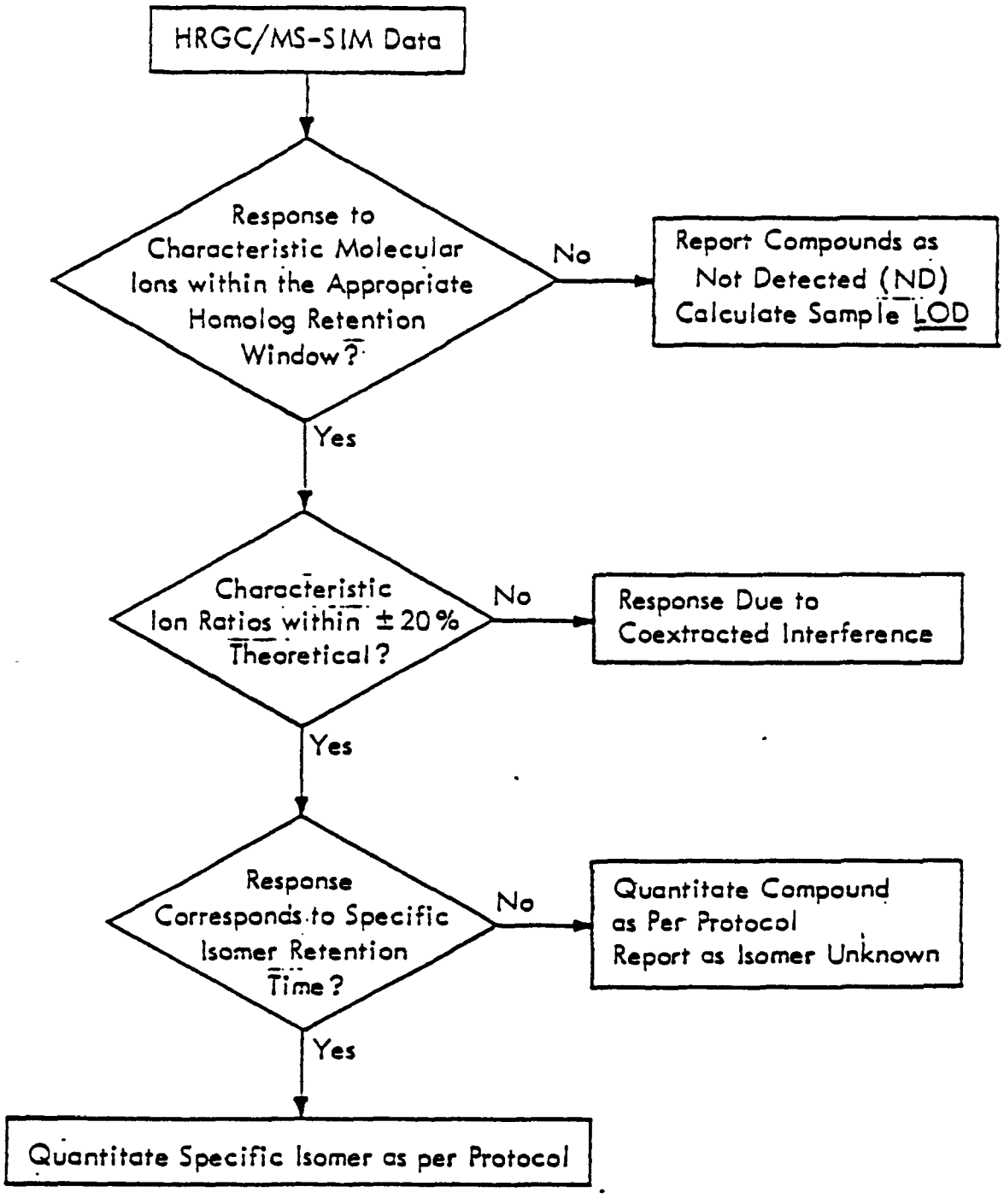

Figure B-1. Qualitative criteria for identifying PCDDs and PCDFs.

Table B-1. Ion Ratios for HRGC/HRMS Analysis of PCDD/PCDF

Compound	Ions monitored	Theoretical ratio	Acceptable range
TCDF	304/306	0.76	0.61 - 0.91
$^{13}C_{12}$-TCDF	316/318	0.76	0.61 - 0.91
TCDD	320/322	0.76	0.61 - 0.91
$^{13}C_{12}$-TCDD	332/334	0.76	0.61 - 0.91
PeCDF	340/342	1.55	1.24 - 1.86
$^{13}C_{12}$-PeCDF	352/354	1.55	1.24 - 1.86
PeCDD	356/358	1.55	1.24 - 1.86
$^{13}C_{12}$-PeCDD	368/370	1.55	1.24 - 1.86
HxCDF	374/376	1.22	0.93 - 1.46
$^{13}C_{12}$-HxCDF	384/386	0.50	0.40 - 0.60
HxCDD	390/392	1.22	0.98 - 1.46
$^{13}C_{12}$-HxCDF	402/404	1.22	0.98 - 1.46
HpCDF	408/410	1.02	0.82 - 1.22
$^{13}C_{12}$-HpCDF	418/420	0.44	0.35 - 0.53
HpCDD	424/426	1.02	0.82 - 1.22
$^{13}C_{12}$-HpCDD	436/438	1.02	0.82 - 1.22
OCDF	442/444	0.87	0.70 - 1.04
OCDD	458/460	0.87	0.70 - 1.04
$^{13}C_{12}$-OCDD	470/472	0.87	0.70 - 1.04

Table B-2. Ion Ratios for HRGC/HRMS Analysis of PBDD/PBDF

Compound	Ions monitored	Theoretical ratio	Acceptable range
TBDF	482/484	0.68	0.54 - 0.82
$^{13}C_{12}$-TBDF	494/496	0.68	0.54 - 0.82
TBDD	498/500	0.68	0.54 - 0.82
$^{13}C_{12}$-TBDD	510/512	0.68	0.54 - 0.82
PeBDF	562/564	1.02	0.82 - 1.22
$^{13}C_{12}$-PeBDF	574/576	1.02	0.82 - 1.22
PeBDD	578/580	1.02	0.82 - 1.22
HxBDF	642/644	1.35	1.08 - 1.62
HxBDD	658/660	1.35	1.08 - 1.62

A_{IS} = the sum of the area of the integrated ion abundances for the labeled PCDD/F used as the internal quantitation standard for the above analyte. For example, for $^{13}C_{12}$-2,3,7,8-TCDD, A_{IS} would be the sum of the integrated ion abundance for m/z 332 and 334.

C_{STD} = concentration of the analyte in pg/µL; and

C_{IS} = concentration of the internal quantitation standard in pg/µL.

Table B-3 provides the pairing of target analytes to internal quantitation standards for determining RRF values for PCDD, PCDF, PBDD, and PBDF compounds.

Relative response factors for the internal quantitation standards (RRF_{IS}). The RRF_{IS} values are calculated from data obtained during the analysis of concentration calibration solutions using the following formula.

$$RRF_{IS} = \frac{A_{IS} \times C_{RS}}{A_{RS} \times C_{IS}} \qquad \text{Eq. B-3}$$

where A_{IS} and C_{IS} are defined as given in Eq. B-2 and

C_{RS} = concentration of the internal recovery standard in pg/µL; and

A_{RS} = the sum of the areas of the integrated ion abundances for the labeled PCDD ($^{13}C_{12}$-1,2,3,4-TCDD or $^{13}C_{12}$-1,2,3,7,8,9-HxCDD). For example, the $^{13}C_{12}$-1,2,3,4-TCDD, A_{RS} would be the sum of the integrated ion abundance for m/z 332 and 334.

The RRF values for the $^{13}C_{12}$-PBDD/PBDF compounds will be calculated using a similar equation. Refer to Table B-1 for pairing of the internal quantitation standards with the appropriate internal recovery standard.

3.3 Concentrations of Sample Components

Figure B-2 presents a schematic for quantitation of PCDD and PCDFs which meet the criteria specific in Section 3. Calculate the concentration of PCDD/Fs or PBDD/Fs in sample extracts using the formula:

$$C_{sample} = \frac{A_{sample} \cdot Q_{IS} \cdot 100}{A_{IS} \cdot RRF \cdot W_{AT} \cdot LC} \qquad \text{Eq. B-4}$$

where: C_{sample} = the lipid adjusted concentration of PCDD or PCDF congener in pg/g;

A_{sample} = sum of the integrated ion abundances determined for the PCDD/PCDF in question;

Table B-3. Target Analyte/Internal Quantitation Standard and Internal Quantitation Standard/Internal Recovery Standard Pairs

Target analyte	Internal standards: Quantitation	Internal standards: Recovery
Chlorinated		
2,3,7,8-TCDD	$^{13}C_{12}$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD
2,3,7,8-TCDF	$^{13}C_{12}$-2,3,7,8-TCDF	$^{13}C_{12}$-1,2,3,4-TCDD
1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,4-TCDD
1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,7,8,9-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
OCDF	$^{13}C_{12}$-OCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
OCDD	$^{13}C_{12}$-OCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
Brominated		
2,3,7,8-TBDD	$^{13}C_{12}$-2,3,7,8-TBDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
2,3,7,8-TBDF	$^{13}C_{12}$-2,3,7,8-TBDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,7,8-PeBDD	$^{13}C_{12}$-1,2,3,7,8-PeBDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,7,8-PeBDF	$^{13}C_{12}$-1,2,3,7,8-PeBDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,4,7,8-HxBDD	$^{13}C_{12}$-1,2,3,7,8-PeBDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
1,2,3,4,7,8-HxBDF	$^{13}C_{12}$-1,2,3,7,8-PeBDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD

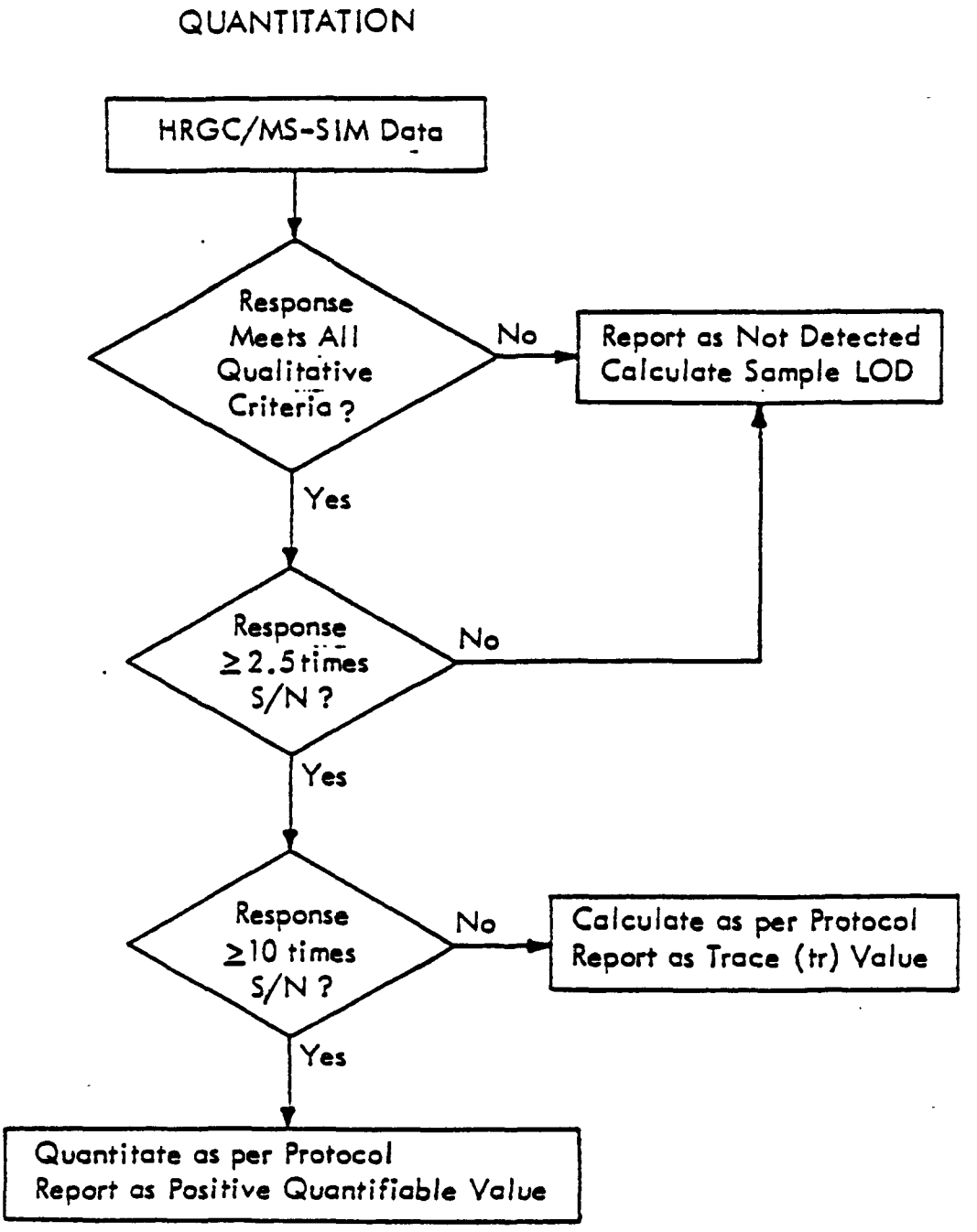

Figure B-2. Procedure for quantitation of PCDDs and PCDFs in human adipose tissue.

A_{IS} = sum of the integrated ion abundances determined for the labeled PCDD/F used as the internal quantitation standard for the above analyte;

Q_{IS} = the amount (total pg) of the labeled internal quantitation standard added to the sample prior to extraction;

RRF = relative response factor of the above analyte relative to its labeled internal quantitation standard determined from the initial triplicate calibration;

W_{AT} = weight (g) of original adipose tissue sample; and

LC = percent extractable lipid determined from Eq. 4-2.

Refer to Table B-1 for pairing of target analytes with the appropriate internal quantitation standard.

Quantitative data will be classified to indicate the intensity of the signal response. Qualifiers will include: not detected, ND (signal-to-noise ratio is less than 2.5); trace, TR (signal-to-noise ratio is greater than or equal to 2.5 but less than 10); and positive quantifiable, PQ (signal-to-noise ratio is greater than or equal to 10).

Because of the lack of analytical standards to demonstrate isomer specificity of the brominated compound, all PBDD and PBDF responses that elute at a retention time of a 2,3,7,8-substituted isomer will be classified as a maximum possible concentration (MPC) for that specific isomer.

Recovery of internal quantitation standards. Calculate the recovery of the labeled internal quantitation standards measured in the final extract using the formula:

$$\text{Internal Quant. Std. Percent Recovery} = \frac{A_{IS} \cdot Q_{RS}}{A_{RS} \cdot Q_{IS} \cdot RRF_{IS}} \cdot 100 \qquad \text{Eq. B-5}$$

where: A_{IS} = sum of the integrated ion abundances determined for the labeled PCDD/PCDF internal quantitation standard in question;

A_{RS} = sum of the integrated ion abundances determined for m/z 332 and m/z 334 of $^{13}C_{12}$-1,2,3,4-TCDD or m/z 390 and m/z 392 of $^{13}C_{12}$-1,2,3,7,8,9-HxCDD (recovery standards);

Q_{RS} = amount (pg) of the respective recovery standard, added to the final extract;

Q_{IS} = amount (pg) of the labeled internal quantitation standard added to the sample prior to extraction; and

- If a response for a specific dioxin or furan congener is qualified as a trace, TR, value (signal-to-noise ratio is greater than or equal to 2.5 but less than 10), the analyst will also provide an estimated method detection limit. This is accomplished by using the observed signal-to-noise ratio on either side of the response and calculating as given in Eq. B-6.

APPENDIX C

METHOD FOR ESTIMATING MEASURES OF UNCERTAINTY

The additive model for analyzing NHATS composite sample data has been presented and discussed in Chapter 7. This model assumes that the concentration for an individual specimen is equal to a linear combination of fixed effects (census region, age group, sex, and race group effects) plus the sum of two independent random effects. The random effects are the effect due to random selection of the MSA and the effect due to random selection of the donor from that MSA. The MSA effect is assumed to have variance σ_m^2, and the donor effect has variance σ_e^2. In addition, random measurement error in the observed concentration for an analyzed composite sample is also present and assumed to have variance σ_γ^2. This appendix discusses how these three measures of uncertainty are estimated and interpreted.

The estimate of σ_γ^2 is obtained from the analysis of control QC samples, detailed in Chapter 6. Control samples were unspiked samples taken from a homogenized bulk sample of human adipose tissue. As a result, the variability in the predicted concentration for unspiked control samples reflects measurement error exclusively. The estimate of σ_γ was obtained from Table 6-3 by multiplying the predicted concentration by the coefficient of variation (as a proportion) for the control samples. The result is equal to the estimated standard deviation of the measured concentration in the control samples. The control sample concentration was nearly equal to the average concentration of the study samples. The estimate of σ_γ is given for each of the compounds in Table C-1, along with the coefficient of variation (in percentage terms) relative to the estimated national average concentration.

As discussed in Chapter 7, estimating the additive model parameters in the prediction process requires an iterative weighted generalized least squares procedure. The procedure is iterative and weighted in nature because the covariance matrix of measured composite concentrations, used in estimating the fixed effect parameters, depends on the three unknown variance terms given above and on the unknown fixed parameters themselves. Starting estimates for these parameters, used to begin the iterative estimation process, are obtained from a mixed model analysis which assumes constant error variances. This approximate model is fitted using the P3V procedure in the BMDP statistical software package. The P3V procedure gives starting values for the fixed effect parameters and estimates for two surrogate variance components. One of the surrogate variance components is due to random selection of MSAs and the other is due to the combined effects of random donor sampling and measurement error.

Table C-1. Estimates of Measurement and Sampling Standard Deviation (in pg/g), and Coefficients of Variation (in percent), for the PCDDs and PCDFs Analyzed in the FY87 NHATS

	Standard deviation and CV (%) due to . . .					
	Measurement error		Sampling MSAs		Sampling individuals	
Analyte	σ_γ	(CV_γ)[a]	σ_m	(CV_m)	σ_e	(CV_e)
2,3,7,8-TCDD	1.77	(33)	0.0	(0)	0.0	(0)
1,2,3,7,8-PeCDD	1.84	(17)	0.0	(0)	2.93	(54)
1,2,3,4,7,8/6,7,8-HxCDD	15.8	(21)	0.0	(0)	0.0	(0)
1,2,3,7,8,9-HxCDD	2.78	(24)	0.0	(0)	0.0	(0)
1,2,3,4,6,7,8-HpCDD	9.52	(9)	14.7	(13)	57.3	(52)
OCDD	57.5	(8)	0.0	(0)	571	(79)
2,3,7,8-TCDF	0.613	(33)	0.346	(18)	0.0	(0)
2,3,4,7,8-PeCDF	4.42	(46)	0.0	(0)	0.0	(0)
1,2,3,6,7,8-HxCDF	4.49	(78)	0.0	(0)	0.0	(0)

[a] CVs (coefficients of variation) are with respect to the estimated national average concentration.

Orban and Lordo (1989) show that the surrogate variance components are linearly related to the variance components σ_γ^2, σ_m^2, and σ_e^2. The variance component estimates are obtained by solving a set of simultaneous linear equations.

Table C-1 lists the estimated standard deviations and the corresponding coefficients of variation (percent of national average) for each of the three random effects. For five of the nine chemicals the sampling standard deviations were estimated to be zero. The formulas for estimating variance components can produce negative estimates. These are replaced by zero. This occurs frequently when measurement variation is large relative to the amount of sampling variation that affects the composite concentrations. However, it does not mean that there is no sampling variation. In fact, of the four chemicals with positive estimates of sampling standard deviations, the sampling errors were found to be quite large.

Two chemicals (1,2,3,4,6,7,8-HpCDD and 2,3,7,8-TCDF) had positive estimates of standard deviations due to random selection of MSAs. The estimates were 13 and 18 percent of their respective national average concentrations. Also, three chemicals (1,2,3,7,8-PeCDD, 1,2,3,4,6,7,8--HpCDD, and OCDD) had positive estimates of standard deviations due to random sampling of individuals. These estimates were between 52 and 79 percent of the national average concentrations.

The total sampling standard deviation due to both sampling of MSAs and individuals can be estimated by

$$\sigma_{samp.} = (\sigma_m^2 + \sigma_e^2)^{1/2} .$$

For example, the total sampling standard deviation for 1,2,3,4,6,7,8-HpCDD is estimated to be

$$59.2 = [(14.7)^2 + (57.3)^2]^{1/2} .$$

On a relative basis this is 54% of the national average concentration of 1,2,3,4,6,7,8-HpCDD.

The statistical uncertainty of the variance component estimates is difficult to establish

because of the approximate nature of the estimation procedure and the relatively small number of composite samples analyzed. Also, the priorities for creating the sampling and compositing designs were aimed primarily at producing accurate estimates of the national average concentrations and determining if there are significant demographic and geographic effects on the average concentrations. To obtain more precise estimates of sampling standard deviations it would be necessary to increase the number of composites or individual specimens that are analyzed and to revise the compositing design objectives.

APPENDIX D

PLOTS OF ESTIMATED CONCENTRATIONS VERSUS SPIKED LEVEL WITH TOLERANCE BOUNDS FOR FY87 NHATS QC SAMPLES

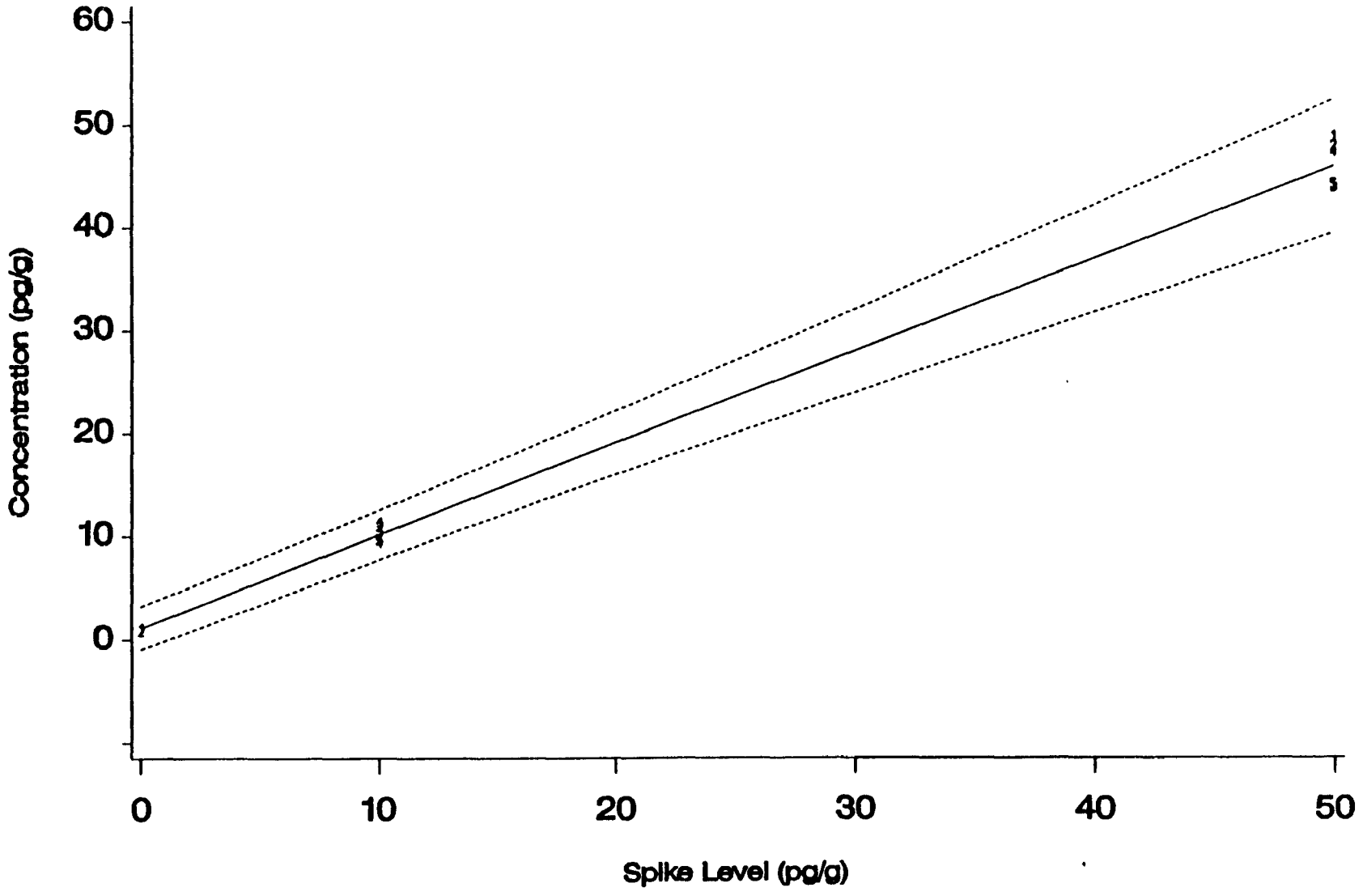

Figure D-1. Plot of Estimated Concentration for Compound 2,3,7,8-TCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

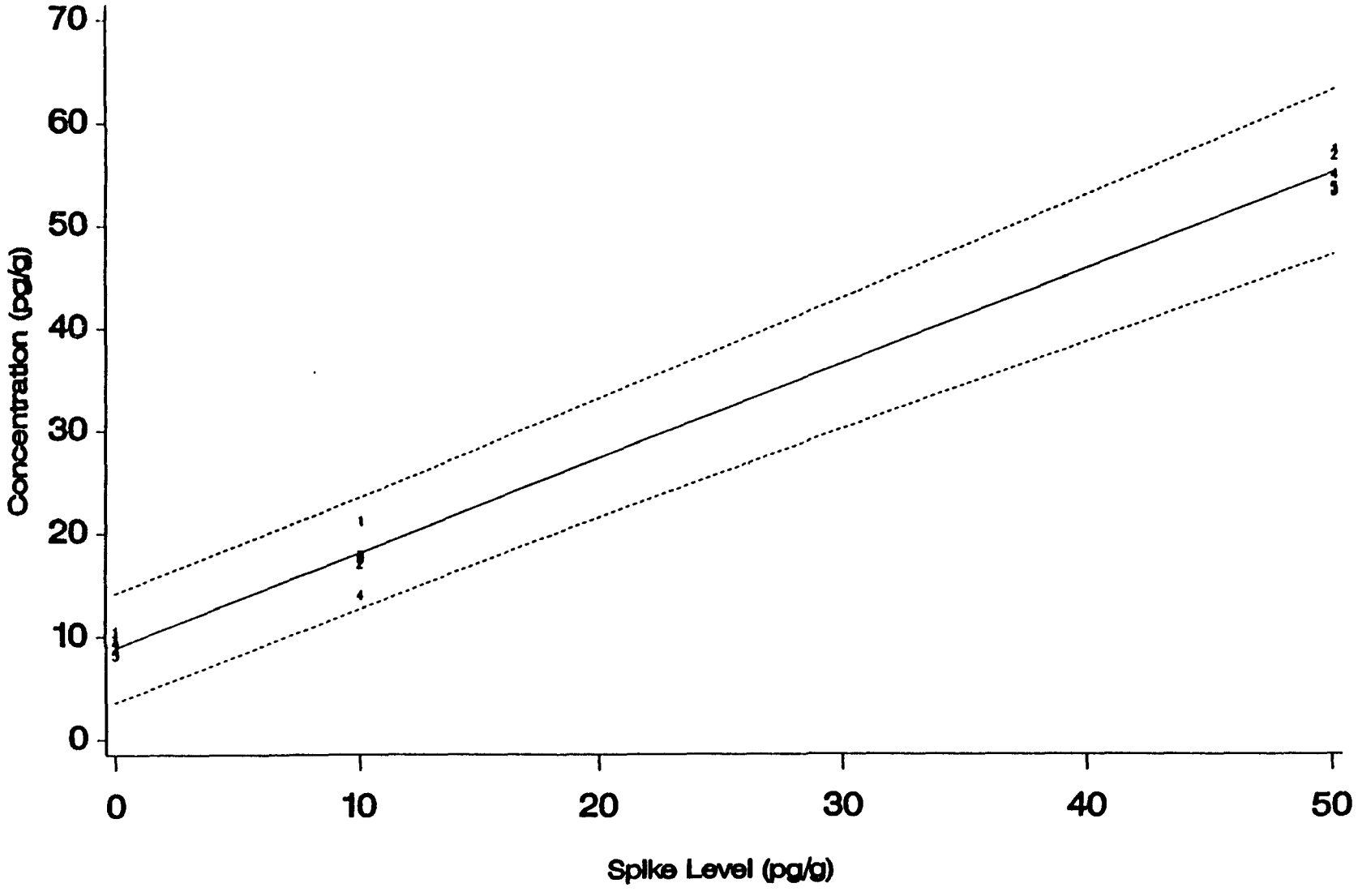

Figure D-2. Plot of Estimated Concentration for Compound 2,3,7,8-TCDD Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

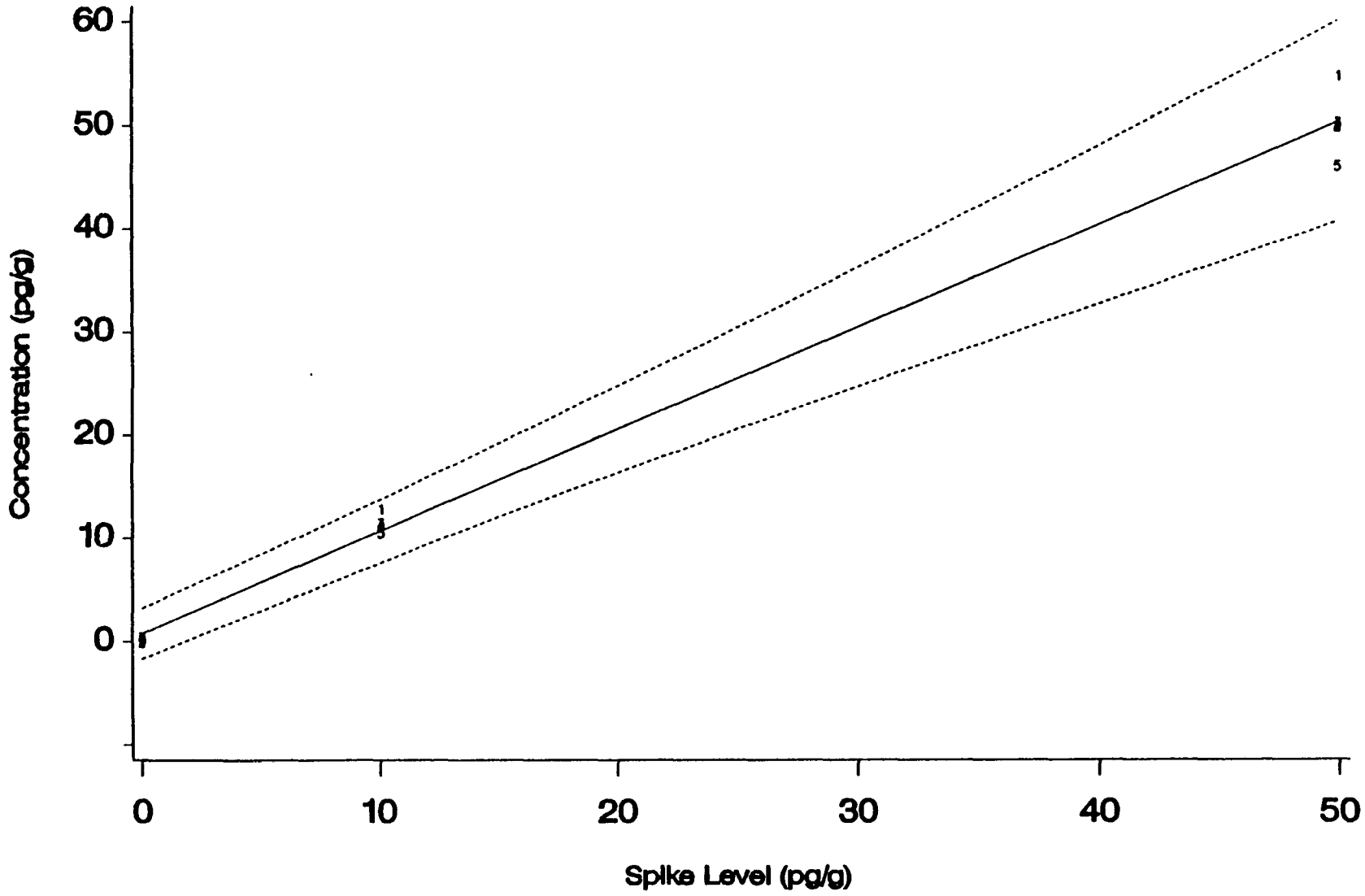

Figure D-3. Plot of Estimated Concentration for Compound 1,2,3,7,8-PeCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

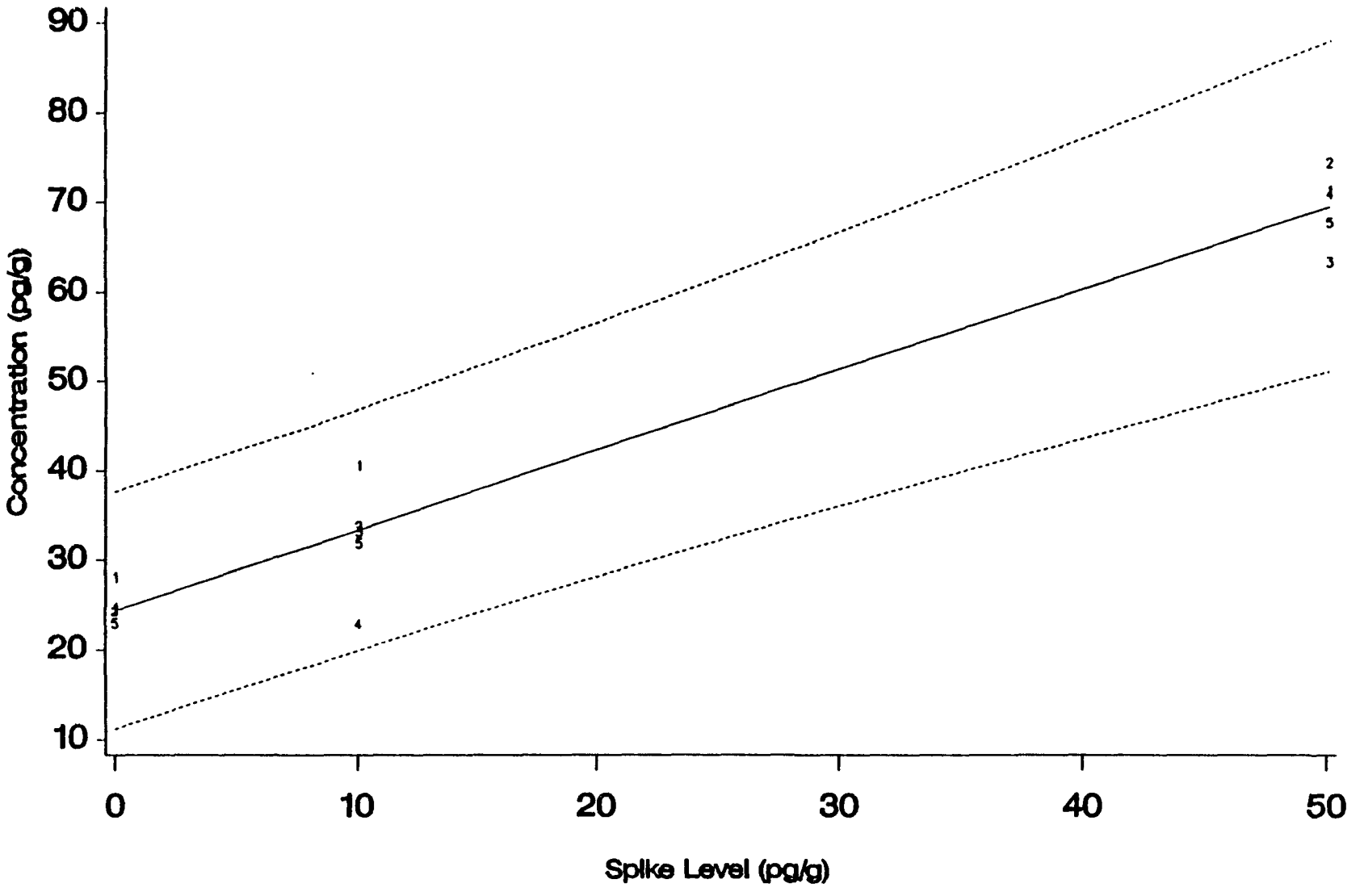

Figure D-4. Plot of Estimated Concentration for Compound 2,3,4,7,8-PeCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

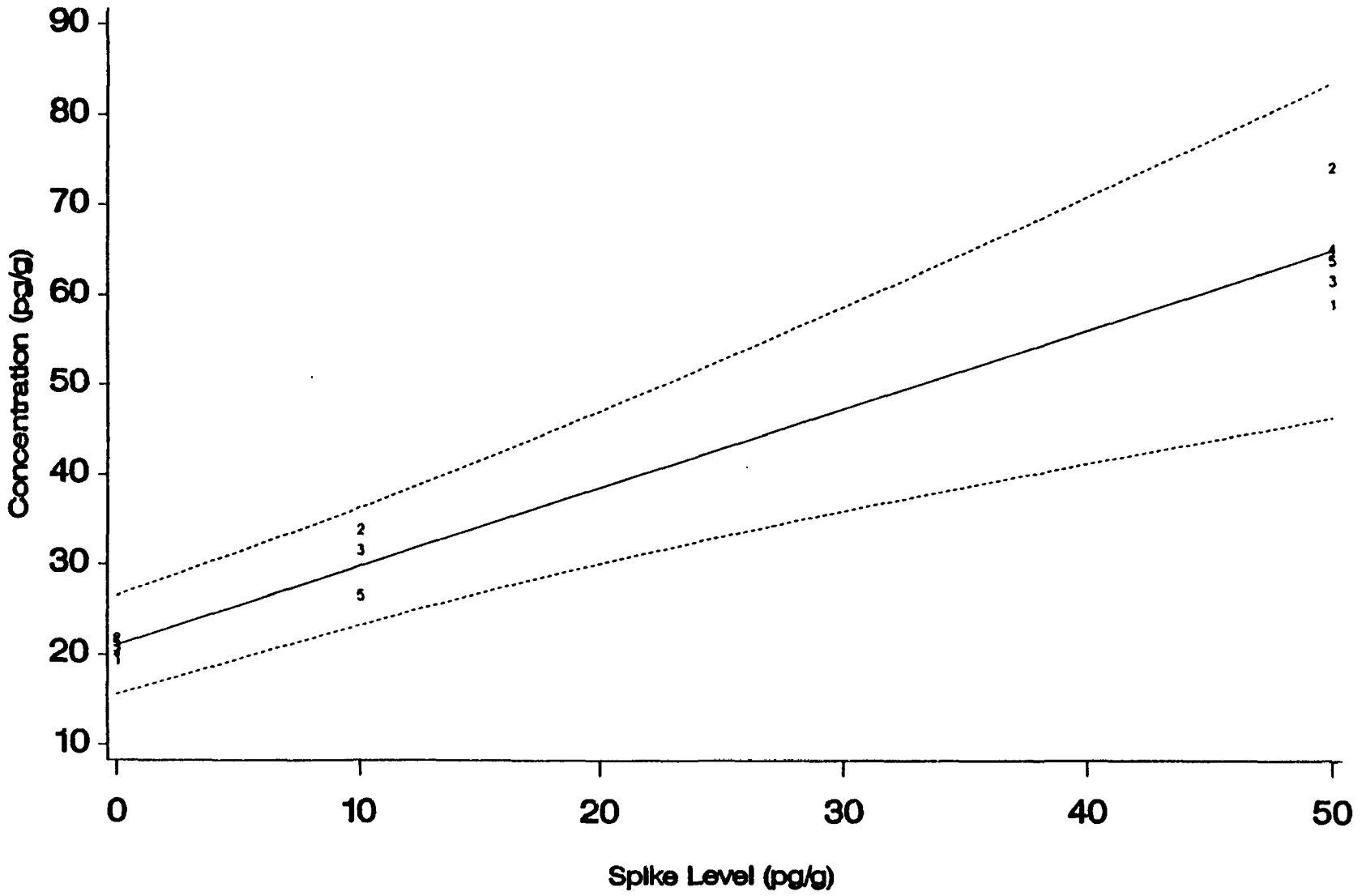

Figure D-5. Plot of Estimated Concentration for Compound 1,2,3,7,8-PeCDD Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

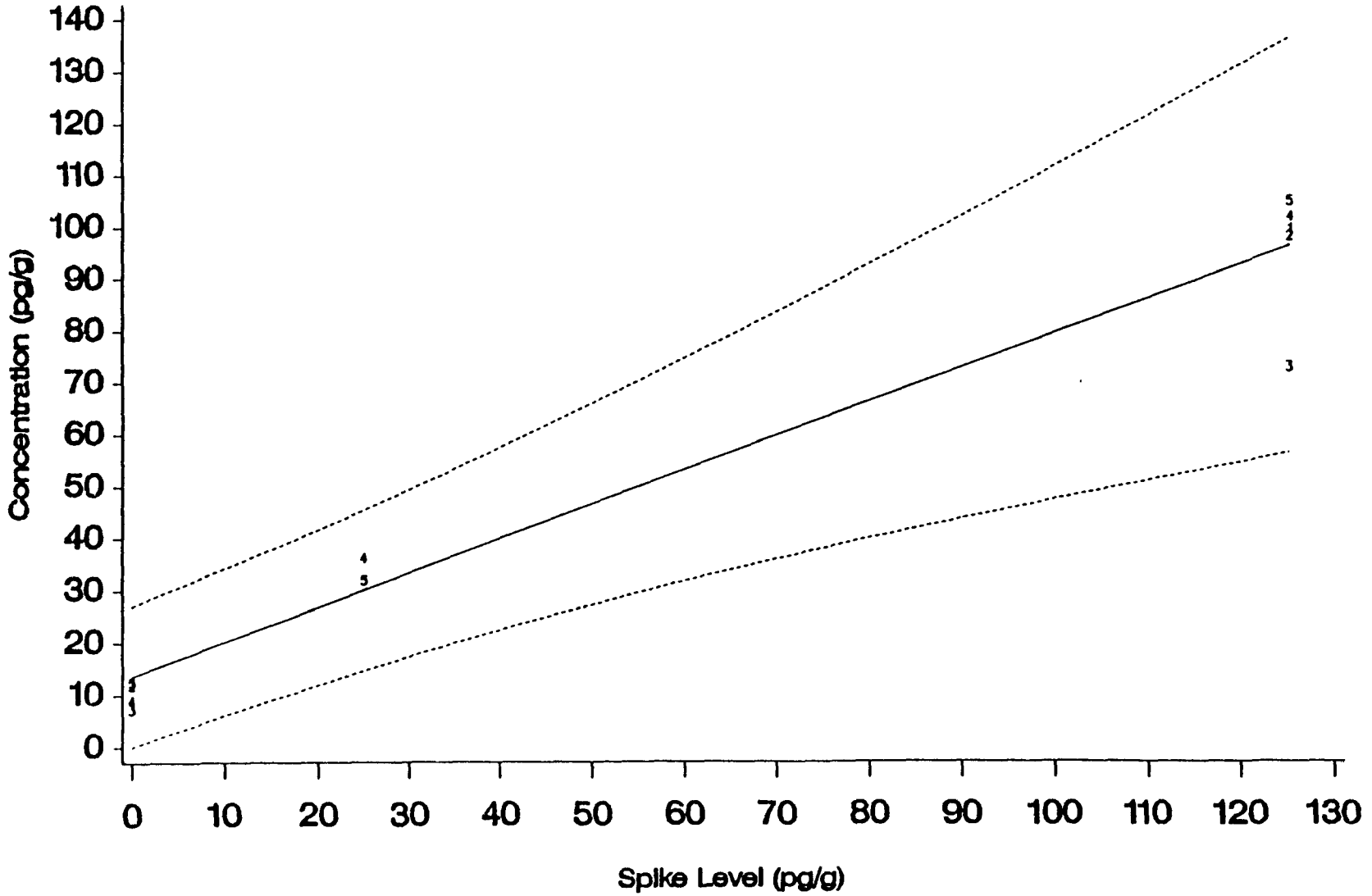

Figure D-6. Plot of Estimated Concentration for Compound 1,2,3,6,7,8-HxCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

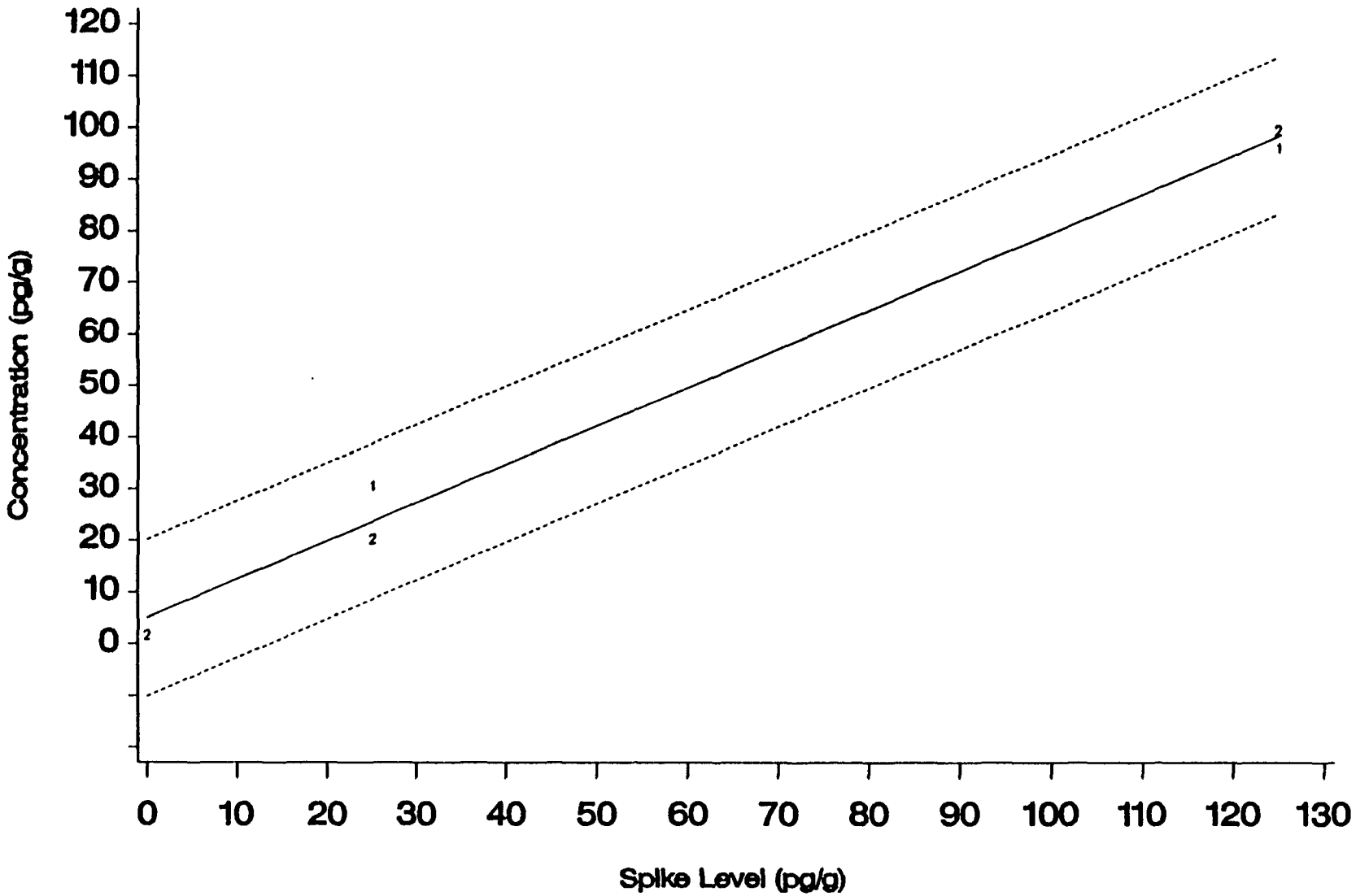

Figure D-7. Plot of Estimated Concentration for Compound 2,3,4,6,7,8-HxCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

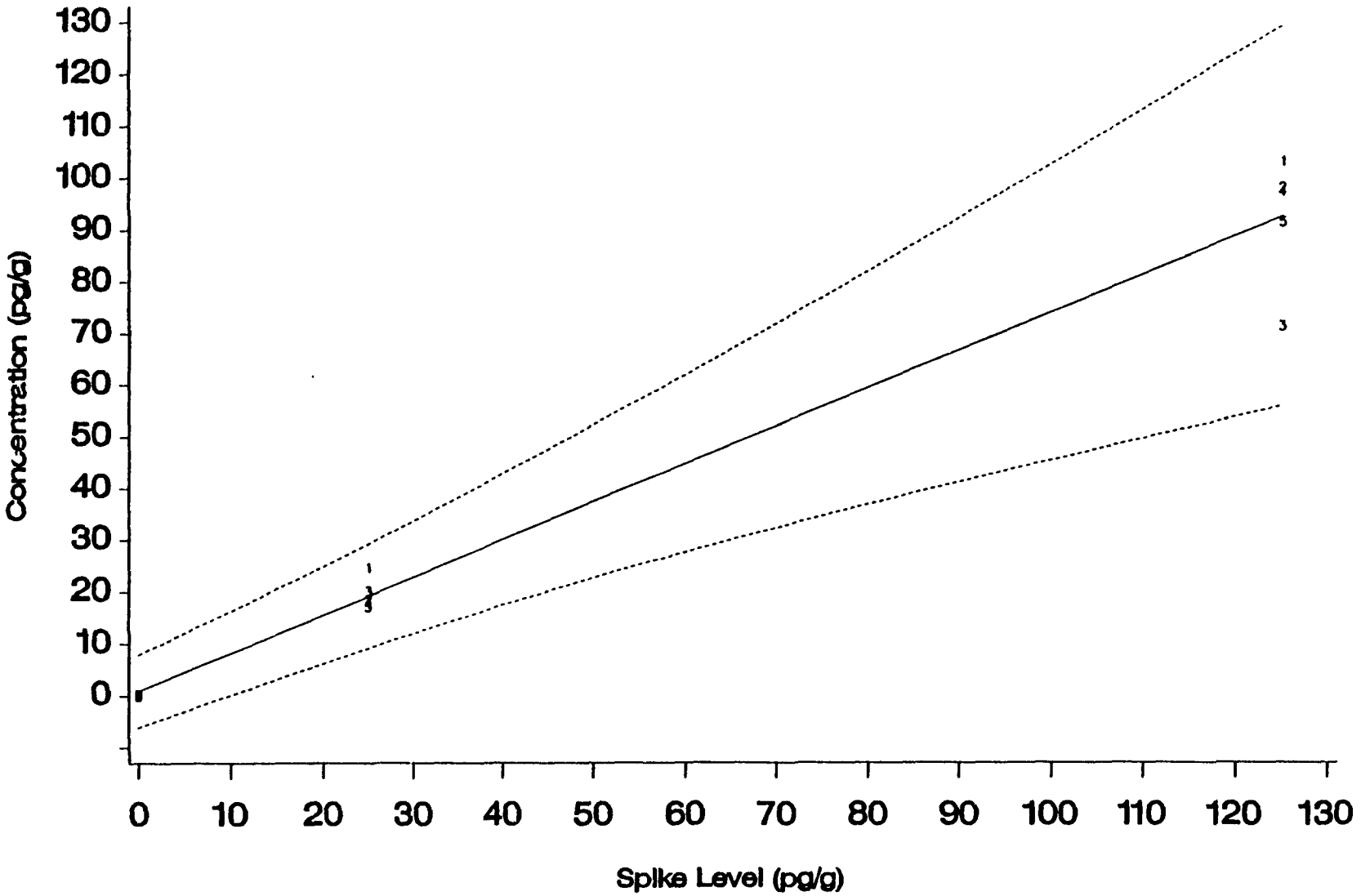

Figure D-8. Plot of Estimated Concentration for Compound 1,2,3,7,8,9-HxCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

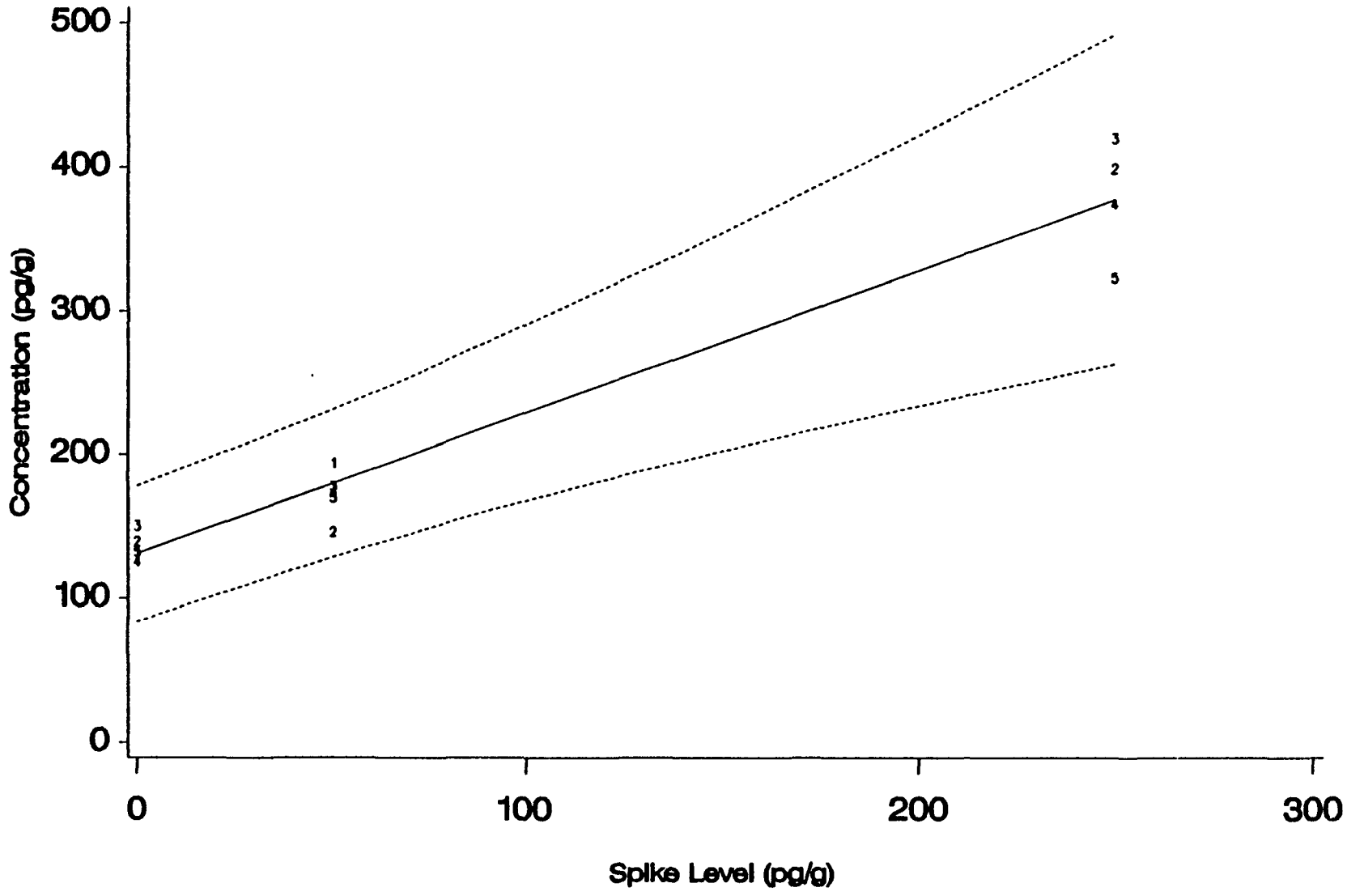

Figure D-9. Plot of Estimated Concentration for Compound 1,2,3,4,7,8/1,2,3,4,6,7,8-HXCDD Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

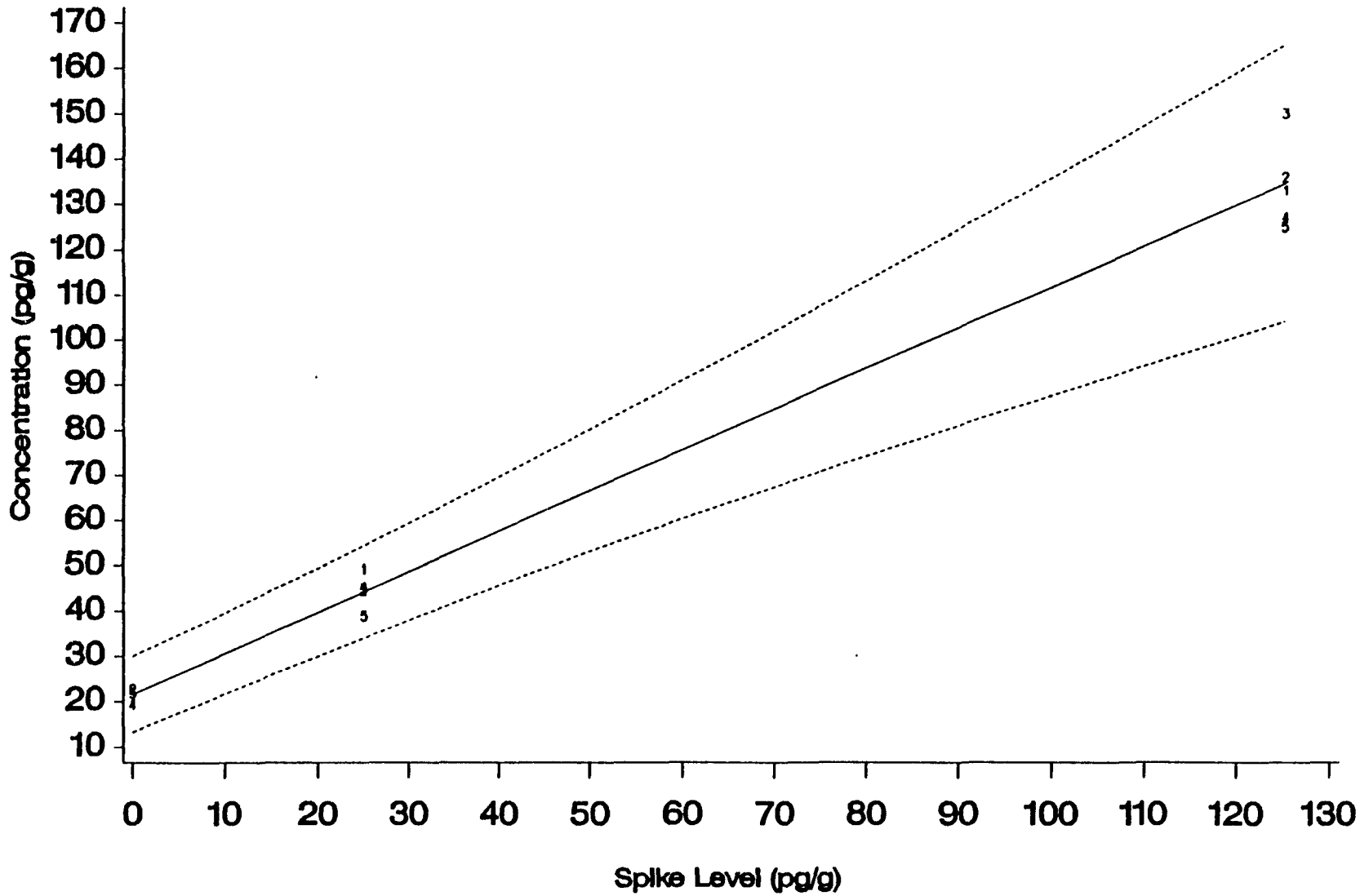

Figure D-10. Plot of Estimated Concentration for Compound 1,2,3,7,8,9-HxCDD Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

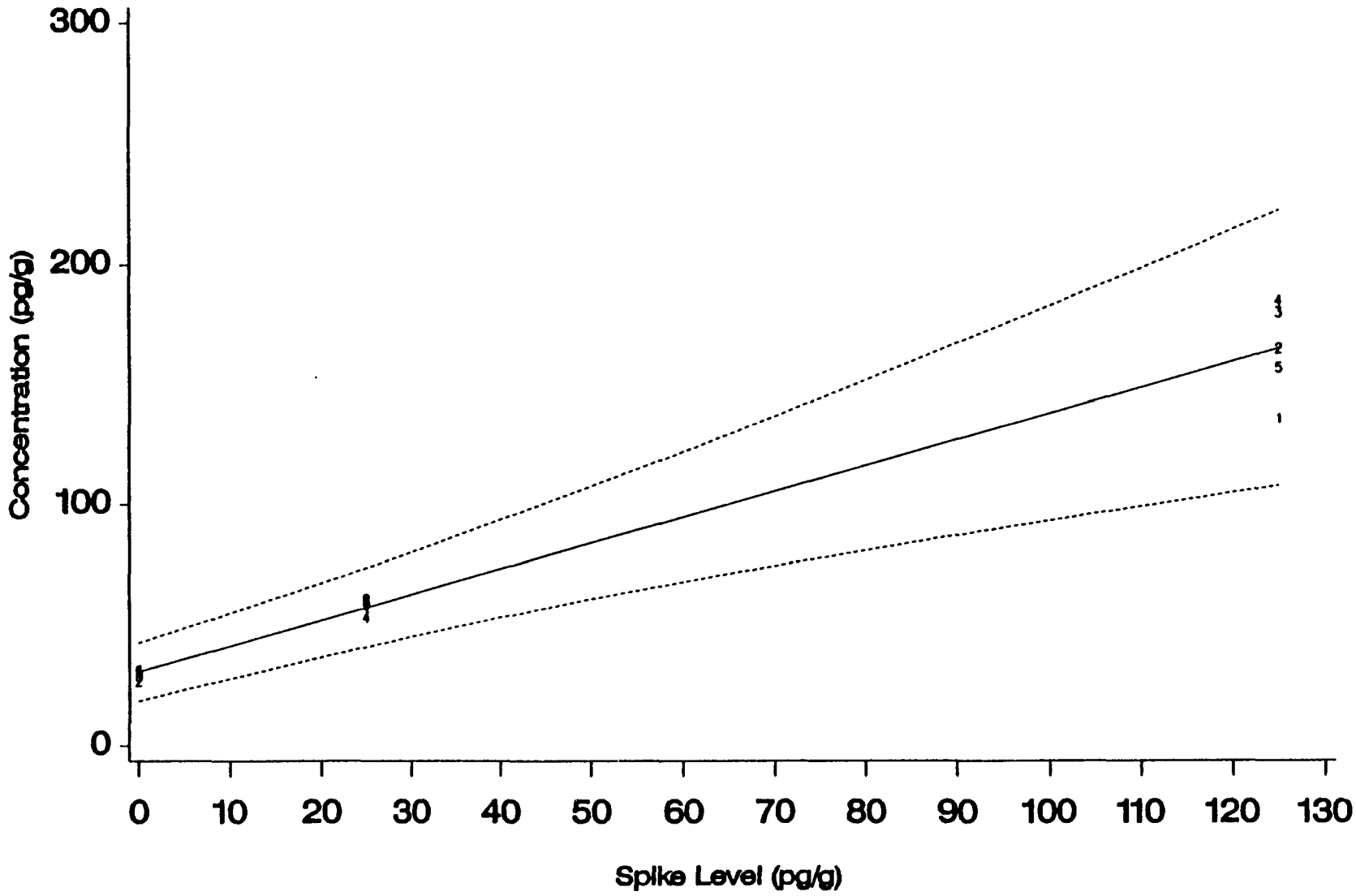

Figure D-11. Plot of Estimated Concentration for Compound 1,2,3,4,6,7,8-HPCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

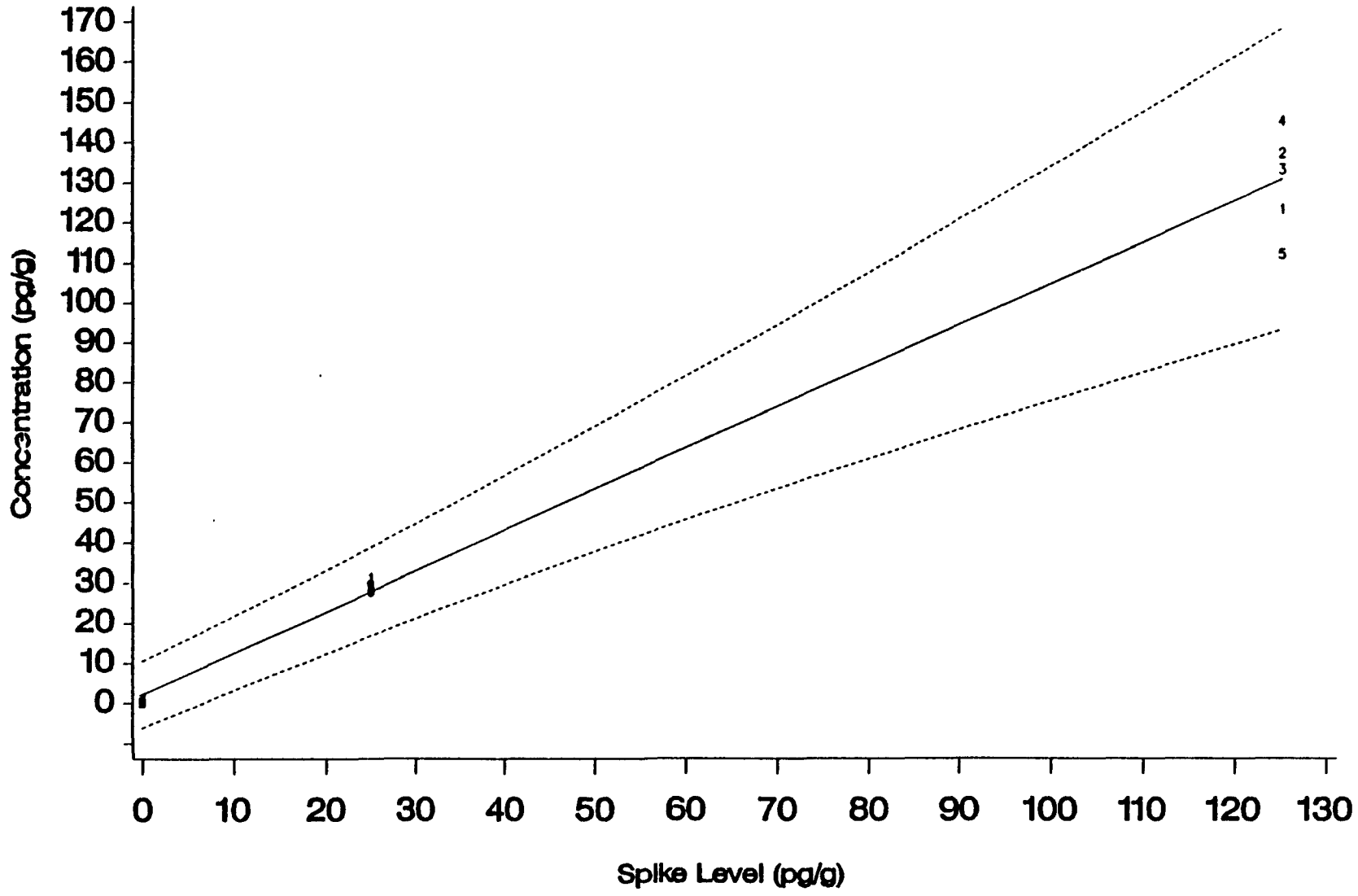

Figure D-12. Plot of Estimated Concentration for Compound 1,2,3,4,7,8,9-HpCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

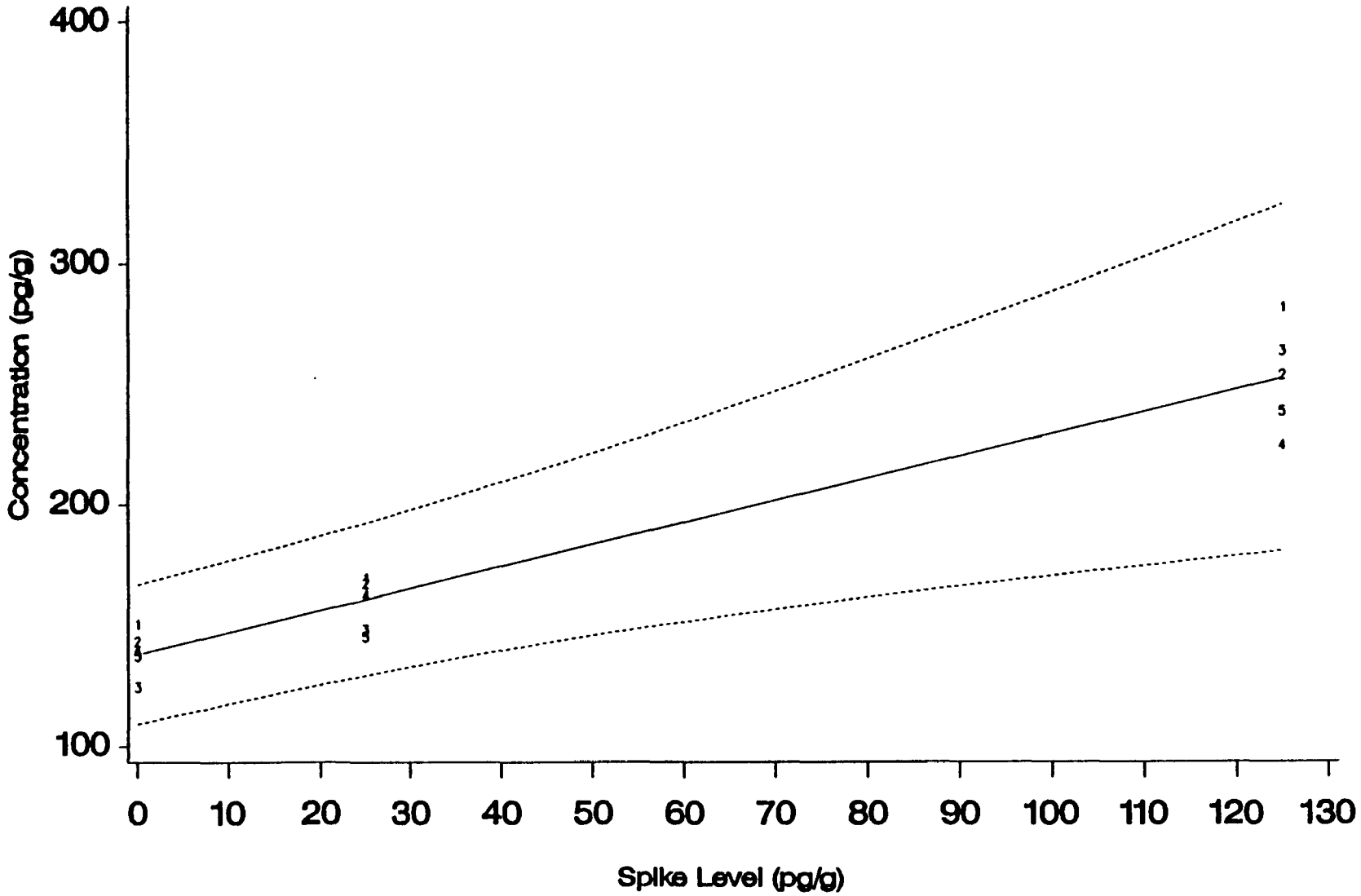

Figure D-13. Plot of Estimated Concentration for Compound 1,2,3,4,6,7,8-HpCDD Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

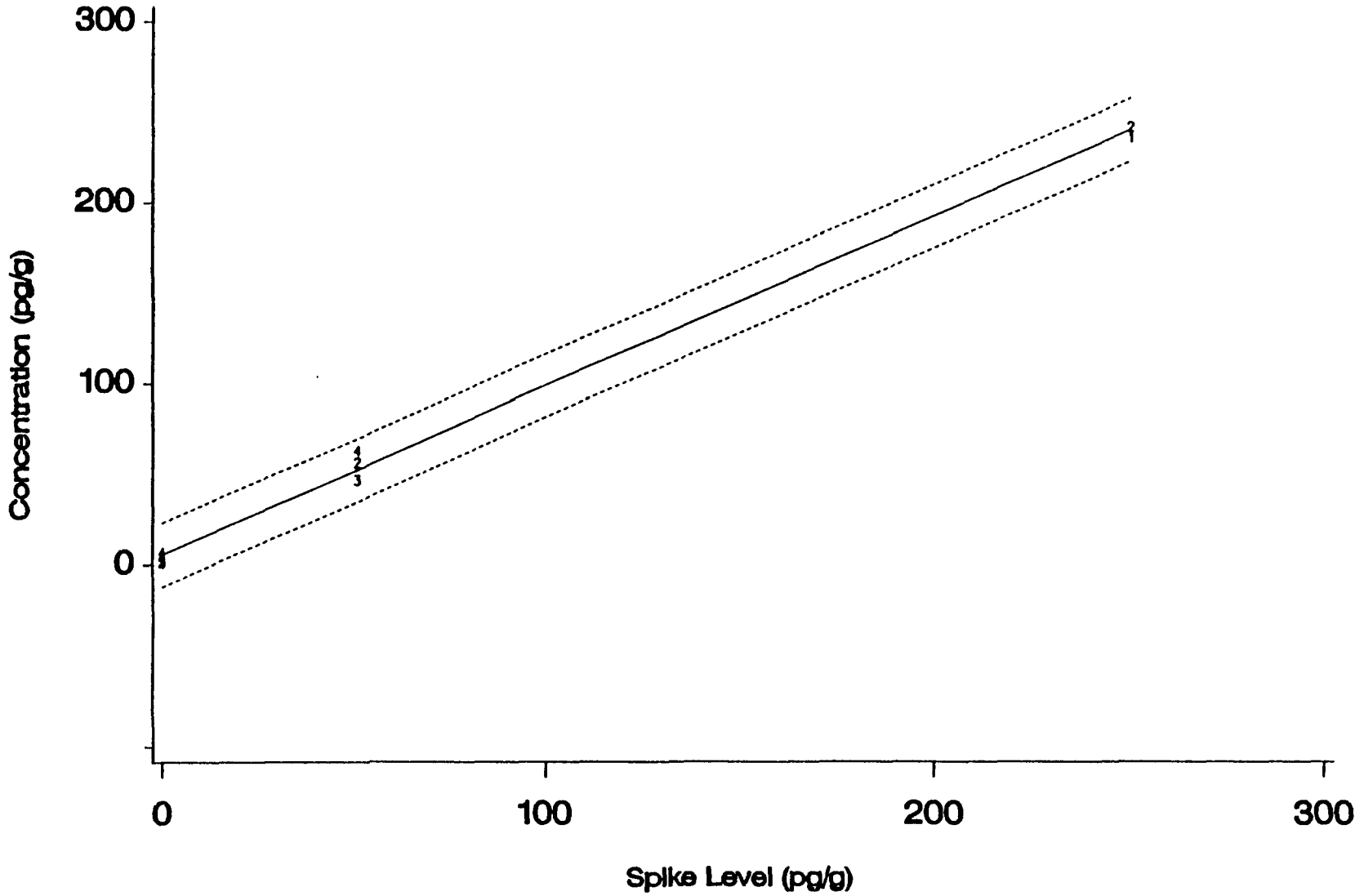

Figure D-14. Plot of Estimated Concentration for Compound 1,2,3,4,6,7,8,9-OCDF Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

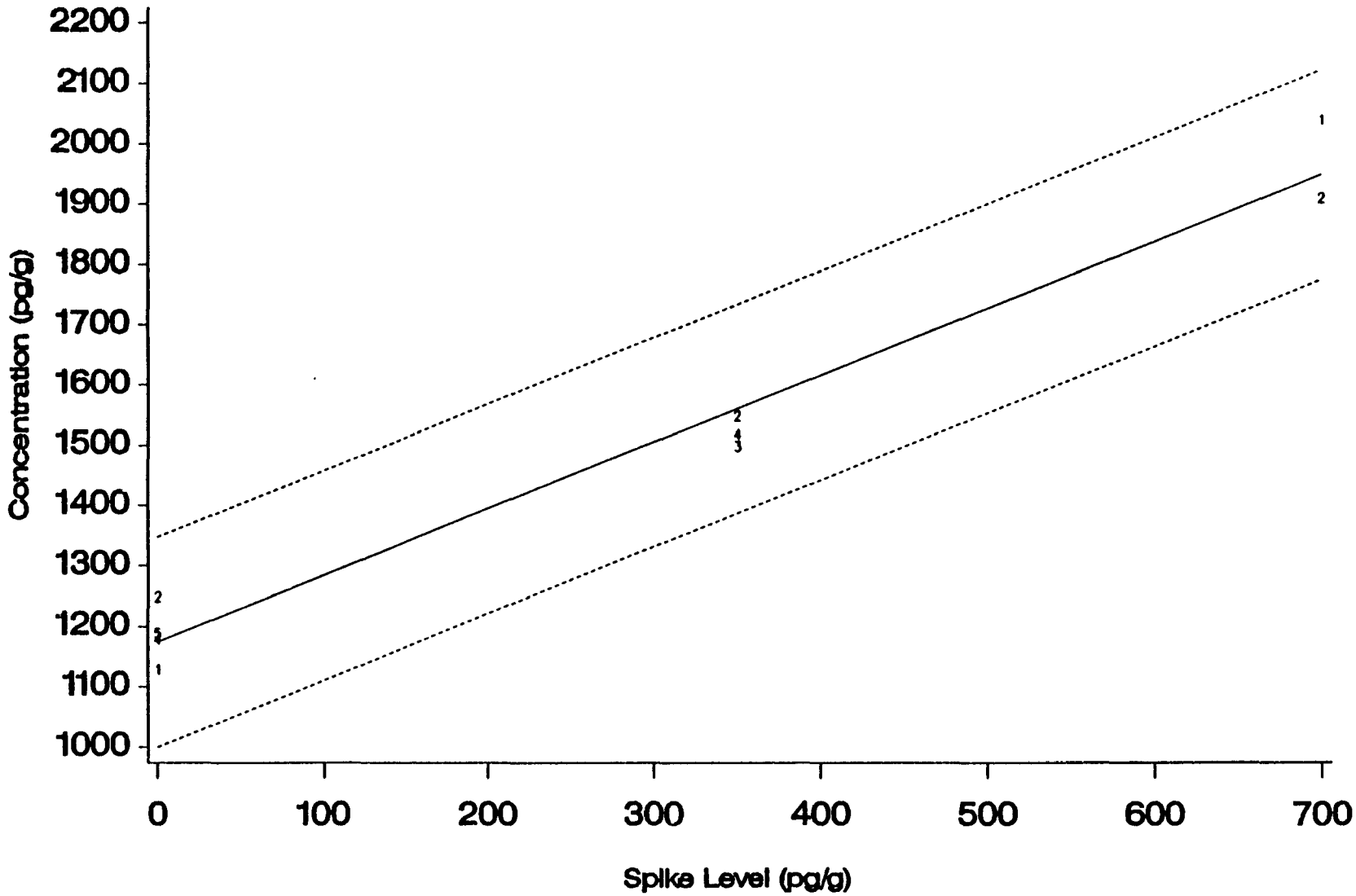

Figure D-15. Plot of Estimated Concentration for Compound 1,2,3,4,6,7,8,9-OCDD Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

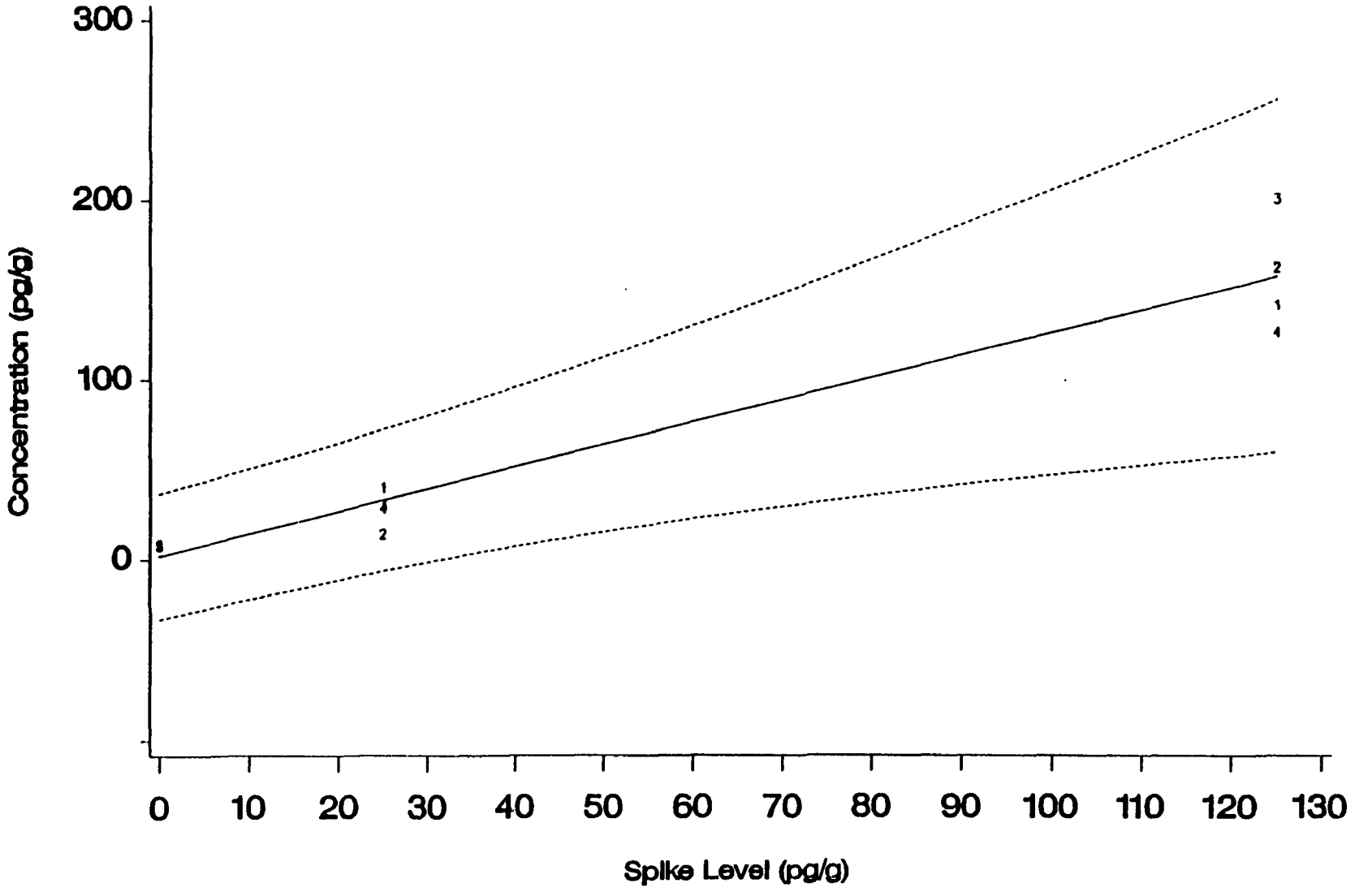

Figure D-16. Plot of Estimated Concentration for Compound 1,2,3,4,7,8-HxCDD Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

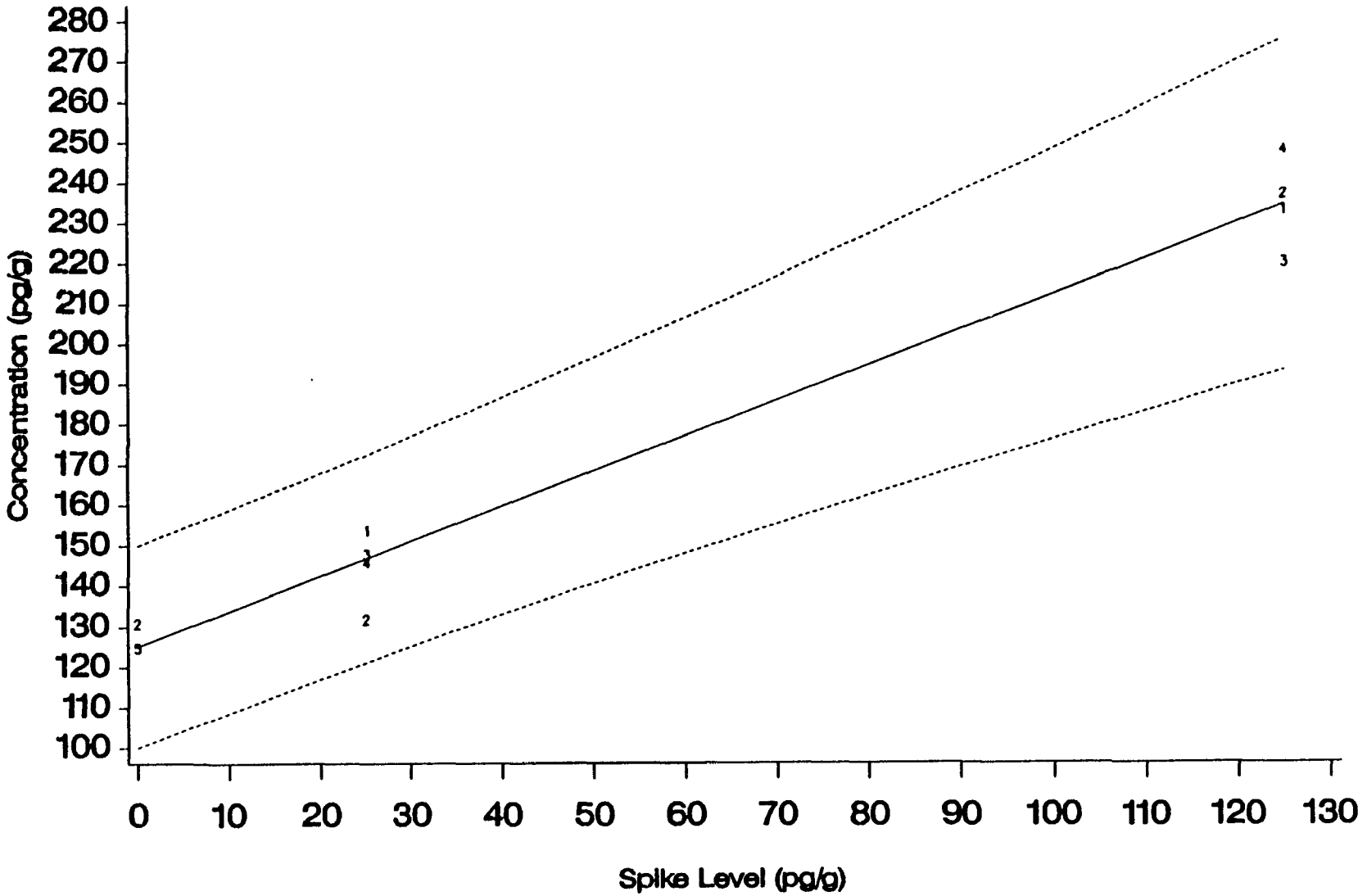

Figure D-17. Plot of Estimated Concentration for Compound 1,2,3,6,7,8-HxCDD Versus Spiked Level with Tolerance Bounds for FY87 NHATS QC Samples.

APPENDIX E

SUPPLEMENTARY DESCRIPTIVE STATISTICS FOR FY87 NHATS PCDDs AND PCDFs

SUPPLEMENTARY DESCRIPTIVE STATISTICS FOR FY87 NHATS PCDDs and PCDFs

Tables E-1 and E-2 give the average measured concentration, standard error, and number of unrestricted measurements for each of the marginal populations defined by the different levels of the four analysis factors. Composites classified as mixed race group or mixed sex group are composites which contain specimens from both white and non-white donors or both male and female donors, respectively. All specimens in each composite are from the same census region and same age group. For example, there were 36 unrestricted measurements of 2,3,7,8-TCDD and the average concentration was 6.60 pg/g. The average was 1.74 pg/g for the five composites containing specimens from the youngest age group (0 to 14 years), 4.33 pg/g from the 12 composites in the middle age group (15 to 44 years), and 9.31 from the 19 composites in the oldest age group (45+ years). The standard errors are all less than 0.6 pg/g. This suggests that the differences between the age group averages are statistically significant. However, the reported significance levels in Chapter 8 were determined by a more rigorous statistical approach.

Table E-1. Composite Averages (pg/g), Standard Errors (pg/g), and Numbers of Composite Samples by Demographic Subpopulation for FY87 NHATS PCDDs

Analyte	Entire nation	Census Region				Age group (years)			Race group			Sex group		
		NE	NC	S	W	0-14	15-44	45 +	White	Mixed	Non-White	Male	Mixed	Female
Population percentages[a]	100	22	26	33	19	23	46	31	83	-	17	49	-	51
2,3,7,8-TCDD	6.60[b]	5.52	7.98	6.86	5.82	1.74	4.33	9.31	5.00	6.92	-	6.02	5.52	8.47
	0.590[c]	1.03	1.38	1.12	1.15	0.407	0.282	0.554	1.41	0.644	-	0.950	0.721	1.25
	36[d]	9	9	11	7	5	12	19	6	30	0	13	12	11
1,2,3,7,8-PeCDD	11.8	10.6	13.9	11.6	8.77	3.43	9.48	18.1	10.5	12.1	-	12.8	9.34	14.9
	1.11	2.64	1.85	2.03	2.00	0.504	0.307	0.999	2.39	1.26	-	2.07	1.24	2.63
	35	8	11	12	4	8	12	15	7	28	0	12	15	8
1,2,3,4,7,8/ 1,2,3,6,7,8-HxCDD	82.2	75.5	96.1	73.8	79.7	26.5	70.8	121	64.6	85.2	-	88.6	59.4	94.8
	6.58	15.8	12.8	9.73	18.1	2.77	2.28	5.47	21.1	6.82	-	11.2	9.55	11.3
	41	7	13	14	7	10	13	18	6	35	0	15	12	14
1,2,3,7,8,9-HxCDD	12.8	10.4	13.5	12.6	15.2	6.20	10.7	17.3	12.6	12.8	-	11.9	11.2	15.2
	0.801	1.70	1.55	1.46	1.30	1.02	0.298	0.811	2.49	0.851	-	1.51	1.26	1.23
	39	9	12	12	6	7	15	17	6	33	0	12	14	13
1,2,3,4,6,7,8-HpCDD	119	131	130	110	110	46.8	99.8	175	108	121	-	125	96.0	137
	8.75	19.7	15.3	14.1	27.9	4.78	5.13	6.24	35.1	8.94	-	12.9	16.7	14.7
	42	7	12	16	7	10	14	18	5	37	0	15	14	13
1,2,3,4,6,7,8,9-OCDD	806	773	856	716	951	247	726	1150	848	800	-	860	609	925
	74.0	170	134	133	194	18.9	59.5	61.8	247	78.8	-	135	116	123
	32	7	11	10	4	8	9	15	4	28	0	10	10	12
1,2,3,4,7,8-HxCDD	10.0	8.81	16.9	4.01	11.5	2.87	6.08	16.9	8.36	10.3	-	6.38	3.11	14.1
	2.72	0.510	7.67	0.721	4.63	0.381	1.07	5.21	5.25	3.07	-	1.36	-	5.06
	16	2	5	6	3	4	5	7	2	14	0	7	1	8
1,2,3,6,7,8-HxCDD	73.8	90.4	96.5	49.6	73.2	24.8	60.8	111	66.9	74.8	-	74.3	19.7	80.1
	9.72	23.6	19.3	10.2	27.0	4.03	3.68	6.92	47.2	9.98	-	15.4	-	13.0
	16	2	5	6	3	4	5	7	2	14	0	7	1	8

[a] Population percentages based on 1980 Census.
[b] Average concentration (pg/g).
[c] Standard error.
[d] Total number of unrestricted samples.

Table E-2. Composite Averages (pg/g), Standard Errors (pg/g), and Numbers of Composite Samples by Demographic Subpopulation for FY87 NHATS PCDFs

Analyte	Entire nation	Census Region				Age group (years)			Race group			Sex group			
		NE	NC	S	W	0-14	15-44	45 +	White	Mixed	Non-White	Male	Mixed	Female
Population percentages[a]	100	22	26	33	19	23	46	31	83	-	17	49	-	51
2,3,7,8-TCDF	2.05[b]	1.91	2.39	1.66	2.52	2.03	1.34	2.50	2.48	1.94	-	2.17	1.94	2.07
	0.141[c]	0.278	0.302	0.184	0.354	0.240	0.098	0.202	0.335	0.150	-	0.226	0.235	0.305
	33[d]	8	8	11	6	9	9	15	7	26	0	12	13	8
1,2,3,7,8-PeCDF	0.323	0.238	0.356	0.381	0.239	0.263	0.244	0.444	0.207	0.342	-	0.306	0.180	0.499
	0.045	0.044	0.097	0.090	0.052	0.072	0.042	0.096	0.060	0.050	-	0.047	0.026	0.120
	43	8	14	14	7	11	16	16	6	37	0	16	14	13
2,3,4,7,8-PeCDF	11.8	12.2	13.4	11.7	7.22	2.75	8.71	17.7	10.2	12.1	-	11.7	8.85	15.1
	1.25	3.31	2.43	2.00	1.97	0.639	0.543	1.74	2.79	1.40	-	1.99	1.28	2.92
	39	7	13	14	5	7	14	18	6	33	0	16	12	11
1,2,3,4,7,8-HxCDF	8.20	7.09	7.38	6.73	10.4	1.56	7.13	12.2	7.05	8.78	-	3.20	8.27	11.4
	1.48	3.11	2.54	-	3.33	-	0.817	2.80	3.10	1.77	-	1.64	1.35	2.87
	9	3	2	1	3	1	5	3	3	6	0	2	4	3
1,2,3,6,7,8-HxCDF	5.73	5.53	6.60	4.90	5.73	1.95	4.63	10.3	5.36	5.81	-	5.72	3.52	8.37
	0.611	1.88	1.17	0.896	1.35	0.176	0.376	0.656	1.45	0.682	-	0.752	0.672	1.34
	37	6	13	12	6	10	15	12	6	31	0	13	13	11
2,3,4,6,7,8-HxCDF	0.707	1.27	0.432	0.579	0.735	0.799	0.281	0.724	2.16	0.514	-	0.783	0.759	0.400
	0.155	0.532	0.145	0.155	-	0.193	0.001	0.310	0.335	0.087	-	0.189	0.364	0.205
	17	4	5	7	1	8	2	7	2	15	0	8	6	3
1,2,3,7,8,9-HxCDF	0.454	0.636	0.415	0.432	0.407	0.604	0.370	0.430	0.559	0.434	-	0.551	0.330	0.491
	0.052	0.236	0.072	0.069	0.105	0.174	0.043	0.058	0.241	0.044	-	0.129	0.034	0.080
	45	7	14	16	8	11	14	20	7	38	0	15	16	14
1,2,3,4,6,7,8-HpCDF	15.9	11.9	21.6	16.6	11.0	9.98	15.9	18.5	10.7	17.4	-	20.2	12.6	14.6
	1.46	4.19	1.95	2.52	2.34	3.51	1.88	2.27	2.98	1.56	-	1.98	2.53	2.50
	27	4	8	8	7	5	10	12	6	21	0	10	10	7
1,2,3,4,7,8,9-HpCDF	0.741	0.752	0.812	0.666	0.755	0.634	0.726	0.811	0.692	0.751	-	0.660	0.712	0.863
	0.043	0.138	0.086	0.062	0.079	0.055	0.048	0.086	0.058	0.051	-	0.045	0.058	0.110
	46	8	14	16	8	11	15	20	8	38	0	15	17	14
1,2,3,4,6,7,8,9-OCDF	2.05	1.05	1.20	3.28	0.908	2.35	2.89	1.33	1.05	2.26	-	1.33	3.59	1.06
	0.604	0.313	0.203	1.31	0.003	0.996	2.07	0.227	0.407	0.721	-	0.175	1.64	0.197
	23	5	6	10	2	7	6	10	4	19	0	9	8	6

[a] Population percentages based on 1980 Census.
[b] Average concentration (pg/g).
[c] Standard error.
[d] Total number of unrestricted samples.

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J.S. Stanley (1) and J. Orban (2)

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1) Midwest Research Institute, 425 Volker Boulevard, Kansas City, Missouri 64110
2) Battelle Columbus Division, 505 King Avenue, Columbus, Ohio 43201

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16. Abstract (Limit: 200 words)

Population estimates of the average levels of polychlorinated dibenzo-*p*-dioxins (dioxins or PCDD) and polychlorinated dibenzofurans (furans or PCDF) were established using 865 human adipose tissue specimens collected in Fiscal Year 1987 through the U.S. Environmental Protection Agency's National Human Adipose Tissue Survey (NHATS). The specimens were composited into 48 unique samples prior to chemical analysis. Estimates of the national average concentrations of levels were made among subpopulations defined by the donors' geographic adipose tissue of the U.S. population was 5.38 pg/g (±0.32); however, the levels increase from 1.98 pg/g (±0.81) in children under 14 years of age to 9.40 pg/g (±0.38) in adults over 45 years old. Significant age effects were determined for all nine of the compounds which were present at quantifiable levels in greater than 90% of all samples. Statistically significant differences based on geographic regions were determined for estimated levels of 2,3,4,7,8-PCCDF with the highest levels in the northeast and the lowest levels in the west. There were no significant differences in the estimated levels from different sexes or race groups for any of the target analytes.

17. Document Analysis a. Descriptors

Polychlorinated dibenzo-*p*-dioxins, polychlorinated dibenzofurans, PCDD, PCDF, 2,3,7,8-TCDD, Human Adipose Tissue, National Human Adipose Tissue Survey (NHATS), Fiscal Year 1987, FY 1987.

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