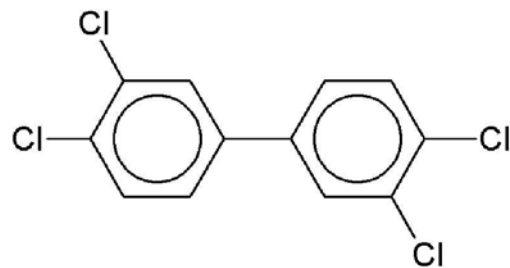
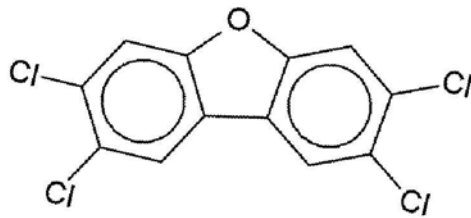
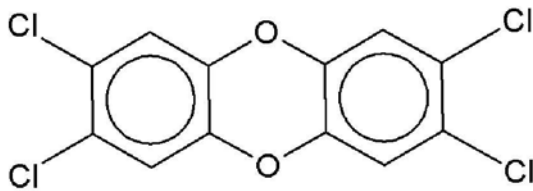




# 2001 National Sewage Sludge Survey Report

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U.S. Environmental Protection Agency  
Office of Water (4303T)  
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## **DISCLAIMER**

This EPA report was prepared by CSC under EPA direction. Questions regarding the 2001 National Sewage Sludge Survey, the survey results, or future plans may be directed to the EPA Biosolids Coordinator:

Rick Stevens  
U.S. Environmental Protection Agency  
Office of Water  
Office of Science and Technology  
Health and Ecological Criteria Division  
Ecological and Health Protection Branch  
1200 Pennsylvania Avenue, NW  
Mail Code: 4304T  
Washington, DC 20460  
202-566-1135  
stevens.rick@epa.gov

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## EXECUTIVE SUMMARY

This report presents the results of the U.S. Environmental Protection Agency's (EPA's) 2001 National Sewage Sludge Survey (NSSS). The survey was conducted jointly by the Health and Ecological Criteria Division (HECD) and the Engineering and Analysis Division (EAD) within the EPA Office of Water's Office of Science and Technology. The objective of the survey was to obtain unbiased national estimates of dioxin and dioxin-like compounds in sewage sludge to enable EPA to perform a multi-pathway exposure assessment and risk assessment of the disposal of sewage sludge through land application.

A total of 113 samples of sludge were collected from 94 publicly owned treatment works (POTWs) in 32 states and the District of Columbia during a seven-week period in February and March 2001. Eighty-nine of these POTWs had a single system for treating and processing their sludge material. Therefore, one final sludge product sample was collected from each of these POTWs. Five of the POTWs had two systems for treating their sludge material; therefore, two final sludge product samples were collected from each of these five facilities (one sample per treatment process). For quality control purposes, field duplicate samples were collected from 15% of the facilities (14 POTWs) sampled. All 113 samples were analyzed for a suite of polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and polychlorinated biphenyls (PCBs) using state-of-the-art EPA analytical methods and project-specific modifications to ensure that the lowest consistent practical sensitivity was achieved. The suite of analytes included the seventeen 2,3,7,8-substituted PCDDs/PCDFs of toxicological concern and the twelve PCB congeners identified as "toxic" by the World Health Organization. The analytical results were reviewed to verify completeness and compliance with the method specifications and study requirements.

The results for each sludge sample were calculated in terms of the toxic equivalents of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD or "dioxin") represented by the various substituted chlorinated dioxins, furans, and PCBs that were present. For the purposes of this calculation, the concentration of any congener that was not detected in the sample was set to one-half the quantitation limit for that congener. This commonly used substitution scheme is conservative relative to protection of the environment, as it assumes that there may be some of the contaminant present below the level at which quantitative measurements can be made. Using this scheme, every sample will have a TEQ value, even if no contaminants were detected in the sample. The lowest possible TEQ value in this study would be 1.6 ng/kg of TCDD on a dry-weight basis.

Sample results ranged from 3 ng/kg to 718 ng/kg of TCDD. The vast majority of the samples had TEQ values in the range of 7 to 55 ng/kg. Only seven results over 100 ng/kg TEQ were reported, and two of those were for field duplicate samples collected as part of the quality assurance program for the survey.

## SECTION 1.0 INTRODUCTION

Section 405(d) of the Clean Water Act (CWA) requires that the U.S. Environmental Protection Agency (EPA) propose, solicit public comments, and promulgate standards for the use or disposal of sewage sludge. These standards are codified at 40 CFR Part 503 (Part 503 Standards). To date, EPA has accomplished this rulemaking activity in phases referred to as “rounds.”

Round One of the Part 503 Standards was promulgated on February 19, 1993 (58 FR 9248). The Round One standards established numerical limits for 10 metals in sewage sludge produced at publicly owned treatment works (POTWs) and managed through land application, surface disposal, and incineration. This rule also established an operational standard for sewage sludge incinerator emissions. These standards were based on a National Sewage Sludge Survey conducted by EPA in 1988–1989 (1989 NSSS) to obtain unbiased national estimates of the concentrations of more than 400 pollutants in sewage sludge collected from 174 wastewater treatment plants that practiced at least secondary wastewater treatment.

On December 23, 1999, EPA proposed Round Two of the Part 503 Standards (64 FR 72045). This proposal sought to establish a numerical standard for 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (dioxin) and dioxin-like compounds (furans) applicable to sewage sludge managed through land application (under Subpart B of the Part 503 rule). In addition, this proposed rule found that numerical standards or management practices were not necessary to protect public health from the presence of dioxins in sewage sludge managed by surface disposal or incineration. The proposal solicited public comments.

The list of pollutants in the 1989 NSSS included the seventeen 2,3,7,8-substituted congeners of chlorinated dibenzo-*p*-dioxins and chlorinated dibenzofurans. PCBs were determined as Aroclors, but no attempt was made to measure the concentrations of the 12 coplanar PCB congeners identified as “toxic” by the World Health Organization. In developing the Round Two rule, EPA relied on estimates of the concentrations of the dioxins and dibenzofurans in sewage sludge from the 1989 NSSS and a national estimate of the 12 coplanar PCB congeners concentrations obtained from a 1995 survey commissioned by the Association of Metropolitan Sewerage Agencies. These estimated concentrations were used as a source term in the risk assessment as the technical basis of the proposed numerical standard and in the proposed rule’s regulatory impact analysis to estimate the costs to the regulated community of complying with the rule. Following proposal of the Round Two rule, EPA received and considered a number of public comments, including comments that strongly urged EPA to conduct a new survey of dioxin and furan concentrations in sewage sludge. Commenters suggested that given the extensive efforts to reduce the sources of dioxins and furans from specific industries, the concentrations of dioxins and furans in sewage sludge at the start of the 21<sup>st</sup> century were likely to be lower than those concentrations found in sewage sludge in the 1989 NSSS. To address these comments, EPA conducted a second NSSS in 2001. The purpose of the 2001 NSSS was to obtain updated, unbiased national estimates of dioxins and dioxin-like compounds in sewage sludge managed by land application. This report describes the sampling and analysis aspects of EPA’s 2001 NSSS and summarizes the analytical results.

EPA developed two documents in preparation for this study: *Sampling Procedures for the 2001 National Sewage Sludge Survey* (Reference 8.1), which contains the study sampling protocol, and *Quality Assurance Project Plan for the 2001 National Sewage Sludge Survey* (Reference 8.2), which identifies study objectives and measurement quality objectives established for the collection and analysis of environmental samples during the study and serves as the study plan. These documents are available separately from EPA.

## SECTION 2.0 STUDY MANAGEMENT AND IMPLEMENTATION

The EPA Office of Science and Technology (OST) is responsible for proposal and promulgation of the Part 503 Standards and for overall management of the 2001 NSSS. Day-to-day responsibility for managing various aspects of the Round Two rulemaking was delegated to the Health and Ecological Criteria Division (HECD) and the Engineering and Analysis Division (EAD) within OST. EAD was responsible for managing all sample collection, laboratory analysis, data verification (data review), and database development activities. Both EAD and HECD were responsible for day-to-day interaction with contractors and with personnel at the POTW facilities visited. HECD was responsible for conducting the data analysis, risk assessment, and environmental assessments necessary to support the rulemaking. The EPA Sample Control Center, operated by CSC (then known as DynCorp) provided cradle-to-grave study support under EAD's direction.

To minimize analytical variability, EAD used a single contract laboratory (Axys Analytical Services) to perform all study analyses. The laboratory has extensive experience in dioxin/furan and PCB analyses and, prior to participation in the study, demonstrated that it could produce data of the quality required for regulatory purposes. CSC facilitated effective communication among all parties involved in the shipment and analysis of samples under this study. CSC's responsibilities also included:

- Drafting the sampling procedures document
- Developing the draft QAPP
- Preparing and distributing field sampling kits
- Participating in sample collection
- Documenting all sample shipments
- Resolving any shipping and analytical problems that arose
- Reviewing laboratory data
- Working with the laboratory and EPA to correct quality control failures, where possible
- Documenting the extent to which data submissions met method acceptance criteria
- Developing a database of study results
- Drafting the study report.

## SECTION 3.0 STUDY OBJECTIVES AND SELECTION OF FACILITIES

### 3.1 STUDY OBJECTIVES

The objective of the survey was to obtain unbiased national estimates of dioxin and dioxin-like compounds in sewage sludge to enable EPA to perform a multi-pathway exposure assessment and risk assessment of the disposal of sewage sludge through land application. Data collected in the study also were to be used in assessing risks associated with surface disposal and incineration of sewage sludges. These risk assessments would serve as the technical basis for determining whether numerical standards for dioxins for these sewage sludge management practices are warranted in the final Part 503 rule.

### 3.2 SELECTION OF FACILITIES

To ensure that EPA obtained a representative sampling of POTW facilities and that data collected could be compared with data from the 1989 NSSS, a total of 101 facilities throughout the contiguous United States were randomly selected from the pool of 174 facilities in the 1989 NSSS for participation in the 2001 NSSS. Seven of the selected facilities were either closed or no longer treated their own sewage sludge and, therefore, were not eligible for sampling. As a result, only 94 facilities were sampled. These 94 facilities were located in 32 states and the District of Columbia and are listed in Table 1, in alphabetical order by state, then city.

**Table 1. Facilities Sampled in 2001 NSSS**

Facility Name	City	State	Facility Name	City	State
Sacramento Regional WWTP	Elk Grove	CA	Metropolitan Council - Metro	Saint Paul	MN
Fallbrook Public Utility District	Fallbrook	CA	Crocker WWTP	Crocker	MO
Manteca WQCF	Manteca	CA	Mason Farm WTP	Carrboro	NC
Central Contra Costa Sanitary District	Martinez	CA	Whiteville WWTP	Whiteville	NC
Fairfield - Suisun Sewer District	Suisun City	CA	Burwell WWTF	Burwell	NE
Boulder - 75th St WWTP	Boulder	CO	Middletown Sewerage Authority	Belford	NJ
Steamboat Springs	Steamboat Springs	CO	Joint Meeting Sewage Treatment	Elizabeth	NJ
Rocky Hill WPCP	Hartford	CT	Passaic Valley Sewerage Commission	Newark	NJ
Waterbury WPCF	Waterbury	CT	Bowery Bay WPC	Corona Queens	NY
DC WASA (Blue Plains)	Washington	DC	Hunt's Point WPC	Corona Queens	NY
Mulberry STP	Mulberry	FL	Cayuga Heights WWTP	Ithaca	NY
Escambia County - Main Street WTP	Pensacola	FL	Brewster WWTP	Mahopac	NY
St. Petersburg SW Treatment Plant	St. Petersburg	FL	NEORS - Southerly	Cleveland	OH
Sunrise Sewage Treatment Plant No.1	Sunrise	FL	Brentwood Estates STP #24	Cuyahoga Falls	OH
R. M. Clayton WPCP	Atlanta	GA	Delphos	Delphos	OH
Buford Westside WPCP	Buford	GA	Massillon	Massillon	OH
Cartersville WPCP	Cartersville	GA	North Olmsted	North Olmsted	OH



**Table 1. Facilities Sampled in 2001 NSSS**

Facility Name	City	State	Facility Name	City	State
Dekalb Co - Snapfinger Cr WPCP	Decatur	GA	Port Clinton	Port Clinton	OH
Garden City WPCP	Garden City	GA	Twin Lakes WWTP	Ravenna	OH
Gwinnett Co Jackson Cr	Lilburn	GA	Thornville	Thornville	OH
Ocmulgee WPCP	Warner Robins	GA	West Carrollton	West Carrollton	OH
Boise	Boise	ID	Blackwell	Blackwell	OK
Belleville STP #1	Belleville	IL	Lebanon	Lebanon	OR
MWRDGC Stickney STP	Cicero	IL	Portland	Portland	OR
Jacksonville STP	Jacksonville	IL	Burnham STP	Burnham	PA
Morris STP	Morris	IL	Downingtown Area Regional Authority	Downingtown	PA
Tolono STP	Westville	IL	Girard Boro	Girard	PA
Evansville STP - Westside	Evansville	IN	Kiski Valley Water Pollution Control	Leechburg	PA
Frankton Municipal STP	Frankton	IN	Philadelphia Water Dept (SW)	Philadelphia	PA
Hammond Municipal STP	Hammond	IN	Philadelphia Water Dept (NE)	Philadelphia	PA
Muncie Sanitary District	Muncie	IN	Allegheny County Sanitary Authority	Pittsburgh	PA
Terre Haute Municipal STP	Terre Haute	IN	Narragansett Bay Commission - Bucklin	Providence	RI
Union City Municipal STP	Union City	IN	Florence - Pee Dee River Plant	Florence	SC
Oakland STP	Topeka	KS	WCRSA/Pelham WWTF	Greenville	SC
Shepherdsville STP	Shepherdsville	KY	Brooking	Brookings	SD
Billerica WWTP	Billerica	MA	Sioux Falls	Sioux Falls	SD
Fall River WWTF	Fall River	MA	Andrews STP	Andrews	TX
Medfield WWTP	Medfield	MA	Del Rio -San Felipe	Del Rio	TX
Pittsfield WWTP	Pittsfield	MA	Navasota, Grimes Co. STP	Navasota	TX
Patapsco WWTP	Baltimore	MD	Orange, Jackson St WWTP	Orange	TX
South Portland WPCF	South Portland	ME	Brazos River Authority (Waco)	Waco	TX
Dowagiac WWTP	Dowagiac	MI	Fredericksburg City STP	Fredericksburg	VA
Iron Mountain - Kingsford WWTP	Kingsford	MI	Augusta County Service Authority	Verona	VA
Genesee County - Ragnone WWTP	Montrose	MI	HRSD - James River STP	Virginia Beach	VA
Port Huron WWTP	Port Huron	MI	HRSD - Chesapeake/Elizabeth STP	Virginia Beach	VA
Wyandotte WWTP	Wyandotte	MI	Metropolitan King County	Renton	WA
Western Lake SSD	Duluth	MN	Greenbrier County PSD No 2	Rainelle	WV

## SECTION 4.0 SAMPLE COLLECTION AND ANALYSIS

### 4.1 SAMPLE COLLECTION

Samples were collected from all 94 facilities between February 5, 2001 and March 20, 2001, by two EPA personnel in the Health and Ecological Criteria Division (HECD) and Engineering and Analysis Division (EAD), and by six CSC Sample Control Center staff. All samples were collected according to the *Sampling Procedures for the 2001 National Sewage Sludge Survey* (Reference 8.1).

Eighty-nine of the sampled POTWs had a single system for treating and processing their sludge material. Therefore, one sample of the final sludge product was collected from each of these facilities. Five of the POTWs had two systems for treating sludge. Therefore, two final sludge product samples were collected from each of these five facilities (one sample from each treatment process). A total of 99 final product samples were collected from the 94 facilities.

A second aliquot of each sample was collected and archived at EAD's chemical repository, operated by Gascoyne Laboratories, for possible future analysis. Split samples were collected for the facility at their request.

EPA's study design called for the collection of a field duplicate sample from 15% of the facilities. A field duplicate sample is a second sample collected at the facility using similar procedures and equipment as the original sample. The results of the field duplicate sample can be compared to the results of the original sample as a means of assessing the overall precision of the sampling and analysis processes.

EPA randomly selected 15 of the original pool of 101 POTWs for collection of field duplicates. Those 15 POTWs are identified in the sampling procedures document (Reference 8.1). However, once the study began, field duplicates were not collected at three of those facilities for various reasons. Therefore, EPA selected 2 more facilities from those that remained to be sampled, such that a total of 14 field duplicate samples ultimately were collected from the pool of 94 POTWs sampled (a 14.9% frequency for field duplicates). The 14 facilities are listed in Table 2, below. The results of the field duplicate samples are discussed in Section 6.4.

**Table 2. Facilities at which Field Duplicates were Collected**

Manteca WQCF, Manteca, CA	Metropolitan Council - Metro, Saint Paul, MN
Buford Westside WPCP, Buford, GA	Crocker WWTP, Crocker, MO
Garden City WPCP, Garden City, GA	Mason Farm WTP, Carrboro, NC
Boise STP, Boise, ID	Portland STP, Portland, OR
Hammond Municipal STP, Hammond, IN	Pee Dee River Plant, Florence, SC
Oakland STP, Topeka, KS	Metropolitan King County, Renton, WA
Shepherdsville STP, Shepherdsville, KY	Greenbrier County PSD No 2, Rainelle, WV

Field duplicates were not identified as such in the traffic reports sent to the laboratory. Therefore, the laboratory was not aware of which samples were field duplicates.

### 4.2 ANALYTES OF INTEREST

Samples were analyzed by Axys Analytical Services (2045 Mills Road West, Sydney, British Columbia, Canada V8L 3S8), using the methods and modifications specified by EPA and described in Section 4.3.

Samples were analyzed for the seven 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin congeners, the ten 2,3,7,8-substituted polychlorinated dibenzofuran congeners, and the full set of 209

coplanar polychlorinated biphenyls (PCBs) congeners. EPA Method 1613B was used to analyze the 17 dioxin and furan compounds and EPA Method 1668A was used for PCB analysis, as shown in Table 3. Method modifications were used to achieve better sensitivity, as described in Section 4.3. Analytical results were reported in the units of nanograms of congener per kilogram of sewage sludge (ng/kg) on a dry-weight basis (100% solids) for all samples.

**Table 3. Analytical Methods and Target Analytes**

Method	Analytes
Method 1613B, Dioxins and Furans by Isotope Dilution High-Resolution Gas Chromatography/Mass Spectrometry	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF
Method 1668A, Polychlorinated Biphenyls by Isotope Dilution High-Resolution Gas Chromatography/Mass Spectrometry*	209 congeners**, including the following 12 congeners identified as toxic by the World Health Organization:  3,3',4,4'-TeCB (PCB 77)      3,3',4,4',5-PeCB (PCB 126) 3,4,4',5-TeCB (PCB 81)      2,3,3',4,4',5-HxCB (PCB 156) 2,3,3',4,4'-PeCB (PCB 105)      2,3,3',4,4',5'-HxCB (PCB 157) 2,3,4,4',5-PeCB (PCB 114)      2,3',4,4',5,5'-HxCB (PCB 167) 2,3',4,4',5-PeCB (PCB 118)      3,3',4,4',5,5'-HxCB (PCB 169) 2',3,4,4',5-PeCB (PCB 123)      2,3,3',4,4',5,5'-HpCB (PCB 189)
*Method 1668A includes all 209 PCB congeners in its list of target analytes. The method is capable of separating 167 individual congeners, including the 12 toxic congeners; the remaining 42 congeners are identified in pairs or groups and reported together. Congeners in each pair or group have the same TEFs.	**All 209 congeners are listed in Table 1 of Method 1668A.

TCDD	Tetrachlorodibenzo- <i>p</i> -dioxin	TCDF	Tetrachlorodibenzofuran	TeCB	Tetrachlorobiphenyl
PeCDD	Pentachlorodibenzo- <i>p</i> -dioxin	PeCDF	Pentachlorodibenzofuran	PeCB	Pentachlorobiphenyl
HxCDD	Hexachlorodibenzo- <i>p</i> -dioxin	HxCDF	Hexachlorodibenzofuran	HxCB	Hexachlorobiphenyl
HpCDD	Heptachlorodibenzo- <i>p</i> -dioxin	HpCDF	Heptachlorodibenzofuran	HpCB	Heptachlorobiphenyl
OCDD	Octachlorodibenzo- <i>p</i> -dioxin	OCDF	Octachlorodibenzofuran		

### 4.3 METHODS MODIFICATIONS

Axys Analytical Services performed analyses for dioxins/furans using Method 1613, Revision B, and for PCB congeners using Method 1668, Revision A, with the modifications listed below:

- *Method 1613B*: Axys achieved MDLs and MLs that were 5 times lower than those specified in Method 1613B. This was accomplished by using a sixth calibration solution, CS0, that contained all method-specified analytes at levels 5 times lower than the levels specified in Table 4 of the method. All other method requirements, including QC acceptance criteria for calibration linearity and laboratory blanks, were met using the modified method.
- *Method 1668A*: Axys employed the 6-point calibration option that encompassed the high sensitivity low calibration point CS-0.2 specified in Table 5 of the method. If interferences were encountered with coplanar congeners 77, 126, or 169, the charcoal column clean-up or DB-1 (or equivalent) GC column was used to resolve interferences.

To achieve the lowest reasonable quantitation limits and to provide relatively consistent quantitation limits across all the sludge samples, the laboratory determined the solids content (e.g.,

percent solids) of an aliquot of each sample prior to taking an aliquot for the analysis of either the dioxins/furans or the PCBs. For all field samples, the laboratory used the protocol specified in Section 11.2.2 of Methods 1613B and 1668A for the determination of percent solids (i.e., the laboratory determined percent solids, as opposed to total suspended solids). The laboratory used the data on the solids content of each sample to determine the appropriate size aliquot that resulted in the extraction and analysis of 10 g of dry solids for each sample (e.g., a liquid sample with 3% solids required the use of a 334-mL aliquot to obtain 10 g of dry solids). Samples that contained 5% solids or greater were prepared and extracted as solid samples, in accordance with the procedures delineated in Sections 11.1.3, 11.5, and 12.3 of Methods 1613B and 1668A.

Some samples were pourable liquids with solids contents that ranged from <1% to as high as 5%. For these samples, the laboratory centrifuged the sample to separate the solids from the aqueous portion of the sample. The treatment of the sample phases after centrifugation depended on the analysis to be conducted. Samples for dioxins/furans were treated differently than those for PCBs, as follows.

- *Preparation of Samples for Dioxin/Furan Analyses:* For dioxin/furan analyses using Method 1613B, the supernatant liquid that resulted from centrifugation was filtered as described in Section 11.4.3 of the method. The filtrate (i.e., liquid phase) was then subjected to separatory funnel extraction as specified in Section 12.1, and the filter and particles were extracted by the Soxhlet-Dean Stark (SDS) extraction procedure specified in Section 12.3. The separatory funnel and SDS extracts were combined, processed, and analyzed as a single extract using the procedures specified in Method 1613B
- *Preparation of Samples for PCB Analyses:* To minimize the risk of losing some low-molecular weight PCB congeners, the supernatant that resulted from the centrifugation step was not filtered as described in Section 11.4.3 of Method 1613B, since that step involved a vacuum filtration. Instead, the supernatant liquid was extracted using a separatory funnel procedure as described in Section 12.1 of Method 1613B, and the extract was combined with the extract from the centrifuged solids, processed, and analyzed as a single extract using the procedures specified in Method 1668A.

## SECTION 5.0 DATA REPORTING AND VALIDATION

Each laboratory data package submitted under this study was reviewed for completeness and compliance with method specifications and subcontract requirements to ensure that the data met the measurement quality objectives of the study. These data reviews were performed by CSC Sample Control Center data review staff trained in procedures for reviewing dioxins/furans and PCB results produced by Method 1613B and 1668A, respectively. Data reviews were performed using a multi-stage review process designed to identify and correct data deficiencies as early as possible, to maximize the amount of usable data generated during the study. This data review process included the following elements.

- *Data Completeness Check* in which all elements in the laboratory submission were evaluated to verify that results for all specified samples were provided, that data were reported in the correct format, and that all relevant information, such as preparation and analysis logs, were included in the data package.
- *Instrument Performance Check* in which the data reviewers verified that calibrations, calibration verifications, standards, and calibration blanks were analyzed at the appropriate frequency and met method or study performance specifications.
- *Laboratory Performance Check* in which the reviewers verified that the laboratory correctly performed the required analytical procedures and was able to demonstrate a high level of precision and accuracy. This check included evaluation of QC elements such as the initial precision and recovery (IPR) and ongoing precision and recovery (OPR) tests, field blanks, method blanks, and reference standards.
- *Method/matrix Performance Check* to discern whether any QC failures were a result of laboratory performance or difficulties with the method or sample matrix. Data evaluated in this stage included labeled compound results. The data reviewers also verified that proper sample dilutions were performed and that necessary sample cleanup steps were taken.

If errors were noted during any of these checks, corrective actions were initiated with the laboratory to resolve the deficiencies identified. After reviewing each data package, the CSC data reviewers prepared written reports in narrative format that described data quality limitations and recommendations concerning data use. These narrative data reports were provided to the EPA Study Manager.

Upon completion of data review, CSC created an analytical database that contained all field sample results from the 2001 NSSS. This database was prepared using Statistical Analysis Software (SAS) and was designed to be consistent with the database format used in EPA's 1989 NSSS. At intervals during database production and again upon completion of the study, CSC ran queries to verify the accuracy of the database. Upon database completion, CSC prepared a Microsoft Access report that contained all data from the 2001 NSSS database in tabular format. This report was provided to the EPA Study Manager.

At EPA's direction, CSC prepared individual reports of facility data and distributed these reports to the participating facilities. The reports contained the results for the seventeen 2,3,7,8-substituted dioxins and furans and the 209 PCB congeners for each sample collected at a given facility, and the EPA sample number(s) associated with the sample(s) collected at that facility. In addition, each facility received a summary of the results across all facilities. The summary report listed each EPA sample number with the Total TEQ for that sample, but did not identify any of the facilities by name. The information in the facility-specific report made it possible for each facility to compare its own results to those of all the other facilities in the survey.

## SECTION 6.0 RESULTS, DATA ANALYSIS, AND DISCUSSION

### 6.1 TOXIC EQUIVALENT CONCENTRATION

Historically, 2,3,7,8-TCDD has exhibited the greatest toxicity in animal studies and therefore has been considered the most toxic of the 210 dioxin and furan compounds. The toxicity of a mixture of dioxin-like compounds is often reported in terms of the toxic equivalents of 2,3,7,8-TCDD, or the TEQ (for the Toxic Equivalent concentration). The toxicity equivalency factor (TEF) is the scaling factor that relates the toxicity of a concentration of a specific dioxin, furan, or PCB compound to a concentration of 2,3,7,8-TCDD. The TEF represents an order of magnitude estimate of the toxicity of the compound of interest, based on a consensus of current research results on these compounds.

The TEQ is calculated as the sums of the products of each PCDD, PCDF, and PCB congener and its respective TEF, as follows:

$$Total\ TEQ = \sum_{x=1}^7 [PCDD_x \times TEF_x] + \sum_{x=1}^{10} [PCDF_x \times TEF_x] + \sum_{x=1}^{11} [PCB_x \times TEF_x]$$

The results from the 2001 NSSS were reported in terms of the total TEQ.

The TEF values used for the 2001 NSSS are shown in Table 4. These values were developed by the World Health Organization (WHO) in 1998 (Reference 8.3) and include non-zero TEFs for the seventeen 2,3,7,8-substituted PCDDs/PCDFs and twelve non-ortho-substituted and mono-ortho-substituted (coplanar) PCBs. The 1998 WHO TEF for 1,2,3,7,8-pentachlorodibenzo-*p*-dioxin (PeCDD) is 1, indicating the current consensus that this compound is as toxic as 2,3,7,8-TCDD. The TEFs for PCBs 156 and 157 are equal, however, these two congeners cannot be completely separated from one another and are reported as the sum of the two congeners in Method 1668A.

In calculating the TEQ for samples in the 2001 NSSS, EPA handled the results for all of the toxic PCDDs, PCDFs, and PCBs that were “not detected” by substituting one-half of the quantitation limit (called the “Minimum Level,” or ML, in Methods 1613B and 1668A) reported by the laboratory for the concentration of that analyte. This commonly used substitution scheme is conservative relative to protection of the environment, in that it assumes that there may be some of the analyte present below the laboratory’s reported quantitation limit. Other common substitution schemes include setting all non-detects to zero or using the quantitation limit (rather than one-half the limit). The use of any non-zero substitution scheme means that *every* sample will have a non-zero TEQ, regardless of whether or not any analytes were detected. Using the WHO TEF values and one-half of the quantitation limits in Table 4, the lowest possible TEQ for a sample in this study would be 1.6 ng/kg (rounded to one decimal place).

**Table 4. Minimum Levels, Toxicity Equivalency Factors for Dioxins, Furans, and PCBs**

Analyte	ML (ng/kg)	1/2 ML (ng/kg)	WHO TEF	TEQ Using ML (ng/kg)	TEQ Using 1/2 ML (ng/kg)
<b>Dioxins</b>					
2,3,7,8-TCDD	0.1	0.05	1	0.1	0.05
1,2,3,7,8-PeCDD	0.5	0.25	1	0.5	0.25
1,2,3,4,7,8-HxCDD	0.5	0.25	0.1	0.05	0.025
1,2,3,6,7,8-HxCDD	0.5	0.25	0.1	0.05	0.025
1,2,3,7,8,9-HxCDD	0.5	0.25	0.1	0.05	0.025
1,2,3,4,6,7,8-HpCDD	0.5	0.25	0.01	0.005	0.0025
OCDD	1	0.5	0.0001	0.0001	0.00005

**Table 4. Minimum Levels, Toxicity Equivalency Factors for Dioxins, Furans, and PCBs**

Analyte	ML (ng/kg)	1/2 ML (ng/kg)	WHO TEF	TEQ Using ML (ng/kg)	TEQ Using 1/2 ML (ng/kg)
<b>Furans</b>					
2,3,7,8-TCDF	0.1	0.05	0.1	0.01	0.005
1,2,3,7,8-PeCDF	0.5	0.25	0.05	0.025	0.0125
2,3,4,7,8-PeCDF	0.5	0.25	0.5	0.25	0.125
1,2,3,4,7,8-HxCDF	0.5	0.25	0.1	0.05	0.025
1,2,3,6,7,8-HxCDF	0.5	0.25	0.1	0.05	0.025
1,2,3,7,8,9-HxCDF	0.5	0.25	0.1	0.05	0.025
2,3,4,6,7,8-HxCDF	0.5	0.25	0.1	0.05	0.025
1,2,3,4,6,7,8-HpCDF	0.5	0.25	0.01	0.005	0.0025
1,2,3,4,7,8,9-HpCDF	0.5	0.25	0.01	0.005	0.0025
OCDF	1	0.5	0.0001	0.0001	0.00005
<b>PCBs</b>					
PCB-77	20.0	10.0	0.0001	0.002	0.001
PCB-81	2.0	1.0	0.0001	0.0002	0.0001
PCB-105	20.0	10.0	0.0001	0.002	0.001
PCB-114	1.0	0.5	0.0005	0.0005	0.00025
PCB-118	20.0	10.0	0.0001	0.002	0.001
PCB-123	2.0	1.0	0.0001	0.0002	0.0001
PCB-126	20.0	10.0	0.1	2	1
PCB-156+PCB-157	2.0	1.0	0.0005	0.001	0.0005
PCB-167	1.0	0.5	0.00001	0.00001	0.000005
PCB-169	1.0	0.5	0.01	0.01	0.005
PCB-189	1.0	0.5	0.0001	0.0001	0.00005
<b>Lowest Possible Toxic Equivalent Concentration (TEQ)</b>				3.26821	1.634105

**6.2 SUMMARY RESULTS**

Table 5 presents the total TEQ and the portions of the TEQ due to the dioxins, furans, and PCBs, for the 113 samples analyzed during the survey, rounded to the nearest whole number. The total TEQ values range from 3 to 718 ng/kg for the samples in this study. Keep in mind that substituting one-half of the quantitation limit for each analyte means that no sample can have a total TEQ less than 1.6 ng/kg (see Table 4 above).

**Note:** Fourteen of the results in Table 5 are for the field duplicate samples collected as part of the quality assurance effort for the survey. The field duplicates are discussed in detail in Section 6.4.

**Table 5. Results for 2001 NSSS Samples, in order of increasing Total TEQ**

EPA Sample Number	Total TEQ	Dioxin TEQ	Furan TEQ	PCB TEQ
57631	3	2	1	0
58105	3	1	0	2
57948	5	3	1	1
57972	5	3	1	1
58026	5	3	1	1
57775	6	4	1	2
57900	6	5	1	1
57780	7	4	1	2
57691	7	4	2	2
57858	8	5	1	2
57859	8	5	1	2

**Table 5. Results for 2001 NSSS Samples, in order of increasing Total TEQ**

EPA Sample Number	Total TEQ	Dioxin TEQ	Furan TEQ	PCB TEQ
57864	8	5	1	2
57738	9	5	1	2
58176	10	1	1	8
58069	10	6	2	1
57961	10	8	2	1
57841	11	8	3	0
57606	11	7	2	2
57798	12	6	1	4
57745	12	7	2	3
58104	12	8	2	1
57984	12	7	3	2
57930	12	7	1	4
57942	13	10	2	0
58032	13	6	4	2
58033	13	7	4	2
57804	13	7	3	3
57978	13	9	4	1
58038	14	9	4	0
58017	14	7	2	4
58014	14	8	2	4
58050	15	10	2	3
57810	15	11	4	1
57624	16	8	2	5
57684	16	12	2	1
57636	16	13	2	1
57601	16	7	3	6
57906	17	13	2	1
57918	17	7	8	2
57878	17	14	2	1
57660	17	10	4	3
57816	17	15	3	0
57828	18	14	4	0
57995	18	9	3	6
57834	18	7	1	10
57846	18	9	4	5
57763	19	11	3	5
57882	19	14	3	2
57943	19	14	3	1
57768	20	16	3	0
57786	20	15	3	1
58008	20	15	4	1
57839	20	7	2	11
57685	20	12	3	6
58122	21	17	4	0
57708	22	16	5	2
58164	23	12	4	8
57895	23	15	3	5
58110	24	12	3	8
58092	24	17	5	1
57757	24	12	5	7
57954	24	14	5	5
58134	24	19	5	0
57894	24	15	3	6
57936	25	19	4	3
57720	26	13	4	8
57678	26	23	3	0
57876	27	22	2	3
58147	27	21	4	2
57955	27	16	6	6



**Table 5. Results for 2001 NSSS Samples, in order of increasing Total TEQ**

EPA Sample Number	Total TEQ	Dioxin TEQ	Furan TEQ	PCB TEQ
57924	27	23	3	1
57990	28	14	5	9
57852	28	24	1	2
57966	29	22	6	1
58116	31	14	6	11
57793	33	22	7	4
57912	34	27	4	3
58099	35	27	5	2
58044	36	14	6	15
57726	36	24	3	9
57655	36	17	8	11
58160	37	27	9	0
57613	38	24	10	4
57666	39	21	7	11
58020	40	22	7	10
58098	40	27	5	8
58074	41	21	8	12
58063	42	25	7	9
57751	42	25	9	7
58140	42	17	13	13
58062	43	26	7	9
57714	46	27	7	13
57702	48	27	9	12
58141	53	22	18	13
57696	53	24	11	18
58080	55	39	10	5
57673	57	36	10	11
58153	58	40	14	4
57870	59	10	45	4
57888	60	22	34	3
57996	63	30	6	27
57618	64	4	2	58
58128	68	44	12	12
57654	74	59	7	8
57732	79	14	9	56
57621	97	28	10	58
57648	110	82	11	17
57998	118	75	15	28
58170	123	119	3	1
57825	313	234	77	1
57824	453	336	114	3
57822	555	449	103	3
57642	718	189	493	36

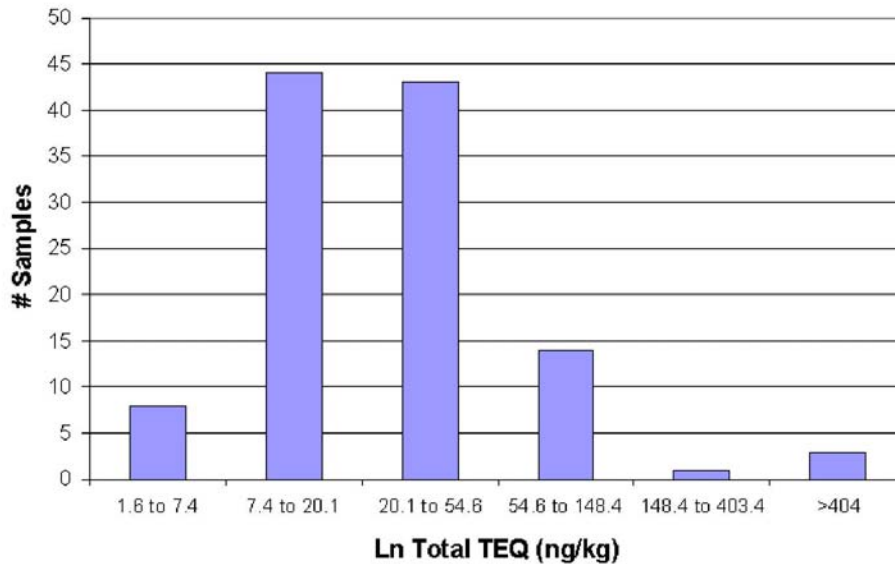
\*All TEQ values were calculated using the WHO TEF values in Table 4. For analytes that were not detected, the concentration of that analyte was set to one-half of the Minimum Level for the analyte in Table 4. All results are reported on a dry-weight basis.

### 6.3 FREQUENCY DISTRIBUTIONS

A frequency distribution of the total TEQ was plotted from the results for all the samples in Table 5 (including the 14 field duplicates). Because the data span a wide TEQ range and are not normally distributed, the total TEQ data were log-transformed using the natural log (ln, base e). The natural logs of the TEQ results range from 1.07 to 6.58, and the span was divided into ranges, based on the first digit on the natural log of the total TEQ. To emphasize the fact that no sample in this study can have a total TEQ value less than 1.6 ng/kg because of the treatment of non-detect values, the first two ranges were combined and the description of the bottom end of lowest range was raised from 0 to 1.6 ng/kg.

Figure 1 shows the frequency distribution based on the log-transformed TEQ data. The concentration ranges used for the log-transformed frequency plot were:

- 1.6 to 7.4 ng/kg (encompassing ln values from 0 to 2)
- 7.4 to 20.1 ng/kg (encompassing ln values from 2 to 3)
- 20.1 to 54.6 ng/kg (encompassing ln values from 3 to 4)
- 54.6 to 148.4 ng/kg (encompassing ln values from 4 to 5)
- 148.4 to 403.4 ng/kg (encompassing ln values from 5 to 6)
- >404 ng/kg (all ln values over 6)



**Figure 1. Frequency of Log-Transformed Total TEQ Results for the 2001 NSSS**

As can be seen, the vast majority of the survey samples have total TEQ values between 7.4 and 54.6 ng/kg. The data are approximately log-normally distributed, which is not unexpected, given the small number of very high values.

A regression of total TEQ and percent solids was performed. The correlation coefficient (r) for the regression was only 0.0608, indicating that there is no apparent relationship of total TEQ to percent solids content of the sludge samples.

#### 6.4 FIELD DUPLICATE RESULTS

Field duplicate samples were collected at 14 facilities. The results for the 14 field duplicate samples and the associated original samples from each facility are shown in Table 6. For each pair of samples, the total TEQ and percent solids results were compared using the relative percent difference (RPD). The RPD is used as the measure of precision because both results from the pair are measured concentrations and there is no “true” concentration to be used in the comparison. The formula for RPD is shown below:

$$RPD = \frac{|Result\ 1 - Result\ 2| \times 100}{\frac{(Result\ 1 + Result\ 2)}{2}}$$

where Result 1 and Result 2 represent the concentrations reported in the two samples in each pair, the vertical bars in the numerator indicate it is the absolute value of the difference, and the factor of 100 converts the value to a percent.

The pairs are presented in Table 6 in order of increasing RPD of the TEQ for each pair. The total TEQ results in this table are expressed to two decimal places solely to illustrate some of the small differences between the samples. However, the whole number TEQ values in Table 5 more appropriately reflect the significant figures in the TEQ calculations.

**Table 6. Field Duplicate Comparisons**

Sample	Total TEQ	RPD (%)	% Solids	RPD (%)
57858	8.08	0.8	17	0.0
57859	8.14		17	
58032	12.80	1.2	29	0.0
58033	12.96		29	
58014	14.09	1.2	14	6.9
58017	13.91		15	
58062	43.01	2.5	67	1.5
58063	41.94		68	
57834	18.36	9.2	14	0.0
57839	20.14		14	
57954	24.04	13.1	81	2.4
57955	27.41		83	
58098	40.49	15.8	0.60	3.3
58099	34.56		0.62	
58140	42.30	22.0	18	5.7
58141	52.74		17	
57824	453.41	36.7	16	6.5
57825	312.68		15	
57990	27.58	40.2	11	37.0
57995	18.36		16	
57942	12.57	41.2	1.7	12.5
57943	19.10		1.5	
57876	26.56	44.4	12	8.0
57878	16.91		13	
57996	63.30	60.4	15	6.5
57998	118.04		16	
58104	11.77	112	14	0.0
58105	3.32		14	

The field duplicate samples largely span the range of concentrations from all samples, with concentrations ranging from 3 to 453 ng/kg. In general, the RPD values reflect those typically seen for field duplicates, with nearly all RPDs  $\leq 40\%$  and all but two RPDs  $\leq 45\%$ . There is no apparent relationship between the RPD of the TEQ in the field duplicates and the mean concentration of those duplicates, since the two largest RPD values, 60.4% and 112%, occur at opposite ends of the concentration range.

The 14 field duplicate pairs were collected by 8 different samplers, two from EPA and six from CSC. There was no apparent effect of sampler experience on RPD values, because two of the pairs with RPDs over 40% were collected by a sampler with over 30 years of experience, while field duplicates that exhibited relatively little difference were collected by samplers with less than 5 years of sampling experience.

The percent solids results for these field duplicates provide additional information. As described in the sampling procedures document (Reference 8.1), most liquid sludge samples were collected directly into the sample containers from a tap or valve in the sludge transfer plumbing. Other than asking the facility staff to mix liquid sludge in any holding tanks and flushing the tap or valve before collecting the

sample, no other efforts were made to composite or homogenize the liquid sludge samples. The two pairs of low-solids sludges (58098/58099 and 57942/57943) exhibited relatively similar percent solids results within each pair, with RPD values of 3.3% and 12.5%, respectively. However the differences in the total TEQ values were substantially greater, at 15.8% and 41.2%, respectively, and may be attributable to the specific conditions of the sampling at each site, as discussed below.

Samples 58098 and 58099 (the field duplicate) were collected from a discharge hose on a storage tank used to fill tankers for land application. The samples were collected immediately after the hose was used to fill a tanker, thereby flushing the hose. However, because another tanker was not available to be filled before the field duplicate sample was collected, the material in the hose was fairly similar to that collected for the initial sample. In contrast, Samples 57942 and 57943 were collected from a large anaerobic digester with an open top. The sampler had to lower a clean bucket into the digester from a catwalk atop the tank to collect each sample. Although the digester had mixers, these results for TEQ and percent solids suggest that the sludge in the tank was not completely homogeneous at the time that the samples were collected.

The solid sludge samples presented more obvious sampling challenges. Many of the solid samples were collected from dewatering devices such as belt presses, filter presses, or centrifuges. These devices are run periodically as the facility removes sludge from the process. Samples collected even a few minutes apart may contain different pollutants or different concentrations depending on the inputs to the treatment plant at the time the sludge was produced, or based on variations in conditions at the time of disposal.

In other cases, sludge is stockpiled at the facility and composted with wood chips, yard waste, and other organic materials. The stockpiles vary greatly in size and complexity. Variations in both the sludge removed from the treatment system and the nature of the amendments used for composting will lead to differences in the pollutants and concentrations. Further, the size of some compost piles makes collection of representative samples difficult. At least one field duplicate pair (57824/57825) was collected at a facility that composted sludge in piles 10 to 12 feet tall, with steeply sloping sides. In this case, small amounts of material were collected from multiple locations around the edges of the pile and composited to form each sludge sample. The field duplicate sample was collected in a similar fashion, but with materials from different locations around the pile. However, the materials that were accessible to the sampler were only those within arm's reach of the sides of the pile and below shoulder height. Thus, the RPD of 36.7% for the total TEQ values in these two samples may represent the variability in the materials in that pile.

## SECTION 7.0 CONCLUSIONS

EPA was successful in collecting and analyzing 113 sewage sludge samples from 94 POTWs for the analysis of dioxins, furans, and PCBs during the 2001 NSSS. The selection of sites for sample collection and the application of state-of-the-art analytical methods and project-specific analytical modifications permitted EPA to develop unbiased national estimates of these pollutants in sewage sludge at the lowest practical levels. These data are suitable for use in a multi-pathway exposure and risk assessment of the land application of sludge that are part of EPA's review of regulations at 40 CFR Part 503.

Results from the survey were calculated in terms of the toxic equivalent concentration (TEQ) of 2,3,7,8-TCDD and ranged from 3 to 718 ng/kg (dry weight). The vast majority of the samples had TEQ values in the range of 7 to 55 ng/kg. Only seven results over 100 ng/kg TEQ were reported, and two of those were for a field duplicate pair collected as part of the quality assurance program for the survey.

## SECTION 8.0 REFERENCES

- 8.1 *Sampling Procedures for the 2001 National Sewage Sludge Survey*, DynCorp, February 2001, 21 pages.
- 8.2 *Quality Assurance Project Plan for the 2001 National Sewage Sludge Survey*, DynCorp, March 2001, 57 pages.
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