

USEPA SUPERFUND NATIONAL NON-ROUTINE ANALYTICAL SERVICES DIOXIN AND CB CONGENER PROGRAM – OVERVIEW OF AVAILABLE SERVICES AND BENEFITS TO USEPA

Holman E¹

¹ U.S. Environmental Protection Agency, 1200 Pennsylvania Ave. MC 5102G, Washington, DC 20460.

Introduction

The National Non-Routine Analytical Services Program of USEPA's Office of Superfund Remediation and Technology Innovation (OSRTI) offers an analytical service that provides data from the analysis of water, soil, sediment, sludge, non-human tissue, ash, oil, and oily matrices for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the dioxin and CB congeners analytical services produce data of known and documented quality. USEPA Regional customers request and receive dioxin and CB congener analyses through preapproved commercial laboratories. In order to participate in the program, laboratories are required to pass a rigorous quality assurance review, including providing SOP's for review and approval, performing blind Performance Evaluation sample analysis, and having an on-site laboratory inspection. The analyses are performed using a standard Statement of Work (SOW), with the dioxin analyses being performed under the DLM02.0 SOW and the CB Congener analyses under CBC01.0.^{1,2} Since the programs inception in 2002, over 3000 samples from potentially hazardous waste sites have been analyzed for a total cost of over \$1.7 million dollars.

The high quality of the data is verified using regional data review procedures and the National Functional Guidelines that were developed by the USEPA OSRTI.³ The program has been used to provide analytical dioxin and CBC data for various high profile sites including the Upper and Mid Columbia River, Passaic River, Escambia Wood Treatment site, and the Hurricane Katrina relief effort. Benefits of the program include the established quality assurance program; a centralized national network of laboratories; a standard SOW that allows for consistency in USEPA data results across regional programs; the standard SOW requirements can also be adjusted to meet USEPA regional requirements; and, finally, because the work is ordered at a bulk rate the program can offer significant cost savings (average cost per sample of \$535 vs. market price of \$900-\$1200).

Materials and Methods

Obtaining a Laboratory

The USEPA regional data requestor sends a file describing the project requirements, including the number of samples, projected sampling dates, and regional specific SOW requirements to the USEPA National Dioxin/CBC Program Manager. This file is reviewed for national consistency and, once finalized, forwarded to the USEPA contracts office. The USEPA contracts office sends the project requirements to each prequalified laboratory which then provide their bids to the contracts office. Each bid is reviewed and the project is awarded to a laboratory based on cost factors, past performance, and technical capability. A laboratory must be in good standing with the program in order to be awarded the project. Samples are then shipped to the appropriate laboratory for analysis after collection.

Extraction and Analysis of Samples

The Statement of Work (SOW) for Multi-Media, Multi-Concentration Dioxins and Furans Analysis (DLM02.0) describes the analytical method for chlorinated dibenzo-p-dioxins and chlorinated dibenzofurans (CDDs/CDFs) analyses. This SOW is based on the EPA Office of Water Method 1613B and defines the means by which 2,3,7,8-substituted CDDs/CDFs are isolated, detected, quantitatively measured, and reported [including the Electronic Data Deliverable (EDD) format].⁴

Analytical quality control and assurance

The CBC01.0 Statement of Work (SOW) for Analysis of Chlorinated Biphenyl (CB) Congeners describes the analytical method for CB Congeners analyses. This SOW is based on the EPA Office of Water Method 1668A and defines the means by which CB congeners are isolated, detected, quantitatively measured, and reported [including the Electronic Data Deliverable (EDD) format].⁵

DLM02.0 and CBC01.0 includes extraction procedures for water, soil, sediment, sludge, tissue (non-human), ash, and oil/oily matrix samples, and employs High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) to analyze the samples.

Each individual task order awarded to the laboratory provides specific requirements for the project. Project requirements will include the data delivery address, projected number of samples to be collected, additional data deliverable format requirements, and adjustments to the SOW for extraction and/or analysis requirements. The standard turnaround time for data from time of sample receipt is 35 days, but this can be adjusted to 7, 14, or 21 days.

Results and Discussion

Part 1A – Dioxin Laboratory Analysis

Between 2002 and October 2005, there were 5 small business laboratories preapproved to participate in the program for analysis of dioxins (under DLM01.4, an earlier version of DLM02.0). From 2005 to present there are 4 small business and 4 large business laboratories that are preapproved to participate in the program for both dioxins and CB Congeners (under DLM02.0 and CBC01.0). Review of their SOP's included the review of their sample receiving, extraction, glassware cleaning, HRGC-MS analysis (including tuning), data reporting and packaging, and training. On-site audits of each laboratory were conducted to confirm that SOP's were being followed as written. In cases where the SOP's were found to be inconsistent with the DLM02.0 and CBC01.0 SOW or where SOP's were not being followed in the laboratory, a recommendation for corrective action was made and resolution confirmed.

Table 2 shows the total number of dioxin samples that have been analyzed under our program for Fiscal Years 2002-2006.

Table 2 – Total Number of Dioxin Samples and Cost for FY02-FY06

	Total Cost	Avg. Cost/Sample	Total # of Dioxin Samples
FY02	\$256,804	\$577	445
FY03	\$302,342	\$558	542
FY04	\$565,429	\$530	1067
FY05	\$316,820	\$520	609
FY06	\$253,105	\$505	501
Grand Total	\$1,694,500	\$536	3164

The average cost per sample has consistently decreased in each year since the programs inception. This is the result of more competition between laboratories, generally increased usage of the program, and an overall decrease in the market price for dioxin analysis. The overall average cost per sample of \$536 represents a cost savings of 40-55% (vs. average market price of \$900-\$1200), saving the government \$1.1-\$2.1 million dollars.

The decreased usage in FY05 is the result of new contracts being awarded and there being a gap in time between when the old contracts expired and new ones awarded. As a result, there was a period of time in FY05 when services were not available through our program. In FY06, we appear to be on track with FY04 and have yet to experience our busiest sampling season projected for summer 2006.

Analytical quality control and assurance

The number of USEPA Regions using the program (the USEPA has 10 Regions) has also increased over the years, with only 3 EPA Regions using the program in 2002 and 7 EPA Regions consistently using the program in 2005 and 2006. As a result, we have expanded the base of our customers and increased the number of hazardous waste site studies supported.

Part 1B – CB Congeners Laboratory Analysis

Table 3 lists the number of CB Congener samples analyzed through the program in FY06.

Table 3 – Total CB Congener Samples and Costs for FY06

	Total Cost	Avg. Cost/Sample	Total # of CB Congener Samples
FY06	\$65,300	\$718	91

The CB Congener analysis program under SOW CBC01.0 was a new service added in FY05/FY06, with 4 small business and 4 large business laboratories preapproved to participate in the program. Only 2 EPA Regions are currently utilizing this program, but we anticipate more Regional customers to take advantage of this service in the future. Again, there is a significant cost savings for samples analyzed through this program, with an average cost of \$718 compared to \$900-\$1200 average market price, giving the government cost savings of 20-40% or \$17-\$34,000.

In addition to routine quality assurance measures (including laboratory control samples, method blanks, and calibration verification standards, and internal standards), performance evaluation samples with known quantities of dioxin and/or CB Congeners are routinely sent to the laboratories for analysis with field samples. This allows us to compare the PE sample results with field sample results and evaluate the laboratories accuracy and precision in the analysis.

Part 2 – Regional Data Review

Once the laboratory has analyzed the field samples and sent the data to the USEPA regional data reviewer, the data is reviewed using the National Functional Guidelines (NFGs) regional data review procedures. By using the NFGs, data is reviewed in a consistent manner across USEPA regions. In some applications, the NFG's may be used as a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. For example, areas where the application of specific SOPs is possible are primarily those in which definitive performance criteria are established. These criteria are concerned with specifications that are not sample dependent; they specify performance requirements that should fully be under a laboratory's control. These specific areas include blanks, calibration standards, Performance Evaluation (PE) standard materials, and instrument performance checks.

The NFGs are intended to assist in the technical review of analytical data generated through the DLM02.0 SOW. Determining contract compliance is not the intended objective of these guidelines. The data review process provides information on analytical limitations of data, based on specific Quality Control (QC) criteria. To provide more specific usability statements, the reviewer must have a complete understanding of the intended use of the data. The NFGs encourage data reviewers to use professional judgment when determining whether data is technically viable and contractually compliant.

A contract laboratory submitting data which are out of specification may be required to rerun samples or resubmit data, even if previously submitted data have been utilized due to program needs. Data which do not meet specified requirements are never fully acceptable. The only exception to this condition is in the area of the requirements for

Analytical quality control and assurance

individual sample analysis; if the nature of the sample itself inhibits the attainment of specifications, appropriate allowances must be made.

Part 3 – Meeting Project Specific Requirements

One of the main benefits of this program is that the standard SOW's can be adjusted to meet the regional data user needs. For example, a recent project required dioxin, CB Congener, and Pesticide analysis of XAD-2 and GFF samples by HR-GCMS. Since this type of analysis (especially the pesticide) was not routinely completed under our program, each laboratory was required to provide an SOP for review detailing how samples would be extracted and analyzed. The laboratory awarded this project was able to show that they had procedures in place to carry out this specialized extraction and analysis. As a result, USEPA was able to obtain the services they needed using the standardized methodology available through this program.

Table 4 lists some of the sites we have provided analytical data for which site decisions have been made. Where appropriate, specific modifications to our SOW are noted, an indication of the flexibility of our program to meet customer needs.

Site Name	Analysis	Modifications to SOW
Passaic River	Dioxin, CB Congener, Pesticides	Addition of Pesticide analyte list, extraction of XAD-2 and GFF samples procedure added.
Hurricane Katrina Relief Effort	Dioxin	Several quick turnaround time projects funded by FEMA for 7, 14, and 21 day TAT (vs. standard 35 day TAT)
Upper Columbia River	Dioxin, CB Congener	Standard analyses; Large quantities of fish, sediment, and water samples being analyzed over 2 year period.
Amco Site	Dioxin	Standard analyses; Routine monitoring analysis being conducted periodically over the last few years.

The overarching goal of the program is to provide data of known and documented quality to support site decisions. We plan to continue to work with the laboratories and Regional customers to continue to improve our services, including a new electronic data deliverable (the Staged Electronic Data Deliverable (SEDD), a universal .xml format) and keeping the analytical methods on the cutting edge of technology.

References

1. EPA. DLM02.0 SOW, May 2005, Exhibits E-H, <http://www.epa.gov/superfund/programs/clp/dlm2.htm>
2. EPA. CBC01.0 SOW, May 2005, Exhibits E-H, <http://www.epa.gov/superfund/programs/clp/cbc1.htm>.
3. EPA National Functional Guidelines for Chlorinated Dioxin/Furan Data Review. EPA-540-R-05-001, 2005, <http://www.epa.gov/superfund/programs/clp/guidance.htm#dioxin>
4. EPA. Method 1613, Revision B. 1994, 40 CFR Part 136, EPA-821-F-97-008.
5. EPA. Method 1668, Revision A. 1999, 40 CFR Part 136, EPA-821-R-00-002