



Multi-Media Dioxin and Furan Analytical Service for Superfund (DLM01.4)

Office of Emergency and Remedial Response
Analytical Operations/Data Quality Center (5204G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites are a series of chlorinated dibenzo-p-dioxins (CDDs) and dibenzofurans (CDFs) that are analyzed using High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS). The Analytical Operations/Data Quality Center (AOC) of EPA's Office of Emergency and Remedial Response (OERR) offers an analytical service that provides data from the analysis of water, soil, sediment, sludge, tissue (not 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 analytical service produces data of known and documented quality.

DESCRIPTION OF SERVICES

The dioxin/furan non-routine analytical service provides a flexible contractual framework for laboratories to apply EPA analytical methods for the isolation, detection, and quantitative measurement of 17 2,3,7,8-substituted tetra- through octa-chlorinated dibenzo-p-dioxins (CDDs) and dibenzofurans (CDFs) in water, soil, sediment, sludge, tissue (no human tissue), ash, oil, and oily matrices. EPA AOC has prequalified laboratories that use the Dioxin/Furan Statement of Work (SOW) DLM01.4 to provide this service. Data evaluation can be performed by the data requestor using National Functional Guidelines provided by EPA AOC. The standard data Turn-around Time (TAT) for this service is 35 days after laboratory receipt of the last sample in the Sample Delivery Group (SDG). This TAT can be changed to meet project-specific requirements.

REQUESTING THIS FLEXIBLE SERVICE

This service can be requested by EPA Regions and other interested parties by submitting a Task Order to EPA AOC. These Task Orders can modify the SOW to meet project-specific requirements (e.g., changes in TAT, detection limits, analyte lists, etc.). The SOW and National Functional Guidelines can be accessed at www.epa.gov/superfund/programs/clp/dlm1.htm.

DATA USES

This analytical service provides data that EPA uses for a variety of purposes such as: determining the nature and extent of contamination at a hazardous waste site; assessing priorities for response based on risks to human health and the environment; determining appropriate clean-up actions; and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including: site inspections; Hazard Ranking System (HRS) scoring; remedial investigation/feasibility studies; remedial design; treatability studies; and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

TARGET COMPOUNDS

The compounds and quantitation limits for which this service is applicable are listed in **Table 1**. For water samples, the lowest reportable quantitation limit is 10 pg/L. For solid samples, the lowest reportable quantitation limit is 1.0 ng/Kg. The specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

METHODS AND INSTRUMENTATION

For water samples, the stable isotopically labeled analogs of 15 of the 2,3,7,8-substituted CDDs/CDFs are spiked into a 1 L sample. Samples with no visible particles are extracted with methylene chloride in a separatory funnel or vacuum-filtered through a glass-fiber filter on top of a solid-phase extraction disk.

Table 1. Target Compound List and Contract Required Quantitation Limits (CRQLs)		
CDD/CDF	Water (pg/L)	Solids (ng/Kg)
2378-TCDD	10	1.0
12378-PeCDD	50	5.0
123678-HxCDD	50	5.0
123478-HxCDD	50	5.0
123789-HxCDD	50	5.0
1234678-HpCDD	50	5.0
OCDD	100	10
2378-TCDF	10	1.0
12378-PeCDF	50	5.0
23478-PeCDF	50	5.0
123678-HxCDF	50	5.0
123789-HxCDF	50	5.0
123478-HxCDF	50	5.0
234678-HxCDF	50	5.0
1234678-HpCDF	50	5.0
1234789-HpCDF	50	5.0
OCDF	100	10

Samples containing visible particles are vacuum-filtered through a glass-fiber filter, the filter is extracted in a Soxhlet/Dean-Stark (SDS) extractor, and the filtrate is extracted with methylene chloride in a separatory funnel.

For soil/sediment samples, the labeled compounds are spiked into a sample containing 10 g (dry weight) of soil/sediments. The soil/sediments are then extracted in an SDS extractor.

For fish and other tissue, a 20 g aliquot of frozen or non-frozen sample is homogenized and a 10 g aliquot is spiked with the labeled compounds. The frozen sample is mixed with sodium sulfate, allowed to dry overnight, and extracted for 12 to 24 hours using methylene

chloride:hexane in a Soxhlet extractor. The non-frozen sample is allowed to equilibrate, then hydrochloric acid and methylene chloride:hexane are added and the bottle is agitated for 12 to 24 hours. In both cases, the extract is evaporated to dryness and the lipid content is determined.

For all samples, the extracts are cleaned and injected with two internal standards to determine percent recoveries of the CDD/CDF congeners. An aliquot of the extract is injected into the High Resolution Gas Chromatograph (HRGC), the analytes are separated by the HRGC and detected by a High Resolution Mass Spectrometer (HRMS). **Table 2** summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include the hardcopy data reporting forms and supporting raw data. Electronic (diskette) deliverables are specified in the Task Order. The laboratory must submit data to EPA within 35 days after laboratory receipt of the last sample in the SDG, or as stated in the Task Order. The EPA Regions then review the data based on project-specific requirements and the National Functional Guidelines.

QUALITY ASSURANCE

The Quality Assurance (QA) process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of the quality required.

During the planning of the data collection program, QA activities focus on defining data and designing a Quality Control (QC) system to measure the quality of data being collected. During the implementation of the data collection effort, QA activities ensure that the QC system is functioning effectively, and the deficiencies uncovered by the QC system are corrected.

After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions.

Each contract laboratory prepares a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, and functional guidelines, as well as the QA/QC activities designed to achieve the data quality requirements for this analytical service.

Table 2. Methods and Instruments

Fraction	Preparation Method	Analytical Instrument
Water - no visible particles	Solid-phase extraction or extraction with methylene chloride in a separatory funnel.	HRGC/HRMS analysis
Water - visible particles	Solid-phase extraction or vacuum filtration/ filter extraction in an SDS extractor. Filtrate extraction with methylene chloride in a separatory funnel.	HRGC/HRMS analysis
Soil/Sediment	Extraction in an SDS extractor.	HRGC/HRMS analysis
Fish and other tissue	Mixed with sodium sulfate, extraction with methylene chloride:hexane in Soxhlet extractor or mixed with hydrochloric acid and methylene chloride:hexane, agitation for 12-24 hours.	HRGC/HRMS analysis

Table 3. Quality Control

QC Operation	Frequency
Initial Calibration	Upon contract award, initial setup of each instrument used, and each time continuing calibration fails to meet the acceptance criteria.
Continuing Calibration Verification	Every 12 hours for each instrument used for analysis and at end of a run.
Internal Standards	Added to all extracts prior to analysis.
Performance Evaluation (PE) Samples	Prepared and analyzed (if provided) with each set of 20 field samples.
Laboratory Control Sample (LCS)	Prepared and analyzed with each group of 20 field samples of a similar matrix in an SDG.
Method Blank	Prepared with each group of 20 field samples or less, or each time samples are extracted.
Window Defining Mixture	Every 12 hours for each instrument used for analysis; precedes Initial and Continuing Calibration.
HRMS System Tune	Every 12 hours.
Isomer Specificity Check	Every 12 hours; may be combined with Window Defining Mixture.
Clean-up Standard	Added to all extracts prior to cleanup.
Gel Permeation Chromatography (GPC) Calibration (optional)	Upon initial setup of instruments, when GC column changed, when channeling occurs, and once every 7 days when samples are cleaned using GPC.

QUALITY CONTROL

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analysis are shown in **Table 3**. A number of optional cleanup procedures are available for this SOW.

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by AOC and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, HRGC/HRMS tape audits, and evaluates laboratory performance through the use of blind performance evaluation samples.

For more information, or to submit suggestions to improve this analytical service, please contact:

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