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Multi-Media, Multi-Concentration Chlorinated Biphenyl Congeners Analytical Service for Superfund (CBC01.2)

Office of Superfund Remediation and Technology Innovation (OSRTI)
Analytical Services Branch (5203P)

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 Biphenyl (CB) Congeners that are analyzed using High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS). The Analytical Services Branch (ASB) of EPA'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 CB congeners analytical service produces data of known and documented quality.

DESCRIPTION OF SERVICES

The CB congeners analytical service provides a flexible contractual framework for laboratories to apply EPA analytical methods for the isolation, detection, and quantitative measurement of 209 2,2',3,3',4,4',5,5',6,6'-substituted mono- through deca-CB congeners in water, soil, sediment, sludge, non-human tissue, ash, oil, and oily matrices. EPA ASB has prequalified laboratories that use the CB congeners Statement of Work (SOW) CBC01.2 to provide this service. The standard data Turnaround 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

The CB congeners analytical service can be requested by EPA Regions and other interested parties by submitting a Task Order (TO) to EPA ASB. These TOs can modify the SOW to meet project-specific requirements [e.g., changes in TAT, detection limits, or Target Compound Lists (TCLs)]. The CB congeners SOW can be accessed at:

http://www.epa.gov/superfund/programs/clp/cbc1.htm

DATA USES

The CB congeners 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 cleanup 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 applicable target compounds and Contract Required Quantitation Limits (CRQLs) for this service are listed in **Table 1**. For water samples, the CRQL is 20 pg/L. For solids and tissue samples, the CRQL is 2.0 ng/kg. For extracts, the CRQL is 1.0 pg/µL. The specific CRQLs are highly matrix-dependent. The CRQLs listed herein are provided for guidance and may not always be achievable.

METHODS AND INSTRUMENTATION

For water samples, the stable isotopically-labeled analogs of the Toxics and labeled Level of Chlorination (LOC) CB congeners are spiked into a 1 L sample. The sample is extracted using solid-phase extraction (SPE), separatory funnel extraction (SFE), or continuous liquid/liquid extraction (CLLE).

For soil/sediment samples, the labeled compounds are spiked into a sample containing 10 g (dry weight) of solids and extracted for 16-24 hours with toluene in a Soxhlet/Dean-Stark (SDS) extractor. The extract is concentrated for cleanup.

For non-human tissue samples, a 20 g aliquot of a sample is homogenized, and a 10 g aliquot is spiked with the labeled compounds. The sample is mixed with anhydrous sodium sulfate, allowed to dry for 12-24 hours, and extracted for 18-24 hours using methylene chloride in an SDS extractor. The extract is evaporated to near dryness, and the lipid content is determined.

For all samples, the extracts are cleaned and concentrated to 20 μL. Labeled internal standards are added to each extract and an aliquot of the extract is injected into the HRGC. The analytes are separated by the HRGC and detected by an HRMS. **Table 2** summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include hardcopy data reporting forms and supporting raw data. Laboratories must also submit the data electronically, referred to as an Electronic Data Deliverable (EDD), within the contract required TAT. Additional information about EDD requirements are provided in Exhibit H of the SOW, located at:

http://www.epa.gov/superfund/programs/clp/cbc1.htm.

EPA then processes the EDD through a web-based data assessment tool – the Electronic Data eXchange and Evaluation System (EXES). EXES provides data users with electronic data assessment/usability reports and spreadsheets within 24 to 48 hours of data receipt. EXES reports also facilitate the transfer of analytical data into client databases. In addition to the data assessment/usability reports, laboratories are provided with a data assessment report documenting instances of noncompliance.

QUALITY ASSURANCE (QA)

The 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 an environmental data collection program, QA activities focus on defining data quality criteria and designing a Quality Control (QC) system to measure the quality of data being generated. 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.

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 in the SOW.

CB Congener[1]	Congener Number	CAS Number	CRQL[4]		
			Water (pg/L)	Other (ng/kg)	Extract (pg/uL)
2,4'-DiCB[2]	PCB-8	34883-43-7	20	2.0	1.0
2,2',5-TrCB[2]	PCB-18	37680-65-2	20	2.0	1.0
2,4,4'-TrCB[2]	PCB-28	7012-37-5	20	2.0	1.0
2,2',3,5'-TeCB[2]	PCB-44	41464-39-5	20	2.0	1.0
2,2',5,5'-TeCB[2]	PCB-52	35693-99-3	20	2.0	1.0
2,3',4,4'-TeCB[2]	PCB-66	32598-10-0	20	2.0	1.0
3,3',4,4'-TeCB[2,3]	PCB-77	32598-13-3	20	2.0	1.0
3,4,4',5-TeCB[3]	PCB-81	70362-50-4	20	2.0	1.0
2,2',4,5,5'-PeCB[2]	PCB-101	37680-73-2	20	2.0	1.0
2,3,3',4,4'-PeCB[2,3]	PCB-105	32598-14-4	20	2.0	1.0
2,3,4,4',5-PeCB[3]	PCB-114	74472-37-0	20	2.0	1.0
2,3',4,4',5-PeCB[2,3]	PCB-118	31508-00-6	20	2.0	1.0
2',3,4,4',5-PeCB[3]	PCB-123	65510-44-3	20	2.0	1.0
3,3',4,4',5-PeCB[2,3]	PCB-126	57465-28-8	20	2.0	1.0
2,2',3,3',4,4'-HxCB[2]	PCB-128	38380-07-3	20	2.0	1.0
2,2',3,4,4',5'-HxCB[2]	PCB-138	35065-28-2	20	2.0	1.0
2,2',4,4',5,5'-HxCB[2]	PCB-153	35065-27-1	20	2.0	1.0
2,3,3',4,4',5-HxCB[3]	PCB-156	38380-08-4	20	2.0	1.0
2,3,3',4,4',5'-HxCB[3]	PCB-157	69782-90-7	20	2.0	1.0
2,3',4,4',5,5'-HxCB[3]	PCB-167	52663-72-6	20	2.0	1.0
3,3',4,4',5,5'-HxCB[2,3]	PCB-169	32774-16-6	20	2.0	1.0
2,2',3,3',4,4',5-HpCB[2]	PCB-170	35065-30-6	20	2.0	1.0
2,2',3,4,4',5,5'-HpCB[2]	PCB-180	35065-29-3	20	2.0	1.0
2,2',3,4',5,5',6-HpCB[2]	PCB-187	52663-68-0	20	2.0	1.0
2,3,3',4,4',5,5'-HpCB[3]	PCB-189	39635-31-9	20	2.0	1.0
2,2',3,3',4,4',5,6-OcCB[2]	PCB-195	52663-78-2	20	2.0	1.0
2,2',3,3',4,4',5,5',6-NoCB[2]	PCB-206	40186-72-9	20	2.0	1.0
DeCB[2]	PCB-209	2051-24-3	20	2.0	1.0
All other non-dioxin-like congeners	-	-	20	2.0	1.0

Abbreviations for chlorination levels:

MoCB = monochlorobiphenyl
DiCB = dichlorobiphenyl
TrCB = trichlorobiphenyl
TeCB = tetrachlorbiphenyl
PeCB = pentachlorobiphenyl
HxCB = hexachlorobiphenyl
HpCB = heptachlorobiphenyl
OcCB = octachlorobiphenyl
NoCB = nonachlorobiphenyl
DeCB = decachlorobiphenyl

2. National Oceanic and Atmospheric Administration (NOAA) Congener of Interest.

3. World Health Organization (WHO) Toxic Congener.

4. The CRQLs in this table are equivalent to the concentration of the low calibration (CS1) standard, assuming that all sample weight, volumes, and cleanup procedures are performed according to Exhibit D of this SOW.

   Specific quantitation limits are highly matrix-dependent. The quantitation limit listed herein is provided for guidance and may not always be achievable.

   These CRQL values are based on the analysis of samples according to the specifications given in Exhibit D. Sample data are reported on a dry weight basis for all non-aqueous samples [except tissues, which are reported on a wet weight basis, along with their Percent Lipid (% Lipid) content].

Table 2. Methods and Instruments

Matrix	Preparation Method	Analytical Instrument
Water	**Using Solid-Phase Extraction (SPE):** Vacuum-filtered through glass fiber on top of an SPE disk; fiber and disk extraction. **Using Separatory Funnel Extraction (SFE):** Filtrate extraction with methylene chloride using a separatory funnel. **Using Continuous Liquid-Liquid Extraction (CLLE):** Filtrate extraction with methylene chloride using a continuous liquid-liquid extractor.	HRGC/HRMS
Soil/Sediment	Extraction with toluene in a Soxhlet/Dean-Stark (SDS) extractor.	HRGC/HRMS
Non-Human Tissue	Extraction with methylene chloride using an SDS extractor for 18-24 hours.	HRGC/HRMS

Table 3. Quality Control (QC)

QC	Frequency
ANALYSES	
HRMS System Tune	Prior to the analysis of calibration standards, samples, and blanks within each 12-hour period.
Window-Defining Mixture (WDM)	Following the HRMS system tune and at the beginning of each 12-hour period for each instrument used for analysis during which standards or samples are analyzed; precedes initial calibration, continuing calibration, and whenever adjustments or instrument maintenance activities are performed that may affect Retention Times (RTs).

Table 3. Quality Control (QC) (Con't)

QC Operation	Frequency
Initial Calibration	Upon contract award, during initial setup of each instrument used prior to analysis of samples and required blanks, and each time calibration verification fails to meet the technical acceptance criteria.
Continuing Calibration Verification (CCV): Mid-Point Calibration Standard (CS3) Relative Response (RR) and Relative Response Factor (RRF)	At the beginning of each 12-hour period after the HRMS system tune during which sample data are collected and at the end of each 12-hour period.
STANDARDS	
Labeled Toxic and Level of Chlorination (LOC) Chlorinated Biphenyls (CBs)	Added to all samples including blanks, LCSs, and PE samples prior to extraction.
Cleanup Standards	Added to all extracts prior to cleanup.
Internal Standards	Added to all extracts prior to analysis.
BLANKS	
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 or less of a similar matrix in an SDG. LCS analysis precedes analysis of samples from the same SDG.
Method Blank	Prepared and analyzed with each group of 20 field samples or less, or each time samples are extracted to determine the level of contamination associated with the processing and analysis of samples. Method Blank analysis precedes analysis of samples from the same SDG.
INSTRUMENT CALIBRATION	
Gel Permeation Chromatography (GPC) Calibration (optional)	Upon contract award, upon initial setup of instruments, when the Gas Chromatography (GC) column is changed, when channeling occurs, and once every 7 days when samples are cleaned using GPC.

PERFORMANCE MONITORING ACTIVITIES

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

CONTACTING EPA

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

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