STATE OF NEW MEXICO
NEW MEXICO WATER QUALITY CONTROL COMMISSION

In the Matter of:
PROPOSED AMENDMENTS TO STANDARDS FOR INTERSTATE AND INTRASTATE WATERS, 20.6.4 NMAC

No. WQCC 20-51 (R)

AMIGOS BRAVOS' NOTICE OF INTENT TO PRESENT REBUTTAL TESTIMONY

Pursuant to 20.6.1.202.A NMAC and the Procedural Order issued in this matter, Amigos Bravos hereby files its Notice of Intent to Present Rebuttal Testimony. As required by the applicable regulations and Procedural Order, Amigos Bravos provides the following information in this notice:

1. Identify the person for whom the witnesses will testify:

The witnesses identified below, Rachel Conn; Jamie C. DeWitt, Ph.D., DABT; David Hope; and Ann Bailey, M.S., will testify on behalf of Amigos Bravos, a New Mexico non-profit water conservation organization dedicated to protecting and restoring the waters of the state.

2. Identify each technical witness the person intends to present for rebuttal testimony, and state the qualifications of that witness, including a description of their educational and work background:

Amigos Bravos intends to present:

- Rachel Conn, Deputy Director for Amigos Bravos, whose educational and work background is set forth in her resume, which is Amigos Bravos' Exhibit 2;
- Jamie C. DeWitt, Ph.D., DABT, Associate Professor in the Department of Pharmacology and Toxicology of the Brody School of Medicine at East Carolina University, whose educational and work background is set forth in her curriculum vitae, which is Amigos Bravos' Exhibit 8;

- David Hope, whose educational and work background is set forth in his curriculum vitae, which is Amigos Bravos' Exhibit 18; and
- Ann K. Bailey, M.S, whose educational and work background is set forth in her curriculum vitae, which is Amigos Bravos' Exhibit 21.

3. Include a copy of the rebuttal testimony of each technical witness in narrative form, and state the estimated duration of the direct oral testimony of that witness:

As required by the Procedural Order, ¶ 3, Amigos Bravos submits the full written rebuttal testimony of:

- Ms. Conn in Exhibit 11,
- Dr. DeWitt in Exhibit 17,
- Mr. Hope in Exhibit 19, and
- Ms. Bailey in Exhibit 22.

Each witness will limit their oral rebuttal testimony at hearing to a summary of their testimony not to exceed 30 minutes, as provided for in the Procedural Order, ¶ 3.

4. Include the text of any recommended modifications to the proposed regulatory change:

A text of the modifications to 20.6.4 NMAC proposed by Amigos Bravos is attached as Exhibit 10 (and revised from the modifications proposed by Amigos Bravos in its Notice of Intent to Present Direct Testimony and filed as Exhibit 1).

5. List and attach all exhibits anticipated to be offered by that person at the hearing:

Below is a list of all direct and rebuttal exhibits to be offered by Amigos Bravos in support of its direct testimony. Amigos Bravos' direct exhibits were filed May 3, 2021. Amigos Bravos' rebuttal exhibits are attached. Both sets of exhibits have a table of contents, accessible

by clicking on the "bookmarks" tab in Adobe Acrobat. Amigos Bravos reserves the right to offer sur-rebuttal exhibits.

Exhibit	Description
	DIRECT
Ex. 1	Amigos Bravos' Proposed Amendments to 20.6.4 NMAC
Ex. 2	Resume of Rachel Conn
Ex. 3	Direct Testimony of Rachel Conn
Ex. 4	USGS-NMED PFAS Sampling Results – Surface Water (Aug.-Sept. 2020)
Ex. 5	Amigos Bravos Valle De Oro Community Water Quality Sampling Results
Ex. 6	NMED - Pharmaceuticals in Water (July 28, 2017)
Ex. 7	Dec. 19, 2006 letter from R. Conn, Amigos Bravos, to S. Barash, EPA, re Effluent-Dependent Waters Strawman Document
Ex. 8	Curriculum Vitae of Jamie C. DeWitt
Ex. 9	Direct Testimony of Jamie C. DeWitt, Ph.D., DABT
	REBUTTAL
Ex. 10	Amigos Bravos' [Revised] Proposed Amendments to 20.6.4 NMAC
Ex. 11	Rebuttal Testimony of Rachel Conn
Ex. 12	NMED Certification of LANL Wastewater Permit
Ex. 13	Draft EPA Wastewater Permit for LANL [portions]
Ex. 14	NMED Certification of LANL Stormwater Permit
Ex. 15	Draft EPA Stormwater Permit for LANL [portions]
Ex. 16	List of Hydrology Protocol Scores
Ex. 17	Rebuttal Testimony of Jamie C. DeWitt, Ph.D., DABT
Ex. 18	Curriculum Vitae of David Hope
Ex. 19	Rebuttal Testimony of David Hope
Ex. 20	Method 1668C Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, U.S. EPA Office of Water (April 2010)
Ex. 21	Curriculum Vitae of Ann K. Bailey, M.S.
Ex. 22	Rebuttal Testimony of Ann K. Bailey, M.S.
Ex. 23	77 Fed. Reg. 29,758, 29,763 (May 18, 2012)

Respectfully submitted,

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Certificate of Service

I certify that a copy of the foregoing pleading was emailed to the following counsel on June 22, 2021:

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AMIGOS BRAVOS' EXHIBIT 10

AMIGOS BRAVOS' [REVISED] PROPOSED AMENDMENTS TO 20.6.4 NMAC[1]

Climate Change

Amigos Bravos proposes to add the following at 20.6.4.6.C NMAC and to delete the New Mexico Environment Department's (NMED) proposed 20.6.4.6.D NMAC:

20.6.4.6 OBJECTIVE:

. . .

C. A further purpose of these surface water quality regulations is to address the inherent threats to water quality due to climate change. The quality of New Mexico surface waters is being affected by climate change. New Mexico's climate is getting hotter and drier, resulting in earlier springs, hotter summers, and less predictable winters. New Mexico is experiencing more intense droughts and a greater proportion of precipitation falling as rain instead of snow. Snowpack is shrinking and earlier snowmelts contribute to lower stream flows at critical times of the year when the reduced availability of water has greater environmental consequences. Increased water temperatures resulting from increased air temperatures tend to lead to lower levels of dissolved oxygen in water, resulting in increased stress on the fish, insects, crustaceans and other aquatic animals that rely on oxygen. More intense precipitation events and increased evaporation rates lead to increased runoff and more pollution, including increased nutrients sediment, and salt that wash into surface waters. Development of New Mexico surface water quality standards should take into account the importance of protecting of water quality in light of climate change.

D.~~C.~~ Pursuant to Subsection A of Section 74-6-12 NMSA 1978, this part does not grant to the water quality control commission or to any other entity the power to take away or modify property rights in water.

~~D. These surface water quality standards serve to address the inherent threats to water quality due to climate change.~~

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[1] Amigos Bravos' proposed changes to the regulations are shown in blue underline, the New Mexico Environment Department's proposed changes are in red, and the existing regulations are in black. Amigos Bravos proposes the proposed change in green underline offered by Communities for Clean Water.

Amigos Bravos proposes to amend NMED's proposed definition of "climate change" at 20.6.4.7.C(4) NMAC as follows:

20.6.4.7 DEFINITIONS: Terms defined in the New Mexico Water Quality Act, but not defined in this part will have the meaning given in the Water Quality Act.

. . .

C. Terms beginning with the letter "C".

. . .

(4) "Climate change" refers to any significant change in the measures of climate lasting for an extended period of time, typically decades or longer, and includes major changes in temperature, precipitation, wind patterns or other weather-related effects. Climate change may be due to natural processes or human-caused changes of the atmosphere, or a combination of the two. Humans are largely responsible for recent climate change.

Contaminants of Emerging Concern

Amigos Bravos proposes to amend NMED's proposed definition of "contaminants of emerging concern" at 20.6.4.7.C(8) NMAC[2] as follows:

20.6.4.7 DEFINITIONS

. . .

C. Terms beginning with the letter "C".

. . .

(8) "Contaminants of emerging concern" or "CECs" refer to water contaminants including, but not limited to, per- and polyfluoroalkyl substances, pharmaceuticals and personal care products that may cause significant ecological or human health effects at low concentrations and are not already considered "toxic pollutants" by the department. CECs are generally chemical compounds that, although suspected to potentially have impacts, may not have regulatory standards, and the concentrations to which negative impacts are observed have not been fully studied.

Amigos Bravos proposes adding the following at 20.6.4.14.F NMAC:

20.6.4.14 SAMPLING AND ANALYSIS

. . .

F. The department may include sampling and monitoring of contaminants of emerging concern as a condition in a federal permit under Section 401 of the federal Clean Water Act.

[2] NMED proposed adding this definition at 20.6.4.7.C**(7)** NMAC, after the definition of "coldwater" at 20.6.4.7.C(5) NMAC and before the definition of "coolwater" at 20.6.4.7.C(6) NMAC, but the definitions are ordered alphabetically, and therefore a new definition for "contaminations of emerging concern" should be placed after the definition of "commission" at 20.6.4.7.C(7) NMAC and "criteria" at 20.6.4.7.C(8) NMAC. Amigos Bravos proposes the definition for "contaminants of emerging concern" to be placed at 20.6.4.7.C**(8)** NMAC, taking into account NMED's new proposed definition for "climate change," which Amigos Bravos supports.

Baseflow and Effluent Dominated

Amigos Bravos proposes that the Commission **not** adopt NMED's new proposed definitions for "baseflow" at 20.6.4.7.B(1) NMAC and "effluent dominated" at 20.6.4.7.E(2) NMAC:

20.6.4.7 DEFINITIONS

. . .

B. Terms beginning with the letter "B".

~~**(1) "Baseflow"** refers to the sustained flow volume of a stream or river. In natural systems, baseflow is comprised from regional groundwater inflow and local shallow subsurface inflow that is temporarily stored in the watershed during snowmelt and rain events and slowly released to the stream or river over time. In effluent dominated systems, baseflow is comprised predominantly from effluent with limited subsurface contributions. Baseflow in both scenarios is critical for sustaining flow in streams and rivers over seasonal and longer timeframes.~~

. . .

E. Terms beginning with the letter "E".

. . .

~~**(2) "Effluent dominated"** refers to a water that has, over a 12-month average, more than three-quarters of its baseflow attributed to discharges from a permitted effluent discharge. Waters that are effluent dominated are of significant value by providing aquatic life and wildlife habitat.~~

Alternatively, Amigos Bravos proposes that the Commission **not** adopt NMED's new proposed definition for "effluent dominated" at 20.6.4.7.E(2) NMAC, and amend NMED's proposed definition for "baseflow" at 20.6.4.7.B(1) NMAC as follows:

20.6.4.7 DEFINITIONS

. . .

B. Terms beginning with the letter "B".

(1) "Baseflow" refers to the sustained flow volume of a stream or river. In natural systems, baseflow is comprised from regional groundwater inflow and local shallow subsurface inflow that is temporarily stored in the watershed during snowmelt and rain events and slowly released to the stream or river over time. ~~In effluent dominated systems, baseflow is comprised predominantly from effluent with limited subsurface contributions. Baseflow in both scenarios is critical for sustaining flow in streams and rivers over seasonal and longer timeframes.~~

Existing Use

Amigos Bravos proposes to amend the current definition of "existing use" as follows:

20.6.4.7 DEFINITIONS

. . .

E. Terms beginning with the letter "E".

. . .

(3) "Existing use" means a use actually attained in a surface water of the state on or after November 28, 1975, whether or not it is a designated use. An existing use can be established by demonstrating that fishing, swimming, or other uses have actually occurred since November 28, 1975; or that the water quality is suitable to allow the use to be attained.

Limited Aquatic Life

Amigos Bravos proposes to delete NMED's proposed changes to the definition of "limited aquatic life," proposing to replace the "ephemeral or intermittent water" with the term "low-flow," and to retain the current definition at 20.6.4.7.L(2):

20.6.4.7

. . .

L. Terms beginning with the letter "L".

(2) "Limited aquatic life" as a designated use, means the surface water is capable of supporting only a limited community of aquatic life. This subcategory includes surface waters that support aquatic species selectively adapted to take advantage of naturally occurring rapid environmental changes, [~~ephemeral or intermittent water,~~] ephemeral or intermittent, ~~low-flow~~, high turbidity, fluctuating temperature, low dissolved oxygen content or unique chemical characteristics.

AMIGOS BRAVOS' EXHIBIT 11

REBUTTAL TESTIMONY OF RACHEL CONN

I. QUALIFICATIONS

My name is Rachel Conn, and I am the Deputy Director for Amigos Bravos, a non-profit water conservation organization dedicated to protecting and restoring the waters of New Mexico. My educational and work background is set forth in Amigos Bravos' Exhibit 2, and in the direct testimony I provided in this matter. *See* Conn Dir. Test. at 1-2 [AB Ex. 3].

II. ADOPTING LANL'S PROPOSALS LIMITING MONITORING METHODS AND THE DEFINITION OF "TOXIC POLLUTANTS" WOULD ALLOW LANL TO EVADE EFFECTIVE MONITORING OF PCBs AND ALL MONITORING FOR PFAS AND WOULD WEAKEN SURFACE WATER QUALITY PROTECTIONS FOR THE STATE

A series of proposals for the Los Alamos National Laboratory ("LANL") from Triad National Security, LLC and the U.S. Department of Energy would severely weaken the Commission's surface water quality standards and the protections they provide to the state's waters and human health and the environment. First, LANL proposes in 20.6.4.14.A NMAC to limit sampling and analysis for purposes of permit compliance and enforcement to methods approved in 40 CFR Part 136 ("Part 136 Methods"). Second, LANL proposes in 20.6.4.7.T(2) NMAC to limit the definition of "toxic pollutants" to the U.S. Environmental Protection Agency's ("EPA") list of toxic pollutants. The combination of these proposals would allow LANL to evade effective monitoring for polychlorinated biphenyls ("PCBs"), evade all monitoring of per- and polyfluoroalkyl substances ("PFAS"), and significantly limit the New Mexico Environment Department's ("NMED") authority to protect New Mexico's surface waters, and should be rejected.

According to EPA:

> PCBs have been demonstrated to cause a variety of adverse health effects. They have been shown to cause cancer in animals as well as a number of serious non-

> cancer health effects in animals, including: effects on the immune system, reproductive system, nervous system, endocrine system and other health effects. Studies in humans support evidence for potential carcinogenic and non-carcinogenic effects of PCBs. The different health effects of PCBs may be interrelated. Alterations in one system may have significant implications for the other systems of the body.[1]

The Water Quality Control Commission ("Commission") has set numeric water quality standards for PCBs which include a standard of 0.014 micrograms per liter ("ug/L") for wildlife habitat, a chronic standard of 0.014 ug/L for aquatic life, and a human health organism only standard of 0.00064 ug/L for aquatic life. 20.6.4.900.J NMAC. The Commission has determined that PCBs are cancer-causing and persistent. *Id.*

According to both Amigos Bravos' experts with expertise in monitoring pollutants, David Hope and Ann Bailey, EPA Method 608.3 (measuring Arochlors), which is a Part 136 Method, is not sufficiently sensitive or sufficiently specific to detect total PCBs at the level of the Commission's numeric standards identified above. However, according to them both, EPA Method 1668C (measuring congeners), which is not a Part 136 Method, does have the sensitivity to detect PCBs at the Commission's numeric limits. Hope Reb. Test. at 3-7 [AB Ex. 19]; Bailey Reb. Test. at 2-5 [AB Ex. 22].

According to Amigos Bravos' toxicological expert, Dr. Jamie DeWitt, whose expertise includes investigation, monitoring, and setting standards for PFAS:

> PFAS that have been studied for their toxicity induce a wide variety of adverse health outcomes in experimental animal models. Epidemiological studies, or studies of people that have been exposed to PFAS through their occupations or from environmental sources such as drinking water, link PFAS exposure to similar adverse health outcomes. These toxicological and epidemiological studies indicate that exposure to PFAS poses a hazard to human health.

DeWitt Dir. Test. at ¶ 17 [AB Ex. 9].

[1] https://www.epa.gov/pcbs/learn-about-polychlorinated-biphenyls-pcbs#healtheffects.

In Dr. DeWitt's opinion, nine PFAS are "toxic pollutants" and others are contaminants of emerging concern ("CECs") that should be monitored. *Id.* at ¶¶ 2(i) & (ii), 29-31. The total number of chemicals classified as PFAS is estimated at nearly 10,000 individual substances. *Id.* at ¶ 13.

NMED has asserted its regulatory authority to require LANL to meet the Commission's numeric standards for PCBs and monitor PFAS, but LANL has objected. On November 30, 2020, pursuant to the Clean Water Act, the New Mexico Water Quality Act, and the Commission's regulations, NMED issued state certifications for two National Pollutant Discharge Elimination System ("NPDES") permits for LANL: Industrial Wastewater NPDES Permit No. NM0028355 ("LANL Wastewater Permit") [AB Ex. 12] and Individual Stormwater NPDES Permit No. NM0030759 ("LANL Stormwater Permit") [AB Ex. 14]. In New Mexico, EPA drafts and issues all NPDES permits while the State of New Mexico, through NMED, must certify that the permits meet state water quality standards. During the certification process, NMED will issue conditions to EPA's draft permits to ensure compliance with state standards.

NMED conditioned the LANL Wastewater Permit on monitoring and compliance of PCBs at a limit of 0.00064 ug/L and monitoring in accordance with EPA Method 1668C. NMED Cert. of LANL Wastewater Permit, p. 5 [AB Ex. 12]. LANL appealed this condition to the NMED Secretary in part on the ground that EPA Method 1668C (the congener method) is not a Part 136 Method, and insists that EPA Method 608.3 (the Aroclor method) be used even though it cannot detect PCBs at the Commission's standard of 0.00064 ug/L.

NMED also conditioned the LANL Wastewater Permit on monitoring 18 PFAS using EPA Method 537.1 and monitoring annually at locations where PFAS are detected above the New Mexico screening level. *Id.* at pp. 2-5. LANL appealed this condition in part on the ground

that the 18 PFAS are not listed "toxic pollutants" in 20.6.4.7.T(2) NMAC and EPA Method 537.1 is not a Part 136 Method. However, there are no constituents listed under the definition of "toxic pollutant" in 20.6.4.7.T(2) NMAC or in the narrative standard for toxic pollutants in 20.6.4.13.F NMAC. These provisions provide a narrative description.

NMED issued its certification on the LANL Stormwater Permit also on November 30, 2020, and LANL also appealed that certification to the NMED Secretary also on December 31, 2020. LANL appealed all ten conditions in NMED's certification including conditions setting a limit of 0.00064 ug/L for PCBs and requiring use of EPA Method 1668C to monitor, again challenging these conditions on the ground that EPA Method 1668C is not a Part 136 Method and again insisting that EPA Method 608.3 be used, even though that method cannot detect total PCBs at all the Commission's numeric limits.

LANL also challenged NMED's condition requiring monitoring of PFAS, again in part based on the ground that PFAS are not "toxic pollutants" and that the required monitoring method, EPA Method 537, is not a Part 136 Method.

Not content to rest on the legal arguments in its appeals, LANL now seeks relief from the Commission and proposes amendments to the state's water quality standards that would buttress its appeal and take away critical protections not only for LANL's surface waters but for all surface waters of the state.

In this proceeding, LANL proposes to amend 20.6.4.12.E and -14.A NMAC to limit sampling and analysis to Part 136 Methods for purposes of compliance. *See* LANL Ex. 1; LANL Ex. 7 at 5-10 (Toll Test.). Presently, the Commission's regulations at 20.6.4.14 NMAC allow sampling in accordance with methods approved by a number of reliable publications. NMED

asserts in its certifications that EPA Method 1668C (testing for PCB congeners) and EPA Method 537 (testing for PFAS) is allowed under various provisions of federal and state law.

From a policy perspective, states should have the authority and flexibility to select reliable sampling and analytical methods in order to ensure compliance with their water quality standards. In this case, the State of New Mexico could not ensure compliance with certain numeric water quality standards for PCB's if it does not have the flexibility to use EPA Method 1668C, a method Amigos Bravos' experts, Mr. Hope and Ms. Bailey, have relied upon for decades and find reliable.

It is important to point out that EPA's **current and draft** LANL Wastewater and Stormwater Permits require LANL to monitor for PCBs using EPA Method 1668C. *See* EPA Draft LANL Wastewater Permit [Ex. 13] and EPA Draft LANL Stormwater Permit [Ex. 15].[2] In EPA's view, therefore, Method 1668C is appropriate and lawful for use for permit compliance and enforcement even though it is not an approved Part 136 Method.

LANL further proposes to limit the definition of "toxic pollutants" at 20.6.4.7.T(2) NMAC to the EPA list of toxic pollutants. *See* LANL Ex. 1. EPA's list of toxic pollutants does not include any PFAS, even though it is widely recognized by the scientific community, including Amigos Bravos' expert Dr. DeWitt, that certain PFAS are toxic pollutants.

Amigos Bravos urges the Commission to reject LANL's transparent efforts to get out from under complying with the Commission's numeric water quality standards for PCB's and all monitoring requirements for PFAS, both of which are necessary to protect public health and the environment. Not only would LANL's proposals undermine the state's ability to protect our

[2] Amigos Bravos provides only the relevant portions of the draft permits, but can provide the entire drafts upon request.

surface waters from pollution from PCBs and PFAS on LANL property, LANL's proposals – limiting acceptable monitoring methods and limiting the list of toxic pollutants – are likely to have wide ranging and unforeseen impacts for the state's surface waters as a whole.

III. THE COMMISSION SHOULD ADD A DEFINITION FOR CONTAMINANTS OF EMERGING CONCERN AND AUTHORIZE NMED TO REQUIRE MONITORING FOR CECs

NMED has proposed to add a definition of "contaminants of emerging concern" or "CECs." LANL and the New Mexico Mining Association ("NMAA") object to NMED's proposal to add a CEC definition. Amigos Bravos supports adding a definition of CEC and proposes to give explicit authority to NMED to monitor for CECs in 20.6.4.14.F NMAC.

CECs are a widely accepted group of potentially harmful contaminants, including by EPA[3], and PFAS are recognized as CECs, including by EPA[4]. Yet, LANL and NMAA would have the Commission ignore this category of water pollutants and reject NMED's proposed definition CECs, further aiding its argument in its appeals that NMED does not have the authority to require monitoring for PFAS. The combination of LANL's proposal to limit toxic pollutants to EPA's list and its opposition to recognizing CECs would mean that the state is rendered powerless to monitor or limit pervasive and persistent contaminants in our surface waters.

Amigos Bravos supports including a definition of CECs in the Commission's regulations, and supports NMED's proposed language with two revisions to make it clear that (1) PFAS can

[3] *See, e.g.*, https://www.epa.gov/wqc/contaminants-emerging-concern-including-pharmaceuticals-and-personal-care-products; https://www.epa.gov/fedfac/emerging-contaminants-and-federal-facility-contaminants-concern.
[4] *See, e.g.*, https://www.epa.gov/newsreleases/epa-delivers-results-pfas-action-plan.

be CECs and (2) "toxic pollutants" and "CECs" represent different categories of contaminants. Amigos Bravos proposes at 20.6.4.7.C(8) NMAC:

> **20.6.4.7 DEFINITIONS**
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> . . .
>
> **C. Terms beginning with the letter "C".**
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> . . .
>
> **(8) "Contaminants of emerging concern" or "CECs"** refer to water contaminants including, but not limited to, per- and polyfluoroalkyl substances, pharmaceuticals and personal care products that may cause significant ecological or human health effects at low concentrations and are not already considered "toxic pollutants" by the department. CECs are generally chemical compounds that, although suspected to potentially have impacts, may not have regulatory standards, and the concentrations to which negative impacts are observed have not been fully studied.[5]

Dr. DeWitt supports both including a definition for CECs and including PFAS as an identified example of CECs. Including PFAS as an example is consistent with EPA's findings that PFAS are CECs numbering in the thousands.[6]. Amigos Bravos also proposes to clarify that CECs that have been identified as toxic pollutants should be treated as such and would, for example, be subject to the Commission's narrative standard for "toxic pollutants" at 20.6.4.13.F NMAC. For example, as Dr. DeWitt testifies, at least nine PFAS have been studied enough to determine that NMED should consider them "toxic pollutants," including the three PFAS compounds listed by the Commission as toxic pollutants in 20.6.2 NMAC. In addition, in the future there may be other individual CECs, such as certain pharmaceuticals and personal care products, where there is sufficient knowledge to determine that these contaminants are toxic to humans and wildlife in accordance with 20.6.4.13.F NAMC. If so, these constituents should be categorized as "toxic

[5] Amigos Bravos' proposed changes are shown in blue underline, NMED's proposed changes are in red, and the existing regulations are in black.

[6] USEPA, EPA Tools and Resources Webinar: Treating Contaminants of Emerging Concern (Mar. 21, 2019) at https://www.epa.gov/sites/production/files/2019-03/documents/2019-03-20_cec_treatment_state_webinar.pdf.

pollutants" and effluent limitations should be established for dischargers with a reasonable potential to discharge these pollutants. In these cases it would no longer be appropriate to consider them of "emerging concern."[7]

Amigos Bravos proposes, with the support of Dr. DeWitt, to expressly give NMED authority to monitor for CECs in permits. We proposed the following amendment:

> **20.6.4.14 SAMPLING AND ANALYSIS**
>
> . . .
>
> **F.** The department may include sampling and monitoring of contaminants of emerging concern as a condition in a federal permit under Section 401 of the federal Clean Water Act.

It is well-established that PFAS are contaminants of emerging concern. Despite the potential harm to human health and the environment, LANL objects to monitoring for them. Giving NMED the express authority to monitor for CECs would help ensure the state has the authority to require LANL and other dischargers to monitor for PFAS and as well as other CECs suspected to be harmful.

IV. THE COMMISSION SHOULD RECOGNIZE THAT CLIMATE CHANGE IS A THREAT TO THE STATE'S SURFACE WATERS, THAT IT SHOULD TAKE CLIMATE CHANGE INTO ACCOUNT WHEN DEVELOPING ITS REGULATIONS, AND THAT IT IS PRIMARILY HUMAN-CAUSED

NMED proposes to add to the objectives of 20.6.4 NMAC a reference to climate change, proposing to add that, "These surface water quality standards serve to address the inherent threats to water quality due to climate change."

As set forth in my direct testimony, while addressing climate change is a critical and necessary goal and while the Commission's water quality standards **should** "serve to address the

[7] The General Criteria under 20.6.4.13 NMAC are also known as "narrative criteria" or "free from" criteria, and are required by EPA regulation where numerical criteria for toxic pollutants cannot be established or to supplement numerical criteria. 40 CFR § 131.11(b)(2).

inherent threats to water quality due to climate change," the standards as currently drafted do not fully accomplish or address this goal, as NMED's proposed language implies.

NMED claims in its direct testimony that the state's antidegradation policy at 20.6.4.8 and -9 NMAC protects the state's waters from the impacts of climate change. *E.g.*, Lemon Test. at 11-12 (11-12/1171of pdf) [NMED Ex. 1]. However, this assertion is not accurate. While the antidegradation policy is an important tool for protecting New Mexico waters from impairment, it does not adequately protect or fully "address" our waters from the impacts of climate change.

For example, the standards as currently drafted have no mechanism in place to understand or track how a changing climate is impacting the hydrology of our waterways. Stream segments are identified by language such as "perennial portions of X water course" yet there is no mechanism to determine if these segments are growing or shrinking and what the cause of any growth or shrinkage may be. A stream segment historically could be perennial, but then become intermittent and the standards for that segment would be downgraded automatically without any analysis why the flow regime has changed and, in particular, without any analysis whether the change is due to climate change and if any mitigation actions could be taken. Put simply, the state has goal posts for protecting our waters that can move without oversight or any real tracking or accountability.

In addition, the state does not have adequate protocols or methodologies in place to determine if changes in temperature, dissolved solids, dissolved oxygen, sediment or turbidity are caused by natural conditions or by anthropogenic sources such as climate change and, therefore, whether exceedances of these parameters are subject to the exemptions at 20.6.4.11.I NMAC, which provides:

> **I. Exceptions**: Numeric criteria for temperature, dissolved solids, dissolved oxygen, sediment or turbidity adopted under the Water Quality Act do

not apply when changes in temperature, dissolved solids, dissolved oxygen, sediment or turbidity in a surface water of the state are attributable to:

(1) natural causes (discharges from municipal separate storm sewers are not covered by this exception.); or

(2) the reasonable operation of irrigation and flood control facilities that are not subject to federal or state water pollution control permitting; major reconstruction of storage dams or diversion dams except for emergency actions necessary to protect health and safety of the public are not covered by this exception.

As explained in my direct testimony, Amigos Bravos proposes to replace NMED's proposed language in the Objective section with the following language. Amigos Bravos also proposes adding language proposed by Communities for Clean Water to our proposed language, which appears in green below:

C. A further purpose of these surface water quality regulations is to address the inherent threats to water quality due to climate change. The quality of New Mexico surface waters is being affected by climate change. New Mexico's climate is getting hotter and drier, resulting in earlier springs, hotter summers, and less predictable winters. New Mexico is experiencing more intense droughts and a greater proportion of precipitation falling as rain instead of snow. Snowpack is shrinking and earlier snowmelts contribute to lower stream flows at critical times of the year when the reduced availability of water has greater environmental consequences. Increased water temperatures resulting from increased air temperatures tend to lead to lower levels of dissolved oxygen in water, resulting in increased stress on the fish, insects, crustaceans and other aquatic animals that rely on oxygen. More intense precipitation events and increased evaporation rates lead to increased runoff and more pollution, including increased nutrients sediment, and salt that wash into surface waters. Development of New Mexico surface water quality standards should take into account the importance of protecting of water quality in light of climate change.

This language provides a more accurate assessment, based on the scientific evidence, how a changing climate is impacting our surface waters and provides better guidance for how the standards should be developed to take climate change into account. In addition, this language, unlike NMED's, does not incorrectly claim that the standards are "addressing" climate change when in fact they are not doing so in any comprehensive manner.

NMED also proposes to add a new definition of "climate change" to the standards at 20.6.4.7.C (4):

> **(4) "Climate change"** refers to any significant change in the measures of climate lasting for an extended period of time, typically decades or longer, and includes major changes in temperature, precipitation, wind patterns or other weather-related effects. Climate change may be due to natural processes or human-caused changes of the atmosphere, or a combination of the two.

NMED states that its proposed definition "is taken almost directly from EPA's definition of climate change". Shelly Test. at 12 (12/1171 of pdf) [Ex. 1]. However, EPA's definition of climate change does not include the language about sources of climate change as proposed by NMED. As found in NMED's Exhibit 33 and on EPA's website, EPA's defines "climate change" as:

> . . . to any significant change in the measures of climate lasting for an extended period of time. In other words, climate change includes major changes in temperature, precipitation, or wind patterns, among others, that occur over several decades or longer.[8]

At this same website, there is a large header with a link that reads "Humans are largely responsible for recent climate change."[9] Amigos Bravos proposes adding a sentence to NMED's proposed definition that clarifies that the sources of climate change are primarily human-caused, and not due to natural processes:

> **(4) "Climate change"** refers to any significant change in the measures of climate lasting for an extended period of time, typically decades or longer, and includes major changes in temperature, precipitation, wind patterns or other weather-related effects. Climate change may be due to natural processes or human-caused changes of the atmosphere, or a combination of the two. Humans are largely responsible for recent climate change.

[8] https://19january2017snapshot.epa.gov/climatechange/climate-change-basic-information_.html.
[9] *Id.*

Scientists agree that humans are the cause of vast majority of current climate change, as I outlined in my direct testimony.[10] Conn Dir. Test. at 5-6 [AB Ex. 3]. This is recognized by the international community as well as by the U.S. climate scientists. According to U.S. Global Change Research Program ("USGCRP"), "human activities have been, and are increasingly, the dominant cause of climate warming."[11] The Intergovernmental Panel on Climate Change ("IPCC"), in its 2018 Special Report on Global Warming of 1.5°C, outlines how human activities have already caused climate warming of 1°C and are likely to cause a warming to 1.5°C by 2030-2050 if current emission levels stay the same.[12] The IPCC find that many of the impacts of climate change "fall disproportionately on the poor and vulnerable (high confidence)."[13] In order to protect our waters and the communities that depend on them, it is essential that the state adequately identify the threat and identify concrete steps to update the Commission's regulations to address the threats posed by climate change to New Mexico's water quality and communities.

V. THE COMMISSION SHOULD DIRECT NMED TO RE-EXAMINE ALL WATER BODIES THAT DO NOT HAVE SECTION 101(a)(2) USES AS REQUIRED BY FEDERAL REGULATION

In preparation for this Triennial Review, NMED prepared an analysis of select waters that currently have the secondary contact use to determine if primary contact is attainable. This analysis, entitled "Existing Use Analysis of Recreational Use for Classified Waters 20.6.4.101-

[10] https://www.ucsusa.org/resources/are-humans-major-cause-global-warming.
[11] USGCRP, Climate Science Special Report: Fourth National Climate Assessment, Volume I (2017) at https://science2017.globalchange.gov/.
[12] IPCC, Special Report: Global Warming of 1.5°C: Summary for Policymakers (2018) at https://www.ipcc.ch/sr15/chapter/spm/.
[13] *Id.* Executive Summary at 51 at https://www.ipcc.ch/site/assets/uploads/sites/2/2019/05/SR15_Chapter1_High_Res.pdf.

20.6.4.899 NMAC" ("EUA") [NMED Ex. 56], details the process by which NMED examined these waterbodies. It is important to note that states are **required** by federal regulations to re-examine every three years any waterbody that does not have a use specified in section 101(a)(2) of the Clean Water Act:

> The State shall also re-examine any waterbody segment with water quality standards that do not include the uses specified in section 101(a)(2) of the Act every 3 years to determine if any new information has become available. If such new information indicates that the uses specified in section 101(a)(2) of the Act are attainable, the State shall revise its standards accordingly.[14]

New Mexico's secondary contact use is not considered by EPA to be a section 101(a)(2) "fishable/swimmable" use and therefore all waterbodies in New Mexico with a secondary contact use must be reexamined every three years to determine if a section 101(a)(2) use is attainable. Similarly, EPA does not consider limited aquatic life to be a section 101(a)(2) use and therefore all waters with a limited aquatic life use also must be re-examined every three years.

However, the EUA study conducted by NMED only looked at waters with secondary contact and did not look at waters with limited aquatic life use. Yet even the analysis of secondary contact waters is incomplete. As NMED acknowledges in its testimony, during its review the state did not evaluate all waters with a secondary contact use: "[l]akes, waterbodies with site specific criteria, and other classified waters undergoing designated use investigations were excluded from the review." Aranda Test. at 7 (40/1171 of pdf) [NMED Ex. 3]. In addition, NMED excluded from analysis waterbodies that did not contain both pH and E. coli data: "Any waterbodies that did not contain both pH and E. coli data were excluded from the analysis and were not included in the proposed recreational use designation change." Aranda Test. at 11 (44/1171 of pdf) [NMED Ex. 3]. NMED also excluded from analysis waterbodies where data

[14] 40 CFR § 131.20(a).

indicated that pH was outside the range for primary contact use. *Id.* As stated above, NMED failed to reexamine waterbodies with the limited aquatic life use. This whittling down of waters that were examined has resulted in an incomplete analysis and does not meet the requirements at 40 CFR § 131.20(a) that require that all waters without section 101(a)(2) uses must be reviewed to determine if those uses are attainable.

Not only is the universe of waterbodies that were examined incomplete, the data that was examined during the process is incomplete. During the EUA process, NMED only looked at water quality data and did not consider other sources of data and information that could demonstrate attainment of uses, such as historical records of swimming. In addition, potential restoration or other planned or existing controls could lead to attainment of a section 101(a)(2) use and therefore a detailed examination of total maximum daily loads ("TMDL") implementation, watershed based planning efforts, and other best management practices should also be part of the process to determine if section 101(a)(2) uses are attainable. The sole criterion that NMED used during its examination was whether there is historical or current chemical water quality data that indicates primary contact use attainment. If there wasn't chemical data that showed primary contact use attainment at least at one point since 1975, then NMED determined that attainment wasn't possible. Examining chemical water quality data is only one component of determining use attainment. The way that NMED went about examining the data was more of an analysis whether or not the primary contact use was actually attained at some point in the past, not whether it may be attainable. For example, there may be a waterbody that has always been impaired for E. coli and yet there is a watershed-based plan and a TMDL that have been completed that show that, if restoration along with point and nonpoint source pollution control

mechanisms are implemented, E. coli levels could be drastically reduced, thus potentially resulting in attainment of not only the secondary contact use but also the primary contact use.

In conclusion, Amigos Bravos supports NMED's proposal to upgrade the designated use from secondary contact to primary contact for the segments they are proposing at 20.6.4.103, -116, -204, -206, and -207 NMAC. However, Amigos Bravos believes that the Commission should direct NMED to conduct the analysis required under the Clean Water Act and re-examine **all** waterbodies that do not have section 101(a)(2) uses.

VI. NON-PERRENIAL WATERS MUST BE BETTER PROTECTED

NMED proposes to move non-perennial waters that are currently protected in classified segments in 20.6.4.101 to 20.6.4.899 NMAC to the non-classified segment at 20.6.4.98 NMAC for intermittent waters. Specifically, NMED proposes to amend 20.6.4.108, -115, -206, -208, -209, -215, -220, -307, and -309 NMAC by removing non-perennial waters from these segments and placing them by default (not expressly) into the non-classified protections for non-perennial waters at 20.6.4.98 NMAC. This represents a downgrading of the aquatic life use from a coldwater or high quality coldwater designated use that applies to the segments listed above to a marginal warmwater aquatic life use that applies to 20.6.4.98 NMAC.

NMED did not present adequate evidence that the current designated uses for these classified non-perennial water bodies are unattainable. Instead, NMED asserted that because there is a lack of water quality data, there are no existing uses for these water bodies. NMED then used this claimed lack of existing uses to justify downgrading the designated use:

> The Department searched readily available water quality data to identify the existing uses for each of the classified non-perennial waters considered for a designated use amendment. The Department's data search using SWQB's in-house database, SQUID, found no applicable data for these waterbodies. Consequently, existing uses could not be established. Since no existing uses were

> established, the implementation of this amendment will not result in the lowering of any known existing use.

Aranda Test. at 18 (51/1171 of pdf) [NMED Ex. 3].

It is concerning that NMED determined that, because there is no chemical water quality data, there are no existing uses in these waterbodies. This logic makes no sense, and is inconsistent with Clean Water Act regulations. The lack of water quality data should not be used as a reason to downgrade water quality standards and should not be used in NMED's procedure for use attainability analyses ("UAAs"). Lack of applicable water quality data should lead to collection of the data before a downgrading is allowed. NMED's rationale is consistent with EPA's framework. According to EPA, federal regulations establish a "rebuttable presumption" for section 101(a)(2) uses. The state's role is to "affirmatively demonstrate" that the uses are not attainable through the UAA. Lack of data does not meet that standard:

> The Water Quality Standards regulations effectively establish a "rebuttable presumption" that the CWA 101(a)(2) uses are attainable and therefore must be assigned to a water body, unless a State or Tribe **affirmatively demonstrates**, with appropriate documentation, that such uses are not attainable.
>
> ***Key Point.*** Along with facilitating achievement of Congress' goals, the "rebuttable presumption" approach preserves the paramount role of States and Tribes in establishing water quality standards and in weighing any available evidence regarding the attainable uses of a particular water body.[15]

(Emphasis added.) Under NMED's approach, it could make the case that any stretch of any river in the state for which there is no chemical water quality data -- even a stretch of the Rio Grande -- doesn't have any existing uses. EPA has anticipated situations where there is a lack of data and provides the following guidance:

> …where data may be limited or inconclusive, EPA expects states and tribes to consider the quantity, quality, and reliability of the different types of available

[15] https://www.epa.gov/wqs-tech/key-concepts-module-2-use#tab-5.

> data to describe the existing use as accurately and completely as possible and to resolve any apparent discrepancies based upon that evaluation.[16]

NMED has not provided any additional information to demonstrate that it considered different types of data for the streams it proposes to downgrade. Instead, NMED relies on the faulty rationale that -- because there isn't any chemical water quality data -- there must not be any existing uses.

Another concern with NMED's approach of classifying waters according to stream flow is that, as the climate continues to warm and more of our waters turn from perennial systems to non-perennial systems, there is the very real potential that the majority of New Mexico's waters will end up by default in 20.6.4.98 NMAC, the non-classified segment in the standards for intermittent waters, which has less protective water quality standards than those in most classified segments. By delineating segments by stream flow, as the current standards do and is expanded upon through the current non-perennial UAA proposal, as perennial waters shrink, protections automatically shrink with them. If NMED delineates segments by stream flow as, for example, "the perennial portions of X River and the perennial portions of the tributaries to X River" -- instead of delineating segments by geographic markers such as "X River from the confluence of Y River to the confluence with Z River" or "X River from the bridge at HWY 1 to the boundary of ABC State Park" -- water quality protections are automatically downgraded during drought conditions as perennial portions shrink.

This is problematic in several ways. First, in some cases changes in flow regimes are a result of non-natural causes, such as increased diversions. Under the current structure of the

[16] EPA tetter September 2008 from Keehner to Smithee at https://www.epa.gov/sites/production/files/2014-10/documents/existinguse-smithee-letter.pdf.

standards there isn't a mechanism to identify a situation where reduced flows are not the result of natural processes, and therefore not subject to the six factors for downgrading a use outlined in 40 C.F.R. § 131.10(g). Second, human-caused climate change, which is the primary cause of the drought conditions New Mexico faces today and expects to experience into the future at intensified levels, is not a natural cause. Waters that are drying up due to climate change shouldn't be downgraded using the low flow condition (one of the six) found at 40 C.F.R. § 131.10(g)(2) [NMED Ex. 22]: "natural, ephemeral, intermittent or low flow conditions or water levels prevent the attainment of the use." In fact, it is unclear from the Non-Perennial UAA before the Commission if the low flow conditions of the waters being proposed to be moved from classified segments to the non-classified 20.6.4.98 NMAC segment are from natural causes or from human-caused impacts of climate change.

Section 7(a)(2) of the federal Endangered Species Act requires EPA to consult with the U.S. Fish and Wildlife Service ("USFWS") to ensure that the proposed downgrading is not likely to jeopardize the continued existence or result in the destruction or adverse modification of habitat. NMED conducted a preliminary review, and claims that downgrading these non-perennial waters will not do either because the proposal is "not amending the natural conditions attainable by these waterbodies," nor will it "alter habitat, only attainable water quality." Aranda Test. at 19 (52/1171 of pdf) [NMED Ex. 3]. This is misleading and inaccurate because water quality actually **is** a critical part of habitat. In fact, one water quality parameter -- dissolved oxygen -- is one of the single most important factors for the suitability of habitat for aquatic organisms. The proposed downgrading will potentially impact aquatic habitat because it will allow more pollution to be discharged to these non-perennial waters as well as potentially

allowing more destructive activities that can result in more pollution discharged and physical impacts to the waterbody.

On a more general note, NMED refers to "perennial uses" and "non-perennial uses" throughout the testimony. This is problematic because the existing uses for a given waterbody are the uses that have occurred in the waterbody since 1975 regardless of whether they are in perennial or non-perennial water bodies and regardless of whether they are lumped into the non-classified segments at 20.6.4.97, 20.6.4.98, or 20.6.4.99 NMAC. It is a false distinction to talk about perennial and non-perennial uses. If the uses are existing or designated in either non-perennial or perennial waters, they must be maintained and protected unless they are proven to be unattainable. Whether they are attainable is not proven simply because the waters are not perennial. In fact, there are likely to be sensitive species emerging, reproducing and rearing in the short periods of time that the non-perennial waters flow. And the Clean Water Act requires states to develop designated uses, water quality criteria and antidegradation policies that protect the most sensitive uses.[17]

This is why the Hydrology Protocol is only one component of a use attainability analysis, because flow is only one of several contributing factors to the uses found in a waterbody. While there may be uses that are more common in perennial waterbodies, such as primary contact, these uses shouldn't be categorized as "perennial" or "non-perennial" uses. For example, here in the arid Southwest, it is common for tributaries that only flow during the spring runoff to be used for swimming during times of high flow but that dry up either wholly or intermittently during the hot dry part of the year. In this case, primary contact would be a use found in a non-perennial waterbody. Each situation can be unique and therefore uses should be determined by what is

[17] 40 CFR § 131.11(a).

occurring in a waterbody and not by stream flow alone. New Mexico should consider applying seasonal uses in non-perennial water bodies where a use exists only part of the year.

VII. "LOW-FLOW" SHOULD NOT REPLACE CURRENT DEFINED TERMS

NMED proposes to replace the terms "intermittent and ephemeral" waters in the limited aquatic life definition with the term "low-flow" at 20.6.4.7.L(2) NMAC. NMED asserts this change will "aid in implementing and applying this aquatic life use, which is not based entirely on the hydrologic regime." Fullam Test. at 5 (64/1171 of pdf) [NMED Ex. 4]. However, using the term "low-flow," which is not defined, will result in applying this non-101(a)(2) use more broadly to include perennial waters, and should be rejected.

VIII. INTERMITTENT LANL WATERS HAVE NOT BEEN ASSIGNED THE PROPER DESIGNATED UES

I participated in the 2003-2005 Triennial Review on behalf of Amigos Bravos. During that Triennial Review, NMED first proposed a new segment for unclassified waters at 20.6.4.98 NMAC ("Segment 98") that included both ephemeral and intermittent waters within one segment. For Segment 98, NMED proposed designated uses of limited aquatic life and secondary contact. During that same Triennial Review, NMED also first proposed a new segment for both ephemeral and intermittent waters on LANL property, at 20.6.4.128 NMAC ("Segment 128"), and NMED proposed that the LANL waters carry the same designated uses as it proposed for Segment 98: limited aquatic life and secondary contact. As discussed above, those uses are not section 101(a)(2) uses.

However, during deliberations, the Commission determined that intermittent waters were able to attain and support a more stringent designated aquatic life use which included chronic criteria because of the "potential long-term exposure of aquatic life to pollutants" in intermittent waters. Fullam Test. at 28 (87/1171 of pdf) [NMED Ex. 4]. Therefore, the Commission split the

initial proposed Segment 98 into two segments: Segment 98 for intermittent waters and Segment 97 for ephemeral waters. The segment for intermittent waters, Segment 98, was assigned more protective standards that included a designated use for marginal warmwater aquatic life and primary contact, both section 101(a)(2) uses. The marginal warmwater designated use includes both chronic aquatic and acute aquatic life criteria, while limited aquatic life includes only acute criteria. However, even though the evidence in support of Segments 98 and 128 was the same, the Commission inexplicably did not assign the same designated uses and criteria to LANL intermittent waters in Segment 128 as it had for intermittent waters for Segment 98, and instead assigned LANL intermittent waters limited aquatic life and secondary contact uses.

When the Commission's amendments from the 2003-2005 Triennial Review were sent to EPA for review and approval, EPA did not approve the designated uses and criteria for Segment 128 because the designated uses -- specifically limited aquatic life and secondary contact -- were not protective of Clean Water Act 101(a)(2) uses and no associated UAA had been developed to support assigning less protective standards.

In response to EPA's rejection, NMED did not separate Segment 128 into two segments -- one for ephemeral and one for intermittent waters -- as had been done for the unclassified ephemeral and intermittent waters. Instead, NMED prepared an after-the-fact UAA to justify the lesser protections in Segment 128 for both intermittent and ephemeral waters. *See* LANL Ex. 18. However, NMED's 2005 UAA was fatally flawed because it was based on an incorrect understanding of Clean Water Act 101(a)(2) uses. The 2005 UAA relied on the erroneous presumption that the presence of fish is the only indicator of a 101(a)(2) use. 2005 UAA for

LANL Waters at 5, 6 [LANL Ex. 18]. In fact, the presence of macroinvertebrates in water is also an indicator of a 101(a)(2) use.[18]

Since that 2005 UAA was completed, NMED has changed its approach on how to determine section 101(a)(2) uses, and has adopted a Hydrology Protocol, which specifically states it is a "guideline to distinguish ephemeral channels from non-ephemeral ones unless there are **aquatic macroinvertebrates** and/or fish, in which case at least one of the Clean Water Act Section 101(a)(2) objectives is attainable and the stream is at least intermittent." Hydrology Protocol at 37 [NMED Ex. 63] (emphasis added). NMED's current Hydrology Protocol correctly acknowledges that if invertebrates are present, section 101(a)(2) uses are present, and the stream in question deserves corresponding marginal warmwater aquatic life, **not** limited aquatic life, protections.

EPA clearly interprets section 101(a)(2) uses as being necessary to protect waters if invertebrates are present, even if fish are not:

> The fact that sport or commercial fish are not present does not mean that water may not be supporting an aquatic life protection function. **An existing aquatic community composed entirely of invertebrates and plants, . . . should still be protected** whether or not such a stream supports a fishery. Even though the shorthand expression "fishable/swimmable" is often used, the actual objective of the act is to "restore and maintain the chemical, physical, and biological integrity of our Nation's waters (section 101(a)." The term "aquatic life" would more accurately reflect the protection of the aquatic community that was intended in Section 101(a)(2) of the Act. [19]

(Emphasis added.)

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[18] Section 101(a)(2) of the Clean Water Act expressly states that "it is the national goal that wherever attainable, an interim goal of water quality which provides for the protection and propagation of fish, **shellfish**, and wildlife and provides for recreation in and on the water be achieved by July 1, 1983; . . . ." 33 U.S.C. § 1251(a)(2) (emphasis added).

[19] EPA Office of Water, Questions and Answers on: Antidegradation at 3 (Aug. 1985) at https://www.epa.gov/sites/production/files/2018-10/documents/questions-answers-antidegradation.pdf.

NMED even acknowledges in this proceeding that its 2005 UAA is flawed for this very reason:

> Although the [2005 NMED] UAA asserts the highest attainable life use for non-perennial waters is limited aquatic life, the [2002] U.S. Fish and Wildlife Service study which "investigated the biological, chemical, and physical characteristics of four intermittent streams on the Los Alamos National Laboratory in New Mexico…to identify suitable living space for fish and benthic macroinvertebrates" found that **"aquatic life is an existing use of these intermittent streams that should be protected." Despite this apparent discrepancy between the UAA and the supporting evidence, EPA approved the designated limited aquatic life use for ephemeral and intermittent waters within LANL as classified under 20.6.4.128 NMAC on September 12, 2007**.

Fullam Test. at 29 (88/1171 of pdf) [NMED Ex. 4] (emphasis added).

For its part, LANL presents an incomplete overview of the history of how protections for LANL waters were established, glazing over the discrepancy in protections for non-LANL and LANL intermittent waters, Meyerhoff Test. at 8-9 [LANL Ex. 2], and takes the position that Segment 128 should continue to apply to both ephemeral and intermittent waters, basing this position on the faulty 2005 UAA. Gallegos Test. at 18, 24 (20/44, 26/44 of pdf) [LANL Ex. 3]; Meyerhoff Test. at 16 (20/37 of pdf) [LANL Ex. 2].During the 2013-2015 Triennial Review, Amigos Bravos proposed to upgrade protections for intermittent waters on LANL property so that LANL intermittent waters would be protected at the same level as all other intermittent waters in the state. I participated in that Triennial Review as well.

Recognizing Amigos Bravos' position had merit, LANL and NMED entered into an agreement with Amigos Bravos – the Joint Stipulation Regarding Proposed Changes to 20.6.4.128 NMAC ("Stipulation") during that Triennial Review. In the Stipulation, Amigos Bravos agreed to withdraw its proposed amendments to upgrade LANL waters in exchange for an agreement from NMED and LANL to engage in a process to review the protections set forth in 20.6.128 NMAC with the goal of reaching agreement on protections for LANL waters

consistent with the Clean Water Act. The Stipulation did not waive the parties' right to propose changes to 20.6.4.128 NMAC at any time in the future.

Since that last Triennial Review, the parties engaged in the data and information-gathering process contemplated in the Stipulation. After engaging in that process, NMED, in its original Petition for this current Triennial Review filed in August 2020, proposed to bring protections for LANL ephemeral and intermittent waters within the same regulatory protections as all other ephemeral and intermittent waters in the state. Specifically, NMED proposed to remove the intermittent waters classification from Segment 128 so that Segment 128, which currently covers intermittent and ephemeral waters, would include only ephemeral waters. NMED then proposed a list of specific waters as ephemeral under Segment 128. NMED also proposed to protect all LANL waters not specifically identified in either 20.6.4. 126 NMAC ("Segment 126"), which are perennial waters on LANL property, or Segment 128 under a new 20.6.4.140 NMAC ("Segment 140"). Unlike Segment 128, this new Segment 140 had section 101(a)(2) protections with designated uses of warmwater aquatic life and primary contact. NMED's proposal in its original Petition reflected the correct analysis for protecting LANL intermittent waters under the Clean Water Act.

Unfortunately, NMED, in its Amended Petition filed in March 2021, significantly reduced protections for intermittent waters on LANL property. Instead of proposing that all LANL waters not identified in Segment 126 or 128 be placed in new Segment 140 (protecting 101(a)(2) uses), NMED proposed to place only three stream segments in Segment 140. These three segments -- portions of Effluent, S-Site, and Two-Mile Canyons – represent all waters that all three parties have agreed should be protected under Segment 140, but do not represent the universe of waters that should be placed in Segment 140, as I have outlined above.

As part of the Stipulation and as outlined in LANL's testimony, Gallegos Test. at 4-6[LANL Ex. 3]), NMED, LANL, and Amigos Bravos applied the Hydrology Protocol to many LANL waters. In summary, LANL conducted 104 Hydrology Protocols[20], 47 of which were conducted with NMED. Of the 47 Hydrology Protocols conducted with NMED, five keyed out as perennial and 21 to intermittent. Yet, NMED only proposes to upgrade three streams, which are within the area covered by six of the Hydrology Protocols, to Segment 140. Therefore, only six of the 21 segments that keyed out to intermittent are being proposed for increased protections for Segment 140. *See* List of LANL Hydrology Scores [AB Ex. 16].

In its Amended Petition, NMED not only significantly reduced the universe of waters proposed for protections under Segment 140, it also weakened two of the proposed designated uses for Segment 140: NMED weakened (1) the aquatic life designated use proposed for Segment 140 from warmwater aquatic life to marginal warmwater aquatic life and (2) the contact use from primary contact to secondary contact. Notably, downgrading from primary contact to secondary contact now means that Segment 140 would not be protective of 101(a)(2) uses.

The decision to downgrade the contact use was apparently based on lack of E. coli data. According to NMED:

> . . . no E. coli data were found for purposes of this analysis for Effluent Canyon, S-Site Canyon, and Two-Mile Canyon. Therefore, the existing recreational use, based on E. coli, was found to be indeterminate at this time based on insufficient evidence and no further analysis of recreational use was conducted. Until further data are available, the existing recreational use is assumed to be at least secondary contact.

[20] LANL states it conducted 117 Hydrology Protocols. Gallegos Test. at 4 (6/44 of pdf) [LANL Ex. 3]. However the field data sent to Amigos Bravos on November 20, 2020 only includes data for 104 Hydrology Protocols.

Fullam Test. at 34 (93/1171 of pdf) [NMED Ex. 4]. However, lack of applicable water quality data should lead to collection of the data before a use that is not protective of section 101(a)(2) uses is assigned. EPA has affirmed there is a rebuttable presumption for section 101(a)(2) uses, and that the state must "affirmatively demonstrate" such uses are not attainable through a UAA before downgrading to a non-101(a)(2) use. Lack of data does not meet that standard.

In closing, while Amigos Bravos concurs with the limited proposal put forth by NMED for Segment 140, this limited proposal does not adequately protect intermittent waters at LANL and **Amigos Bravos retains its right to propose changes to 20.6.4.128 NMAC and other LANL segments at any time in the future.**

This concludes my rebuttal testimony, which is accurate to the best of my knowledge.

6/21/21

Rachel Conn — Date

AMIGOS BRAVOS' EXHIBIT 12

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Deputy Secretary

Original via FedEx-Copy via Electronic Mail

November 30, 2020

Mr. Charles Maguire, Director
Water Quality Protection Division (6WD)
U. S. Environmental Protection Agency
1201 Elm Street, Suite 500
Dallas, Texas 75202

Re: State Certification Los Alamos National Laboratory Industrial Wastewater
NPDES Permit No. NM0028355

Dear Director Maguire:

Enclosed, please find the state certification for the following proposed National Pollutant Discharge Elimination System (NPDES) permit NM0028355, Los Alamos National Laboratory Industrial Wastewater Permit. Comments and conditions are enclosed on separate sheets.

The U.S. Environmental Protection Agency (EPA) proposes to regulate discharges under the above referenced NPDES Individual permit. A state Water Quality Certification is required by the federal Clean Water Act (CWA) Section 401 to ensure that the action is consistent with state law (New Mexico Water Quality Act, New Mexico Statutes Annotated [NMSA] 1978, Sections 74-6-1 to -17) and complies with the State of New Mexico Water Quality Standards at 20.6.2 and 20.6.4 New Mexico Administrative Code (NMAC), Water Quality Management Plan and Continuing Planning Process, including Total Maximum Daily Loads (TMDLs), and Antidegradation Policy.

Pursuant to State regulations for permit certification at 20.6.2.2001 NMAC, EPA jointly with the New Mexico Environment Department (NMED) issued a public notice of the draft permit and announced a public comment period posted on the NMED web site at https://www.env.nm.gov/surface-water-quality/public-notices/ on November 27, 2019. The NMED public comment period ended on November 2, 2020. NMED received comments from the Buckman Direct Diversion Board and the Permittees, which were considered in this certification.

Sincerely,

for

ATTACHMENT A

Shelly Lemon, Bureau Chief
Surface Water Quality Bureau

cc: (w/ enclosures)
Ms. Evelyn Rosborough, USEPA (6WDPN) via e-mail
Mr. Brent Larsen, USEPA (6WDPE) via e-mail
Mr. Isaac Chen, USEPA (6WDPE) via e-mail
Mr. Michael Hazen, ESHQSS, Triad National Security, LLC by email
Mr. Enrique Torres, EPC-DO, Triad National Security, LLC by email
Mr. Michael Saladen, EPC-CP, Triad National Security, LLC by email
Ms. Taunia Van Valkenburg, EPC-CP, Triad National Security, LLC by email
Ms. Jennifer Griffin, EPC-CP, Triad National Security, LLC by email
Mr. Michael Weis, USDOE NA-LA by email
Ms. Karen Armijo, USDOE NA-LA by email
Buckman Direct Diversion Board, via luke@egolflaw.com

Mr. Ken McQueen, Regional Administrator
Environmental Protection Agency
1201 Elm Street, Suite 500
Dallas, TX 75202

November 30, 2020

STATE CERTIFICATION

RE: **NM0028355, Los Alamos National Laboratory Industrial Wastewater**

Dear Regional Administrator McQueen:

The Cabinet Secretary of the New Mexico Environment Department (NMED) has delegated signatory authority for state certifications of federal Clean Water Act permits to the Surface Water Quality Bureau Chief. NMED examined the proposed NPDES permit referenced above. The following conditions are necessary to assure compliance with the applicable provisions of the Clean Water Act Sections 208(e), 301, 302, 303, 306, and 307 and with appropriate requirements of State law. Compliance with the terms and conditions of the permit and this certification will provide reasonable assurance that the permitted activities will be conducted in a manner which will not violate applicable water quality standards or the water quality management plan and will be in compliance with the antidegradation policy.

The State of New Mexico

( ) certifies that the discharge will comply with the applicable provisions of Sections 208(e), 301, 302, 303, 306 and 307 of the Clean Water Act and with appropriate requirements of State law

(x) certifies that the discharge will comply with the applicable provisions of Sections 208(e), 301, 302, 303, 306 and 307 of the Clean Water Act and with appropriate requirements of State law upon inclusion of the following conditions in the permit (see attachments)

( ) denies certification for the reasons stated in the attachment

( ) waives its right to certify

In order to meet the requirements of State law, including water quality standards and appropriate basin plan as may be amended by the water quality management plan, each of the conditions cited in the draft permit and the State certification shall not be made less stringent, unless changes are in response to formal comments received by EPA and discussed with NMED prior to the finalization of the draft permit.

NMED reserves the right to amend or revoke this certification if such action is necessary to ensure compliance with the State's water quality standards and water quality management plan.

Please contact Sarah Holcomb at (505) 819-9734 if you have any questions concerning this certification. Comments and conditions pertaining to this draft permit are attached.

Sincerely,

Shelly Lemon, Bureau Chief
Surface Water Quality Bureau

State of New Mexico Comments and Conditions on the Proposed NPDES Permit
Los Alamos National Laboratory Industrial Wastewater
NM0028355
November 30, 2020

The following conditions are necessary to ensure that discharges allowed under the National Pollutant Discharge Elimination System (NPDES) permit protect State of New Mexico surface water quality standards (WQS) adopted in accordance with Section 303 of the Clean Water Act (CWA) and the New Mexico Water Quality Act (NMSA 1978, §§ 74-6-1 to -17). State of New Mexico (State) WQS are codified in Title 20, Chapter 6, Part 4 of the New Mexico Administrative Code (20.6.4 NMAC), *Standards for Interstate and Intrastate Surface Waters*, as amended by the New Mexico Water Quality Control Commission (WQCC) on May 22, 2020 and most recently approved by the U.S. Environmental Protection Agency (EPA or USEPA) as of July 24, 2020. Additional state WQS are published in Title 20, Chapter 6, Part 2 of the New Mexico Administrative Code (20.6.2 NMAC), *Ground and Surface Water Protection*, as amended by the WQCC most recently on December 21, 2018.

NPDES regulations at 40 CFR § 122.44(d)(l)(i) require that permit "…limitations must control all pollutants or pollutant parameters... which the Director determines are or may be discharged at a level which will cause, have the reasonable potential to cause, or contribute to an excursion above any State water quality standard…"

40 CFR § 124.53(e) states that, "State certification shall be in writing and shall include: (1) Conditions which are necessary to assure compliance with the applicable provisions of CWA Sections 208(e), 301, 302, 303, 306 and 307 and with appropriate requirements of State law…"

Conditions of Certification:

Condition # 1:
Facilities at outfalls 001, 13S, 027, 022, 055, and 051 (which incorporate facilities operating under NAICS codes listed in the *Final Rule [June 22, 2020]* for TRI Reporting [noted above]) shall monitor and report PFAS in effluent once during the first year of coverage, or when the facility next discharges if no discharge occurs during the first year. Samples shall be analyzed by an accredited lab for all 18 PFAS analytes using EPA Method 537.1 (EPA 2018), and the DoD Quality Systems Manual Method 5.3 (2019) as guidance. Method and analysis shall be sufficiently sensitive to evaluate the New Mexico screening level for PFOA and PFOS.

The PFAS screening level in New Mexico is indicated below. The screening level is not a standard of quality and purity for the surface waters of New Mexico but allows detection and further evaluation of the existence of PFAS in discharges to determine if more attention is warranted.

PFAS Screening Level for New Mexico*	
PFOA + PFOS	0.070 ug/L

* Concentrations of PFOA and PFOS are summed before being compared to the screening level.

If PFOA and/or PFOS are detected above the New Mexico screening level, additional monitoring and reporting shall occur annually and in accordance with the same parameters and methods as required for the first sampling event. In addition, the permittee should take corrective action and identify ways to minimize, reduce, and eliminate PFAS from the industrial activity through product substitution and/or

additional best management practices and operational controls. Results of past monitoring and any corrective actions taken should be documented by the permittee.

The permittee shall submit monitoring results for all 18 PFAS analytes under EPA Method 537.1, as required, to NMED at the following address:

Point Source Program Manager
Surface Water Quality Bureau
New Mexico Environment Department
P.O. Box 5469
Santa Fe, NM 87502-5469

Background for Condition #1

New Mexico regulations (Standards for Interstate and Intrastate Surface Waters) under 20.6.4.13(F) NMAC state: Except as provided in 20.6.4.16 NMAC, surface waters of the state shall be free of toxic pollutants from other than natural causes in amounts, concentrations or combinations that affect the propagation of fish or that are toxic to humans, livestock or other animals, fish or other aquatic organisms, wildlife using aquatic environments for habitations or aquatic organisms for food, or that will or can reasonably be expected to bioaccumulate in tissues of fish, shellfish and other aquatic organisms to levels that will impair the health of aquatic organisms or wildlife or result in unacceptable tastes, odors or health risks to human consumers of aquatic organisms.

New Mexico regulations (Ground and Surface Water Protection) under 20.6.2.7(T)(2)(s) NMAC lists the following perfluorinated chemicals (PFCs) as toxic pollutants: perfluorohexane sulfonic acid (PHHxS), perfluorooctane sulfonate (PFOS), and perfluorooctanoic acid (PFOA).

The EPA revised the Emergency Planning and Community Right-to-Know Act (EPCRA) Section 313 list of reportable chemicals covered by the Toxics Release Inventory (TRI) to include the 172 per- and polyfluoroalkyl substances (PFAS) added by the National Defense Authorization Act.[1]

The following is a list of North American Industrial Classification System (NAICS) codes from EPA's Final Rule (June 22, 2020) that may be potentially affected by TRI reporting requirements:[2]

- *Facilities included in the following NAICS manufacturing codes (corresponding to Standard Industrial Classification (SIC) codes 20 through 39): 311*, 312*, 313*, 314*, 315*, 316, 321, 322, 323*, 324, 325*, 326*, 327, 331, 332, 333, 334*, 335*, 336, 337*, 339*, 111998*, 211130*, 212324*, 212325*, 212393*, 212399*, 488390*, 511110, 511120, 511130, 511140*, 511191, 511199, 512230*, 512250*, 519130*, 541713*, 541715* or 811490*. *Exceptions and/or limitations exist for these NAICS codes.*
- *Facilities included in the following NAICS codes (corresponding to SIC codes other than SIC codes 20 through 39): 212111, 212112, 212113 (corresponds to SIC code 12, Coal Mining (except 1241)); or 212221, 212222, 212230, 212299 (corresponds to SIC code 10, Metal Mining (except 1011, 1081, and 1094)); or 221111, 221112, 221113, 221118, 221121, 221122, 221330 (limited to facilities that combust coal and/or oil for the purpose of generating power for distribution in commerce) (corresponds to SIC codes 4911, 4931, and 4939, Electric Utilities); or 424690, 425110, 425120 (limited to facilities previously classified in SIC code 5169, Chemicals and Allied Products, Not Elsewhere Classified); or 424710 (corresponds to SIC code 5171, Petroleum Bulk Terminals and Plants); or 562112 (limited to facilities primarily engaged in solvent recovery services on a contract or fee basis (previously classified under SIC code 7389, Business Services, NEC)); or 562211, 562212, 562213, 562219, 562920 (limited to facilities regulated under the Resource Conservation and Recovery Act, subtitle C, 42 U.S.C. 6921 et seq.) (corresponds to SIC code 4953, Refuse Systems).*

- *Federal facilities.*

Information prepared by the EPA and the Agency for Toxic Substances and Disease Registry (ATSDR) demonstrates that PFAS are toxic and can pose hazards to human health and the environment.[3,4] In EPA's PFAS Action Plan[5] program update, dated February 2020, the Agency recommends using a screening level of 40 parts per trillion (0.040 ug/L) to determine if PFOA and/or PFOS is present at a site and may warrant further attention.

PFAS has been detected in nearly all environmental media. However, there is very limited data on industrial wastewater discharges of PFAS into the environment, in part due to the fact that relatively few facilities have NPDES permit limits or monitoring requirements for PFAS. The EPA identified only 13 industrial facilities that reported PFAS discharges on discharge monitoring reports (DMRs) in 2016 even though the EPA has identified several categories of industry that are likely to discharge PFAS, such as airports, military bases, fire-fighting equipment manufacturers, organic chemical manufacturers, paper and paperboard manufacturers, tanneries and leather treaters, textiles and carpet manufacturers, semiconductor manufacturers, household cleaning product manufacturers, petroleum refining, and landfills.[6]

Other states' PFAS guidance for various surface and groundwater screening levels are indicated in the tables below.[7,8]

Surface Water PFAS Guidelines in Other States				
	Oregon (ug/L)*	Michigan (ug/L)** DWS/not DWS	Minnesota (ug/L) Rivers	Alaska, Montana (ug/L)***
PFHpA	300	-	-	-
PFOA	24	0.420/12	2.7	0.070
PFOS	300	0.011/0.012	0.007	0.070
PFOSA	0.2	-	-	-
PFNA	1	-	-	-

* The Oregon DEQ wastewater initiation levels were adopted into rule (OAR 340-045-0100, Table A) in 2011. The PFAS are 5 chemicals on a list of 118 persistent priority pollutants for water that Oregon DEQ developed in response to state legislation. *Municipal wastewater treatment plants with effluent exceeding initiation levels are required to develop a pollution prevention plan that becomes a part of their NPDES permit*.
** Michigan's advisory levels are designed to protect human health (non-cancer values) and are based on whether the surface water is a drinking water source (DWS) or not.
*** For these states, concentrations of PFOA and PFOS are summed before being compared to the screening level.

Groundwater PFAS Guidelines in Other States						
	Maine (ug/L)*	New Jersey (ug/L)	New Hampshire (ug/L)**	Colorado, Rhode Island, Delaware (ug/L)*	Illinois (ug/L) ***	Minnesota (ug/L) ****
PFHpA	-	-	-	-	-	-
PFOA	0.400	0.010	0.012	0.070	0.021	0.035
PFOS	0.400	0.010	0.015	0.070	0.014	0.027
PFOSA	-	-	-	-	-	-
PFNA	-	-	0.011	-	0.021	-

* For these states, concentrations of PFOA and PFOS are summed before being compared to the screening level.
** Proposed rulemaking in New Hampshire covers 4 PFAS, and includes PFHxS = 0.018 ug/L.
*** Proposed rulemaking in Illinois covers 5 PFAS, and includes PFHxS = 0.140 ug/L and PFBS = 140 ug/L.
**** Health-based values (not maximum contaminant levels, or MCLs).

States use a variety of methods to test PFAS analytes in different media. The most widely used are EPA Method 537 (2008, applies to 14 PFAS) and EPA Method 537.1 (2018, applies to 18 PFAS). Some labs perform modifications, like using isotope dilution, to these methods for use in other matrices besides drinking water to account for lower reporting limits or greater accuracy. For example, modifications to Method 537.1 can be applied for non-drinking water media.[7]

Monitoring these toxic contaminants helps provide information about whether they are present in discharges to better control and mitigate PFAS in the environment. As stated on EPA's PFAS website,[9] *"PFAS can be found in living organisms, including fish, animals, and humans, where PFAS have the ability to build up and persist over time." Due to the characteristics of these contaminants (i.e., persistence in the environment and the human body, and evidence that exposure to PFAS can lead to adverse human health effects), NMED advocates taking a proactive approach and establishing PFAS sampling and reporting requirements to assure protection of New Mexico's surface waters, public health and the environment.*

1 *https://www.epa.gov/toxics-release-inventory-tri-program/list-pfas-added-tri-ndaa*
2 *https://www.federalregister.gov/documents/2019/12/04/2019-26034/addition-of-certain-per--and-polyfluoroalkyl-substances-community-right-to-know-toxic-chemical*
3 *https://www.epa.gov/ground-water-and-drinking-water/drinking-water-health-advisories-pfoa-and-pfos*
4 *https://www.atsdr.cdc.gov/pfas/pfas_fact_sheet.html*
5 *https://www.epa.gov/sites/production/files/2020-01/documents/pfas_action_plan_feb2020.pdf*
6 *EPA Office of Water, Preliminary Effluent Guidelines Program Plan 14, October 2019, EPA-821-R-19-005*
7 *https://www.ecos.org/documents/ecos-white-paper-processes-and-considerations-for-setting-state-pfas-standards/*
8 *http://pfas-1.itrcweb.org*
9 *https://www.epa.gov/pfas/basic-information-pfas#health*

Condition # 2:
USEPA must continue the requirement in the draft permit to include a monitoring and compliance maximum discharge limit for Polychlorinated Biphenyls (PCBs) of 0.00064 micrograms per Liter (µg/L). The State requires that monitoring and reporting of PCBs be performed in accordance with USEPA published Method 1668C or later revisions. Pursuant to 20.6.4.14(A)(3) NMAC, Method 1668C is a State approved method for testing surface wastewater discharges. Additionally, Method 1668C has a Minimum Quantification Level (MQL) set at or below the applicable and limiting State WQS found in 20.6.4.900(J)(1) NMAC. Further supporting this requirement is that Method 1668C is the only known and least restrictive and readily available laboratory wastewater sampling method that can reasonably assure that the proposed discharges do not exceed the WQS limits of 20.6.4.900(J)(1) NMAC.

For Outfall 03A027 add footnote: EPA published congener Method 1668 Revision and detection limits shall be used for reporting purposes. The permittee is allowed to develop an effluent specific MDL in accordance with Appendix B of 40 CFR Part 136 (instructions in Part II.A of this permit).

Outfall 051 has recently discharged and according to representative effluent characteristics submitted in the application there may be a reasonable potential for the effluent to exceed state WQS and EPA should add an effluent limitation for PCBs at Outfall 051.

Background for Condition #2
Below, NMED provides an explanation for why specific PCB monitoring conditions are necessary for State certification. The following table summarizes the applicable PCB numeric criteria from 20.6.4.900(J)(1) NMAC for the receiving waters of this permit action:

		Aquatic Life			
Pollutant	Wildlife Habitat	Acute	Chronic*	Human Health-Organism Only	Type of Pollutant

PCBs	0.014 µg/L	2 µg/L	0.014 µg/L	0.00064 µg/L	Chronic, Persistent

*Note: * Chronic Aquatic Life Criterion does not apply to Segment 20.6.4.128 with a designated use of Limited Aquatic Life*

As PCBs are identified as a persistent pollutant the HH-OO criteria applies to both the coldwater aquatic life use in Segment 20.6.4.126 and the limited aquatic life use in Segment 20.6.4.128, consistent with 20.6.4.11(G) NMAC. USEPA reasonable potential analysis in the Fact Sheet determined that the PCB effluent characteristics at Outfalls 001, 13S and 027 have a reasonable potential to exceed State WQS. The point source discharge permit condition is calculated to meet numeric criteria based on a modified harmonic low flow per State WQS 20.6.4.11 NMAC and as consistent with the New Mexico Implementation Plan (2012).

The following is a summary of a portion of the monitoring and effluent limitation conditions for PCBs in Part I.A of the Draft Permit for Outfalls 001, 13S and 051:

		Concentration		Loading		
		Monthly Average	Daily Maximum	Monthly Average and Daily Maximum	Frequency	Sample Type
				lbs/day		
001	Total PCB (µg/l)	0.00064	0.00064	Report	1/Year	24-hr Composite
13S	Total PCB (µg/l)	0.00064	0.00064	Report	1/Year	24-hr Composite
027	Total PCB (µg/l)	0.00064	0.00064	Report	1/Quarter	Grab

As noted above and below, the Aroclor method is not sufficiently sensitive to assure that the Permittees will comply with the applicable effluent limit for PCBs contained within the permit and thus cannot be used for monitoring or compliance purposes under state law. The following demonstrates the MDL and MQL limits of several PCB testing methods:

Method	MDL	MQL
EPA Method 608 (Aroclor)	0.065 µg/L	0.02145 µg/L
EPA Method 625	30 µg/L	99 µg/L
SM 6410 B	30 µg/L	99 µg/L
EPA Method 1668C	7-30 pg/L	23-99 pg/L (0.000023-0.000099 µg/L)

Notes: EPA Method 1668 Revision A became Revision C in the May 18, 2012 Federal Register notice of 40 CFR Part 136.

The Aroclor method's MQL is two orders of magnitude above the effluent limitation provided in this draft permit as necessary to comply the State WQS. As documented above, the congener method, EPA Method 1668C, is the only method with a sufficiently sensitive detection limit below State WQS for Total PCBs and therefore must be used when it has been determined that PCBs *"are or may be discharged at a level which will cause, have the reasonable potential to cause, or contribute to an excursion above"* State WQS. Again, this condition constitutes *"monitoring requirements necessary to assure that any applicant for a Federal license or permit will comply with any applicable effluent limitations"* consistent with the provisions of the CWA Section 401(d). 33 U.S.C. §1341 (d).

The State received comments from the Permittees. By their letter dated October 28, 2020, Los Alamos National Laboratory (LANL) provided arguments to support the use of the PCB congener method (EPA

Method 1668C) for reporting purposes but not for enforcement or compliance purposes. As detailed below, the State considered these arguments but found them insufficient to support LANL's proposition:

1. *"NMED may only include reference methods that are approved by EPA under 40 CFR Part 136 for determining compliance with effluent limitations. 40 CFR § 136.1 requires the use of EPA Methods 608 or 625 or Standard Methods 6410.B for determining compliance with effluent limits in NPDES permits."* LANL further cites the May 18, 2012 Federal Register publication of the USEPA decision to defer consideration of inclusion of EPA Method 1668C as a 40 CFR Part 136 method in support of this comment.

The State respectfully disagrees. As noted above, the State is requiring this condition in order to assure compliance with the applicable effluent and state water quality limitation which can only be achieved by use of EPA Method 1668C. This conditional action, as previously stated, is consistent with the provisions of the CWA for State Certification at 401(d) and in accordance with 20.6.2.2001 NMAC and 20.6.4.14(A)(3) NMAC.

Furthermore in reviewing USEPA's action in May 2012, to defer adoption of EPA Method 1668C, they included as part of their discussion that "EPA is still evaluating the large number of public comments and intends to make a determination on the approval of this method [1668C] at a later date…[and t]his decision does not negate the merits of this method for the determination of PCB congeners in regulatory programs or for other purposes when analyses are performed by an experienced laboratory." (FR, Vol. 77, No.97, page 29763)

2. *"LANL is the only known facility in New Mexico where use of the Congener Method 1668 is required to determine compliance with an NPDES permit limit."*

LANL is correct that it is the only facility where the use of USEPA Method 1668C is required for compliance purposes, however there is a very specific reason for this. LANL is the only facility whose discharge has been shown to have a reasonable potential to exceed State WQS for PCBs. The State also notes that LANL is not the only NPDES permittee in New Mexico subject to the specific use of USEPA Method 1668C. For example, six other NPDES permits are required to use this method for monitoring and reporting only. These discharge to waters where PCBs have been identified as a probable cause of a water quality impairment, but there was insufficient data to determine if the discharge had a reasonable potential to exceed State WQS or may contribute to a listed impairment. Therefore, based on these facts, use of Method 1668C is the least restrictive means known by the State to assure that the proposed activity will not exceed or contribute to the degradation of state water quality.

Condition #3:

EPA must revise the publicly noticed Reasonable Potential analysis to include all relevant monitoring data submitted as part of the reapplication package and supplemental information updates and comments from the Permittees per the process in the *New Mexico Implementation Guidance (2012)*. As it stands, the public noticed versions of Reasonable Potential analysis for each outfall covered under this permit are not correctly reflected in the draft permit, and according to the Permittees' comments, also are not reflective of monitoring data they submitted or contain other inaccuracies. NMED requires that once revised, EPA discuss the results of the revisions with the Department prior to finalizing the draft permit to ensure that the permit is technically sound and meets the requirements of State law, including the *Standards for Interstate and Intrastate Waters* at 20.6.4 NMAC. NMED reserves the right to revoke and reissue certification if necessary, to ensure compliance with water quality standards.

Based on NMED's review of the Reasonable Potential (RP) spreadsheets public noticed with the draft permit and data submitted to EPA by the Permittees, it appears that limitations for Thallium and PCBs are

necessary at several outfalls. Monitoring requirements shall exist in the final permit at outfalls where there is an impairment in the receiving waterbody, regardless of whether RP exists.

Outfall	Added Limits/Monitoring	Monitoring Frequency
001	Limit for thallium; monitoring for temperature – compliance schedule ok.	1/year
13S	Limit for thallium; monitoring for gross alpha	1/year
03A027	No additional limits or monitoring.	N/A
03A048	No RP for limits but monitoring for all impairments: gross alpha; cyanide; total mercury; PCBs; total selenium	1/year
03A113	EPA did not evaluate RP for PCBs at this outfall. A limit appears necessary.	1/year
03A160	EPA did not evaluate RP for PCBs at this outfall. A limit appears necessary.	1/year
03A181	It appears no RP spreadsheet was drafted for this outfall. Based on data, RP must be determined for copper and PCBs.	1/year
03A199	RP for thallium exists. EPA did not evaluate RP for PCBs.	1/year
03A022	EPA did not evaluate RP for PCBs. Monitoring requirements must stay in the permit for copper.	1/year
05A055	No additional limits or monitoring.	N/A
051	RP exists for Thallium. EPA did not evaluate RP for PCBs.	1/year

Background for Condition #3:

Below is a comparison of the effluent limitations in the administratively continued permit, water quality impairments as noted in the State of New Mexico CWA §303(d) Integrated List, notes on changes at the facility, pollutants detected in the effluent, and exceedances noted in 2015-2020 monitoring as compared to limits in the proposed permit. From this review, it appears that the following limits should either be added or modified in the final permit. Although RP exists for thallium at multiple outfalls EPA did not place limits into the draft permit.

Outfall Number	Description	Receiving Stream - WQ Segment	Impairments	Changes to Facility	Impaired pollutants detected (2C) (ug/L)	RP	2015-2020 monitoring	Metals Monitoring/Limit in 2020 Permit	Needed Limitations or Monitoring in Final Permit based on RP
001	Power Plant, SWWS, SERF, SCC, NMHFL	Sandia Canyon - 126	Aluminum, Total; Copper, Dissolved; Polychlorinated Biphenyls (PCBs); Temperature	added SCC, future add TA55	Cu 5.45, Al <19.3, PCB <0.0422, Temp, Thallium =0.442	Cu, Zn, PCB, Tl	Exceed PCB	Total Aluminum-report, Total Copper, Zinc, PCB	Thallium; monitoring for temp – compliance schedule ok.
13S	SWWS	Canada del Buey - 128	Alpha Particles; Polychlorinated Biphenyls (PCBs)		α <1.16 PCB<0.0333, Tl =0.6	PCB	No discharge	PCB	Thallium; monitoring for gross alpha (1/year)
03A027	SERF	Sandia Canyon - 126	Aluminum, Total; Copper, Dissolved; Polychlorinated Biphenyls (PCBs); Temperature		Cu 3.15, Al <19.3, PCB <0.0354, Temp	Cu, Zn	Exceed PCB and Cu limit	Total Aluminum, T Copper, PCB, Temperature, Zinc, Phosphorus	No additional limits or monitoring.
03A048	LANSCE	Los Alamos Canyon - 128	Alpha Particles; Cyanide; Mercury, Total; Polychlorinated Biphenyls (PCBs); Selenium, Total		α <1.85, CN<1.67, Hg <0.067, Se <2, PCB <0.0354		No exceed	Phosphorus	No RP for limits but monitoring for all impairments (1/year).
03A113	LEDA	Sandia Canyon - 128	Alpha Particles; Aluminum, Total; Mercury, Total; Polychlorinated Biphenyls (PCBs)		α=2.95, Al<19.3, Hg=0.011, PCB <0.354		Exceed WQS Cu 1x	Total Mercury, Alpha, Total Aluminum, Phosphorus	EPA did not evaluate RP for PCBs at this outfall. A limit appears necessary.
03A160	NMHFL	Ten Site Canyon - 128	Alpha Particles; Polychlorinated Biphenyls (PCBs)		α<0.96, PCB<0.0343	Cr6, Hg, Se, Cy	exceed Cy WQS, 2 exceed Cu WQS	Phosphorus, Mercury, Selenium, Cyanide, Chromium 6	EPA did not evaluate RP for PCBs at this outfall. A limit appears necessary.
03A181	TA-55	Mortandad Canyon - 128	Alpha Particles; Copper, Dissolved; Mercury, Total; Polychlorinated Biphenyls (PCBs)	future to SWWS?	α <0.772, Cu=3.24, Hg<0.067, PCB<0.0378		Cu 0.002	Phosphorus	It appears no RP spreadsheet was drafted for this outfall. Based on data, RP must be determined for copper and PCBs.
03A199	LDCC	Tributary to Sandia Canyon - 126	Aluminum, Total; Copper, Dissolved; Polychlorinated Biphenyls (PCBs); Temperature		Temp, Tl 0.282, Al=<19.3, Cu=3.15, PCB<0.0354		ok	Total Aluminum, T Copper, Temperature, Zn, P	RP for Thallium exists. EPA did not evaluate RP for PCBs.

Outfall Number	Description	Receiving Stream - WQ Segment	Impairments	Changes to Facility	Impaired pollutants detected (2C) (ug/L)	RP	2015-2020 monitoring	Metals Monitoring/Limit in 2020 Permit	Needed Limitations or Monitoring in Final Permit based on RP
03A022	Sigma	Mortandad Canyon - 128	Alpha Particles; Copper, Dissolved; Mercury, Total; Polychlorinated Biphenyls (PCBs)	new heat exchanger	α <1.14, Cu=5.46, Hg<0.067, PCB<0.0351		above WQS for copper	Dissolved Copper-report	EPA did not evaluate RP for PCBs. Monitoring requirements must stay in the permit for copper (1/year).
05A055	HEWTF	Canon de Valle - 128	Alpha Particles		not present	Al, Cu, Pb, Se, Zn	No discharge	TNT, RDX, perchlorate, Aluminum, Copper, Lead, Selenium, Zinc	No additional limits or monitoring.
051	RLWTF	Mortandad Canyon - 128	Alpha Particles; Copper, Dissolved; Mercury, Total; Polychlorinated Biphenyls (PCBs)		α=2.22, Cu=11, PCB<0.0378, Hg <0.067	Cu		Dissolved Copper	RP exists for Thallium. EPA did not evaluate RP for PCBs.

Comments that are not Conditions of Certification:

Comment 1: There appears to be a typo in Footnote 5 for Outfall 001. NMED proposes revision to delete last sentence "6T3 Temperature of 20°C (68°F) shall not be exceeded for six or more consecutive hours in a 24-hour period on more than three consecutive days. ~~Daily maximum temperature shall be determined by 6T3 temperature record when 6T3 temperature~~ ."

Comment 2:
Please ensure that all of the notices of change submitted by LANL since the 2019 NPDES Permit Re-Application was submitted on March 26, 2019 are incorporated.

- Revision 3 to Outfall 03A048 fact sheet to add a Chlorine monitoring system, submitted July 14, 2020 (EPC-DO: 20-222)
- Revision 3 to the Outfall 001 Flow Diagram which addresses improvements made to reduce the temperature of effluent discharged to the outfall as follows:
    - Piping modification to allow for effluent stored in the Reuse Tank to be routed (as needed) to the power plant cooling tower prior to discharge.
    - Piping modification to allow for blowdown associated with the Strategic Computing Complex (SCC) Cooling Towers to be routed to the Reuse Tank where (as needed) it can either be recycled to SERF or routed to the power plant cooling tower prior to discharge.

  This change will not increase the volume or impact the effluent quality (i.e., no new chemicals) other than to reduce the temperature. This change was submitted as a notice of change on July 16, 2020 (EPC-DO: 20-221).
- Renovation of the power plant. This change was submitted as a notice of change on November 26, 2019 (EPC-DO: 19-430). This will increase the volumes at Outfall 001 as indicated below, and were incorporated into the antidegradation calculations.

Table 5
Potential New Future Flow Rates and Frequencies for Discharges to Outfall 001

Potential Future Source	Frequency		Flow Rates and Volumes				
	Days/Week	Months	Average (MGD)	Maximum (MGD)	Average Volume (GPD)	Maximum Volume (GPD)	Duration (days)
SCC Cooling Towers [a, b]	7.0	12	0.074	0.201	74,436	201,056	365
Power Plant Co-Generation Renovation	7.0	12	0.170	0.220	169,920	220,320	365
~~TA-55-006 Cooling Towers [c]~~	~~7.0~~	~~12~~	~~0.009~~	~~0.032~~	~~9,365~~	~~31,986~~	~~365~~
Future Outfall 001 Total [c]	7.0	12	~~0.189~~ 0.311	~~0.439~~ 0.751	~~199,320~~ 310,595	~~438,586~~ 752,463	365

a. See the permit section provided for Outfall 03A027 for a schematic showing this change.
b. Cooling tower blowdown calculated for the operation of 15 towers.
~~b.~~ c. Total volume estimate for four source facilities: SWWS Effluent; SERF Effluent; SCC Cooling Towers; and Power Plant Co-Generation Renovation. All four facilities are hydraulically connected and eventually discharge water to Outfall 001 regardless of flow path.

- Startup of 5 additional Cooling Towers at the SCC. This modification was included as a future change in the 2019 NPDES Permit Application submitted March 26, 2019 (see EPC-DO: 19-106).

AMIGOS BRAVOS' EXHIBIT 13

Region 6
1201 Elm Street, Suite 500
Dallas, Texas 75270-2102

NPDES Permit No. NM0028355

AUTHORIZATION TO DISCHARGE UNDER THE NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM

In compliance with the provisions of the Clean Water Act, as amended, (33 U.S.C. 1251 et. seq; the "Act"),

Triad National Security, LLC Los Alamos National Laboratory PO Box 1663, K491 Los Alamos, New Mexico 87544	AND	U.S. Department of Energy Los Alamos Area Office, A316 3747 West Jemez Road Los Alamos, NM 87544

are authorized to discharge from a facility located at Los Alamos,

to receiving waters named: Perennial portion of Sandia Canyon in Waterbody Segment No. 20.6.4.126, and Mortandad Canyon, Canada del Buey, Los Alamos Canyon, ephemeral portion of Sandia Canyon, Ten Site Canyon, and Canon de Valle, in Waterbody Segment No. 20.6.4.128 of the Rio Grande Basin,

in accordance with this cover page and the effluent limitations, monitoring requirements, and other conditions set forth in Parts I [Requirements for NPDES Permits], II [Other Conditions], III [Standard Conditions for NPDES Permits], and IV [Sewage Sludge Requirements] hereof.

This permit, prepared by Isaac Chen, Environmental Engineer, Permitting Section (6WDPE), supersedes and replaces NPDES Permit No. NM0028355 issued August 12, 2014, then modified March 27, 2015, with an expiration date of September 30, 2019.

This permit shall become effective on

This permit and the authorization to discharge shall expire at midnight,

Issued on

_____
Charles W. Maguire
Director
Water Division (6WQ)

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PART I - REQUIREMENTS FOR NPDES PERMITS

A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS

OUTFALL 001

Discharge Type: Continuous
Latitude 35°52'26"N, Longitude 106°19'09"W (TA-3-22)

During the period beginning the effective date of the permit and lasting through the expiration date of the permit (unless otherwise noted), the permittee is authorized to discharge Power Plant waste water from cooling towers, boiler blowdown drains, demineralizer backwash, R/O reject, and including treated sanitary wastewater effluent from the Sanitary Wastewater System (SWWS) Facility, recycled sanitary effluent from the Sanitary Effluent Reclamation Facility (SERF), and treated cooling tower blowdown from the Strategic Computing Complex (SCC) to Sandia Canyon, and the discharge creates a perennial portion of Sandia Canyon, Segment Number 20.6.4.126 of the Rio Grande Basin.

Such discharges shall be limited and monitored by the permittee as specified below:

EFFLUENT CHARACTERISTIC	DISCHARGE LIMITATIONS				MONITORING REQUIREMENTS	
	CONCENTRATION (mg/L, unless stated)		LOADING (Lbs/day, unless stated)		FREQUENCY	SAMPLE TYPE
	MONTHLY AVERAGE	DAILY MAXIMUM	MONTHLY AVERAGE	DAILY MAXIMUM		
Flow (MGD)	***	***	Report	Report	Continuous	Record
TSS	30	100	Report	Report	1/Month	24-hr Composite
BOD (*1)	30	45	73	109	1/Month	24-hr Composite
E. Coli (#/100 ml) (*2)	126	410	***	***	2/Month	Grab
Total Residual Chlorine	***	0.011 (*3)	***	***	1/Week	Grab
Total Recoverable Aluminum	Report	Report	***	***	1/Year	Grab
Total Copper	0.0087	0.0087	***	***	1/Year	Grab
Total Zinc	0.126 (*4)	0.126 (*4)	***	***	1/Year	Grab
6T3 Temperature (°C)	20°C (*5)	***	***	***	1/Hour	Grab (or Continuous Record)
Total PCB (µg/l) (*6)	0.00064	0.00064	Report	Report	1/Year	24-hr Composite

pH (Standard Unit)	Range from 6.6 to 8.8	***	***	1/Week	Grab

EFFLUENT CHARACTERISTICS	DISCHARGE MONITORING	MONITORING REQUIREMENTS	
WHOLE EFFLUENT TOXICITY (*7) (7-day Chronic Static Renewal)	VALUE	MEASUREMENT FREQUENCY	SAMPLE TYPE
Ceriodaphnia dubia (Limit)	100%	1/6-Months	24-Hr Composite
Pimephales promelas	Report	1/5-Years	24-Hr Composite

FOOTNOTES

*1 BOD monitoring is required when discharges of treated sanitary waste occur at Outfall 001.

*2 Geometric mean. Effluent limitations and monitoring requirements only apply when effluent from Outfall 13S is rerouted and discharged at Outfall 001.

*3 Effluent limitation for TRC is the instantaneous maximum and cannot be averaged for reporting purposes.

*4 Effluent limitations take effective on the date of three years from the effective date of the permit.

*5 6T3 Temperature of 20°C (68°F) shall not be exceeded for six or more consecutive hours in a 24-hour period on more than three consecutive days. Daily maximum temperature shall be determined by 6T3 temperature record when 6T3 temperature.

*6 EPA published congener Method 1668 Revision and detection limits shall be used. [The permittee is allowed to develop an effluent specific MDL in accordance with Appendix B of 40 CFR Part 136 (instructions in Part II.A of this permit).] Human health-based limitations.

*7 Critical dilution 100%, and the dilution series are 32%, 42%, 56%, 75%, 100%. See Part II, Section G. Whole Effluent Toxicity (7-Day Chronic Testing). WET limit applies to Ceriodaphnia dubia. WET monitoring only applies to Pimephales promelas.

SAMPLING LOCATION(S)

Samples taken in compliance with the monitoring requirements specified above shall be taken at the following location(s): following final treatment and prior to or at the point of discharge from Outfall 001.

NO DISCHARGE REPORTING

If there is no discharge event at this outfall during the sampling month, place an "X" in the NO DISCHARGE box in the Discharge Monitoring Report. Electronic DMR reporting will use the appropriate "No Discharge" or "NODI" codes such as NODI code C= No discharge.

FLOATING SOLIDS, OIL AND GREASE

There shall be no discharge of oils, scum, grease and other floating materials that would cause the formation of a visible sheen or visible deposits on the bottom or shoreline, or would damage or impair the normal growth, function or reproduction of human, animal, plant or aquatic life.

D. APPLICATION

A complete copy of application with original officer signature for permit renewal shall be sent to EPA and either a paper copy or an electronic copy shall be sent to New Mexico Environment Department (NMED) at the mailing address listed in Part III of this permit.

AMIGOS BRAVOS' EXHIBIT 14

NEW MEXICO
ENVIRONMENT DEPARTMENT

Harold Runnels Building
1190 Saint Francis Drive, PO Box 5469
Santa Fe, NM 87502-5469
Telephone (505) 827-2855
www.env.nm.gov

Michelle Lujan Grisham
Governor

Howie C. Morales
Lt. Governor

James C. Kenney
Cabinet Secretary

Jennifer J. Pruett
Deputy Secretary

Original via FedEx-Copy via Electronic Mail

November 30, 2020

Mr. Charles Maguire, Director
Water Quality Protection Division (6WD)
U. S. Environmental Protection Agency
1201 Elm Street, Suite 500
Dallas, Texas 75202

Re: State Certification Los Alamos National Laboratory Individual Stormwater Permit, NM0030759

Dear Mr. Maguire:

Enclosed, please find the state certification for the following proposed National Pollutant Discharge Elimination System (NPDES) permit NM0030759, Los Alamos National Laboratory Individual Stormwater Permit. If any, comments and conditions are enclosed on separate sheets.

U.S. Environmental Protection Agency (USEPA) proposes to regulate discharges under the above-referenced NPDES Individual Permit. A state Water Quality Certification is required by the federal Clean Water Act (CWA) Section 401 to ensure that the action is consistent with state law (New Mexico Water Quality Act, New Mexico Statutes Annotated (NMSA) 1978, Sections 74-6-1 to -17,) and complies with State of New Mexico Water Quality Standards, Water Quality Management Plan and Continuing Planning Process, including Total Maximum Daily Loads (TMDLs), and Antidegradation Policy.

Pursuant to State regulations for permit certification at 20.6.2.2001 New Mexico Administrative Code (NMAC), USEPA jointly with NMED issued a public notice of the draft permit and announced a public comment period posted on the NMED web site at https://www.env.nm.gov/surface-water-quality/public-notices/ on November 30, 2019. The NMED public comment period ended on November 2, 2020. NMED received comments from Amigos Bravos, the Buckman Direct Diversion Board and from a private citizen, which were considered in this certification.

Sincerely,

Sarah Holcomb Digitally signed by Sarah Holcomb Date: 2020.11.30 11:32:34 -07'00' for

Shelly Lemon, Bureau Chief
Surface Water Quality Bureau

Mr. Ken McQueen, Regional Administrator
Environmental Protection Agency
1201 Elm Street, Suite 500
Dallas, TX 75202

November 30, 2020

STATE CERTIFICATION

RE: **Los Alamos National Laboratory Individual Stormwater Permit, NM0030759**

Dear Regional Administrator McQueen:

The Cabinet Secretary of the New Mexico Environment Department (NMED) has delegated signatory authority for state certifications of federal Clean Water Act permits to the Surface Water Quality Bureau Chief. NMED examined the proposed NPDES permit referenced above. The following conditions are necessary to assure compliance with the applicable provisions of the Clean Water Act Sections 208(e), 301, 302, 303, 306, and 307 and with appropriate requirements of State law. Compliance with the terms and conditions of the permit and this certification will provide reasonable assurance that the permitted activities will be conducted in a manner which will not violate applicable water quality standards and the water quality management plan and will be in compliance with the antidegradation policy.

The State of New Mexico:

( ) certifies that the discharge will comply with the applicable provisions of Sections 208(e), 301, 302, 303, 306 and 307 of the Clean Water Act and with appropriate requirements of State law

(X) certifies that the discharge will comply with the applicable provisions of Sections 208(e), 301, 302, 303, 306 and 307 of the Clean Water Act and with appropriate requirements of State law upon inclusion of the following conditions in the permit (see attachments)

( ) denies certification for the reasons stated in the attachment

( ) waives its right to certify

In order to meet the requirements of State law, including water quality standards and appropriate basin plan as may be amended by the water quality management plan, each of the conditions cited in the draft permit and the State certification shall not be made less stringent, unless changes are in response to formal comments received by USEPA and discussed with NMED prior to the finalization of the draft permit.

The Department reserves the right to amend or revoke this certification if such action is necessary to ensure compliance with the State's water quality standards and water quality management plan.

Please contact Sarah Holcomb at (505) 827-2798, if you have any questions concerning this certification. Comments and conditions pertaining to this draft permit are attached.

Sincerely,

Shelly Lemon, Bureau Chief
Surface Water Quality Bureau

upstream into Starmers gulch and Starmers spring and Water canyon from Area-A canyon upstream to State Route 501.
A. Designated Uses: *coldwater aquatic life, livestock watering, wildlife habitat and secondary contact.*
B. Criteria: *the use-specific numeric criteria set forth in 20.6.4.900 NMAC are applicable to the designated uses.*

20.6.4.128 NMAC. ***RIO GRANDE BASIN - Ephemeral and intermittent portions of watercourses within lands managed by U.S. department of energy (DOE) within LANL, including but not limited to: Mortandad canyon, Cañada del Buey, Ancho canyon, Chaquehui canyon, Indio canyon, Fence canyon, Potrillo canyon and portions of Cañon de Valle, Los Alamos canyon, Sandia canyon, Pajarito canyon and Water canyon not specifically identified in 20.6.4.126 NMAC. (Surface waters within lands scheduled for transfer from DOE to tribal, state or local authorities are specifically excluded.)***
A. Designated Uses: *livestock watering, wildlife habitat, limited aquatic life and secondary contact.*
B. Criteria: *the use-specific criteria in 20.6.4.900 NMAC are applicable to the designated uses, except that the following segment-specific criteria apply: the acute total ammonia criteria set forth in Subsection K of 20.6.4.900 NMAC (salmonids absent).*

NPDES regulations at 40 CFR 122.44(d)(l)(i) require that permit "*...limitations must control all pollutants or pollutant parameters... which the Director determines are or may be discharged at a level which will cause, have the reasonable potential to cause, or contribute to an excursion above any State water quality standard...*"

NPDES regulations at 40 CFR 124.53(e)(2) require:
When the State certifies a draft permit instead of a permit application, any conditions more stringent than those in the draft permit which the State finds necessary to meet the requirement listed in paragraph (e)(l) of this section. For each more stringent condition, the certifying State agency shall cite the CWA or State law references upon which that condition is based.

The following conditional certification includes references to USEPA's "Procedures for Implementing NPDES Permits in New Mexico – NMIP." The NMIP establishes procedures to effectively incorporate state WQS and total maximum daily loads (TMDLs) into NPDES permits. The State of New Mexico Statewide Water Quality Management Plan and Continuing Planning Process (WQMP/CPP), approved by the WQCC on September 21, 2020 and USEPA on October 23, 2020 states, among other things, *"as the current NPDES permitting authority for NM, EPA Region 6 develops effluent limitations and schedules of compliance in accordance with the NMIP, which is based on applicable federal regulations and guidance."* The current version of the NMIP prepared by USEPA Region 6 Permits Branch in consultation with the NMED Surface Water Quality Bureau (SWQB) is dated March 15, 2012.

This certification includes a number of appendices to assist in organizing information related to the conditions included below. These appendices include the following information:

- Appendix 1: Documentation of sampler moves during the Sampling Implementation Plan investigation in 2016-2018.
- Appendix 2: Proposed SSD guidance flow chart
- Appendix 3: Sites proposed for deletion that NMED must conditionally include
- Appendix 4: Sites conditioned for addition to the draft permit
- Appendix 5: TALs conditioned for addition to the draft permit
- Appendix 6: Sediment Decision Tree

requirement of State law set forth in such certification, and shall become a condition on any Federal license or permit subject to the provisions of this section." 33 U.S.C. §1341(d) (emphasis added).

State certification regulations, which implement NMSA 1978, § 74-6-5(B), state that, "[t]he purpose of such certification is to reasonably ensure that the permitted activities will be conducted in a manner that will comply with applicable water quality standards, including the antidegradation policy, and the statewide water quality management plan." 20.6.2.2001(A) NMAC. In addition to these, other federal code provisions apply to how, when and to what extent the state can issue its certification. NPDES regulations found at 40 CFR 122.44 require that permit, "[l]imitations must control all pollutants or pollutant parameters...which the Director determines are or may be discharged at a level which will cause, have the reasonable potential to cause, or contribute to an excursion above any State water quality standard." 40 CFR 122.44(d)(1)(i). NPDES regulations at 40 CF. 122.44 generally provide that the State requesting a condition in an NPDES permit should first consider existing controls on point and non-point sources, variability of the pollutant, sensitivity of species to the toxin(s), and the potential dilution of the receiving waters. 40 CFR 122.44(d)(1)(ii). Next, the USEPA must then include the effluent limit for the specific pollutant. 40 CFR 122.44(d)(1)(iii)-(iv). Additionally, where the state proposes a term or condition that is more stringent than included in the draft permit, the state must cite the specific CWA or state law reference on which it is based. 40 CFR 124.53(e)(2).

Among the information presented in USEPA's Fact Sheet, language in the Draft Permit, and Public and Permittee comments, were issues concerning PCB monitoring, effluent limitations, reporting and required methods in the permit action. Below, NMED provides an explanation for why specific PCB monitoring conditions are necessary for State certification. The following table summarizes the applicable PCB numeric criteria from 20.6.4.900.J(2) NMAC for the receiving waters of this permit action:

		Aquatic Life			
Pollutant	Wildlife Habitat	Acute	Chronic*	Human Health-Organism Only	Type of Pollutant
PCBs	0.014 µg/L	2 µg/L	0.014 µg/L	0.00064 µg/L	Chronic, Persistent

*Note: * Chronic Aquatic Life Criterion does not apply to Segment 20.6.4.128 with a designated use of Limited Aquatic Life*

Although the Aroclor Method is the only EPA approved method for PCBs in 40 CFR 136.3, this method is not sufficiently sensitive to assure that the Permittees will comply with the applicable effluent limit for PCBs contained within the permit and thus cannot be used for monitoring or compliance purposes under state law. The following demonstrates the method detection limit (MDL) and method quantitation limit (MQL) limits of several PCB testing methods:

Method	MDL	MQL
EPA Method 608 (Aroclor)	0.065 µg/L	0.02145 µg/L
EPA Method 625	30 µg/L	99 µg/L
SM 6410 B	30 µg/L	99 µg/L
EPA Method 1668C	7-30 pg/L	23-99 pg/L (0.000023-0.000099 µg/L)

Notes: EPA Method 1668 Revision A became Revision C in the May 18, 2012 Federal Register notice of 40 CFR Part 136.

The Aroclor method's MQL is an order of magnitude above the effluent limitation provided in this draft permit as necessary to comply the State WQS. As documented above, the congener method, EPA Method

NMED has specific concerns that a static list of monitoring locations and parameters as proposed in this permit is not appropriately protective of state Water Quality Standards. Under the requirements of both the administratively continued Individual Permit and the 2005 and 2016 Compliance Orders on Consent executed by NMED and the U.S. Department of Energy (2005 and 2016 Consent Order), the Permittees in collaboration with NMED conducted investigations concerning the history of sites covered under this permit. As a result, there is more available information about constituents present at these sites, as well as information about how these sites were used. It is not clear that all of this new information was used to inform the monitoring requirements in the draft permit. Furthermore, additional information will be acquired during the upcoming five-year permit term. Due to the scope and complexity of sites and site information related to this permit, a static list of monitoring locations and parameters should not be used.

In the 401 Certification on the publicly noticed draft permit in 2015, NMED required the development of a Sampling Implementation Plan (SIP) for the permittees to investigate each SWMU under this permit concurrent with available and newly collected soil data under the 2016 Consent Order in order to better inform Clean Water Act permitting requirements that will protect water quality in the receiving waters of the Plateau. NMED and LANL have worked together to complete this task for most of the sites under the administratively continued permit, which resulted in approximately 37% (147 out of 405 SWMUs) requiring the addition of various constituents to the monitoring suite based on historic industrial activities at the site. Additionally, there were 55 out of 250 (22%) sampler moves required during the SIP in order to appropriately monitor certain sites and obtain representative samples, and there were 27 out of 250 (11%) investigational samplers required in order to capture runoff from sites where one sampler was not adequate, or the sampling mechanism shifted to looking at run-on versus runoff characteristics at the site. However, many sites did not have comprehensive soil sampling information available because investigations under RCRA had not been completed yet. Appendix 1 shows the sites where samplers were moved/adjusted, and the sites where investigational samplers were added, or the sampling mechanism was changed to a run-on/runoff setup. The SIP must reflect a dynamic, adaptive process to update sampling suites based on new information with the **approval** of EPA and/or NMED. The Permittees have also requested a mechanism for feedback on determinations where Pollutants of Concern are no longer an issue at a site.

The current draft permit seems to allow for the Permittees to modify Target Action Levels (TALs) and Background Threshold Values (BTVs) values during the term of the permit (through the SIP process) without any prior approval or involvement from EPA or NMED. This is not appropriate. TALs should be and are based (as a conservative measure) on water quality standards, and BTVs should be set to a static number and updated with each permit term as appropriate. The only number that could potentially change is the composite BTV that is derived for each site during the annual SIP process. That is based on the ratio of pervious to impervious area.

Condition #3 (SIP Changes and Approval):
The Permittees must consult with NMED prior to sending SIP updates to EPA for approval. The SIP must also be publicly noticed for 30 days. EPA must add an approval process for proposed SIP changes to monitoring locations (beyond small location changes needed to address erosion) or constituent suite additions.

EPA must also specify that site deletions and monitoring deletions are not allowed without modifying the permit as required by 40 CFR 122.62(a)(2) unless it is considered a minor modification under 122.

Background for Condition #4 (Monitoring Requirements):

For aluminum, the criteria are based on analysis of total recoverable aluminum in a sample that is filtered to minimize mineral phases as specified by the department.

Condition #4 (Monitoring requirements):

TALs must be added to sites based on additional information. For example, if the receiving waterbody is impaired for a specific constituent, and that constituent was a material historically managed at the site, the constituent should be monitored in stormwater runoff. Specific information on various TAL updates is required below.

1. The draft permit indicates that sampler locations should be updated based on the annual SIP process. The draft permit must also indicate that TALs should be added or updated where appropriate based on additional information (e.g., soil data, impairment determinations).

2. Based on review of site histories and available soil screening data collected under the 2016 Consent Order, NMED requires that the TALs noted in Appendix 5 of this Certification are added to appropriate sites covered by this permit, as outlined in Appendix 1 to this certification. These TALs are reflective of current water quality standards that are applicable to the current water quality designated uses in segments 20.6.4.126 and 20.6.4.128 NMAC.

3. Consistent with the updated hardness data submitted with the Permittees' comments, the TAL table in Appendix C of the draft permit must be adjusted slightly to the following:

Major Canyon	Dissolved Hardness (mg/L)	Total Recoverable Aluminum (ug/L)	Dissolved Cadmium (ug/L)	Dissolved Chromium III (ug/L)	Dissolved Copper (ug/L)	Dissolved Lead (ug/L)	Dissolved Nickel (ug/L)	Dissolved Silver (ug/L)	Dissolved Zinc (ug/L)
Ancho	37.2	883	0.71	253	5.0	22	203	0.6	65
Chaquehui	26.9	566	0.54	194	4.0	15	154	0.3	48
Los Alamos/ Pueblo	33.5	765	0.65	233	5.0	19	186	0.5	59
Mortandad	29.5	643	0.58	210	4.0	17	167	0.4	53
Pajarito	30.2	664	0.59	214	4.0	17	170	0.4	54
Sandia	43.0	1077	0.8	285	6.0	25	229	0.8	74
Water/ Cañon de Valle	47.7	1241	0.88	311	7.0	29	250	0.90	82

4. In the proposed permit, in Part I.B (Applicable Target Action Levels), the following footnote should be added to the TAL table for monitoring requirements to specify sample collection procedures for total recoverable aluminum:

   The acute and chronic aquatic life criteria for aluminum are based on analysis of total recoverable aluminum in a sample that is filtered to minimize mineral phases as specified by the department. If stream turbidity is greater than 30 NTUs, the sample must be filtered using a 10-µm filter prior to acidification. If there are equipment problems prohibiting the measurement of turbidity in the field and the water has any cloudiness as determined by visual inspection, then the total recoverable aluminum sample should be filtered using a 10-µm filter.

Background for Condition #5:

DP	20.6.4.128	• Los Alamos Canyon to grade control: PCBs, total recoverable aluminum, adjusted gross alpha • Grade control to upper LANL bnd: dissolved copper, PCBs, total recoverable aluminum, adjusted gross alpha	No changes
Fence	20.6.4.128	Not assessed.	No changes
Graduation	20.6.4.98	Pueblo Canyon to headwaters: PCBs, dissolved copper	No changes
Los Alamos	20.6.4.128	• DP to Upper LANL boundary: PCBs, total recoverable cyanide, total recoverable selenium, adjusted gross alpha, total mercury • NM-4 to DP Canyon: adjusted gross alpha, PCBs, total recoverable aluminum, total recoverable cyanide, radium 226+228, total mercury	No changes
Mortandad	20.6.4.128	within LANL: adjusted gross alpha, PCBs, dissolved copper, total mercury	No changes
North Fork Ancho	20.6.4.128	Ancho Canyon to headwaters: adjusted gross alpha, PCBs	No changes
Pajarito	20.6.4.126 (Arroyo de la Delfe to Starmers), 20.6.4.128	• Arroyo de la Delfe to Starmers Spring: fully supporting • Within LANL above Starmers Gulch: total recoverable aluminum, adjusted gross alpha • Lower LANL boundary to Two Mile: PCBs, total recoverable aluminum, adjusted gross alpha, total recoverable cyanide, dissolved copper • Two Mile to Arroyo de la Delfe: PCBs, dissolved silver, dissolved copper, adjusted gross alpha	No changes
Potrillo	20.6.4.128	above Water Canyon: adjusted gross alpha	No changes
Pratt	20.6.4.128	Not assessed.	No changes
Pueblo	20.6.4.98	• Acid Canyon to headwaters: PCBs, total recoverable aluminum, adjusted gross alpha, dissolved copper • Los Alamos Canyon to Los Alamos WWTP: adjusted gross alpha, PCBs, total recoverable aluminum, total recoverable selenium • Los Alamos WWTP to Acid Canyon: PCBs, adjusted gross alpha	No changes
Rendija	20.6.4.98	Guaje Canyon to headwaters: Not assessed	No changes
Sandia	20.6.4.126 (Sigma to Outfall 001), 20.6.4.128	• Sigma Canyon to NPDES Outfall 001: total recoverable aluminum, PCBs, dissolved copper, temperature • within LANL below Sigma: PCBs, total recoverable aluminum, adjusted gross alpha, total mercury, dissolved copper	No changes

(B) Effluent limits developed to protect a narrative water quality criterion, a numeric water quality criterion, or both, are consistent with the assumptions and requirements of any available wasteload allocation for the discharge prepared by the State and approved by EPA pursuant to 40 CFR 130.7.

(2) Attain or maintain a specified water quality through water quality related effluent limits established under section 302 of CWA;

(3) Conform to the conditions to a State certification under section 401 of the CWA that meets the requirements of §124.53 when EPA is the permitting authority. If a State certification is stayed by a court of competent jurisdiction or an appropriate State board or agency, EPA shall notify the State that the Agency will deem certification waived unless a finally effective State certification is received within sixty days from the date of the notice. If the State does not forward a finally effective certification within the sixty day period, EPA shall include conditions in the permit that may be necessary to meet EPA's obligation under section 301(b)(1)(C) of the CWA;

(4) Conform to applicable water quality requirements under section 401(a)(2) of CWA when the discharge affects a State other than the certifying State;

(5) Incorporate any more stringent limitations, treatment standards, or schedule of compliance requirements established under Federal or State law or regulations in accordance with section 301(b)(1)(C) of CWA;

(6) Ensure consistency with the requirements of a Water Quality Management plan approved by EPA under section 208(b) of CWA;

(7) Incorporate section 403(c) criteria under part 125, subpart M, for ocean discharges;

(8) Incorporate alternative effluent limitations or standards where warranted by "fundamentally different factors," under 40 CFR part 125, subpart D;

(9) Incorporate any other appropriate requirements, conditions, or limitations (other than effluent limitations) into a new source permit to the extent allowed by the National Environmental Policy Act, 42 U.S.C. 4321 et seq. and section 511 of the CWA, when EPA is the permit issuing authority. (See §122.29(c)).

Additional site-related constituents for evaluation in stormwater

According to 40 CFR 122.44(d)(1)(vi), if there are known constituents being discharged from a facility that have the reasonable potential to cause or contribute to a narrative water quality standard violation where a State has not developed accompanying numeric water quality criteria, EPA must develop effluent limits for those pollutants. Through data review of stormwater data publicly available through IntellusNM, NMED SWQB has observed that the following pollutants are being discharged in amounts that may contribute to exceedances of the narrative criteria at 20.6.4.13(F) and (G) NMAC, Toxic Pollutants and Radioactivity, respectively. The dataset obtained from IntellusNM was targeted to stormwater samples and organized by canyon, where a geometric mean of the available data was calculated. Some datasets did not have much information, such as lithium (data for only one canyon was available).

Specific to perfluorinated compounds

New Mexico regulations (Standards for Interstate and Intrastate Surface Waters) under 20.6.4.13(F) NMAC state: Except as provided in 20.6.4.16 NMAC, surface waters of the state shall be free of toxic pollutants from other than natural causes in amounts, concentrations or combinations that affect the propagation of fish or that are toxic to humans, livestock or other animals, fish or other aquatic organisms, wildlife using aquatic environments for habitations or aquatic organisms for food, or that will or can reasonably be expected to bioaccumulate in tissues of fish, shellfish and other aquatic organisms to levels that will impair the health of aquatic organisms or wildlife or result in unacceptable tastes, odors or health risks to human consumers of aquatic organisms.

Other states' PFAS guidance for various surface and groundwater screening levels are indicated in the tables below.[7,8]

Surface Water PFAS Guidelines in Other States				
	Oregon (ug/L)*	Michigan (ug/L)** DWS/not DWS	Minnesota (ug/L) Rivers	Alaska, Montana (ug/L)***
PFHpA	300	-	-	-
PFOA	24	0.420/12	2.7	0.070
PFOS	300	0.011/0.012	0.007	0.070
PFOSA	0.2	-	-	-
PFNA	1	-	-	-

* The Oregon DEQ wastewater initiation levels were adopted into rule (OAR 340-045-0100, Table A) in 2011. The PFAS are 5 chemicals on a list of 118 persistent priority pollutants for water that Oregon DEQ developed in response to state legislation. *Municipal wastewater treatment plants with effluent exceeding initiation levels are required to develop a pollution prevention plan that becomes a part of their NPDES permit.*

** Michigan's advisory levels are designed to protect human health (non-cancer values) and are based on whether the surface water is a drinking water source (DWS) or not.

*** For these states, concentrations of PFOA and PFOS are summed before being compared to the screening level.

Groundwater PFAS Guidelines in Other States						
	Maine (ug/L)*	New Jersey (ug/L)	New Hampshire (ug/L)**	Colorado, Rhode Island, Delaware (ug/L)*	Illinois (ug/L) ***	Minnesota (ug/L) ****
PFHpA	-	-	-	-	-	-
PFOA	0.400	0.010	0.012	0.070	0.021	0.035
PFOS	0.400	0.010	0.015	0.070	0.014	0.027
PFOSA	-	-	-	-	-	-
PFNA	-	-	0.011	-	0.021	-

* For these states, concentrations of PFOA and PFOS are summed before being compared to the screening level.

** Proposed rulemaking in New Hampshire covers 4 PFAS, and includes PFHxS = 0.018 ug/L.

*** Proposed rulemaking in Illinois covers 5 PFAS, and includes PFHxS = 0.140 ug/L and PFBS = 140 ug/L.

**** Health-based values (not maximum contaminant levels, or MCLs).

States use a variety of methods to test PFAS analytes in different media. The most widely used are EPA Method 537 (2008, applies to 14 PFAS) and EPA Method 537.1 (2018, applies to 18 PFAS). Some labs perform modifications, like using isotope dilution, to these methods for use in other matrices besides drinking water to account for lower reporting limits or greater accuracy. For example, modifications to Method 537.1 can be applied for non-drinking water media.[7]

Monitoring these toxic contaminants helps provide information about whether they are present in stormwater discharges to better control and mitigate PFAS in the environment. As stated on EPA's PFAS website,[9] *"PFAS can be found in living organisms, including fish, animals, and humans, where PFAS have the ability to build up and persist over time." Due to the characteristics of these contaminants (i.e., persistence in the environment and the human body, and evidence that exposure to PFAS can lead to adverse human health effects), NMED advocates for taking a proactive approach and establish PFAS sampling and reporting requirements to assure protection of New Mexico's surface waters, public health and the environment.*

1 https://www.epa.gov/toxics-release-inventory-tri-program/list-pfas-added-tri-ndaa
2 https://www.govinfo.gov/content/pkg/FR-2020-06-22/html/2020-10990.htm, or

Constituents	**CAS Number**	**Other Evaluated Criteria**	**National Recommended Water Quality Criteria (µg/L)[2]**			**Highest Canyon Concentration (geomean) observed in stormwater (µg/L, unless otherwise noted)**
			HH-water + organism	**HH-OO**	**NMED WQS ug/L**	
Thorium[9]	7440-29-1					No stormwater data
Tungsten	7440-33-7					No stormwater data
Anthracene	120-12-7				40,000	0.064849
Benzo(a)anthracene	56-55-3				0.18	0.06 (across plateau but data shows individual exceedances of the standard)
Benzo(b)fluoranthene	205-99-2				0.18	0.06 (across plateau but data shows individual exceedances of the standard)
Benzo(k)fluoranthene	207-08-9				0.18	0.03 (across plateau but data shows individual exceedances of the standard)
Bis (2-ethylhexyl) phthalate	117-81-7				22	1.03 (but individual exceedances of the standard)
Chrysene	218-01-9				0.18	0.08 (across plateau but individual exceedances of the standard)
Dibenzo(a,h)anthracene	53-70-3				0.18	0.07 (across plateau but individual exceedances of the standard)
Ethylbenzene	100-41-4				2,100	No data
Tetrachloroethylene	127-18-4				33	No data

(A) If additional sampling determines that it is unlikely that PFAS exist in a permittee's stormwater discharge, if the permittee provides facility data that demonstrate PFAS are unlikely to be present in the stormwater discharge, or there are no available, accredited laboratories capable of performing the required PFAS analysis; or
(**B**) If additional sampling demonstrates that the pollutant concentration is lower than the screening level or the permittee is subject to duplicative or more stringent PFAS requirements.
However, to be exempted for these reasons, the permittee must submit documentation to NMED for approval.

NMED requires EPA to consider if these observed pollutants may contribute to exceedances of the Toxic Pollutants and Radioactivity narrative criteria cited above. Through this evaluation, EPA may set additional TALs, or add these constituents for evaluation through the SIP process described in Condition #1.

Background for Condition #7:
EPA administered National Pollutant Discharge Elimination System (NPDES) permit programs under 40 CFR 122.26(a)(ii),122.26(b)(12) and (14), and 122.26(g) require the following:

> *§122.26 Storm water discharges*
> *122.26(a) Permit requirement. (1) Prior to October 1, 1994, discharges composed entirely of storm water shall not be required to obtain a NPDES permit except:*
> *(ii) A discharge associated with industrial activity (see §122.26(a)(4));*
>
> *122.26(b)(12) Significant materials includes, but is not limited to: raw materials; fuels; materials such as solvents, detergents, and plastic pellets; finished materials such as metallic products; raw materials used in food processing or production; hazardous substances designated under section 101(14) of CERCLA; any chemical the facility is required to report pursuant to section 313 of title III of SARA; fertilizers; pesticides; and waste products such as ashes, slag and sludge that have the potential to be released with storm water discharges.*
>
> *122.26(b)(14) Storm water discharge associated with industrial activity means the discharge from any conveyance that is used for collecting and conveying storm water and that is directly related to manufacturing, processing or raw materials storage areas at an industrial plant. The term does not include discharges from facilities or activities excluded from the NPDES program under this part 122. For the categories of industries identified in this section, the term includes, but is not limited to, storm water discharges from industrial plant yards; immediate access roads and rail lines used or traveled by carriers of raw materials, manufactured products, waste material, or by-products used or created by the facility; material handling sites; refuse sites; sites used for the application or disposal of process waste waters (as defined at part 401 of this chapter); sites used for the storage and maintenance of material handling equipment; sites used for residual treatment, storage, or disposal; shipping and receiving areas; manufacturing buildings; storage areas (including tank farms) for raw materials, and intermediate and final products; and areas where industrial activity has taken place in the past and significant materials remain and are exposed to storm water. For the purposes of this paragraph, material handling activities include storage, loading and unloading, transportation, or conveyance of any raw material, intermediate product, final product, by-product or waste product. The term excludes areas located on plant lands separate from the plant's industrial activities, such as office buildings and accompanying parking lots as long as the drainage from the excluded areas is not mixed with storm water drained from the above described areas. Industrial facilities (including industrial facilities that are federally, State, or municipally owned or operated that*

are linked to Certificates of Completion received from the NMED Hazardous Waste Bureau under the Consent Order.

While NMED concurs with the deletion of some of these SMAs (as noted in the appendices), the rest of the sites must be kept on the permit until they can be evaluated using the method in the Sampling Implementation Plan (SIP) and the proposed method to utilize soil screening data to characterize a site. During the SIP review that occurred in 2016-2018, NMED reviewed site history and soil sampling information (if available) to determine the appropriateness and thoroughness of the original monitoring requirements in the 2009 permit as referenced in Condition #1. At many of these SMA/SWMU/AOC combinations, there were additional constituents of concern identified through history review and soil sampling information that indicated that additional stormwater monitoring would be useful to characterize the site more appropriately.

The Permittees submitted comments with subsets of other requests to delete sites from the permit. The Permittees' Attachment 6 to their comments details a request to delete sites based on the receipt of Certification of Completion (CoCs) from the NMED Hazardous Waste Bureau, which NMED Surface Water Quality Bureau has stated many times in the past is not acceptable because the RCRA process does not evaluate impacts on surface water quality requirements.

The Permittees also submitted Attachment 7 to their comments with a proposal to move sites that are deferred for cleanup under the 2016 Consent Order to Long Term Stewardship until internal discussions can be arranged to identify how to handle these sites between DOE NNSA as the landlord of active operations, and DOE EM as the responsible party for legacy contamination cleanup. If EPA cannot move these sites to Sector AD of the MSGP for permit coverage, then moving these sites to Long Term Stewardship under the final permit would be acceptable as long as the Permittees are still required to maintain BMPs that mitigate stormwater impacts from legacy activities and contamination.

The Permittees also submitted Attachment 8 to their comments with another proposal for sites to be deleted that met the administratively continued permit's requirement for deletion of sites when they had collected two stormwater samples and saw results that were below TALs. Because of the evaluations during the SIP process and the ability to evaluate soil data to provide a more comprehensive understanding of the presence of pollution at these sites, EPA must rely on the results of the SIP process as presented in this certification with respect to keeping sites on the permit.

NMED notes the following four categories of deleted sites:

- **Requests to delete sites because the property has been transferred and is no longer owned by DOE**: the Permittees have deeded land over to Los Alamos County, private citizens, or the Forest Service when they have received a certificate of completion without controls from NMED HWB. The Permittees request indicated that they no longer have access to these locations to be able to perform maintenance and/or collect samples. However, NMED asked the Permittees to investigate the lease agreements or other transfer of ownership paperwork in order to confirm that this is the case. Until NMED SWQB knows the details of these land transfers, no sites in this category should be deleted from the permit because the agreements may still assign liability for legacy contaminant cleanup to the Permittees.
- **Active sites**: There are a number of "active" sites for which the Permittees have requested deletion from the permit. To define "active", these are sites that are still used for day to day operations at the laboratory, including a number of firing sites. Active sites in this category should be transferred to require coverage under the MSGP. Prior to the existence of this individual stormwater permit, these firing sites were covered under Sector K (Hazardous Waste) of the MSGP; however, Sector K monitoring requirements do not adequately characterize the

Background for Condition #8:

EPA administered National Pollutant Discharge Elimination System (NPDES) permit programs under 40 CFR 122.26(a)(ii),122.26(b)(12) and (14) require the following:

> *§122.26 Storm water discharges*
> *122.26(a) Permit requirement. (1) Prior to October 1, 1994, discharges composed entirely of storm water shall not be required to obtain a NPDES permit except:*
> *(ii) A discharge associated with industrial activity (see §122.26(a)(4));*
>
> *122.26(b)(12) Significant materials includes, but is not limited to: raw materials; fuels; materials such as solvents, detergents, and plastic pellets; finished materials such as metallic products; raw materials used in food processing or production; hazardous substances designated under section 101(14) of CERCLA; any chemical the facility is required to report pursuant to section 313 of title III of SARA; fertilizers; pesticides; and waste products such as ashes, slag and sludge that have the potential to be released with storm water discharges.*
>
> *122.26(b)(14) Storm water discharge associated with industrial activity means the discharge from any conveyance that is used for collecting and conveying storm water and that is directly related to manufacturing, processing or raw materials storage areas at an industrial plant. The term does not include discharges from facilities or activities excluded from the NPDES program under this part 122. For the categories of industries identified in this section, the term includes, but is not limited to, storm water discharges from industrial plant yards; immediate access roads and rail lines used or traveled by carriers of raw materials, manufactured products, waste material, or by-products used or created by the facility; material handling sites; refuse sites; sites used for the application or disposal of process waste waters (as defined at part 401 of this chapter); sites used for the storage and maintenance of material handling equipment; sites used for residual treatment, storage, or disposal; shipping and receiving areas; manufacturing buildings; storage areas (including tank farms) for raw materials, and intermediate and final products; and areas where industrial activity has taken place in the past and significant materials remain and are exposed to storm water. For the purposes of this paragraph, material handling activities include storage, loading and unloading, transportation, or conveyance of any raw material, intermediate product, final product, by-product or waste product. The term excludes areas located on plant lands separate from the plant's industrial activities, such as office buildings and accompanying parking lots as long as the drainage from the excluded areas is not mixed with storm water drained from the above described areas. Industrial facilities (including industrial facilities that are federally, State, or municipally owned or operated that meet the description of the facilities listed in paragraphs (b)(14)(i) through (xi) of this section) include those facilities designated under the provisions of paragraph (a)(1)(v) of this section.*

When the 2016 Consent Order was initially developed, there was a list of 2093 total Solid Waste Management Units (SWMUs) that were added to the Consent Order. The number of SWMUs that were subsequently included on this permit (405 SWMUs) were a subset of that initial list chosen based on the Permittees' assessment of whether or not the site would actually discharge stormwater. During the SIP process, NMED reviewed stormwater monitoring data and site histories, and observed that the predictions used to first select sites for inclusion on the permit was not accurate in predicting which sites would actually produce runoff. NMED noted additional SWMUs or AOCs that may need to be added to the IP in order to adequately protect surface waters from legacy activities that have yet to be mitigated/reclaimed/remediated. These sites are noted in Appendix 4, along with a description of the legacy activity and the constituents that would be of concern in stormwater runoff from the site.

(4) Certification. The no exposure certification must require the submission of the following information, at a minimum, to aid the NPDES permitting authority in determining if the facility qualifies for the no exposure exclusion:

(i) The legal name, address and phone number of the discharger (see §122.21(b));

(ii) The facility name and address, the county name and the latitude and longitude where the facility is located;

(iii) The certification must indicate that none of the following materials or activities are, or will be in the foreseeable future, exposed to precipitation:

(A) Using, storing or cleaning industrial machinery or equipment, and areas where residuals from using, storing or cleaning industrial machinery or equipment remain and are exposed to storm water;

(B) Materials or residuals on the ground or in storm water inlets from spills/leaks;

(C) Materials or products from past industrial activity;

(D) Material handling equipment (except adequately maintained vehicles);

(E) Materials or products during loading/unloading or transporting activities;

(F) Materials or products stored outdoors (except final products intended for outside use, e.g., new cars, where exposure to storm water does not result in the discharge of pollutants);

(G) Materials contained in open, deteriorated or leaking storage drums, barrels, tanks, and similar containers;

(H) Materials or products handled/stored on roads or railways owned or maintained by the discharger;

(I) Waste material (except waste in covered, non-leaking containers, e.g., dumpsters);

(J) Application or disposal of process wastewater (unless otherwise permitted); and

(K) Particulate matter or visible deposits of residuals from roof stacks/vents not otherwise regulated, i.e., under an air quality control permit, and evident in the storm water outflow;

(iv) All "no exposure" certifications must include the following certification statement, and be signed in accordance with the signatory requirements of §122.22: "I certify under penalty of law that I have read and understand the eligibility requirements for claiming a condition of "no exposure" and obtaining an exclusion from NPDES storm water permitting; and that there are no discharges of storm water contaminated by exposure to industrial activities or materials from the industrial facility identified in this document (except as allowed under paragraph (g)(2)) of this section. I understand that I am obligated to submit a no exposure certification form once every five years to the NPDES permitting authority and, if requested, to the operator of the local MS4 into which this facility discharges (where applicable). I understand that I must allow the NPDES permitting authority, or MS4 operator where the discharge is into the local MS4, to perform inspections to confirm the condition of no exposure and to make such inspection reports publicly available upon request. I understand that I must obtain coverage under an NPDES permit prior to any point source discharge of storm water from the facility. I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gathered and evaluated the information submitted. Based upon my inquiry of the person or persons who manage the system, or those persons directly involved in gathering the information, the information submitted is to the best of my knowledge and belief true, accurate and complete. I am aware there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations."

Condition #9 (No exposure qualifications):

40 CFR 122.26 (g) requires that Permittees claiming "no exposure" of industrial materials to stormwater must complete and sign a certification that there are no discharges of contaminated stormwater. The

First, New Mexico's water quality standard for adjusted gross alpha specifically takes the exemption under the AEA into account, hence the "adjustment" to gross alpha. NMED SWQB notes that the Permittees were monitoring total gross alpha under the administratively continued permit and never provided an adjusted measurement to demonstrate that values above the TAL were caused by materials exempt under the AEA. NMED recognizes that the Permittees have documented the presence of a significant number of AEA-exempted nuclides in soil data from sites covered in this permit, however they have not demonstrated that removing these constituents would result in gross alpha levels compliant with the State Water Quality Standard.

Secondly, while NMED recognizes that there may be naturally elevated levels of gross alpha within the permit area it is not appropriate to simply remove this TAL. Additionally, as the Permittees currently refer to background, they refer to the background calculated as an Upper Tolerance Limit (UTL). Rather, the proposed permit provides a mechanism through the site contributing test to address background levels of pollutants, and the Permittees can conduct run-on/runoff monitoring at Sites where natural sources of gross alpha are an issue.

NMED asserts that it is appropriate to utilize adjusted gross alpha as a TAL under this permit to ensure that this permit is protective of State Water Quality Standards. If EPA decides to remove the TAL for adjusted gross alpha from the final permit, NMED SWQB reserves the right to revoke and amend this certification as necessary.

Condition #10 (TAL for adjusted gross alpha):
EPA must retain the TAL for adjusted gross alpha in the final permit. Permittees are encouraged to investigate run-on/run-off evaluations as allowed in the SIP for SMAs where they believe naturally occurring conditions may be contributing to TAL exceedances. Additionally, if the measurement of total gross alpha shows exceedances of the adjusted gross alpha standard after investigation of run-on sources, permittees may need to collect data to evaluate adjusted gross alpha data instead of relying on total gross alpha data.

2. NMED believes that with the flexibility afforded to the Permittee in the proposed Site-Specific Demonstration (SSD) that there is no need for the alternative compliance request provision in the proposed permit. NMED suggests that it be removed to provide clarity on the Permittees' path to compliance, especially considering EPA's resources and ability to respond to alternative compliance requests (EPA did not approve a single alternative compliance request during the previous permit term). The language included in the draft permit providing automatic approval of alternative compliance requests is not appropriate and should be removed.
3. The Permittees submitted Alternative Compliance Requests for 81 sites to EPA under the administratively continued permit that were not approved or dealt with otherwise. These sites should all be addressed via the SSD process before any determinations are made to delete the sites from the permit.
4. NMED Surface Water Quality Bureau and NMED Hazardous Waste Bureau worked with the Permittees to develop a sediment removal decision tree that accounted for both hazardous waste and surface water regulatory requirements for removal of sediments accumulated in stormwater retention facilities. NMED includes this decision tree as supplemental information to this certification to assist in decision making regarding maintenance of BMPs required under this permit. The decision tree is attached as Appendix 6.
5. New Mexico Water Law codified at 19.16.2.15(B) NMAC requires that for water retained for longer than 96 hours, there must be a water right associated with that water. If the water infiltrates or is otherwise discharged, no water right is required. NMED is unclear that the permit requirements as written adequately convey that additional requirement with respect to BMPs such as retention berms and sediment ponds.
6. NMED received comments indicating that a mass balance approach should be taken regarding calculation of pollutant contributions from a site by requiring that flow measurements are taken in addition to water quality data. This would require the Permittees to install additional water quality equipment at every single SMA and would be burdensome. Additionally, no other stormwater permit issued in New Mexico requires mass loading calculations. The approach laid out by EPA to calculate the pollutant contribution by calculating the pollutant concentration upstream and subtracting it from the pollutant concentration downstream, and setting that value less than the TAL is appropriate, considering that the TALs are already conservatively set at the water quality standard.

$$(2)\ \frac{[V(\text{runoff})^* \text{ total catchment area}] - [V(\text{run-on \& precipitation})^* \text{Non-site area}]}{(\text{site area})} \leq \text{TAL}$$

7. Permittees requested in their comments to amend the above formula for the SSD process to the following:

   "Composite BTV = [(% impervious SMA area * 90th percentile developed landscape BTV) + (% pervious SMA area * 95-95 UTL 90th percentile undeveloped landscape BTV)]/ 100%"

   And they provide the following rationale:
   "The Permittees have worked diligently with EPA, NMED, and CCW regarding the development of storm water BTVs, particularly with respect to investigating data stability, data quality, and selecting sampling locations for background that are upwind of the Laboratory yet have similar elevation gradients, soil types, geologic formations, and vegetative cover (Windward, SEP DQO/DQA Document, 2017). During a series of webinars and meetings between September 2018 and January 2019, the Permittees and stakeholders discussed various statistical approaches to use for BTVs, with the Permittees proposing the 95-95 upper tolerance limit (UTL) as the most appropriate statistic for the intended use and population parameters of the background dataset.

and Background Threshold Values are listed in Appendix B and Appendix C to this permit, respectively."

12. Part 1.C.3(c) of the permit states that a site may be requested to be placed in the long term stewardship category if "storm water sample results are greater than HH-OO based TALs, but below Wildlife Habitat TALs for discharges to non-perennial streams." The Permittees have requested the ability to delete sites that fall into this category. NMED believes these sites should be kept on the permit in the long term stewardship category because we are concerned that the discharge of stormwater containing pollutants that may meet criteria for Wildlife Habitat immediately at the location of the site, may accumulate in sediments and be carried further downstream in subsequent storm events and deposited into the Rio Grande (especially in the case of Los Alamos Canyon) where there is a drinking water use and the aquatic life uses that trigger the lower HH-OO criteria.

13. The Permittees request in Part 1.C.3 to add language allowing them to place RCRA deferred sites into long term stewardship. Generally, their suggestion is acceptable to NMED, but should be clarified that BMPs should still be installed and maintained at these sites to prevent any pollutants of concern from migrating from the site. Some RCRA deferred sites are still active (i.e. firing sites that may have residual contamination from historic activities) and could alternatively be covered under Sector AD of the MSGP, so NMED asks EPA Region 6 to consider that approach for these sites as well.

14. The Permittees request that EPA delete the first sentence of the last paragraph of Part 1.C.4 because they state that there will no longer be stormwater discharges associated with industrial activity. NMED respectfully disagrees and asserts that if the installed permanent control measures are the reason that site-associated pollutants are no longer being discharged in stormwater, then maintenance requirements should exist. EPA should not delete this requirement for certification of maintenance of those permanent control measures from this permit.

15. In Part I.C.6(a), the draft permit states that if soil disturbance occurs within the Site-affected media, storm water samples collected following these activities shall be monitored for the entire suite of pollutants listed in Appendix B for that site. However, soil disturbance is not defined in this permit. NMED offers the following for clarification. Referencing other CWA stormwater permits, the Construction General Permit defines earth moving as clearing, grading and excavating activities. If any of these activities occur but are not part of BMP installation or are outside of the catchment area of a BMP within site-related media, the Permittees shall reinitiate sampling using the entire suite of pollutants listed in Appendix B for that site.

16. The permit currently states in Part I.D.1(a) that the Permittees may collect run-on and run-off data for comparison at a site to determine what the site's contribution is to pollutant loading in runoff. However, the permit does not specifically require the Permittees to do so in a paired sampling setup. Due to the major variabilities between storm events and the differing abilities for a storm to transport sediment and associated pollutants, NMED strongly recommends that EPA modify the language to require that run-on/run-off monitoring is matched from the same storm event. It would not be appropriate to compare monitoring data from a 3-year event to a 100-year event.

17. Part I.D.1(b)(ii) has a note, which states that if surface runoff from a site will penetrate deeper than three feet, the Permittees may not use this approach; this section talks about removal and replacement of three feet of surface soil with clean fill. The Permittees have requested to delete this note from the permit, but in light of the Permittees' request to use green infrastructure methods to mitigate runoff, there could be situations where green infrastructure allows the

watershed-based measure could be considered for compliance under the permit. This approach is not appropriate in all situations. There are some canyons upstream on the Plateau that are perennial and have more stringent water quality standards allocated to them. If a watershed approach were to be used and did not account for those higher quality waters upstream, then those waterbodies could potentially be degraded. NMED SWQB is concerned about the potential use of this approach without more clarification and guidance. NMED suggests the following language:

"While a watershed approach may be appropriate, Permittees must institute control measures with the understanding that upstream waters, higher in the canyons, may have more stringent water quality standards which must still be protected."

20. NMED agrees with deletion of the following sites:
    a. 00-011(c) [R-SMA-2.05]: This was an alleged former mortar impact site, but evidence of the use of the site for its alleged purpose was never found (evidence of UXO, ordnance, MD, MEC or impact scars).
    b. C-00-020 [R-SMA-0.5]: This was an alleged former mortar impact site, but evidence of the use of the site for its alleged purpose was never found (evidence of UXO, ordnance, MD, MEC or impact scars).
    c. 16-030(c) [CDV-SMA-1.4]: This site was former roof drains from a rest house building at TA-16 that has now been removed. It was never used for the management of hazardous constituents and was never comingled with another process. One stormwater sample has been collected at this SMA and showed a TAL exceedance for silver. This TAL exceedance is clearly associated with another SWMU in this SMA.
    d. 35-016(m) [PRATT-SMA-1.05]: This was a formerly NPDES permitted outfall that never discharged. It was meant to discharge noncontact cooling water from a sodium reactor in support of a cooling system. The sodium reactors were never installed and the cooling tower never operated and there was no discharge.
    e. C-46-001 [CDB-SMA-1]: This was a one-time mercury spill outside of building 46-75. According to the Permittees, the spill was cleaned up immediately and soil samples taken at the site do not show elevated levels of mercury (above background levels). A stormwater sample taken at the SMA sampler did not show TAL exceedances for mercury.
    f. 35-004(h) [PRATT-SMA-1.05]: This was a former hazardous waste satellite accumulation area. Soil was removed in this area to 15 feet and backfilled with clean soil.

AMIGOS BRAVOS' EXHIBIT 15

Region 6
1201 Elm Street, Suite 500
Dallas, Texas 75270-2102

NPDES Permit No. NM0030759

AUTHORIZATION TO DISCHARGE UNDER THE NATIONAL POLLUTANT DISCHARGE ELIMINATIONSYSTEM

In compliance with the provisions of the Clean Water Act, as amended,
(33 U.S.C. 1251 et. seq; the "Act"),

Los Alamos National Laboratory (LANL), managed and owned by Permittees

Newport News Nuclear BWXT-Los Alamos, LLC and U.S. Department of Energy
600 Sixth Street
Los Alamos, New Mexico 87544

Office of Environmental Management
Los Alamos Field Office
P.O. Box 1663
Los Alamos, New Mexico
87545-1663

is authorized to discharge storm water associated with industrial activities from specified solid waste management units (SWMUs) and areas of concern (AOCs) (as identified in Appendix A and referred to herein as “Sites”) from the facility located at Los Alamos, New Mexico, to receiving waters named:

Tributaries or main channels of Mortandad Canyon, Canada del Buey, Los Alamos Canyon, DP Canyon, Sandia Canyon, Ten Site Canyon, Canyon de Valle, Water Canyon, Ancho Canyon, Bayo Canyon, Chaquehui Canyon, Fence Canyon, Pajarito Canyon, Twomile Canyon, Threemile Canyon, Potrillo Canyon, Pueblo Canyon, and Rendija Canyon, in Water Body Segment No. 20.6.4.98, 20.6.4.126 or 20.6.4.128 of the Rio Grande Basin,

in accordance with this cover page and monitoring requirements, and other conditions set forth in the Requirements for NPDES Permits and Appendices, hereof.

This permit, prepared by Isaac Chen, Environmental Engineer, Permitting Section (6WDPE), supersedes and replaces the administratively continued NPDES Permit No. NM0030759 issued February 13, 2009, then modified September 30, 2010, with an expiration date of March 31, 2014.

This permit shall become effective on

This permit and the authorization to discharge shall expire at midnight,

Issued on

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Charles W. Maguire
Director
Water Division

APPENDIX C
STORM WATER BACKGROUND THRESHOLD VALUES (BTVS)

Total, unless indicated	CAS No.		MQL (µg/l)(*1)	ATAL (µg/l)(*2)	MTAL (µg/l)(*3)
RADIOACTIVITIES					
Ra-226 and Ra-228 (pCi/l)				30	---
METALS					
Aluminum, total recoverable	7429-90-5		2.5	---	(*4)
Antimony, dissolved (P)	7440-36-0		60	640	---
Arsenic, dissolved (P)	7440-38-2		0.5	9	340
Boron, dissolved	7440-42-8		100	5000	---
Cadmium, dissolved	7440-43-9		1	---	(*4)
Chromium, dissolved	18540-29-9		10	---	(*4)(*5)
Cobalt, dissolved	7440-48-4		50	1000	---
Copper, dissolved	7440-50-8		0.5	---	(*4)
Lead, dissolved	7439-92-1		0.5	---	(*4)
Mercury, total	7439-97-6		0.005	0.77	---
Nickel, dissolved (P)	7440-02-0		0.5	---	(*4)
Selenium, total recoverable	7782-49-2		5	5	20
Silver, dissolved	7440-22-4		0.5	---	(*4)
Thallium, dissolved (P)	7440-28-0		0.5	0.47	---
Vanadium, dissolved	7440-62-2		50	100	---
Zinc, dissolved	7440-66-6		20	---	(*4)
CYANIDE					
Cyanide, total recoverable	57-12-5		10	5.2	22
DIOXIN					
2,3,7,8-TCDD (P)	1746-01-6		0.00001	5.1E-08	---
SEMIVOLATILE COMPOUNDS					
Pentachlorophenol	87-86-5		5	---	19
Benzo(a)pyrene (P)	50-32-8		5	0.18	---
Hexachlorobenzene (P)	118-74-1		5	0.0029	---
PESTICIDES					
Aldrin (P)	309-00-2		0.01	0.0005	3

(*3) MTAL stands for Maximum Target Action Level.

(*4) Hardness-dependent metals target action levels. See Table C-1 below.

(*5) While the 20.6.4 New Mexico Administrative Code (NMAC) aquatic life standard is for chromium III, analyzing this in storm water is operationally infeasible because of the 24-hr preservation requirement. Therefore, for the purposes of this Permit, total dissolved chromium will be analyzed and compared to the hardness-dependent criteria (see Table C-1 below).

(*6) Method 1668 Revision C or the most current revision of the Congener Method shall be used for PCB analysis. Per Appendix C of 2010 Permit, the MQLs for PCB congeners 4/10, 5/8, 6, 7/9, 11, 12/13, 14, and 15 will be 50 pg/l, and the MQLs for all other PCB Congeners will be 25 pg/l. If adjusted Reporting Limits (RL) are used to adjust MQLs due to laboratory's contemporary ambient background, such adjusted RL shall be updated no less than once per 6 mo. If laboratory method blank, field blank, or trip blank subtraction are used in calculation of sample analytical result, supporting document shall be submitted with the Annual Report.

(*7) If the stream reach that an SMA drains to is classified as ephemeral (per the Clean Water Act 303(d)/305(b) Integrated Report), the total PCB wildlife habitat surface water quality criterion (0.014 µg/l from 20.6.4 NMAC) will be used as the ATAL; if the stream reach that an SMA drains to is classified as intermittent or perennial, the total PCB human health-organism aquatic life criterion (0.00064 µg/l) will be used as the ATAL.

AMIGOS BRAVOS' EXHIBIT 16

Date	Stream Name	Site ID	Map Legend	Assessment Unit	Evaluators	NMED in Attendance	Latitude	Longitude	Dec Lat	Dec Long	Drought Index	Weather Now	Weather Past 48 hours	Heavy rain in past 48 hours?	Stream Modifications	Diversions	Discharges	Water in Channel	Fish	Macroinvertebrates	Filamentous Algae/Periphyton	Diff in Vegetation	Rooted Upland Plants	Sinuosity	Floodplain Dimensions	Pool/Riffle Seq	Particle Size/Sorting	Hydric Soils	Sediment on Plants/Debris	Seeps and Springs	Iron Oxidizing Bacteria	Total Points	FPA Width/Bankfull Width	Comments
8/9/19	Ancho Canyon	Ancho Canyon Above E275-Site 1	Ad-5	NM-9000.A_54	Sam Loftin, Robert Gallegos, Jennifer Fullam, Kris Barrios	Yes	35°46'58"N	106°14'50"W	35.78277778	-106.2472222	0-1	60% Cloud Cover	Intermittent showers-at TA-49 Gage Station: .47" on 8/6/19	No	Yes-Site located below State Road 4. Culverts are present below State Road 4.	No	No	0	0	0	0	0	1	1	1.5	0	1	0	0.5	0	0	5	1.37	
9/13/19	Ancho Canyon	Above Confluence	Ac-7.5	NM-9000.A_046	Brad Schilling, Kris Barrios, Karen Armijo, Alethea Banar	Yes	35°47'31"N	106°15'56"W	35.79194444	106.2655556	0-1	2% Cloud Cover	40% Cloudy- no rain recorded in previous 3 days at TA-49 gage.	No	No	No	No	0	0	0	0	1	2	1	1.5	1	1	0	0	0	0	7.5	1.79	Vegetation present:Ponderosa pine, juniper, New Mexico Olive, upland grasses, indian rice grass, Artemisia, aster, skunkbrush, oak, chenopodes, New Mexico locust, Red Rocket, Mullen, wild onion, horseweed, penstemon. 1.10-Limited sorting, majority of streambed composed of sand.
9/13/19	Ancho Canyon	Below TA-49 MET Tower	Ab-7.5	NM-9000.A_046	Brad Schilling, Kris Barrios, Karen Armijo, Alethea Banar	Yes	35°48'40"N	106°17'18"W	35.81111111	106.2883333	0-1	2% Cloud Cover	40% Cloudy- no rain recorded in previous 3 days at TA-49 gage.	No	No	No	No	0	0	0	0	1	1.5	1	1.5	1	1.5	0	0	0	0	7.5	2.05	Vegetation present: Ponderosa pine, oak, rose, upland grasses, artemesia, wild onion, aster, composites, barberry, stinging nettle, solidago, juniper, skunkbrush. 1.6-Presence of rooted plants is variable within reach.
9/13/19	Ancho Canyon	Below Drought Study Area	Aa-6.5	NM-9000.A_046	Brad Schilling, Kris Barrios, Karen Armijo, Alethea Banar	Yes	35°49'13"N	106°18'22"W	35.82027778	106.3061111	0-1	95% Cloud Cover	40% Cloudy- no rain recorded in previous 3 days at TA-49 gage.	No	No	No	No	0	0	0	0	1	1	1	1.5	1	1	0	0	0	0	6.5	2.02	Vegetation present: Oak, New Mexico Olive, New Mexico Locust, rose, ragweed, upland grasses, skunkbrush, barberry, chokecherry, virginia creeper, poison ivy. 1.5-Channel bottom is covered in leaf litter.
8/8/19	Arroyo de la Delfe	Site 1	Ab-4	NM-128.A_16	Brad Schilling, Brian Iacona, Robert Gallegos, Jennifer Fullam, Kris Barrios	Yes	35°51'25"N	106°20'19"W	35.85694444	-106.3386111	0-1	40% Cloud Cover	Intermittent showers/ 30% Cloud Cover	No				0	0	0	0	1	2	1								4		Stopped due to lightning
8/16/19	Arroyo de la Delfe	Arroyo de la Delfe Downstream of Bulldog Spring	Ac-25	NM-128.A_16	Brian Iacona, Brad Schilling, Jennifer Fullam	Yes	35°51'25"N	106°20'4"W	35.85694444	-106.3344444	0-1	Clear/Sunny	Intermittent showers- at TA-6 Gage Station: .01" on 8/14 and .11" on 8/15	No	No	No	No	6	0	3	2	3	3	2	3	1	0.5	0	0	1.5	0	25	3.13	
8/16/19	Arroyo de la Delfe	Site 1-Upstream of Kieling Spring	Aa-7.5	NM-128.A_16	Brian Iacona, Brad Schilling, Jennifer Fullam	Yes	35°51'25"N	106°20'20"W	35.85694444	-106.3388889	0-1	Clear/Sunny	Intermittent showers- at TA-6 Gage Station: .01" on 8/14 and .11" on 8/15	No	No	No	No	0	0	0	0	0.5	2	1	1.5	1	1.5	0	0	0	0	7.5	1.73	
8/22/19	Canada del Buey	Canada del Buey Above E225	CBb-3	NM-128.A_00	Brad Schilling, Marwin Shendo, Kelkenny Bileen, Cesar Dominguez	No	35°50'1"N	106°14'22"W	35.83361111	106.2394444	0-1	Clear/Sunny	Intermittent showers- TA-54 Rain Gage Station: 01" on 8/21/2019	No	No	No	No	0	0	0	0	0.5	0	1	1.5	0	0	0	0	0	0	3	1.6	
9/13/19	Canada del Buey	Above E229.3	CBc-6.5	NM-128.A_00	Brian Iacona, Kelkenny Bileen, Bryan Dail, Josh Faulconer	No	35°49'41.7"N	106°13'21"W	35.82825	106.2225	0-1	5% Cloud Cover	40% Cloudy	No	Yes-Road crossing and culvert approximately 200 yards upstream of assessment site. Gage station E229.3 is downstream from site.	No	No	0	0	0	0	0	2	1	3	0	0	0	0.5	0	0	6.5	4.7	Vegetation present: Shrubby cinquefoil, juniper, ponderosa pine, sage, cheatgrass
9/13/19	Canada del Buey	Below E218	CBa-13	NM-128.A_00	Brian Iacona, Kelkenny Bileen, Bryan Dail, Josh Faulconer	No	35°51'20"N	106°16'42"W	35.85555556	106.2783333	0-1	5% Cloud Cover	40% Cloudy	No	Yes-Site is below gage E218.	No	No	2	0	0	0	2	1	1	1.5	3	1.5	0	1	0	0	13	2.31	Vegetation present:aspen, willow, oak, upland grasses. 1.1-Moist sediment and standing water in metal portion of gage station. Note*-Steep banks and evidence of flooding (trees/boulders in channel) at this segment
8/23/19	Canon de Valle	Canon de Valle Above E262	CVf-11.5	NM-128.A_01	Robert Gallegos, Jennifer Fullam, Sam Loftin, Marwin Shendo	Yes	35°49'56"N	106°18'17"W	35.83222222	106.3047222	0-1	40% Cloud Cover	Intermittent showers- TA-49 Rain Gage Station: .03" on 8/21/2019	No	No	No	No	0	0	0	0	1	2	1	1.5	3	3	0	0	0	0	11.5	1.26	NPDES Outfall 03A185 (now inactive) is located on the mesa top nearby. Vegetation found at the site: box elder, chokecherry, poison ivy, mullen, douglas fir, gamble oak.
8/29/19	Canon de Valle	Canon de Valle Below TA-15 ACO	CVd-8.5	NM-128.A_01	Sam Loftin, Marwin Shendo, Brad Schilling, Karen Armijo, Jennifer Fullam	Yes	35°50'34"N	106°18'43"W	35.84277778	106.3119444	0-1	Clear/Sunny	Intermittent showers-TA-6 Rain Gage had .14 inches on 8/27/2019.	No	No	No	No	0	0	0	0	1	1	1	1.5	2.5	1.5	0	0	0	0	8.5	1.43	Vegetation found at the site: clover, oak, ponderosa, mullen, chokecherry
9/6/19	Canon de Valle	Below CDV 1.7	CVb-6	NM-128.A_02	Brad Schilling, Sam Loftin	No	35°51'2"N	106°20'43"W	35.85055556	106.3452778	0-1	Clear/Sunny	Clear/Sunny	No	Yes-CDV 1.7 Retention berm approximately 100 yards upstream	No	No	0	0	0	0	0	2	1	1.5	0	1.5	0	0	0	0	6	1.35	
9/6/19	Canon de Valle	Below E253 and SEP Controls	CVa-2.5	NM-128.A_02	Brad Schilling, Sam Loftin	No	35°51'18"N	106°20'57"W	35.855	106.3491667	0-1	Clear/Sunny	Clear/Sunny	No	Yes-SEP Retention Berm 100 yards upstream of assessment site.	No	No	0	0	0	0	0	1	0	1.5	0	0	0	0	0	0	2.5	1.76	Vegetation present: Ponderosa Pine.
9/6/19	Canon de Valle	Below E256	CVc-8	NM-128.A_01	Brad Schilling, Sam Loftin	No	35°51'4"N	106°19'53"W	35.85111111	106.3313889	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	2	1	1	1.5	1	1.5	0	0	0	0	8	1.84	Vegetation present: Oak, rose, chokecherry, chamisa, locust, ponderosa pine, nettles, box elder
9/26/19	Canon de Valle	Below Fish Ladder	CVe-11	NM-128.A_01	Isaac Cadiente, Karen Armijo, Kelkenny Bileen, Robert Gallegos, Bryan Dail	No	35°50'23"N	106°18'47"W	35.83972222	106.3130556	0-1	10% Cloud Cover	Clear/Sunny	No	No	No	No	0	0	0	0	1	1	1	1.5	3	3	0	0.5	0	0	11	1.31	Vegetation present: Poison ivy, mullen, spruce, fir, alder, ponderosa, redgrasses, berries, maiden's hair, alfalfa. A spring is documented as being downstream (unknown if flowing or not). No known active discharges, Above perennial reach of CdV; Fish Ladder is Outfall 055
9/19/19	Chaquehui Canyon	Above E340	Cc-6.5	NM-128.A_03	Brian Iacona, Alethea Banar, Marc Gallegos, Lauren Beers	No	35°46'47"N	106°15'1"W	35.77972222	106.2502778	0-1	15% Cloud Cover	Clear/Sunny	No	No	No	No	0	0	0	0	0	2	1	1.5	2	0	0	0	0	0	6.5	1.28	Vegetation present: Pinon, juniper, artemesia, wavyleaf oak, upland grasses, clover, Prickly pear, yucca, mullen, composites, snakeweed, New Mexico Olive, skunkbrush, Mountain mahogany, ponderosa pine.
9/19/19	Chaquehui Canyon	Below E338	Cb-4.5	NM-128.A_03	Brian Iacona, Alethea Banar, Marc Gallegos, Lauren Beers	No	35°46'10"N	106°15'7"W	35.76944444	106.2519444	0-1	15% Cloud Cover	Clear/Sunny	No	No	No	No	0	0	0	0	0	2	1	1.5	0	0	0	0	0	0	4.5	1.27	Vegetation present: Ponderosa pine, pinon, juniper, New Mexico Olive, Apache Plume, New Mexico locust, skunkbrush, gooseberry, artemesia, aster, tansy mustard, chenododes, snakeweed, upland grasses, mullen, ragweed, wavy leaf oak. Stream channel drops over a cliff ~20 yards below evaluation site.
9/26/19	Chaquehui Canyon	Headwaters above E338	Ca-4.5	NM-128.A_03	Brad Schilling, Marwin Shendo, Alethea Banar, Marc Gallegos, Cesar Dominguez	No	35°46'49"N	106°15'42"W	35.78027778	106.2616667	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	0	2	1	1.5	0	0	0	0	0	0	4.5	1.34	Vegetation present: Ponderosa pine(near channel), Pinon, Juniper, Skunkbrush, Artemesias, chenopodes, Aster, New Mexico Olive, Gooseberry, Apache Plume, Tumbleweed, Cheatgrass, Mullen, Upland Grasses, Composites, Helianthus, Boutalua grass, Wavy leaf oak, New Mexico Locust, Indian Rice Grass, Sand cherry.
9/26/19	Chaquehui Canyon	Tributary North of E338-2	Cd-8	NM-128.A_03	Brad Schilling, Marwin Shendo, Alethea Banar, Marc Gallegos, Cesar Dominguez	No	35°46'27"N	106°14'54"W	35.77416667	106.2483333	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	0	2	2	1.5	1	1	0	0.5	0	0	8	1.74	Vegetation present: Ponderosa (near channel), Pinon, Juniper, Gambel Oak, New Mexico locust, New Mexico Olive, Skunkbrush, Sand Cherry, Fourwing saltbush, Apache Plume, Snakeweed, Artemesias, Mullen, Threadleaf, Tansy Mustard, Upland Grasses, Brome, Cheatgrass, Composites, Tumbleweed, Indian Ricegrass. 1.7-Sinuosity score due to large boulders present. 1.10-Limited sorting-mostly sand and boulders.
9/26/19	Chaquehui Canyon	Tributary North of E338-1	Ce-7	NM-128.A_03	Brad Schilling, Marwin Shendo, Alethea Banar, Marc Gallegos, Cesar Dominguez	No	35°46'19"N	106°14'46"W	35.77194444	106.2461111	0-1	5% Cloud Cover	Clear/Sunny	No	No	No	No	0	0	0	0	1	2	1	1.5	1	0	0	0.5	0	0	7	1.76	Vegetation present: Ponderosa (near channel), Pinon, Juniper, Apache Plume, Wavyleaf oak, New Mexico Oliver, Chamisa, Skunkbrush, New Mexico Locust, Artemesias, Antelope Sage, Composites, Sand Cherry, Upland Grasses, Tansy Mustard. 1.12-Mostly debris, little sediment.
7/11/19	DP Canyon	DP Site 1	De-7.5	NM-128.A_10	Brian Iacona, Robert Gallegos, Allison Chan, Bryan Dail	No	35°52'26"N	106°15'36"W	35.87388889	-106.26	0-1	5% Cloud Cover	Clear/Sunny	No	Yes-gabions located downstream of site (associated with gage station E040); grade control structure is located upstream of site.	No	No	0	0	0	0	1	1	1	1.5	0.5	2	0	0.5	0	0	7.5	1.42	
10/3/19	DP Canyon	Below DP Spring	Dd-17	NM-128.A_10	Brian Iacona, Brad Schilling, Jennifer Fullam, Sam Loftin, Joshua Faulconer, Diana Aranda	Yes	35°52'28"N	106°15'46"W	35.87444444	106.2627778	0-1	Clear/Sunny	Clear/Sunny	No	Yes-Grade Control Structure located upstream of assessment site.	No	No	1	0	0	2	1	2	2	1.5	3	3	0	0	1.5	0	17	1.26	Stream modification-Grade Control Structure located upstream of assessment site. Vegetation present: Apricot, New Mexico Olive, gambel oak, currant, virginia creeper, brickle bush, chamisa, ponderosa pine, mullen, worm wood, meadow fescue. 1.1-DP spring is weeping down rock wall but not pooling or flowing downstream.
10/17/19	DP Canyon	Below E038	Da-10.5	NM-128.A_14	Sam Loftin, Marwin Shendo, Brad Schilling, Luke Simms, Joshua Falconer	No	35°52'44"N	106°16'37"W	35.87888889	106.2769444	0-1	20% Cloud Cover	Clear/Sunny	No	No	No	No	0	0	0	0	0	2	2	1.5	1	3	1	0	0	0	10.5	1.53	
10/17/19	DP Canyon	Below DP Grade Control	Dc-26	NM-128.A_10	Sam Loftin, Brad Schilling, Luke Simms, Joshua Falconer	No	35°52'39"N	106°16'14"W	35.8775	106.2705556	0-1	30% Cloud Cover	Clear/Sunny	No	Yes-Below Grade Control Structure	No	No	6	0	2	3	2	3	1	3	3	1.5	0	1.5	0	0	26	4.4	Vegetation Present-Willow, smooth brome, oak, ponderosa, barberry, locust.
10/17/19	DP Canyon	Above Grade Control	Db-20.5	NM-128.A_14	Sam Loftin, Brad Schilling, Luke Simms, Joshua Falconer	No	35°52'40"N	106°16'20"W	35.87777778	106.2722222	0-1	10% Cloud Cover	Clear/Sunny	No	Yes- Grade Control Structure is located downstream of assessment site.	No	No	3	0	2	2	3	3	2	3	1	0	0	1.5	0	0	20.5	32.4	Vegetation Present-Smooth brome, red top, meadow fescue. 1.1-Area above stream assessment site is dry, area below is wet.
9/5/19	Effluent Canyon	Below 051 Outfall and SEP Grade Control	Ea-15	N/A	Marwin Shendo, Sam Loftin, Brad Schilling, Jennifer Fullam, Cesar Dominguez	Yes	35°51'54"N	106°17'52"W	35.865	106.2977778	0-1	Clear/Sunny	Clear/Sunny	No	Yes-Below SEP Grade Control Structure	No	Yes-Downstream of outfall 051(not currently discharging); 21,345 gallons of effluent discharged from outfall on a single day in June 2019.	2	0	1	0	1.5	2	2	1.5	3	1.5	0	0.5	0	0	15	1.28	Vegetation present: barberry, chokecherry, virginia creeper, gamble oak, rush, douglas fir, orchard grass; riparian vegetation in patches - uneven distribution. SEP GCS retains water.
8/1/19	Fence Canyon	Fence Canyon Above Potrillo Confluence	Fd-3.5	NM-128.A_04	Brad Schilling, Brian Iacona, Robert Gallegos, Marwin Shendo	No	35°48'33"N	106°13'43"W	35.80916667	-106.2286111	0-1	5% Cloud Cover	Clear/Sunny	No	Yes-State Road 4 and substation are located upstream of this site.	No	No	0	0	0	0	0.5	0.5	1	1.5	0	0	0	0	0	0	3.5	1.32	
8/30/19	Fence Canyon	Below Minie	Fb-9	NM-128.A_04	Kelkenny Bileen, Kris Barrios, Brad Schilling, Brian Iacona	Yes	35°49'39"N	106°16'26"W	35.8275	106.2738889	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	1	0	0	0	1	1	1	1.5	1.5	1.5	0	0.5	0	0	9	1.87	Storm Water IP berms are present above the IP sampler. 1.1-Standing pools of water in bedrock (plunge pools) and moist sediment in pools. 1.9-Mostly pools and riffles; hard to identify
8/30/19	Fence Canyon	Above State Road 4	Fc-2.5	NM-128.A_04	Kelkenny Bileen, Kris Barrios, Brad Schilling, Brian Iacona	Yes	35°48'48"N	106°14'37"W	35.81333333	106.2436111	0-1	20% Cloud Cover	Clear/Sunny	No	No	No	No	0	0	0	0	0	0	1	1.5	0	0	0	0	0	0	2.5	1.84	
10/17/19	Fence Canyon	Below Minie	Fa-11.5	NM-128.A_04	Robert Gallegos, Brian Iacona, Kelkenny Bileen, Kris Barrios, Alethea Banar	Yes	35°49'39"N	106°16'26"W	35.8275	106.2738889	0-1	50% Cloud Cover	Clear/Sunny	No	Yes-Upstream of evaluation site are active firing site and storm water control berms across the channel.	No	No	2	0	0	0	1.5	2	2	1.5	1	0	1.5	0	0	0	11.5	1.57	Vegetation Present-Ponderosa Pine, spruce, juniper, gambel oak, apache plume, NM Olive, NM locust, gooseberry, bearberry, tumbleweed, artemesia, mullen, upland grasses, composites, patches of sedges (not consistently along channel)
8/29/19	Fish Ladder	Fish Ladder Below Spring	FLa-9	N/A	Sam Loftin, Marwin Shendo, Brad Schilling, Karen Armijo, Jennifer Fullam	Yes	35°50'42"N	106°19'39"W	35.845	106.3275	0-1	<10% Cloud Cover	Intermittent showers-TA-6 Rain Gage had .14 inches on 8/27/2019.	No	no	No	No	0	0	0	0	2	2	1	1.5	1	1.5	0	0	0	0	9	1.45	Vegetation found at the site: thrushes, rushes, chokecherry, barberry, sedge, oak, locusts, mullen, ponderosa
9/26/19	Fish Ladder	Above CdV Confluence	FLb-8.5	N/A	Isaac Cadiente, Karen Armijo, Kelkenny Bileen, Robert Gallegos, Bryan Dail	No	35°50'39"N	106°19'21"W	35.84416667	106.3225	0-1	Clear/Sunny	Clear/Sunny	No	No	No	Yes-Active permitted Outfall (055) at TA-55, no discharge occurring-zero discharge location.	0	0	0	0	1	2	1	1.5	1	1.5	0	0.5	0	0	8.5	1.43	Vegetation Present: Gambel Oak, red top grasses, Pine, spruce, asters, low lying ferns, maiden hair. Note: No distinct riparian vegetation present. Chunks of charcoal are in stream bed. 1.10-Some sorting observed. Note: Active permitted NPDES Outfall 055 @ TA-55- no discharge occurring-zero discharge location.
8/1/19	Indio Canyon	Indio Canyon Above State Road 4	Ia-5	NM128.A_05	Brad Schilling, Brian Iacona, Robert Gallegos, Marwin Shendo	No	35°48'18"N	106°14'53"W	35.805	106.2480556	0-1	15% Cloud Cover	Clear/Sunny	No	No	No	No	0	0	0	0	0	2	1	1.5	0	0	0	0.5	0	0	5	1.34	
9/13/19	Indio Canyon	Above I1EP3	Ib-5	NM128.A_05	Brian Iacona, Kelkenny Bileen, Bryan Dail, Josh Faulconer	No	35°48'30.4"N	106°15'30.3"W	35.80844444	106.2583333	0-1	10% Cloud Cover	40% Cloudy- no rain recorded in previous 3 days at TA-49 gage.	No	No	No	No	0	0	0	0	1	1	1	1.5	0	0	0	0.5	0	0	5	1.33	
7/11/19	Los Alamos Canyon	LA Site 2	Lh-24.5	NM 9000.A_006	Brian Iacona, Robert Gallegos, Allison Chan, Bryan Dail	No	35°52'10"N	106°14'4"W	35.86944444	-106.2344444	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	5.5	0	3	2	2	3	1	1.5	2.5	1.5	2	0.5			24.5	1.27	
7/11/19	Los Alamos Canyon	LA Site 3	Le-18	NM-9000.A_63	Brian Iacona, Robert Gallegos, Allison Chan, Bryan Dail	No	35°52'20"N	106°15'39"W	35.87222222	-106.2608333	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	6	0	1	0	0.5	3	0.5	1.5	2.5	1.5	1	0.5	0	0	18	2.13	
7/11/19	Los Alamos Canyon	LA Site 1	Li-12.5	NM 9000.A_006	Brian Iacona, Robert Gallegos, Allison Chan, Bryan Dail	No	35°52'3"N	106°13'1"W	35.8675	-106.2169444	0-1	Clear/Sunny	Clear/Sunny	No	Upstream- LA Weir and Gage Station E050; Down Stream-Large culverts	No	No	2	0	0.5	0	2	1.5	0	1.5	0.5	2	2	0.5	0	0	12.5	1.58	
8/9/19	Los Alamos Canyon	Los Alamos Canyon below E026-Site 1	Lb-22.5	NM-9000.A_63	Sam Loftin, Robert Gallegos, Jennifer Fullam, Kris Barrios	Yes	35°52'48"N	106°19'29"W	35.88	-106.3247222	0-1	95% Cloud Cover	Intermittent showers- at TA-6 Gage Station: .84" on 8/6, .47" on 8/7, and .05" on 8/8	No	Yes-Los Alamos Reservoir breached upstream of site. Culverts upstream on West Road.	No	No	6	0	2	0	2	3	1	1.5	2.5	3	0	1.5	0	0	22.5	1.78	
8/9/19	Los Alamos Canyon	Los Alamos Canyon above E030-Site 2	Ld-13.5	NM-9000.A_63	Sam Loftin, Robert Gallegos, Jennifer Fullam, Kris Barrios	Yes	35°52'21"N	106°16'3"W	35.8725	-106.2675	0-1	70% Cloud Cover	Intermittent showers- at TA-6 Gage Station: .84" on 8/6, .47" on 8/7, and .05" on 8/8	No	Yes-Upstream, TA-2 hardened channel; IP Site LA-SMA-2 retention ponds upstream	No	No	4	0	1	0	1	2	1	1.5	1	1.5	0	0.5	0	0	13.5	2.35	
10/3/19	Los Alamos Canyon	Below E026	La-11.5	NM-9000.A_63	Brian Iacona, Brad Schilling, Jennifer Fullam, Sam Loftin, Joshua Faulconer, Diana Aranda	Yes	35°52'48"N	106°19'29"W	35.88	106.3247222	0-1	Clear/Sunny	Clear/Sunny	No	Yes-Assessment site is downstream of Los Alamos Reservoir and stream gage E026.	No	No	0	0	0	0	2	3	1	1.5	2	1.5	0	0.5	0	0	11.5	1.33	Note: Skate Spring is identified upstream on the Intellus map, but no evidence of flow from spring into assessed reach. Vegetation present- alder, narrowleaf cottonwood, ponderosa pine, willow, white fir, douglas fir, box elder, Rocky Mountain Maple, gambel oak, burdock, virginia creeper.
10/3/19	Los Alamos Canyon	Above E030	Lc-6.5	NM-9000.A_63	Brian Iacona, Brad Schilling, Jennifer Fullam, Sam Loftin, Joshua Faulconer, Diana Aranda	Yes	35°52'21"N	106°16'13"W	35.8725	106.2702778	0-1	Clear/Sunny	Clear/Sunny	No	Yes-Road crossing upstream of assessment site.	No	No	0	0	0	0	1	2	1	0	1	1.5	0	0	0	0	6.5	1.13	Vegetation present: Willow, ponderosa pine, red top grass, juniper, pinon, sweet clover, horseweed, wormwood, oak, russian olive, douglas fir.
10/3/19	Los Alamos Canyon	Below DP Confluence	Lf-20	NM-9000.A_006	Brian Iacona, Brad Schilling, Jennifer Fullam, Sam Loftin, Joshua Faulconer, Diana Aranda	Yes	35°52'23"N	106°15'26"W	35.87305556	106.2572222	0-1	Clear/Sunny	Clear/Sunny	No	Yes-Stream Gage station E030 is located upstream of assessment site.	No	Yes	5	0	0	3	2	3	1	1.5	1	1.5	0	0.5	1.5	0	20	1.34	Site is downstream of Los Alamos County well/outfall Otowi-4 and recent flow is evident. 1.1-Downstream of spring, flow is evident in channel with some dry sections (possible subsurface flow). Note-Staining is evident in stream channel from unknown source. May be from LAC outfall. Vegetation present: New Mexico Olive, smooth brome, juniper, narrowleaf cottonwood, virginia creeper, ponderosa pine, New Mexico locust, oak, teasel, alder, dock, cockleburr, sweet clover.
10/17/19	Los Alamos Canyon	LA-5.19 Spring	Lg-16.5	NM-9000.A_006	Robert Gallegos, Kris Barrios, Luke Simms, Joshua Faulconer, Marwin Shendo	Yes	35°52'7"N	106°14'19"W	35.86861111	106.2386111	0-1	Clear/Sunny	Clear/Sunny	No	No	No	Yes-NPDES Outfall 048 is upstream of the assessment site. Los Alamos County well Otowi #4 is upstream of the assessment site and regularly discharges.	1.5	0	0	0	1.5	2.5	1	1.5	2	3	1.5	0.5	1.5	0	16.5	1.65	1.11-Hydric soils are isolated in study reach.
8/29/19	Martin Springs	Martin Springs Canyon 3 at MSC16-06293	MSb-8	N/A	Sam Loftin, Marwin Shendo, Brad Schilling, Karen Armijo, Jennifer Fullam	Yes	35°50'26.952"N	106°19'59.016"W	35.84082	106.33306	0-1	70% Cloud Cover	Intermittent showers-TA-6 Rain Gage had .14 inches on 8/27/2019.	No	No	No	No	1	0	0	0	1.5	2	1	1.5	1	1	0	0	0	0	8	1.83	
8/29/19	Martin Springs	Below Martin Springs	MSa-16	N/A	Sam Loftin, Marwin Shendo, Brad Schilling, Karen Armijo, Jennifer Fullam	Yes	35°50'31"N	106°20'10"W	35.84194444	106.3361111	0-1	90% Cloud Cover	Intermittent showers-TA-6 Rain Gage had .14 inches on 8/27/2019.	No	No	No	No	2	0	2	2	1.5	2	1	1.5	1	0	0	1.5	1.5	0	16	2.22	Benthic Macroinvertebrates founds: mayfly, mosquito, water beetle. Vegetation found at the site: rushes, roses, grasses(differ between reach and upland), gamble oak, thistle.
9/5/19	Mortandad Canyon	Above E201	Mc-6.5	NM-9000.A_042	Marwin Shendo, Sam Loftin, Brad Schilling, Jennifer Fullam, Cesar Dominguez	Yes	35°51'50"N	106°16'43"W	35.86388889	106.2786111	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	2	1	1	1.5	0	0.5	0	0.5	0	0	6.5	1.56	Vegetation present: sumac, virginia creeper, New Mexico Olive, box elder, clover, grasses. 1.1-Moist sediment found under rocks. 1.10-Limited sorting observed. 1.13- No springs documented. Discharges Note: Assessment site is downstream of outfalls 051 and 03A181.
9/5/19	Mortandad Canyon	Above Sediment Traps	Md-5	NM-9000.A_042	Marwin Shendo, Sam Loftin, Brad Schilling, Jennifer Fullam, Cesar Dominguez	Yes	35°51'39"N	106°16'14"W	35.86083333	106.2705556	0-1	Clear/Sunny	Clear/Sunny	No	Yes-Ten Site Canyon Sedimentation Basin	No	No-Outfalls 051, 160, and 181 Upstream of assessment site.	0	0	0	0	1	1	1	1.5	0	0	0	0.5	0	0	5	2.16	Vegetation Present: Geranium, mullen sumac, ponderosa pine, gramma grass
9/5/19	Mortandad Canyon	Above E204	Me-2.5	NM-9000.A_042	Marwin Shendo, Sam Loftin, Brad Schilling, Jennifer Fullam, Cesar Dominguez	Yes	35°51'23"N	106°14'46"W	35.85638889	106.2461111	0-1	Clear/Sunny	Clear/Sunny	No	Yes-Mortandad Sediment Traps upstream	No	Yes-Outfalls 051, 160, and 181 upstream of assessment site.	0	0	0	0	0	0	1	1.5	0	0	0	0	0	0	2.5	1.3	Vegetation present: sumac, gramma grass, juniper 1.8-Wide active floodplain
9/6/19	Mortandad Canyon	Below SEP Upper Grade Control	Ma-19.5	NM-9000.A_042	Brad Schilling, Sam Loftin	No	35°52'11"N	106°19'3"W	35.86972222	106.3175	0-1	20% Cloud Cover	Clear/Sunny	No	Yes-SEP Grade Control Structure is 100 yards upstream of assessment site.	No	No	4	0	3	3	2	2	1	1.5	1	0	0	0.5	0	1.5	19.5	1.5	Vegetation present: Oaks, red top, little blue stem, brickle bush, rushes. 1.10-Bedrock channel present.
9/6/19	Mortandad Canyon	Below Confluence with Effluent	Mb-14.5	NM-9000.A_042	Brad Schilling, Sam Loftin	No	35°51'55"N	106°17'44"W	35.86527778	106.2955556	0-1	Clear/Sunny	Clear/Sunny	No	No	No	Yes-NPDES Outfalls 03A081 and 051 are upstream of assessment site.	0	0	0	0	2	3	2	1.5	3	3	0	0	0	0	14.5	1.43	
8/2/19	North Ancho Canyon	North Ancho Above Point 88	NAa-8.5	NM-9000.A_055	Robert Gallegos, Sam Loftin, Brad Schilling, Kelkenny Bileen, Alethea Banar	No	35°48'35.6"N	106°16'19"W	35.80988889	-106.2719444	0-1	15% Cloud Cover	10% Cloud Cover	No	No	No	No	0	0	0	0	0	2	1.5	1.5	1	1.5	0	1	0	0	8.5	1.25	
8/2/19	North Ancho Canyon	North Ancho at Y Below SEP	NAc-4.5	NM-9000.A_055	Robert Gallegos, Sam Loftin, Brad Schilling, Kelkenny Bileen, Alethea Banar	No	35°47'56.51"N	106°15'35.35"W	35.79903056	-106.2597222	0-1	2% Cloud Cover	10% Cloud cover	No	Yes-Supplemental Environmental Project Watershed Enhancement Control Upstream	No	No	0	0	0	0	0	2	0	1.5	0	1	0	0	0	0	4.5	1.29	
8/2/19	North Ancho Canyon	North Ancho Above Building 69	NAd-4	NM-9000.A_055	Robert Gallegos, Sam Loftin, Brad Schilling, Kelkenny Bileen, Alethea Banar	No	35°47'36.5"N	106°15'10.5"W	35.79347222	-106.2527778	0-1	Clear/Sunny	10% Cloud Cover	No	Yes-Supplemental Environmental Project Watershed Control Upstream	No	No	0	0	0	0	0	2	0.5	1.5	0	0	0	0	0	0	4	1.24	
8/2/19	North Ancho Canyon	North Ancho Below Building 57	NAb-3.5	NM-9000.A_055	Robert Gallegos, Sam Loftin, Brad Schilling, Kelkenny Bileen, Alethea Banar	No	35°48'27"N	106°15'52"W	35.8075	-106.2644444	0-1	10% Cloud Cover	10% Cloud Cover	No	Yes-Culvert under road upstream of evaluation area	No	No	0	0	0	0	0	2	0.5	0	0	1	0	0	0	0	3.5	1.15	
8/8/19	Pajarito Canyon	Pajarito Above Starmers Site 2	Pb-28	NM128.A_07	Brad Schilling, Brian Iacona, Robert Gallegos, Sam Loftin, Alethea Banar, Jennifer Fullam, Kris Barrios	Yes	35°51'35"N	106°20'13"W	35.85972222	-106.3369444	0-1	50% Cloud Cover	Intermittent showers/ 30% Cloud Cover	No	Yes-Two large culverts under Anchor Ranch Road upstream of evaluation area.	No	No	6	0	2	2	3	3	2	1.5	3	1.5	0	1	1.5	1.5	28	2.1	
8/8/19	Pajarito Canyon	Pajarito Above Starmers Site 1	Pa-14	NM128.A_07	Brad Schilling, Brian Iacona, Robert Gallegos, Sam Loftin, Alethea Banar, Jennifer Fullam, Kris Barrios	Yes	35°51'43"N	106°20'22"W	35.86194444	-106.3394444	0-1	30% Cloud Cover	Intermittent showers/ 30% Cloud Cover	No	Yes-Two large culverts under Anchor Ranch Road upstream of evaluation area.	No	No	1	0	0	0	0.5	2	2	1.5	3	3	0	1	0	0	14	1.5	
8/16/19	Pajarito Canyon	Pajarito Above Two Mile Site 1	Pc-24	NM-128.A_06	Brian Iacona, Brad Schilling, Jennifer Fullam	Yes	35°51'23"N	106°19'41"W	35.85638889	-106.3280556	0-1	Clear/Sunny	Intermittent showers- at TA-6 Gage Station: .01" on 8/14 and .11" on 8/15	No	No	No	No	6	0	3	3	2	3	1	1.5	3	1.5	0	0	0	0	24	2.13	
8/16/19	Pajarito Canyon	Pajarito Above Two Mile Site 2	Pd-12	NM-128.A_06	Brian Iacona, Brad Schilling, Jennifer Fullam	Yes	35°51'20"N	106°19'2"W	35.85555556	-106.3172222	0-1	Clear/Sunny	Intermittent showers- at TA-6 Gage Station: .01" on 8/14 and .11" on 8/15	No	No	No	No	1	0	1	1	1.5	2	1	1.5	1	2	0	0	0	0	12	1.56	
8/16/19	Pajarito Canyon	Pajarito Above Two Mile Site 3 by E243	Pe-8	NM-128.A_06	Brian Iacona, Brad Schilling, Jennifer Fullam	Yes	35°51'14"N	106°17'52"W	35.85388889	-106.2977778	0-1	50% Cloud Cover	Intermittent showers- at TA-6 Gage Station: .01" on 8/14 and .11" on 8/15	No	No	No	No	0	0	0	0	1	2	1	1.5	1	1.5	0	0	0	0	8	1.48	
8/22/19	Pajarito Canyon	Pajarito Below Three Mile	Pi-10	NM-128.A_08	Brad Schilling, Marwin Shendo, Kelkenny Bileen, Cesar Dominguez	No	35°50'21"N	106°15'54"W	35.83916667	106.265	0-1	Clear/Sunny	Intermittent showers- TA-54 Rain Gage Station: 01" on 8/21/2019	No	No	No	No	0	0	0	0	0.5	2	1	1.5	1	3	0	1	0	0	10	1.2	
8/22/19	Pajarito Canyon	Pajarito Below Wetlands South of Pajarito Road	Pj-8.5	NM-128.A_08	Brad Schilling, Marwin Shendo, Kelkenny Bileen, Cesar Dominguez	No	35°49'43"N	106°14'34"W	35.82861111	106.2427778	0-1	Clear/Sunny	Intermittent showers- TA-54 Rain Gage Station: 01" on 8/21/2019	No	Yes-site is located approximately 200 yards downstream of culvert.	No	No	0	0	0	0	0.5	2	1	1.5	1	2	0	0.5	0	0	8.5	1.2	
8/22/19	Pajarito Canyon	Pajarito Below Dam	Pf-8.5	NM-128.A_08	Brad Schilling, Marwin Shendo, Kelkenny Bileen, Cesar Dominguez	No	35°51'12"N	106°17'30"W	35.85333333	106.2916667	0-1	Clear/Sunny	Intermittent showers- TA-6 Rain Gage Station: .3" on 8/20/2019	No	Yes-Site is downstream of Pajarito Dam.	No	No	0	0	0	0	0.5	2	1	1.5	1	1.5	0	1	0	0	8.5	1.23	
8/22/19	Pajarito Canyon	Pajarito Above Three Mile	Ph-7.5	NM-128.A_08	Brad Schilling, Marwin Shendo, Kelkenny Bileen, Cesar Dominguez	No	35°50'26"N	106°16'3"W	35.84055556	106.2675	0-1	Clear/Sunny	Intermittent showers- TA-54 Rain Gage Station: 01" on 8/21/2019	No	No	No	No	0	0	0	0	0.5	2	0.5	1.5	1	1.5	0	0.5	0	0	7.5	1.3	
8/22/19	Pajarito Canyon	Pajarito Directly Above E245.5	Pg-7	NM-128.A_08	Brad Schilling, Marwin Shendo, Kelkenny Bileen, Cesar Dominguez	No	35°50'46"N	106°16'29"W	35.84611111	106.2747222	0-1	Clear/Sunny	Intermittent showers- TA-6 Rain Gage Station: .3" on 8/20/2019	No	No	No	No	0	0	0	0	0.5	0.5	1	1.5	1	1	0	1.5	0	0	7	1.83	
8/22/19	Pajarito Canyon	Pajarito Above E250	Pk-6.5	NM-128.A_08	Brad Schilling, Marwin Shendo, Kelkenny Bileen, Cesar Dominguez	No	35°49'26"N	106°13'43"W	35.82388889	106.2286111	0-1	Clear/Sunny	Intermittent showers- TA-54 Rain Gage Station: 01" on 8/21/2019	No	No	No	No	0	0	0	0	0.5	2	1	1.5	1	0	0	0.5	0	0	6.5	1.38	
8/1/19	Potrillo Canyon	Potrillo Below Fence Confluence	POf-6	NM-128.A_09	Brad Schilling, Brian Iacona, Robert Gallegos, Marwin Shendo	No	35°48'31"N	106°13'39"W	35.80861111	106.2275	0-1	5% Cloud Cover	Clear/Sunny	No	Yes-State Road 4 is upstream of this site.	No	No	0	0	0	0	0.5	2	1	1.5	0	0.5	0	0.5	0	0	6	1.55	
8/30/19	Potrillo Canyon	Below PT4.2	POc-2	NM-128.A_09	Kelkenny Bileen, Kris Barrios, Brad Schilling, Brian Iacona	Yes	35°49'27"N	106°14'55"W	35.82416667	106.2486111	0-1	Clear/Sunny	Clear/Sunny	No	Yes-Storm Water IP retention control structure approximately 50 yards upstream.	No	No	0	0	0	0	0	1	1	0	0	0	0	0	0	0	2	1.037	
8/30/19	Potrillo Canyon	Above Lower Slobbovia	POb-6.5	NM-128.A_09	Kelkenny Bileen, Kris Barrios, Brad Schilling, Brian Iacona	Yes	35°49'48"N	106°15'42"W	35.83	106.2616667	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	1	1	2	1.5	0	1	0	0	0	0	6.5	1.56	
8/30/19	Potrillo Canyon	Below Fire Break	POd-2.5	NM-128.A_09	Kelkenny Bileen, Kris Barrios, Brad Schilling, Brian Iacona	Yes	35°49'15"N	106°14'34"W	35.82083333	106.2427778	0-1	15% Cloud Cover	Clear/Sunny	No	Yes-IP berm and fire break upstream from assessment site.	No	No	0	0	0	0	0	0	1	1.5	0	0	0	0	0	0	2.5	1.59	
8/30/19	Potrillo Canyon	Above E267	POe-4.5	NM-128.A_09	Kelkenny Bileen, Kris Barrios, Brad Schilling, Brian Iacona	Yes	35°48'48"N	106°14'1"W	35.81333333	106.2336111	0-1	15% Cloud Cover	Clear/Sunny	No	Yes-IP berm and fire break upstream from assessment site.	No	No	0	0	0	0	1	1	0.5	1.5	0	0	0	0.5	0	0	4.5	2.5	1.7-Minimal bends observed.
9/19/19	Potrillo Canyon	Below TA-15 ACO	POa-8	NM-128.A_09	Brad Schilling, Cesar Dominguez, Kelkenny Bileen, Robert Gallegos	No	35°50'16"N	106°17'53"W	35.83777778	106.2980556	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	1	2	2	1.5	1	0	0	0.5	0	0	8	2.09	Vegetation present: Upland grasses, oak, gramma grass, pine grass, wolftail, ponderosa
6/26/19	Sandia Canyon	S-Site 1	Sc-5	NM-128.A_11	Amanda White, Isaac Cadiente, Brian Iacona, Robert Gallegos, Bryan Dail, Alethea Banar	No	35°51'43.056"N	106°14'20.6514"W	35.86196	-106.23907	0-1	<5% Cloud Cover	Clear/Sunny	No	Yes, Upstream-Soil cement channel and dissipation structure at TA-72 shooting range. Downstream-SEP Grade Control Structures	No	No	0	0	0	0	1	1	1	1.5	0	0.5	0	0	0	0	5	1.75	
7/18/19	Sandia Canyon	Sandia Canyon Below Sigma	Sa-17.5	NM-128.A_11	Amanda White, Isaac Cadiente, Marwin Shendo, Brad Schilling, Robert Gallegos	No	35°52'00"N	106°16'18"W	35.86666667	-106.2716667	0-1	5% Cloud Cover	on 7/16/19 .02" at E203 and .04" at TA-53	No	No stream modifications in transect. However, there is a culvert at Sigma Canyon.	No	No	2	0	1.5	2	2	2.5	1	1.5	2	2	0	1	0	0	17.5	1.93	
7/18/19	Sandia Canyon	Sandia Canyon Below E124	Sb-3	NM-128.A_11	Amanda White, Isaac Cadiente, Marwin Shendo, Brad Schilling, Robert Gallegos	No	35°51'52"N	106°15'37"W	35.86444444	-106.2602778	0-1	Clear/Sunny	on 7/16/19 .02" at E203 and .04" at TA-53	No	Yes-Culverts from road upstream and downstream	No	No	0	0	0	0	1	1	0.5	0	0	0.5	0	0	0	0	3	1.11	
8/22/19	Sandia Canyon	Sandia Canyon Below SEP	Sd-6.5	NM-128.A_11	Brian Iacona, Robert Gallegos, Karen Armijo, Alethea Banar	No	35°51'39"N	106°14'15"W	35.86083333	106.2375	0-1	Clear/Sunny	Intermittent showers- TA-53 Gage Station: 01" on 8/21/2019	No	Yes-SEP watershed control upstream of evaluation area. Soil cement channel and dissipation structure at TA-72 shooting range.	No	No	0	0	0	0	0.5	1.5	1	3	0	0	0	0.5	0	0	6.5	3.37	Closer to State Road 4 the channel becomes very diffuse.
8/22/19	Sandia Canyon	Sandia Above E125	Se-6.25	NM-128.A_11	Brian Iacona, Robert Gallegos, Karen Armijo, Alethea Banar	No	35°51'33"N	106°13'34"W	35.85916667	106.2261111	0-1	Clear/Sunny	Intermittent showers- TA-53 Gage Station: 01" on 8/21/2019	No	Yes-Upstream-Two culverts under East Jemez Road. SEP control (jax) upstream of site.	No	No	0	0	0	0	0.5	1.5	1	1.5	0.5	0.75	0	0.5	0	0	6.25	1.58	
9/5/19	Ten Site Canyon	Below Spring TS-1.42	TSa-7.5	NM-128.A_17	Marwin Shendo, Sam Loftin, Brad Schilling, Jennifer Fullam, Cesar Dominguez	Yes	35°51'42"N	106°17'43"W	35.86166667	106.2952778	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	1	2	2	1.5	0	0.5	0	0.5	0	0	7.5	2.46	Outfall 160 is located upstream of site but is not presently set up to discharge to the environment (all discharges go to SWWS). Vegetation present: juniper, gamble oak, russian olive, ponderosa pine, willow, smooth brome. 1.10-Limited sorting observed 1.11-Moist conditions present. 1.13- TS1.42 is present but is dry at time of assessment.
9/5/19	Ten Site Canyon	Above E201.5	TSb-6.5	NM-128.A_17	Marwin Shendo, Sam Loftin, Brad Schilling, Jennifer Fullam, Cesar Dominguez	Yes	35°51'38"N	106°16'32"W	35.86055556	106.2755556	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	1	2	1	1.5	0	0.5	0	0.5	0	0	6.5	1.62	Vegetation Present: mullen, gamble oak, virginia creeper, locust, forbs. 1.10- Limited sorting observed. Discharges Note: Outfall 160 is in vicinity of assessment site but discontinued discharges in 2018. Alluvial wells B-15 and A-6 were near this assessment site.
7/12/19	Three Mile	3 Mile-1	TMd-24.5	NM-9000.A_091	Amanda White, Allison Chan, Bryan Dail, Robert Gallegos	No	35°50'20"N	106°16'22"W	35.83888889	106.2727778	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	5	0	2	3	2.5	2	1.5	2	1	0.5	3	0.5	1.5	0	24.5	2.53	
7/12/19	Three Mile	3 Mile-2	TMc-16.5	NM-9000.A_091	Amanda White, Allison Chan, Bryan Dail, Robert Gallegos	No	35°50'29"N	106°16'42"W	35.84138889	106.2783333	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	3	1.5	1.5	2	1.5	0	0.5	3	0.5	1.5	1.5	16.5	1.96	Neither spring nor channel were flowing at time of assessment.
9/19/19	Three Mile	Above N3B R-45 FP	TMa-5	NM-9000.A_91	Brad Schilling, Cesar Dominguez, Kelkenny Bileen, Robert Gallegos	No	35°50'59"N	106°18'10"W	35.84972222	106.3027778	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	1	0	1	3	0	0	0	0	0	0	5	2.8	Vegetation present: Meadowgrass, red top, New Mexico Locust, yarrow, pinegrass.
9/19/19	Three Mile	Above Three Mile Spring	TMb-9	NM-9000.A_91	Brad Schilling, Cesar Dominguez, Kelkenny Bileen, Robert Gallegos	No	35°50'40"N	106°16'54"W	35.84444444	106.2816667	0-1	Clear/Sunny	Clear/Sunny	No	No	No	No	0	0	0	0	1	2	2	1.5	1	1	0	0.5	0	0	9	1.75	Vegetation present: Box elder, poison ivy, red top, oak, ponderosa, New Mexico locust, privit. 1.10-Partial sorting
10/17/19	Tributary to Water Canyon	Above E261	TWa-9	N/A	Robert Gallegos, Brian Iacona, Kelkenny Bileen, Kris Barrios, Alethea Banar	Yes	35°49'52"N	106°18'28"W	35.83111111	106.3077778	0-1	Clear/Sunny	Clear/Sunny	No	No-Note: SR501 is in upper part of canyon.	No	No	0	0	0	0	1.5	2	2	1.5	1	1	0	0	0	0	9	1.69	Vegetation Present-Ponderosa pine, spruce, fir, gambel oak, water birch, ash, rose, NM locust, yarrow, mullen, vetch, upland grasses, virginia creeper, aster, composites, bedstraw, bearberry. 1.5-No distinct riparian corridor.
9/12/19	Two Mile Canyon	Below Anderson Spring Above North Fork Confluence	Tc-8	NM-128.A_15	Brad Schilling, Alethea Banar, Karen Armijo	No	35°52'2"N	106°19'36"W	35.86722222	106.3266667	0-1	Clear/Sunny	Intermittent Showers-TA-6 Rain Gage Recorded .06" on 9/10 and .03" on 9/11	No	No	No	No	0	0	0	0	1	2	1	1.5	1	1.5	0	0	0	0	8	1.85	Vegetation present: Ponderosa pine, fir, spruce, oak, upland grasses, hops, artemesia, aster, ragweed
9/12/19	Two Mile Canyon	Below Anderson Spring	Tb-25	NM-128.A_15	Brad Schilling, Alethea Banar, Karen Armijo, Brian Iacona	No	35°52'5"N	106°19'51"W	35.86805556	106.3308333	0-1	Clear/Sunny	Intermittent Showers-TA-6 Rain Gage Recorded .06" on 9/10 and .03" on 9/11	No	Yes-Culvert under West Jemez Road upstream of evaluation area. Utility corridor with gabion structure across stream channel upstream of evaluation area.	No	No	6	0	3	2	3	3	2	1.5	3	0	0	0	1.5	0	25	1.45	Vegetation present: Oak, ponderosa pine, hops, barberry, rumex, thistle, yarrow, rose, spruce, morning glory, New Mexico locust, big golden pea, orchardgrass, mullen, solidago. *Riparian vegetation starts at Anderson Spring.
9/12/19	Two Mile Canyon	Above SM-30 Spring	Td-8	NM-128.A_15	Brad Schilling, Alethea Banar, Karen Armijo, Brian Iacona	No	35°52'23"N	106°19'54"W	35.87305556	106.3316667	0-1	Clear/Sunny	Intermittent Showers-TA-6 Rain Gage Recorded .06" on 9/10 and .03" on 9/11	No	Yes-Culverts under West Jemez Road upstream of evaluation area.	No	No	0	0	0	0	1	2	1	1.5	1	1.5	0	0	0	0	8	2.22	Vegetation present:Oak, ponderosa pine, artemesia, ragweed, upland grasses, fir, mulllen, chenopodes, one section of sedges on upland bank. 1.1-soil moisture observed from recent rain events
9/12/19	Two Mile Canyon	Below SM-30 Spring	Te-12.5	NM-128.A_15	Brad Schilling, Alethea Banar, Karen Armijo	No	35°52'15"N	106°19'40"W	35.87083333	106.3277778	0-1	Clear/Sunny	Intermittent Showers-TA-6 Rain Gage Recorded .06" on 9/10 and .03" on 9/11	No	Yes-Land bridge with large culvert upstream of evaluation area.	No	No	1	0	0	0	1	2	1	1.5	2	3	0	1	0	0	12.5	2	Vegetation present: Ponderosa pine, fir, spruce, barberry, rose, gooseberry, hops, aster, upland grass,oak, clover. 1.1- A few pools exist upstream of evaluation area due to recent rain events-the channel is dry with moist sediments under rocks, no evidence of base flow. Note*-substantial impervious areas northeast of canyon bottom from urban development contributing to flashiness of stream flow.
9/12/19	Two Mile Canyon	Above Hanlon Spring	Ta-10	NM-128.A_15	Brad Schilling, Alethea Banar, Karen Armijo, Brian Iacona	No	35°52'9"N	106°20'18"W	35.86916667	106.3383333	0-1	Clear/Sunny	Intermittent Showers-TA-6 Rain Gage Recorded .06" on 9/10 and .03" on 9/11	No	Yes-Culvert under West Jemez Road upstream of evaluation area.	No	No	0	0	0	0	1	2	1	3	1	1.5	0	0.5	0	0	10	2.65	Vegetation present: Oak, ponderosa pine, spruce, barberry, chenopodes, hops, ninebark, upland grasses, aster, artemesia, solidago, composites.
9/12/19	Two Mile Canyon	Below Confluence	Tf-18	NM-128.A_15	Jennifer Fullam, Robert Gallegos, Sam Loftin	Yes	35°52'4"N	106°19'25"W	35.86777778	106.3236111	0-1	Clear/Sunny	Intermittent Showers-TA-6 Rain Gage Recorded .06" on 9/10 and .03" on 9/11	No	No	No	No	4	0	2	2	1	2	1	0	3	1.5	0	0	1.5	0	18	1.07	Vegetation present: Chokecherry, grasses, fescue, redtop. 1.7-Sinuosity is inhibited by narrow canyon. 1.10-Incised channel-no difference between channel and floodplain.
9/12/19	Two Mile Canyon	Below TA-59	Tg-20.5	NM-128.A_15	Jennifer Fullam, Robert Gallegos, Sam Loftin	Yes	35°52'3"N	106°19'11"W	35.8675	106.3197222	0-1	Clear/Sunny	Intermittent Showers-TA-6 Rain Gage Recorded .06" on 9/10 and .03" on 9/11	No	No	No	No	4	0	2	3	2	2	1	1.5	3	1.5	0	0.5	0	0	20.5	1.54	1.3-Macroinvertebrates observed: beetles, mayfly, redworm
9/12/19	Two Mile Canyon	TA-55 Confluence	Th-19	NM-128.A_15	Jennifer Fullam, Robert Gallegos, Sam Loftin	Yes	35°51'30"N	106°18'9"W	35.85833333	106.3025	0-1	Clear/Sunny	Intermittent Showers-TA-54 Rain Gage Recorded .06" on 9/10	No	No	No	No	4	0	2	2	2	2	1	1.5	3	1	0	0.5	0	0	19	2.45	Vegetation present: Oak, clover, red top, daisy, box elder, rose, poison ivy, thistle. 1.3-Caddisfly casing, worm, mayfly. 1.10-Limited sorting.
9/12/19	Two Mile Canyon	Above E244	Ti-10.5	NM-128.A_15	Jennifer Fullam, Robert Gallegos, Sam Loftin	Yes	35°51'21"N	106°17'45"W	35.85583333	106.2958333	0-1	Clear/Sunny	Intermittent Showers-TA-54 Rain Gage Recorded .06" on 9/10	No	No	No	No	0	0	0	1	2	2	1	1.5	1	1.5	0	0.5	0	0	10.5	1.5	Vegetation present-Evening primrose, orchard grass, ponderosa pine, box elder, red top, thistle, yarrow, New Mexico locust
8/1/19	Water Canyon	Water Canyon Above Indio (E263)	Wj-10	NM128.A_13	Brad Schilling, Brian Iacona, Robert Gallegos, Marwin Shendo	No	35°48'19"N	106°14'52"W	35.80527778	106.2477778	0-1	5% Cloud Cover	Clear/Sunny	No	Yes-State Road 4 is upstream of this site.	No	No	1	0	0.5	0	0.5	1	0.5	1.5	2	2	0	1	0	0	10	1.2	
8/22/19	Water Canyon	Water Canyon Above E265	Wk-9	NM-128.A_13	Brian Iacona, Robert Gallegos, Karen Armijo, Alethea Banar	No	35°48'19"N	106°14'37"W	35.80527778	106.2436111	0-1	Clear/Sunny	Intermittent showers- TA-54 Rain Gage Station: 01" on 8/21/2019	No	Yes-Large culvert under State Road 4 upstream of evaluation area.	No	No	0	0	0	0	1	1	1.5	1.5	1	2	0	1	0	0	9	1.26	Seeps and Springs present in upper part of Assessment Unit but several miles upstream.
8/23/19	Water Canyon	Water Canyon Below CdV Confluence	Wg-12.5	NM-128.A_13	Robert Gallegos, Jennifer Fullam, Sam Loftin, Marwin Shendo	Yes	35°49'47"N	106°17'60"W	35.82972222	106.3	0-1	Clear/Sunny	Intermittent showers- TA-49 Rain Gage Station: .03" on 8/21/2019	No	No	No	No	0	0	0	0	2	2	2	1.5	2	3	0	0	0	0	12.5	2.13	Wet zone with iron deposits found during assessment (see site photos). Vegetation found at the site: smooth brome, virginia creeper, box elder, willow, mullen, NM Locust, orchard grass, narrowleaf cottonwood, raspberry, rush.

1

AB Ex. 16

Date	Stream Name	Site ID	Map Legend	Assessment Unit	Evaluators	NMED in Attendance	Latitude	Longitude	Dec Lat	Dec Long	Drought Index	Weather Now	Weather Past 48 hours	Heavy rain in past 48 hours?	Stream Modifications	Diversions	Discharges	Water in Channel	Fish	Macroinvertebrates	Filamentous Algae/Periphyton	Diff in Vegetation	Rooted Upland Plants	Sinuosity	Floodplain Dimensions	Pool/Riffle Seq	Particle Size/Sorting	Hydric Soils	Sediment on Plants/Debris	Seeps and Springs	Iron Oxidizing Bacteria	Total Points	FPA Width/Bankfull Width	Comments
8/23/19	Water Canyon	Water Canyon Above E262.5	Wh-11	NM-128.A_13	Robert Gallegos, Jennifer Fullam, Sam Loftin, Marwin Shendo	Yes	35°49'32" N	106°17'4" W	35.82555556	106.2844444	0-1	Clear/Sunny	Intermittent showers- TA-49 Rain Gage Station: .03" on 8/21/2019	No	No	No	No	0	0	0	1	2	2	1	1.5	2	1.5	0	0	0	0	11	1.49	Vegetation found at the site: upland grasses, grape, NM olive, virginia creeper, ponderosa, clove, gamble oak.
8/23/19	Water Canyon	Water Canyon Above CdV Confluence	Wf-10.5	NM-128.A_13	Robert Gallegos, Jennifer Fullam, Sam Loftin, Marwin Shendo	Yes	35°49'51" N	106°18'23" W	35.83083333	106.3063889	0-1	70% Cloud Cover	Intermittent showers- TA-49 Rain Gage Station: .03" on 8/21/2019	No	No	No	No	0	0	0	0	2	1	1	1.5	3	2	0	0	0	0	10.5	1.26	Vegetation Present at the site: sweet clover, ret top, white pine, rubeckia, alser, vox, barberry
8/23/19	Water Canyon	Water Above SR-4	Wi-4.5	NM-128.A_13	Robert Gallegos, Jennifer Fullam, Sam Loftin, Marwin Shendo	Yes	35°49'7" N	106°15'51" W	35.81861111	106.2641667	0-1	Clear/Sunny	Intermittent showers- TA-49 Rain Gage Station: .03" on 8/21/2019	No	No	No	No	0	0	0	0	1	2	1	0	0	0	0	0.5	0	0	4.5	1.12	
8/29/19	Water Canyon	Below Area A	Wb-16.5	NM-128.A_13	Kelkenny Bileen, Cesar Dominguez, Robert Gallegos, Brian Iacona	No	35°50'3" N	106°20'9" W	35.83416667	106.3358333	0-1	Clear/Sunny	Intermittent showers-TA-49 had .05 inches on 8/27/2019.	No	No	No	No	2	0	2	0	2	3	2	1.5	2	2	0	0	0	0	16.5	2.34	Caddisfly casing and live mayfly found. 1.10-Very few pools were observed.
8/29/19	Water Canyon	Below TA-11	Wc-11	NM-128.A_13	Kelkenny Bileen, Cesar Dominguez, Robert Gallegos, Brian Iacona	No	35°49'44" N	106°19'17" W	35.82888889	106.3213889	0-1	60% Cloud Cover	Intermittent showers-TA-49 had .05 inches on 8/27/2019.	No	No	No	No	0	0	0	0	1.5	2.5	2	1.5	2	1.5	0	0	0	0	11	2.33	1.6-Very few plants in thalweg.
8/29/19	Water Canyon	At E260	Wd-9	NM-128.A_13	Kelkenny Bileen, Cesar Dominguez, Robert Gallegos, Brian Iacona	No	35°49'50" N	106°18'27" W	35.83055556	106.3075	0-1	60% Cloud Cover	Intermittent showers-TA-49 had .05 inches on 8/27/2019.	No	No	No	No	0	0	0	0	1.5	1	1.5	1.5	2	1.5	0	0	0	0	9	1.3	
10/17/19	Water Canyon	At E260	We-10.5	NM-128.A_13	Robert Gallegos, Brian Iacona, Kelkenny Bileen, Kris Barrios, Alethea Banar	Yes	35°49'50" N	106°18'27" W	35.83055556	106.3075	0-1	50% Cloud Cover	Clear/Sunny	No	No-Note: SR501 is in upper part of canyon.	No	No	1	0	0	0	1.5	2	1	1.5	2	1.5	0	0	0	0	10.5	1.21	Vegetation Present-Ponderosa pine, spruce, fir, water birch, rose, gambel oak, aspen, NM locust, mullen, vetch, upland grasses, thistle, gooseberry, bearberry, composites, equisetum, raspberry, artemesia, virginia creeper, ash, yarrow.
10/23/19	Water Canyon	Above 501	Wa-21	NM-128.A_12	Brian Iacona, Robert Gallegos	No	35°50'18" N	106°21'38" W	35.83833333	106.3605556	0-1	Clear/Sunny	Clear/Sunny	No	Yes- Widening of channel. Matting and grade control below assessment site.	No	No	5	0	2	2	2.5	2	1	1.5	2	1.5	0	0.5	1.5	0	21	2.28	Vegetation present-Sedges, willow, red top, cattails, timothy. 1.1-Water flow is present below gcs; dry channel with standing pools above gcs. 1.3-Waterbug and caddis found. 1.10-Limited sorting. 1.11-No hydric soils found but standing water observed in hyporheic zone with digging to 4 inches.

Where NMED was present
located within three segments proposed in 140
Scored as Perennial (>19 Score)
Scored as Intermittent (9-19 Score)

AMIGOS BRAVOS' EXHIBIT 17

REBUTTAL TESTIMONY OF JAMIE C. DeWITT, PH.D., DABT

Q: What is your name?

A: Jamie DeWitt

Q: Dr. DeWitt, you provided direct testimony in this matter on behalf of Amigos Bravos?

A: Correct.

Q: Since then, have you reviewed portions of the notices of intent to submit direct testimony filed by the New Mexico Environment Department (NMED), Triad National Security, LLC/U.S. Department of Energy (Triad/DOE), New Mexico Mining Associations (NMMA), and San Juan Water Association (SJWA)?

A. Yes. I reviewed portions of their notices of intent related to the subject matter of my direct testimony.

Contaminants of Emerging Concern

Q: In your direct testimony, you supported NMED's proposal to include a new definition in the surface water quality regulations for "contaminants of emerging concern," correct?

A: Correct. I support adding a definition for "contaminants of emerging concern" or "CECs" and adding "per- and polyfluoroalkyl substances" or "PFAS" as an example of CECs. I also support adding a qualifier to the definition of "CECs" to clarify that CECs are compounds distinct from the defined regulatory category of "toxic pollutants." The definition for CECs, at 20.6.4.C(7) NMAC, that I supported in my direct testimony provides that:

> "Contaminants of emerging concern" or "CECs" refer to water contaminants including, but not limited to, per- and polyfluoroalkyl substances, pharmaceuticals and personal care products that may cause significant ecological or human health effects at low concentrations and are not considered "toxic pollutants" by the department. CECs are generally chemical compounds that, although suspected to potentially have impacts, may not have regulatory standards, and the concentrations to which negative impacts are observed have not been fully studied.

Q: What is the basis for that opinion?

A: CECs are generally chemical compounds that, although suspected to potentially have impacts, may not have regulatory standards, and the concentrations at which negative impacts are observed have not been fully studied. The inclusion of PFAS as CECs is supported by the

U.S. PFAS Action Plan Program Update (U.S. EPA, 2020a[1]), which refers to PFAS as "emerging contaminants." In light of the prevalence of PFAS, their persistence in environmental media, and their potential for harm to human health and the environment, it is appropriate to highlight these compounds as examples of CECs in NMED's regulatory definition.

Q: Dr. DeWitt, you also opined in your direct testimony that NMED should have the authority to require monitoring for CECs in federal permits, correct?

A: Yes. I support the following language proposed by Amigos Bravos at 20.6.4.14.F NMAC:

> **20.6.4.14 SAMPLING AND ANALYSIS**
> . . .
> **F.** The department may include sampling and monitoring of contaminants of emerging concern as a condition in a federal permit under Section 401 of the federal Clean Water Act.

Q: What is the basis for your opinion?

A: NMED should have the authority to study these compounds and add to the science community's body of knowledge by requiring dischargers to establish baseline and monitor and assess. Monitoring and characterization data can further our understanding of the prevalence of these compounds in surface waters, identify levels of PFAS and other CECs to which humans and other living organisms are exposed, and provide data for development of mitigation and management strategies that can potentially prevent harm to human and ecological health.

Q: You've reviewed the portions of Triad/DOE, NMMA, and SJWA notices of intent objecting to adding a definition of "contaminants of emerging concern" to the surface water quality regulations, correct?

A: Yes, I have.

Q: What is your response to their objections?

Their objections to adding a definition are based in large measure on NMED's proposal to add CECs to the narrative standard for toxic pollutants at 20.6.4.13.F(1) NMAC to which they object on the ground that too many unidentified constituents could be banned. Because they object to including CECs in the narrative standard for toxic pollutants, they object to adding a definition for CECs because there would then be no other reference in the regulations to CECs.

However, I support authorizing NMED to require monitoring for CECs, for the reasons set forth above and in my direct testimony, and therefore support adding CECs as a defined term in the regulations.

[1] I have included a list of references referred to in my testimony at the end of my testimony.

Definition of "Toxic Pollutants"

Q: Dr. Dewitt, you have expertise in the toxicity of per- and polyfluoroalkyl substances?

A: Yes, I set forth my credentials in my direct testimony.

Q: And you gave your opinion that certain PFAS should be considered "toxic pollutants" under the surface water quality regulations, correct?

A: That is correct.

Q: Specifically, you testified that the following nine PFAS should be considered "toxic pollutants", correct?

- **Perfluorooctanoic acid (PFOA),**
- **Perfluorooctane sulfonate (PFOS),**
- **Perfluorohexane sulfonic acid (PFHxS),**
- **Perfluorononanoic acid (PFNA),**
- **Perfluorobutane sulfonate (PFBS),**
- **Fluorotelomer sulphonic acid 8:2 (8:2 FTS),**
- **N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA),**
- **N-methylperfluorooctane sulfonamidoacetic acid (NMeFOSAA), and**
- **Perfluorooctanesulfonamide (PFOSA or FOSA).**

A: Correct.

Q: Triad/DOE in its technical testimony, and NMMA in non-technical comments, propose a new definition for "toxic pollutants." At 20.6.4.7.T(2) NMAC, they propose:

> **(2) "Toxic pollutant"** means those pollutants, or combination of pollutants~~, including disease-causing agents, that after discharge and upon exposure, ingestion, inhalation or assimilation into any organism, either directly from the environment or indirectly by ingestion through food chains, will cause death, shortened life spans, disease, adverse behavioral changes, reproductive or physiological impairment or physical deformations in such organisms or their offspring~~ ***listed by the EPA Administrator under section 307(a) of the federal Clean Water Act, 33 U.S.C. § 1313(a) or in the list below***.

Dr. DeWitt, have you reviewed the U.S. Environmental Protection Agency's (EPA) list of "toxic pollutants", and are any of the nine PFAS that you have identified as "toxic pollutants" on the EPA list?

A: I have reviewed the EPA list, and none of the nine PFAS I believe should be categorized as toxic pollutants under New Mexico surface water quality regulations are on the EPA list.

Q: What explains the fact that these compounds that you as an expert believe are "toxic pollutants" are not on EPA's list?

The EPA has demonstrated a commitment to collecting scientific evidence about PFAS that will allow it to determine if individual PFAS, groups of PFAS, or all PFAS as a class may cause significant harm to human or ecological health, even at low concentrations. This commitment is demonstrated by EPA's PFAS Action Plan (U.S. EPA, 2019), the establishment of an EPA Council on PFAS (U.S. EPA, 2021), and the funding of PFAS research grants through National Priorities awards (U.S. EPA, 2020b). The EPA also has demonstrated a commitment to protecting human or ecological health from PFAS by the establishment of lifetime health advisory levels for PFOA and PFOS in drinking water (U.S. EPA, 2016a,b), and the creation of health risk assessment documents for additional PFAS (U.S. EPA, 2018, 2021b).

Unfortunately, the EPA is often slow to add substances to the Clean Water Act; the last amendment to the list of toxic pollutants was made in 1981. Numerous factors beyond the scope of my opinion likely contribute to the lack of additional substances to the Clean Water Act. Individual states are not prohibited from enacting regulations that are more stringent than the EPA under the Clean Water Act. The Clean Water Act establishes national minimum standards but states may protect their waters more rigorously than the minimum standards under the Clean Water Act (ELI, 2013). As stated in my opinion, the State of Colorado developed translational levels for five individual PFAS (PFOA, PFOS, PFHxS, PFNA, and PFBS) as well as four PFAS that can parent constituents that can degrade to PFOA or PFOS (NEtFOSAA, NMeFOSAA, PFOSA/FOSA, and 8:2 FTS). Other states within the U.S. have set maximum contaminant levels (MCLs) for some of these PFAS in drinking water -- for example, Michigan has MCLs for PFOA, PFOS, PFHxS, PFNA, and PFBS; EGLE, 2020 -- giving additional support to the toxicity of these PFAS.

Q: Do you have experience working with states that are examining how they should regulate PFAS?

A: Yes, in 2019, I was a member of the Science Advisory Workgroup (SAW) to the Michigan PFAS Action Response Team (MPART), which was charged with advising the State of Michigan on Maximum Contaminant Level recommendations for PFAS. The job of the SAW was to work with members of the MPART to evaluate available occurrence data on PFAS in Michigan and available toxicological and epidemiological data on PFAS to propose MCLs. While the deliberations of the SAW with the MPART are confidential, one outcome of the SAW was to recommend MCLs for seven individual PFAS: PFOA, PFOS, PFHxS, PFNA, PFBS, PFHxA, and HFPO-DA. These MCL recommendations were later enacted into law by the State of Michigan. Additionally, based on the similarity in toxicity for the long-chain PFAS, the SAW recommended using the health-based value derived for PFNA (6 ng/L) as a screening level for all other long-chain PFAS included on the EPA Method 537.1 analyte list for which the SAW did not develop an individual health-based value (MSAW, 2019).

Just as under the Clean Water Act, under the Safe Water Drinking Act, states may establish MCLs for contaminants that have not been regulated by the EPA under the Safe Water Drinking Act (Congressional Research Service, 2021). I also am a member of the Secretaries Science Advisory Board, which is advisory to the Department of Health and Human Services and Department of Environmental Quality in North Carolina. I also am a member of the Tennessee PFAS External Advisory Group, which is advisory to the Tennessee Departments of Environment and Conservation and Health. Both of the advisory bodies have discussed PFAS and actions that the state(s) could and should take with respect to PFAS.

Q: Based on your experience in Michigan and your overall education and experience as a toxicologist, do you have an opinion whether states like New Mexico should have the authority to pass more stringent regulations governing toxic pollutants than EPA?

A: Yes, I do.

Q: What is your opinion?

My opinion is that states like New Mexico should have the authority to pass more stringent regulations than the EPA governing toxic pollutants. My opinion also is consistent with the State of Michigan, which is:

> "Unfortunately, we do not have federal drinking water standards, despite knowing they are in our drinking water and that some PFAS have been associated with adverse health effects. Recognizing that the USEPA is still likely several years away from providing any leadership on PFAS drinking water standards, Michigan, like other states, was left to develop our own." (MSAW, 2019)

Sampling and Analysis

Q: Currently, under the Commission's regulations, NMED may use various sampling methods listed in 20.6.4.14 NMAC to monitor for constituents. Triad/DOE propose to limit sampling and analysis of constituents for purposes of compliance with standards and certification of federal permits to methods approved in 40 CFR Part 136.[2] Have you reviewed 20.6.4.14 NMAC, on sampling and analysis?

[2] Triad/DOE propose:

> **20.6.4.14 SAMPLING AND ANALYSIS:**
>
> **A.** ***CFR Part 136 approved methods shall be used to determine compliance with these standards and in Section 401 certifications under the federal Clean Water Act. In all other cases, sampling*** ~~Sampling~~ and analytical techniques shall conform with methods described in the following references unless otherwise specified by the commission pursuant to a petition to amend these standards:
>
> **(1)** "*Guidelines Establishing Test Procedures For The Analysis Of Pollutants Under The Clean Water Act*," 40 CFR Part 136 or any test procedure

A: Yes.

Q: And are you familiar with 40 CFR Part 136?

A: I am aware of 40 CFR Part 136 in that I have read it and familiarized myself with its contents. I do not routinely refer to it, use it, or follow it in my everyday work.

Q: If the Commission were to adopt Triad/DOE's proposal, what are the implications for sampling and analysis for PFAS?

A: 40 CFR 136 does not include methods for sampling or analytical techniques specifically for PFAS, which would limit NMED's ability to monitor for PFAS in surface waters. However, the EPA has published several test methods that are applicable for drinking water and/or surface waters. These include EPA Method 537.1: "Determination of selected PFAS in drinking water by SPE and LC/MS/MS" (2018/2020); EPA Method 537: "Determination of selected PFAS in drinking water by SPE and LC/MS/MS" (2009); EPA Method 533: "Determination of PFAS in drinking water by isotope dilution anion exchange SPE and LC/MS/MS" (2019); and EPA Method 8327: "PFAS using external standard calibration and MRM LC/MS/MS" (2019).

Q: Currently, do the Commission's regulations authorize appropriate sampling for PFAS?

A: Yes. It appears as if water sampling for PFAS done by the Commission are using EPA Method 537.1 (NMED MOA, 2019). This is a method developed and published by the EPA.

Q: Method 537.1 is approved by EPA's Office of Research and Development, correct?

---

approved or accepted by EPA using procedures provided in 40 CFR Parts 136.3(d), 136.4, and 136.5;

(2) *Standard Methods For The Examination Of Water And Wastewater*, latest edition, American public health association;

(3) *Methods For Chemical Analysis Of Water And Waste*, and other methods published by EPA office of research and development or office of water;

(4) *Techniques Of Water Resource Investigations Of The U.S. Geological Survey*;

(5) *Annual Book Of ASTM Standards*: volumes 11.01 and 11.02, water (I) and (II), latest edition, ASTM international;

(6) *Federal Register*, latest methods published for monitoring pursuant to Resource Conservation and Recovery Act regulations;

(7) *National Handbook Of Recommended Methods For Water-Data Acquisition*, latest edition, prepared cooperatively by agencies of the United States government under the sponsorship of the U.S. geological survey; or

(8) *Federal Register*, latest methods published for monitoring pursuant to the Safe Drinking Water Act regulations.

A: Yes.

Q: Is it valid to use Method 537.1 to test for PFAS in surface water?

A: While U.S. EPA Method 537.1 was developed for measuring PFAS in drinking water, it can be modified to be applied to surface waters. For example, in Hopkins et al. (2018), EPA Method 537 was modified for the measurement of PFAS in surface waters. These EPA methods offer guidelines that laboratories can follow and apply to matrices other than drinking water. Kotlarz et al. (2020) used EPA Method 537.1 to guide analysis of PFAS in blood in people who had consumed drinking water contaminated with PFAS.

Q: Do you have an opinion whether the Commission should adopt Triad/DOE's proposal to limit sampling and analysis, for purposes of compliance and federal permits, to EPA's guidelines as that proposal applies to sampling and analysis for PFAS?

A: As Triad/DOE's proposal to limit sampling and analysis, for purposes of compliance and federal permits, to EPA's guidelines under 40 CFR 136, does not include a method for sampling or analytical techniques specific to PFAS, I disagree that this approach will be protective of human or ecological health from effects of PFAS in surface waters.

Conclusion

Q: Is the testimony you've provided accurate to the best of your knowledge:

A: Yes.

Q: Thank you for your testimony.

A: You're welcome.

Jamie DeWitt, Ph.D., DABT

6/19/21
Date

List of References

Congressional Research Service. 2021. Regulating contaminants under the Safe Drinking Water Act (SDWA). https://crsreports.congress.gov/product/pdf/R/R46652/8. Accessed June 2021.

EGLE (Michigan Department of Environment, Great Lakes, and Energy). 2020. Michigan adopts strict PFAS in drinking water standards. https://www.michigan.gov/egle/0,9429,7-135-3308_3323-534660--,00.html. Accessed June 2020.

ELI (Environmental Law Institute). 2013. State Constraints: State-imposed limitations on the authority of agencies to regulated waters beyond the scope of the Federal Clean Water Act. An ELI 50-state study. https://www.eli.org/sites/default/files/eli-pubs/d23-04.pdf. Accessed June 2020.

Hopkins ZR, Sun M, DeWitt JC, and Knappe DRU. 2018. Recently detected drinking water contaminants GenX and other per- and polyfluoroalkyl ether acids. *Journal of the American Water Works Association.* 110:13-28.

Kotlarz N, McCord J, Collier D, Lea SC, Strynar M, Lindstrom AB, et al. 2020. Measurement of novel, drinking water-associated PFAS in blood from adults and children in Wilmington, North Carolina. *Environmental Health Perspectives*. 127: 77005. doi: 10.1289/EHP6837.

MSAW (Michigan Science Advisory Workgroup). 2019. Health-based drinking water value recommendations for PFAS in Michigan. https://www.michigan.gov/documents/pfasresponse/Health-Based_Drinking_Water_Value_Recommendations_for_PFAS_in_Michigan_Report_659258_7.pdf.

NMED MOA (Memorandum of Agreement). 2019. Memorandum of agreement between the New Mexico Environment Department and the United States Geological Survey. Amendment #1. https://www.env.nm.gov/pfas/wp-content/uploads/sites/21/2019/04/2020-12-09-A1-USGS-PFAS-MOA-21-667-2080-0001_USGS-Executed.pdf. Access June 2020.

U.S. EPA. 2016a. Drinking water health advisory for perfluorooctanoic acid (PFOA). *U.S. Environmental Protection Agency, Office of Water, Health and Ecological Criteria Division*. Washington, DC.

U.S. EPA. 2016b. Drinking water health advisory for perfluorooctane sulfonate (PFOS). *U.S. Environmental Protection Agency, Office of Water, Health and Ecological Criteria Division*. Washington, DC.

U.S. EPA. 2018. Human health toxicity values for hexafluoropropylene oxide (HPFO) dimer acid and its ammonium salt (CASRN 13252-13-6 and CASRN 62037-80-3). https://www.epa.gov/sites/production/files/2018-11/documents/genx_public_comment_draft_toxicity_assessment_nov2018-508.pdf. Accessed June, 2021.

U.S. EPA (Environmental Protection Agency). 2019. EPA's Per- and Polyfluoroalkyl Substances (PFAS) Action Plan. https://www.epa.gov/sites/production/files/2019-02/documents/pfas_action_plan_021319_508compliant_1.pdf. Accessed June 2020.

U.S. EPA (Environmental Protection Agency). 2020a. EPA PFAS Action Plan: Program Update February 2020a. https://www.epa.gov/sites/production/files/2020-01/documents/pfas_action_plan_feb2020.pdf. Accessed June 2020.

U.S. EPA (Environmental Protection Agency). 2020b. National Priorities: Per- and polyfluoroalkyl substances. Recipients list. https://cfpub.epa.gov/ncer_abstracts/index.cfm/fuseaction/recipients.display/rfa_id/640/records_per_page/ALL. Accessed June 2020.

U.S. EPA (Environmental Protection Agency). 2021a. EPA Administrator Regan establishes new council on PFAS. https://www.epa.gov/newsreleases/epa-administrator-regan-establishes-new-council-pfas. Accessed June 2020.

U.S. EPA (Environmental Protection Agency). 2021b. Human health toxicity values for perfluorobutane sulfonic acid (CASRN 375-73-5) and related compound potassium perfluorobutane sulfonate (CASRN 29420-49-3). *Office of Research and Development, Center for Public Health and Environmental Assessment.*

AMIGOS BRAVOS' EXHIBIT 18

David Hope, P.Chem.
Professional Chemist

General:
David Hope has worked in the chemistry industry for over 40 years. He is a founding partner and Chief Executive Officer for Pacific Rim Laboratories Inc (PRL), whose clients span the globe. He also acts as QA Manager, Lab Director and Marketing Manager for the lab, which is ISO17025 accredited. His first-hand experience includes bench chemistry, instrumental analysis, method auditing and data interpretation for all methods (including PCB methods) offered by Pacific Rim Labs. David has been a significant part of dozens of scientific peer-reviewed publications as author or analytical services provider and has presented at numerous international conferences on the analysis of PCBs and other persistent organic contaminants. He has provided many large-scale projects with quality analytical services.

Education and Professional

- **Professional Chemist in the Province of British Columbia, Canada (P.Chem.)**
- **B.Sc. Chemistry, University of Victoria, BC, 1980**
-

Career History:

Pacific Rim Laboratories Inc.	Jan. 2003 – present	Founding partner and CEO; Method QA/QC
AXYS Analytical Services Ltd.	Mar 2001 – Sept. 2002	President.
BC Environment Industry Association	Mar 1999 – Feb. 2001	Executive Director.
Philip Analytical Services Corp.	Jan. 1996 – Feb. 1999	General Manager, Burnaby, BC laboratory
Zenon Environmental Inc.	Jan. 1989 – Dec. 1995	General Manager, Burnaby, BC laboratory.
Seakem Oceanography Ltd.	Sep. 1980 – Jan. 1989	GC/MS operator.

Career Highlights

1. Implemented methods for the analysis of dioxins and furans in soil, water and tissue in three labs.
2. Chaired the Organic Methods Committee for the BC Ministry of Environment Methods Manual (1994).
3. Expert witness testimony on the “Fate of Oil in the Marine Environment” (1998)
4. Organized and led trade missions to Japan, Korea, Hong Kong (1999-2002)
5. Director, BC Environment Industry Association (1994 – 1999)
6. First Vice-Chair, Canadian Environment Industry Association (2002)
7. Chair, Asia Working Group, Trade Team Canada Environment (2002-03)

8. President, Canadian Council of Independent Laboratories (2009-2011)
9. Director, Canadian Council of Independent Laboratories (2005-present)
10. Professional chemist (P.Chem.), member of the Association of the Chemical Profession of British Columbia

Selection of National and International Projects Managed

PRL has had an ongoing contract with the Canadian Food Inspection Agency (CFIA) for the last eleven years. This involves analyzing meat, dairy, oil and vegetation samples for dioxins, PCB and PAH. We are trusted to monitor the Canadian food supply!

Since 2006, we have been analyzing water, sediment and biota from sites on the Uruguay River for dioxins, PCB and PAH. This data was used by Uruguay at the World Court in The Hague in defense of accusations from Argentina that they were polluting the river. In short, our data has stood up to international scrutiny in a court of law. Pulp Mills on the River Uruguay (Argentina v. Uruguay) https://www.icj-cij.org/en/case/135

One of the results from the World Court ruling was a suggestion that Argentina and Uruguay set up a joint commission to continue monitoring the Uruguay River and the effects of the pulp mill built at Fray Bentos. PRL has been the contract lab for Comisión Administradora del Río Uruguay (CARU) since 2011 and routinely analyze dioxins, PCB, PAH, OCP and NP.

PRL was contracted by IZSM (Naples, Italy) to analyzed soil and ambient air samples for PCB, PAH, PBDE and organochlorine pesticides by high resolution mass spectrometry. To date, 975 soil samples and 1130 ambient air samples (PUF, rainfall, dust) have been reported.

Academic Publications:

SELECTION of PEER-REVIEWED JOURNALS & VOLUMES

2021

QU C., DE VIVO B., ALBANESE S., FORTELLI A., SCAFETTA N., LI J., **HOPE D.**, CERINO P., PIZZOLANTE A., QI S. and LIMA A., 2021. *Highly spatial-resolved measurements of passive-sampler derived air concentrations of persistent organic pollutants in the Campania region, Italy: Implications for sources and human exposure*. Environmental Pollution, X, XX-XX. Doi: XXXXX (Accepted)

DE VIVO B., ALBANESE S., LIMA A., QU C., FORTELLI A., GUARINO A., ZUZOLO D., ESPOSITO M, PIZZOLANTE A, CERINO P., **HOPE D.**, POND P., CICCHELLA D., 2021b. *Monitoraggio geochimico-ambientale dei suoli della Regione Campania. Il Piano Campania Trasparente. Volume 2. Composti Organici Persistenti: Idrocarburi Policiclici Aromatici, Policlorobifenili, Pesticidi. Distribuzione nei suoli superficiali*. ARACNE Editrice, Roma. ISBN: 978-88-255-4107-6, 320 p. http://www.aracneeditrice.it/index.php/pubblicazione.html?item=9788825541076

DE VIVO B., ALBANESE S., CICCHELLA D., QU C., FORTELLI A., GUARINO A., **HOPE D.**, POND P., ESPOSITO M, CERINO P., PIZZOLANTE A, LIMA A., 2021c. *Monitoraggio geochimico-ambientale della matrice aria della Regione Campania. Il Piano Campania Trasparente. Volume 3. Composti Organici Persistenti in PUF (Filtri Passivi di Poliuretano) e W&D (Deposimetri Passivi di Umido/Secco). Idrocarburi policiclici aromatici (IPA) Policlorobifenili (PCB), Pesticidi (OCP) e Eteri di Polibromobifenili (PBDE). Distribuzione nella matrice aria.* ARACNE Editrice, Roma. ISBN….., XXX pag**. (In press)**

2019

QU C., ALBANESE S., LIMA A., **HOPE D.**, POND P., FORTELLI A., ROMANO N., CERINO P., PIZZOLANTE A. and DE VIVO B., 2019. *The occurrence of OCPs, PCBs, and PAHs in the soil, air, and bulk deposition of*

the Naples metropolitan area, southern Italy: Implications for sources and environmental processes. Environment International, 124, 89-97. Doi: 10.1016/j.envint.2018.12.031. **(PS)**

QU C., ALBANESE S., LI J., CICCHELLA D., ZUZOLO D., **HOPE D.**, CERINO P., PIZZOLANTE A., DOHERTY A. L., LIMA A. and DE VIVO B., 2019. *Organochlorine pesticides in the soils from Benevento provincial territory, southern Italy: spatial distribution, air-soil exchange and implications for environmental health*. Science of Total Environment, 674, 159-170. Doi: 10.1016/j.scitotenv.2019.04.029.

QI P., QU C., ALBANESE S., LIMA A., CICCHELLA D., **HOPE D**., CERINO P., PIZZOLANTE A., ZHENG H., LI J. and DE VIVO B., 2019. *Investigation of polycyclic aromatic hydrocarbons in the soils from Caserta provincial territory, southern Italy: spatial distribution, source apportionment and risk assessment*. J. Hazardous Materials, 383, 121-158. Doi: 10.1016/j.jhazmat.2019.121158.

2015

HOPE D, POND P, MUDALIGE WA, DEL POZO J, WRIGHT M *Recent advances in lowering the cost of dioxin analysis*. Organohalogen Compounds, Vol. 77, 668-671 (**2015**).

2014

HOPE D, POND P, MUDALIGE WA, DEL POZO J, WRIGHT M *Inexpensive rapid method for POPS analysis of food using ASE and tandem acid silica/carbon columns*, Organohalogen Compounds Vol. 76, 796-799 (**2014**).

2009

POND P, HOPE D, MA E. *Improved PCB congener specific analysis by HRGC-HRMS*, Organohalogen Compounds, Vol. 71, 1214-1219 (**2009**).

2008

CHAPTER SIX – *Gas chromatographic methods of chemical analysis of organics and their quality control*. Environmental Geochemistry, Site Characterization, Data Analysis and Case Histories, (**2008**), Pages 119-133.

Selection of ABSTRACTS TO CONFERENCES

2021

DE VIVO B., LIMA A., CICCHELLA D., QU C., **HOPE D.**, FORTELLI A., CERINO P., ESPOSITO M, PIZZOLANTE A., ALBANESE S., 2021. *Potentially Toxic Metals and Persistent Organic Pollutants high resolution monitoring at regional and local scale in the soil, air, and bulk deposition of the Campania Region, southern Italy*. Goldschmidt2021, Lyons (France), 4-9 July 2021

2020

DE VIVO B., LIMA A., QU C., **HOPE D.**, FORTELLI A., THIOMBANE M., CERINO P., PIZZOLANTE A., ALBANESE S., 2020. *Potentially toxic metals and persistent organic pollutants high resolution monitoring at regional and local scale in the soil, air, and bulk deposition of the Campania Region, southern Italy: sources, environmental processes and health issues***.** GeoHealth 2020, Bari, 1-5/9/2020.

2019

QU C., ALBANESE S., LIMA A., **HOPE D.**, POND P., FORTELLI A., CERINO P., PIZZOLANTE A., DE VIVO B., 2019. *The occurrence of OCPs, PCBs, and PAHs in the soil, air, and bulk deposition of the Naples metropolitan area, southern Italy: Implications for sources and environmental processes.* Dioxin Conference 2019, Kyoto. "39th International Symposium on Halogenated Persistent Organic Pollutants"

2018

HOPE, K., FIEDLER H, POND P, **HOPE D.**. Polychlorinated Biphenyls in Major Foodstuffs on the Canadian Market Dioxin Conference 2018, Krakow. "38th International Symposium on Halogenated Persistent Organic Pollutants"

WRIGHT M, **HOPE D**, POND P, HOPE K, DEL POSO J– *Moving from ASTM 5790-95 to Isotope Dilution for OCPs using GC-MS/MS* Dioxin Conference 2018, Krakow. "38th International Symposium on Halogenated Persistent Organic Pollutants"

2017

DE VIVO B., QU C., ALBANESE S., LIMA A., **HOPE D.**, FORTELLI A., ROLANDI R., CERINO P. and PIZZOLANTE A., 2017. *The occurrence of OCPs, PCBs, and PAHs in the soil, air, and particle deposition of provincial and metropolitan Naples areas, Italy: Implications for potential risk and environmental cycling*. Dioxin 2017, Vancouver. "37th International Symposium on Halogenated Persistent Organic Pollutants"

HOPE, K., Wright M, **HOPE D.**. PCBs in Bottled Water and Water Storage Containers Including Teflon. Dioxin 2017, Vancouver. "37th International Symposium on Halogenated Persistent Organic Pollutants"

2016

DE VIVO B., QU C., ALBANESE S., LIMA A., **HOPE D.**, FORTELLI A., ROLANDI R., PIZZOLANTE A., ESPOSITO M., GALLO A., NICODEMO F. and CERINO P., 2016. *A high spatial resolution project of polyurethane foam-based air samples for monitoring persistent organic pollutants in the atmosphere of the Campania Region, southern Italy*. DIOXIN 2016 FIRENZE, "36th International Symposium on Halogenated Persistent Organic Pollutants".

DE VIVO B., ALBANESE S., QU C., CICCHELLA D., ZUZOLO D., LIMA A., **HOPE D.**, ESPOSITO M., GALLO A., PIZZOLANTE A., NICODEMO F. and CERINO P., 2016. *A region-wide soil characterization for PAHs, OCPs and PCBs: the Campania case study*. DIOXIN 2016 FIRENZE, "36th International Symposium on Halogenated Persistent Organic Pollutants"

2014

POND P, **HOPE D**, MUDALIGE WA, DEL POZO J, *Evaluation of TSQ8000 (GC-MS/MS) for PCDD/F analysis.* DIOXIN 2014 MADRID, "34th International Symposium on Halogenated Persistent Organic Pollutants"

Selection of SEMINAR LECTURES

2020

Webinar: *Running a Commercial ISO 17025 POPs Lab During a Pandemic*

2018

Canadian Trace Organic Workshop, Vancouver *Organochlorine Pesticides by GC-MS/MS*

2017

ThermoFisher Scientific Workshop, DIOXIN 2017, Vancouver. *GC-MS/MS Workflow for POPs and PAH*
ThermoFisher Scientific Workshop, DIOXIN 2017, Vancouver. *Using Magnetic Sector DFS with DualData XL in a Commercial Dioxin Lab*
ThermoFisher Scientific Workshop, Boston. *Using Magnetic Sector DFS with DualData XL in a Commercial Dioxin Lab*
ThermoFisher Scientific Workshop, BFR2017, York. *Using Magnetic Sector DFS with DualData XL in a Commercial Dioxin Lab*
Webinar. *Using Magnetic Sector GC-HRMS in a Commercial Dioxin Lab*
SANIPES International Workshop, Lima. *A Deeper Look into the Life of a Dioxin and POPs Lab – Tips From the Experts: Pacific Rim Laboratories Inc.*

2016

ThermoFisher Scientific Workshop, DIOXIN 2016 Florence, Italy *Analyzing Organochlorine Pesticides (OCPs) by Magnetic Sector GC/HRMS*
Webinar *A Deeper Look into the Life of a Dioxin and POPs Lab – Tips From the Experts: Pacific Rim Laboratories Inc.*
Canadian Trace Organic Workshop, Saskatoon, *Analysis of PAH and Alkylated PAH by GC-MS/MS*
2016 Pacific Northwest Snowfighters Conference, Portland, *PCB 101*

2015

ThermoFisher Scientific Workshop, Prague. *Recent Advances in Lowering the Cost of POPs Analysis*

2014

ThermoFisher Scientific Workshop, Bremen Low Cost Dioxin Analysis - *Simplified Manual Sample Clean-up using Disposable Preparation Columns Combined with Thermo Scientific TSQ 8000 GC-MS/MS*

2013

Brominated Flame Retardant Conference BFR2013, San Francisco *HRMS PBDE Analysis on a Thermo DFS*
ThermoFisher Scientific Workshop, BFR2013, San Francisco *HRMS Analysis of PAH and Alkylated PAH*
Western Canada Trace Organic Workshop, Vancouver *Analysis of Tributyltin in Biota*

2012

ThermoFisher Scientific Workshop, Venice, Italy *Advances in HRMS PBDE Analysis*
Spokane River Forum, Spokane, *Analytical Methods for Analysis of PCB*

2011

ThermoFisher Scientific Workshop, Niagara-on-the-Lake, Improved PCB Congener Analysis By HRMS
Spokane River Forum, Spokane, *PCB 101*

AMIGOS BRAVOS' EXHIBIT 19

REBUTTAL TESTIMONY OF DAVID HOPE

Q: Please state your name?

A: David Hope.

Q: Mr. Hope, what is your educational background?

A: I have a Bachelor of Science with a Chemistry Major from the University of Victoria, 1980.

Q: Would you please describe your professional experience?

A: I started out as a bench chemist in 1980, mainly interested in extraction and analysis of petroleum hydrocarbons and polycyclic aromatic hydrocarbons (PAH). In the mid-80's my fascination with PCBs began as I worked to develop methods for analysis of polychlorinated biphenyl (PCBs) in soil from a former industrial site in Victoria Harbor. We did not have access to written methods other than what was available in the literature. We extracted soil with a mix of dichloromethane and methanol, purified the extract using Florisil, and then analyzed on a Gas Chromatography – Electron Capture Detector (GC/ECD). We used PCB209 as a surrogate. That method is not far off from what is used today.

In 1988, I developed methods for the analysis of polychlorinated dibenzo(p)dioxins and dibenzofurans, more commonly referred to as "dioxins". This is where I first started working at part per trillion levels in soil, and part per quadrillion levels in water. The work was all by low resolution mass spectrometry, but still quite cutting edge for its time.

After 10 years as a lab chemist I moved into a lab management role. I stayed closely involved with the organic chemistry lab, especially with the developments related to dioxin analysis.

In 2003, I brought together three other partners to form Pacific Rim Laboratories Inc. Our motto was to become the most diversified (niche) high resolution mass spectrometry (HRMS) lab in North America. We are an International Organization for Standardization (ISO) 17025 accredited lab, and also accredited by Washington State Department of Ecology (WDOE).

Initially, we only analyzed 18 of the 209 PCB congeners. Then, in 2004, there was a bid opportunity out of WDOE for 209 congener PCB analysis using EPA 1668A. We were a small four-person lab at the time, so I worked intimately with the lab staff in implementing the method for water, soil/sediment and fish tissue. Data processing software was not available at the time for the detailed calculations required by EPA 1668, so we moved everything over to spreadsheets and completed the calculation there. Level IV data packages were reviewed and accepted by WDOE.

In 2006, we won a contract with the government of Uruguay to analyzed dioxins, PCB and PAH in water and sediment samples. Subsequently, and unbeknown to us, our data was presented in the World Court by Uruguay in defense of an accusation from Argentina that a pulp mill on the

Uruguay River (this is the border between the two) was polluting the river. As we are an ISO 17025 laboratory, the Court accepted our data without question. In the final ruling, the Court did not find evidence of pollution, and recommended that the two countries monitor the river jointly. The Comision Administradora Rio Uruguay remains our client to this day.

The SPB-Octyl column specified in Method 1668 resolves only 162 of the 209 congeners. In 2007, at the Dioxin Conference in Tokyo, a colleague told us of this amazing GC column that could separate all 209 PCB congeners. He had only been using it on a GC/ECD and wanted to see if we could get similar results on a high resolution mass spectrometer. Some months later we received the column and subsequently published a poster at the 2009 Dioxin Conference on "Improved PCB Congener Specific Analysis by HRGC-HRMS". We were able to resolve 192/209 congeners. To this date, no one has published a method for separation of more than 192 congeners by HRMS.

Following this publication, I have been invited to talk on PCBs and other trace organic topics at a number of conferences and workshops.

- Western Canada Trace Organics Workshop (2009)
- Thermo Scientific POPs Symposium (Niagara-on-the-Lake, 2011; Venice 2012; Cairns 2012; San Francisco 2013, Bremen 2014, Prague 2015, Boston 2017, Vancouver 2017)
- Spokane River Forum (2011, 2012)
- In 2014, co-authored and presented two papers for the Dioxin2014 conference – Inexpensive Rapid Method for POPs Analysis of Food Using ASE and Tandem Acid Silica / Carbon Columns; and Evaluation Of TSQ8000 (GC-MS/MS) For PCDD/F Analysis. That was followed that up at Dioxin 2015 with a paper titled "Recent Advances in Lowering the Cost of Dioxin Analysis."

In 2015, I was invited by the Instituto Zooprofilatico Spementale de Mezzogiorno, Naples, Italy, to help them implement a program for identifying sources of persistent organic pollutants in the province of Campania. The project involved 1000 soil samples and 750 ambient air, water (rainfall) and dust samples collected over a period of three years. All samples were analyzed for PCB, Organochlorine Pesticides and PAH, while selected air samples were also analyzed for Polybrominated Diphenylethers and phthalates. Numerous conference posters, refereed publications and a three volume book, all of which I am a co-author, have come out of this work and are still coming out.

Q: Is Amigos Bravos Exhibit 18 an accurate copy of your curriculum vitae?

A: Yes

Q: Mr. Hope, would you please summarize the expert opinions you will provide in your testimony?

A: Yes. In my testimony, I will describe the methodology of two U.S. Environmental Protection Agency (EPA) methods for sampling and analyzing polychlorinated biphenyls or PCBs: EPA Method 608.3, testing for Aroclors, and EPA Method 1668C, testing for congeners.

I will testify that, in my opinion, EPA Method 608.3 is not sufficiently sensitive to detect PCBs at the New Mexico Water Quality Control Commission's ("Commission") numeric water quality standards for wildlife and for chronic and human health organism only standards for aquatic life, and that EPA Method 1668C is able to detect PCBs at the state's numeric limits.

I will testify that, in my opinion, the State of New Mexico should not limit itself to use of sampling methods approved by EPA in 40 CFR Part 136 (Part 136 Methods) for purposes of compliance with permits. Currently the Commission's regulations authorize use of other sampling methods from reliable sources. There are other sampling and analysis methods, such as EPA Method 1668C, that accurately detect pollutants, and should be available to the State to monitor the discharge of pollutants into New Mexico's surface waters.

Q: Mr. Hope, what are polychlorinated biphenyls or PCBs?

A: PCB is the generic term used to refer to a group of 209 individual molecules (congeners) that have 1-10 chlorine atoms on a biphenyl backbone (biphenyl is two benzene molecules joined together by a single bond). For the most part, PCBs were manufactured chemicals. Their production ceased in North America in 1977, but continued in other parts of the world until 1993. Aroclor is a trade name used to describe the product manufactured by Monsanto. Aroclors were sold under eight names – 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1268. The last two digits refer to the percentage of chlorine in the product (*i.e.*, Aroclor 1221 has 21% chlorine and Aroclor 1260 has 60% chlorine). In Japan, a similar product was manufactured and sold under the tradename Kanaclor. Approximately 1.3-1.5 million tons were produced globally, about half of that in the USA. Fifty percent of the remaining production was imported to the USA.

Q: What dangers or risks do PCBs in water present to humans and other living organisms?

A: I am not a toxicologist and this is not my field of expertise. However, 12 PCB congeners have been labeled as dioxin-like PCBs by the World Health Organization and given Toxic Equivalency Factors (TEF). These TEF relate the toxicity of the congener to that of 2,3,7,8-Tetrachlorodibenzo(p)dioxin (TCDD), one of the most dangerous chemicals we know. For example, PCB126 has a TEF of 0.1, or is 10% as toxic as TCDD.

Q: What experience do you and your laboratory have sampling and analyzing PCBs in water media?

A: As a commercial laboratory, we do not collect samples. We only analyze samples submitted by clients. PCBs were part of the first set of analytes for which Pacific Rim Labs was accredited in 2003. In 2004, we implemented EPA 1668. In the last 5 years, 2016-2020, we have averaged 1210 PCB samples per year, of which 383 were water samples.

Q: Are you familiar with EPA Methods 1668C, testing for congeners, and 608.3, testing for Aroclors, in PCBs? A: Yes.

Q: Would you explain how each of these methods works, and the differences between the two?

A: Although there are many PCB water methods, I will limit my comparison to 608.3 and 1668C.

Method 608 was first written in 1984 for the combined analysis of organochlorine pesticides and PCBs. It used packed columns and electron capture detectors. Method 608.3 was published in 2016. It has modernized the method calling for capillary columns and any halogen specific detector. Not much else has changed as far as extraction or quantitation techniques are concerned. It was and is an Aroclor method. Method 608.3 is a Part 136 Method.

The time between when a sample is collected and when the extraction must be started is called the hold time. The hold time in 608.3 is seven day, while 1668C lists a hold time of one year and states "There are no demonstrated maximum holding times associated with the CBs in aqueous, solid, semi-solid, tissue, or other sample matrices. If stored in the dark at less than 6 ºC, aqueous samples may be stored for up to one year." The shorter hold time for 608.3 is caused by including organochlorine pesticides together with PCBs. The only similarity between the two is that they all contain chlorine.

High resolution mass spectrometry was commercially available since the 1960's, but distribution was limited to universities and government labs. It became a required instrument for dioxin labs in the early 1990's. At first, HRMS PCB methods were limited to the 12 dioxin-like PCBs. Then Method 1668A was written in December 1999, the first method to look at all 209 PCB congeners on an individual basis. The latest version, 1668C was published in April 2010. Many of the significant updates were around quality control acceptance criteria. Extraction and instrumentation remained the same.

First, let me divide the methods into two parts – extraction and instrumental analysis. In each of these methods, the extraction is relatively the same. A 1 L water sample is either extracted with a solvent (liquid:liquid extraction), a continuous liquid:liquid extractor or by solid phase extraction. The extract can be purified through column chromatography using Florisil and/or washed with sulfuric acid. It is then concentrated to a small volume and analyzed gas chromatography coupled to various detectors.

Where the extraction differs is in the use of surrogates and internal standards. A surrogate is similar to the chemicals you are analyzing and is expected to behave in a similar fashion, and therefore its recovery in the final analysis can give you an indication of the quality of the result. As an extreme example, if your method does not recover any surrogate, then the analytical result you get for PCB concentration is not valid.

An internal standard is a chemical added to your extracted sample, just prior to injection on the GC. It is then used to quantify the results.

Method 608.3 requires 1-4 surrogates added to the water sample prior to extraction. They are:

- Dibutyl chlorendate
- Tetrachloro-m-xylene
- 4,4'-Dibromobiphenyl
- Decachlorobiphenyl (PCB209)

Any of these compounds not added as a surrogate can later be added as an internal standard. Surrogate recovery acceptance limits are not specified by the method but rather determined by each lab.

Method 1668 fortifies every sample prior to extraction with 27 carbon-13 labeled PCB standards. These standards are not just similar to PCBs, they are PCBs! However, as the twelve carbon atoms have been replaced with carbon-13 (one extra neutron) they have a different molecular weight and are therefore easily distinguishable from the carbon-12 analytes on a mass spectrometer (this would not be true on a halogen selective detector). The 27 standards consist of the first and last eluting PCBs in each level of chlorination, plus all dioxin-like PCBs. After extraction, three more carbon-13 labeled PCB standards are added. Then, prior to injection on the GC, five more carbon-13 labeled PCB standards are added. These are referred to as "Recovery Standards" because they are used to calculate the recovery of the other 30 standards. The 27 standards added at the beginning of analysis are used to quantify the results. The theory is that any loss of these standards during work-up would be at the same rate of loss for the corresponding PCBs. Lastly, the three standards added after extraction, give you an idea of the losses caused during extraction. Acceptable recoveries for each of these standards is dictated by the method.

Method 608.3 requires GC coupled with a halogen (chlorine, bromine, fluorene) specific detector, usually an ECD. These are very sensitive for chlorine, with sensitivity increasing at a rate significantly greater than the increase in number of chlorines. The ECD cannot give you any information on the number of chlorines present in a molecule. The method relies on a 30 m GC column to separate the congeners, however it does not resolve them all. It is possible that co-eluting congeners have the same or differing levels of chlorination (*i.e.*, a pentaCB could be coeluting with another pentaCB, or a hexaCB). For confirmation of results, a second 30 m column of different polarity is used.

Standard methods of quantitation involve comparing the area of a known concentration of internal standard and an analyte of interest, also of known concentration, from a calibration standard. This is true for PCB analysis as well. However, as the Aroclors have multiple components, they result in multiple peaks in the chromatogram. Method 608.3 requires a multipoint calibration using a combination of Aroclors 1016 and 1260, and a single point for all other Aroclors. For each Aroclor, five peaks from the chromatogram are used as indicative of that Aroclor. We determine relative response factors for each of those from the calibration standards. Next, you must visually observe the sample chromatogram and compare it with the Aroclor standards. If it looks like Aroclor 1242, then you use the response factors from an Aroclor 1242 standard to quantify the sample. It can sometimes be difficult to determine which Aroclor resembles the sample. This is especially true if the PCB has been exposed to environmental conditions for an extended period of time. In that case it is left up to operator judgment.

Method 1668C uses GC coupled with high resolution mass spectrometry. It is an extremely sensitive technique. PCBs are separated on a 60 m column, and then into their level of chlorination by mass spectrometry. Depending on which GC column is used between 162 and 182 peaks can be resolved. A multi-point calibration (0.2-2000 pg/µL) is analyzed containing the 27 native PCBs corresponding to the 13C-PCB internal standards. Relative response factors are determined. All other congeners are quantified based on response factors determined from a single standard containing all 209 congeners. Two 13C-monochlorobiphenyls are used to quantify the 3 monochlorobiphenyl congeners; two 13C-dichlorobiphenyls are used to quantify the 12 dichlorobiphenyl congeners; two 13C-trichlorobiphenyls are used to quantify the 24 trichlorobiphenyl congeners, and so on. It gets a bit more complicated when we include the dioxin-like PCBs into the equation – tetra through heptachlorobiphenyl! The lowest level calibration standard is at 0.2 picograms per microliter (pg/µL). This corresponds to a concentration of 20 pg/L (0.00002 micrograms per liter (µg/L)) per congener when extract is concentrated to a 0.1 mL final volume. Method 1668C is not a Part 136 Method.

The minimum level for Method 608.3 is 95 nanograms per liter (ng/L) (0.095 µg/L) and MDL is 65 ng/L (0.065 µg/L). The minimum level for Method 1668C is 20-200 pg/L (0.00002-0.0002 µg/L) and MDL is 7-77 pg/L (0.000007-0.000077 µg/L).

Q: Are you familiar with the Commission's numeric water quality standards for PCB's?

A: Yes.

Q: What are those standards?

A: The standards are below, set forth in a chart, with a legend following:

Pollutant	CAS Number	DWS	WH	Aquatic Life			Type
				Acute	Chronic	HH-OO	
Polychlorinated Biphenyls (PCBs)	1336-36-3	0.50 µg/L	0.014 µg/L	2 µg/L	0.014 µg/L	0.00064 µg/L	C, P

DWS: domestic water supply
WH: wildlife habitat
HH-OO: human health organism only
C: cancer causing
P: persistent

Q: These numeric criteria are set forth at 20.6.4.900.J NMAC of the Commission's water quality standards, correct?

A: Yes.

Q: Can EPA Method 608.3, testing for Aroclors, detect PCBs at the numeric levels of the Commission's standards?

A: Definitely not for the Aquatic Life Human Health-Organism Only, or for the Wildlife Habitat or Aquatic Life Chronic. The method detection limit specified in Method 608.3 is 65 ng/L or 0.065 µg/L. The method would be acceptable for Domestic Water Supply or Aquatic Life Acute testing.

Q: Can EPA Method 1668C, testing for congeners, detect PCBs at the numeric levels of the Commission's standards?

A: Yes.

Q: In conducting that work, to what extent does your laboratory utilize EPA Method 1668C?

A: We use 1668C exclusively for testing PCBs.

Q: What is your expert opinion regarding the validity, accuracy, and sensitivity of EPA Method 1668C?

A: EPA 1668C is the definitive method for low level PCB analysis (*i.e.*, anything <0.1 µg/L). It is sensitive and reproducible. The quality control measures far exceed anything found in EPA 608.3. Regulations talk about concentrations of PCB. EPA 608.3 provides concentrations of Aroclors, which are a subset of PCBs (there are PCBs that can be present in water that are not found in Aroclors). EPA 1668C includes all PCBs.

Q: Mr. Hope, is EPA Method 1668C a method published by the EPA Office of Water?

A: Yes. The EPA Office of Water published the method in April 2010. The published method, "Method 1668C Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS," is Amigos Bravos' Exhibit 20.

Q: Mr. Hope, based on your knowledge and experience, including your experience with states and local governments, do you believe that states should have the flexibility to use sampling and analysis methods in addition to Part 136 Methods?

A: Yes.

Q: Is the testimony you've provided accurate to the best of your knowledge?

A: Yes.

David Hope

21 June 2021
Date

AMIGOS BRAVOS' EXHIBIT 20

United States
Environmental Protection
Agency

Method 1668C
Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS

April 2010

U.S. Environmental Protection Agency
Office of Water
Office of Science and Technology
Engineering and Analysis Division (4303T)
1200 Pennsylvania Avenue, NW
Washington, DC 20460

EPA-820-R-10-005

Method 1668C Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS April 2010

The Office of Science and Technology (OST) in EPA's Office of Water developed Method 1668C (Method 1668C; the "Method") for use in Clean Water Act (CWA) programs. EPA is publishing this Method for users who wish to measure PCBs as congeners now, and in 2010, EPA expects to publish a proposal in the *Federal Register* for public comment to add this Method to other CWA Methods published at 40 CFR Part 136.

This Method determines chlorinated biphenyl congeners in environmental samples by isotope dilution and internal standard high-resolution gas chromatography/high-resolution mass spectrometry, HRGC/HRMS. EPA developed this Method for use in wastewater, surface water, soil, sediment, biosolids and tissue matrices. Other applications and matrices may be possible, which may or may not require modifications of sample preparation, chromatography, etc.

EPA used the results of an interlaboratory validation study of Method 1668A, a peer review of that study, user suggestions and additional interlaboratory data to write this version, 1668C, of Method 1668. Method 1668C, the validation study report, *Method1668A Interlaboratory Validation Study Report* (EPA-821-08-021), and the addendum describing the revised QC acceptance criteria, *Method 1668A Interlab Study Report Addendum*, are available at EPA's CWA methods website at www.epa.gov/waterscience/methods.

This "C" version of Method 1668 revises the quality control (QC) acceptance criteria in EPA Method 1668B to allow the upper recovery limit for some congeners to be above 100 percent, to revise the estimated method detection limits (EMDLs) and estimated minimum levels of quantitation (EMLs) to MDLs and MLs, and to makes other changes summarized below. The QC acceptance criteria developed in the interlaboratory method validation study of 1668A, and published in version B of the Method, did not allow the upper recovery limit for some congeners to be above 100 percent. The criteria have been revised based on data from the interlaboratory study and data from two laboratories with extensive experience in use of Method 1668A. TestAmerica, Knoxville, Tennessee and AXYS Analytical Services, Ltd., Sidney, British Columbia, Canada provided this new data. These two laboratories and Battelle-Columbus provided MDLs for the congeners and congener groups, which EPA pooled and used to replace the EMDLs and EMLs in Table 2 of Method 1668B with the MDLs and MLs in Method 1668C.

The detection limits and quantitation levels in this Method are usually dependent on the level of interferences and laboratory background levels rather than instrumental limitations. The method detection limits (MDLs) and minimum levels of quantitation (MLs) in Table 2 are concentrations at which a congener can be measured with no interferences present. In water, MDLs range from approximately 7 to 30 parts per quadrillion (picograms per liter, pg/L).

Interface, Inc. and CSC prepared this Method under EPA Contract EP-C-06-085. AXYS Analytical provided the single-lab data in Method 1668A that was later replaced by multi-lab data from laboratories that participated in EPA's inter-laboratory validation of 1668A (six labs for water and tissue, four for biosolids).

Summary of changes between EPA Method 1668B (January 2009) and 1668C (April 2010)

- Additional information on the concentration of extracts has been included in Section 4.2.

- The following note has been added to Section 10.1, "RTs, RRTs, and RRT limits may differ slightly from those in Table 2." This statement has also been added to the footnotes to Table 2.

- The note in Section 10.2.1 has been modified to inform the analyst that careful selection of the grade and purity of PFK may help minimize interferences with the dichlorobiphenyl secondary quantitation ion.

- The diluted combined 209 congener solution is now used for calibration verification, in place of the VER-3 solution. This allows all verification tests to be performed with a single solution.

- Section 17.2.1 has been changed to clarify that concentrations of native compounds other than those in the native toxics/LOC standard, in the labeled cleanup standard, and in the labeled injection internal standard (except for labeled CB 178) should be determined using the response factors from Section 10.5 or Section 15.4.2.3.

- Section 17.6.5 has been added to provide information on the use of optional data qualifier flags for reporting coeluting congeners.

- Based on data from the interlab validation study and data from two laboratories, the QC acceptance criteria in Table 6 have been revised to be consistent among tests for calibration verification (VER), initial precision and recovery (IPR), on-going precision and recovery (OPR), and labeled compound recovery from samples.

- Reference 22 has been added to cite the Addendum to the interlaboratory validation study report.

- Sections 1.3, 4.1, 4.6, 9.1.2.1, 9.5.2, 10.3.3, 17.6.1.4.1, 17.6.1.4.2, 17.6.1.4.3, and Table 2 been revised to change estimated method detection limits (EMDLs) and estimated minimum levels of quantitation (EMLs) to MDLs and MLs.

- Reference 23 has been added to cite the MDL data from AXYS, TestAmerica-Knoxville, and Battelle-Columbus, and to explain how these data were processed to produce the pooled MDLs in Table 2.

- A sentence was added to Section 11.4.2.1 to require weighing the sample bottle after emptying, and to determine the volume using the density of water.

- ML definition revised to cite the ML procedure.

- A note was added to Section 10.3.3 to state that MDLs and MLs lower than those in Table 2 may be established per Section 17.6.1.4.1.

- Section 17.6.1.4.1 expanded to state how MDLs and MLs lower than those in Table 2 may be established.

- A footnote was added to Table 2 to cite Reference 23.

Summary of changes between EPA Method 1668A (8-20-03) and 1668B (January 2009) (excluding typographical and grammatical error corrections, and section insertions or deletions necessitated by the following changes).

- Based on the interlaboratory validation study, single-laboratory QC acceptance criteria are replaced with interlaboratory criteria (Table 6). A new footnote 1 to Table 6 references the EPA interlaboratory study report, and the other footnote numbers are incremented.

- Section 1.5, the performance-based discussion, describes additional flexibility to modify CWA Methods that is allowed by 40 CFR Part 136.6.

- Section 2.5.2 now indicates that internal standards are the labeled congeners spiked into the sample.

- Section 2.5.3 now indicates that injection internal standards are labeled compounds spiked into the extract.

- Section 5.4 is an added section on biohazards.

- Section 7.8 notes that Method 1668A part numbers are valid for Method 1668B.

- Section 8.1 allows use of alternate sample collection techniques, if documented.

- Section 8.2 adds that one liter, or a larger or smaller volume of sample, may be collected.

- Section 12.3 adds a note to indicate that SDS extraction may cause loss of some mono- through tri-chloro congeners.

- Section 12.5.6 states that a macro concentration device is to be used to concentrate extracts, and deletes the requirement for collection of the extract in a round-bottom flask because any macro concentration device may be used.

- Section 16.2 requires an expert spectrometrist to determine analyte presence when an interference precludes meeting the signal-to-noise requirement for dichloro-CB congeners.

- Section 21 cites the validation studies, and that performance data are in the interlaboratory validation study report.

- Reference 1 was updated to the 2006 World Health Organization paper on toxicity equivalency factors.

- References 4 and 17 add titles to the papers in these references.

- Reference 21 cites the Method 1668A Interlaboratory Validation Study Report.

- Tables 2 and A-1 revised the elution order for congeners 107-109.

- Table 4 defines the solutions containing congeners 107, 108, and 109.

- Table 6 contains revised QC acceptance criteria for performance tests, and footnote 1 to Table 6 references the Method 1668A Interlaboratory Validation Study Report.

- Table 7 adds footnote 2 to require meeting the 10:1 signal-to-noise specification at the CS-2 calibration level.

Summary of corrections and changes to EPA Method 1668A as of August 20, 2003 (excluding typographical and grammatical error corrections, and section insertions or deletions necessitated by the following changes).

- Throughout: All references to IUPAC have been deleted. We have been informed that IUPAC does not assign congener numbers. Therefore, all references to congeners by number are to "congener number." The congener naming system given by Guitart, *et al.* (Guitart R., Puig P., Gomez-Catalan J., Chemosphere 27 1451-1459, 1993) has been used in EPA Method 1668A since its inception and continues in this version.

- Sections 2.1.3, 12.4.2., 12.4.3, 12.4.5, and 12.4.9: Hexane has be deleted from the extraction solvent for fish and other tissue to preclude loss of the more volatile CBs.

- Section 7.7: A note has been added to reference the two known suppliers of labeled compounds.

- Section 7.15: A statement has been added to include certified reference materials (CRMs) from the National Resource Council of Canada.

- Sections 8.2.3, 8.3.2, and 8.4.2: The preservation temperature for shipment of samples has been changed to <6 °C to encompass the 4 ± 2 °C used by some organizations (e.g., USGS).

- Section 8.2.3: The requirement to preserve aqueous samples with sulfuric acid has been deleted because PCBs are stable in environmental samples, and the storage temperature for aqueous samples has been changed to <6 °C.

- Section 9.1.2.1: A statement has been added that a modification may be used routinely after it has been demonstrated to meet the QC acceptance criteria of the performance tests, so long as the other requirements in the Method are met (e.g., labeled compound recovery).

- Section 10.1.2.3: The word "approximately" has been inserted in the requirement to meet the retention times in Table 2 to reflect that slight changes in GC columns will produce slightly different retention times.

- Section 10.1.2.4: A statement has been added to indicate that the absolute and relative retention times in Table 2 were obtained under the GC conditions given in Section 10.1.1.

- Section 10.2.2: The text has been changed to clarify that the deviation between each monitored exact m/z and the theoretical m/z (Table 7) must be less than 5 ppm.

- Section 10.5: The text has been corrected to state that the diluted combined 209 congener solution (Section 7.10.2.2 and Table 5) is used for single-point calibration of the Native Toxics/LOC CBs.

- Section 12.4: A note has been added to allow use of a separate aliquot for percent lipid determination.

- Section 12.4.1: The minimum time required to dry the sample has been reduced from 12-24 hours to 30 minutes.

- Section 15.6: A requirement has been added to analyze one or more aliquots of solvent after the OPR if the CBs would be carried into the Method blank.

- Section 16.4: RRT QC limits may be based on the limits in Table 2 or limits developed from calibration data.

- Section 17.2.2: The units have been corrected to ng/mL

- Section 17.4: A multiplier of 1000 has been inserted in the equation to convert ng in extract to pg in sample.

- Section 18.5: A section has been added to suggest that the carbon column should be used if interferences preclude identification and/or quantitation of the Toxics.

- Table 2: The relative retention times have been changed to correct errors and reference each compound to the correct retention time and quantitation reference. The RT and RRT windows have been adjusted to attempt to unambiguously identify each congener in the presence of other congeners. Footnotes 7 and 8 have been revised to reflect this changes.

- Table 3: Units for the diluted combined 209 congener solution have been corrected to ng/mL as have the concentrations of the native compounds in the diluted combined 209 congener solution.

- Table 6: The lower QC acceptance criteria limit for the labeled monochloro- and dichloro-CBs has been lowered for the IPR, OPR, and recovery from samples to reflect that these compounds can be lost by evaporation.

- Table 7: Cl-3 scan descriptors have been added to Function 2 and the m/z types for the 13C12 Cl-4 PCBs have been corrected in Function 4.

- Table 8: The m/z's forming the ratio, the ratio, and the QC limits have been corrected for decachlorobiphenyl.

- Table A1: The header has been corrected to delete reference to EMDLs and EMLs.

Disclaimer

Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

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Method 1668C
Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS

April 2010

1.0 Scope and Application

1.1 Method 1668C (the Method) is for determination of chlorinated biphenyl congeners (CBs) in wastewater and other matrices by high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

1.1.1 The CBs that can be determined by this Method are the 12 polychlorinated biphenyls (PCBs) designated as toxic by the World Health Organization (WHO): congeners 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189. The Method also determines the remaining 197 CBs, approximately 125 of which are resolved adequately on an SPB-octyl gas chromatographic column to be determined as individual congeners. The remaining approximately 70 congeners are determined as mixtures of isomers (co-elutions).

1.1.2 The 12 PCBs designated as toxic by WHO (the "Toxics"; also known as dioxin-like PCBs; DLPCBs), and the earliest and latest eluted congener at each level of chlorination are determined by the isotope dilution quantitation technique; the remaining congeners are determined by the internal standard quantitation technique.

1.1.3 This Method allows determination of the PCB toxicity equivalent (TEQ_{PCB}) for the Toxics in a sample using toxicity equivalency factors (TEFs; Reference 1) and allows unique determination of 19 of 21 CBs of interest to the National Oceanic and Atmospheric Administration (NOAA; Reference 2). A second-column option is provided for resolution of the two toxic PCB congeners (congener 156 and 157) that are not resolved on the SPB-octyl column and for resolution of other CB congeners.

1.1.4 This Method also allows estimation of homolog totals by level of chlorination (LOC) and estimation of total CBs in a sample by summation of the concentrations of the CB congeners and congener groups.

1.1.5 The list of 209 CBs (Table 1) identifies the Toxics, the CBs of interest to NOAA, and the LOC CBs.

1.2 EPA developed this Method for use in Clean Water Act (CWA) programs and for wastewater, surface water, soil, sediment, biosolids and tissue matrices. Other applications and matrices may be possible, which may or may not require modifications of sample preparation, chromatographic conditions, etc. Method 1668C is a revision of previous versions of Method 1668 all of which are based on a compilation of methods from the technical literature (References 3 and 4), and EPA's dioxins and furans Method, Method 1613.

1.3 The detection limits and quantitation levels in this Method are usually dependent on the level of interferences and laboratory background levels rather than instrumental limitations. The method detection limits (MDLs; 40 CFR 136, appendix B) and minimum levels of quantitation (MLs; 68 FR 11790) in Table 2 are the levels at which the CBs can be determined with no interferences present. The MDL for CB 126 in water is 16 pg/L (picograms-per-liter; parts-per-quadrillion).

1.4 The GC/MS portions of this Method are for use only by analysts experienced with HRGC/HRMS or under the close supervision of such qualified persons. Each laboratory that uses this Method must demonstrate the ability to generate acceptable results using the procedure in Section 9.2.

1.5 This Method is "performance-based," which means that you may make modifications without additional EPA review to improve performance (e.g., overcome interferences, or improve the sensitivity, accuracy or precision of the results) provided that you meet all performance criteria in this Method. Requirements for establishing equivalency are in Section 9.1.2, and include 9.1.2.2.3 – explaining the reason for your modifications. For CWA uses, additional flexibility is described at 40 CFR 136.6. You must document changes in performance, sensitivity, selectivity, precision, recovery, etc., that result from modifications within the scope of Part 136.6, and Section 9 of this Method, and how these modifications compare to the specifications in this Method. Changes outside the scope of Part 136.6 and Section 9 of this Method may require prior review or approval.

2.0 Summary of Method

Flow charts summarize procedures for sample preparation, extraction, and analysis for aqueous and solid samples, multi-phase samples, and tissue samples (Figures 1, 2 and 3, respectively.)

2.1 Extraction

2.1.1 Aqueous samples (samples containing less than one percent solids) – Stable isotopically labeled analogs of the Toxics and labeled LOC CBs 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).

2.1.2 Solid, semi-solid, and multi-phase samples (excluding tissue) – The labeled compounds are spiked into a sample containing 10 g (dry weight) of solids. Samples containing multiple phases are pressure filtered and any aqueous liquid is discarded. Coarse solids are ground or homogenized. Any non-aqueous liquid from multi-phase samples is combined with the solids and extracted in a Soxhlet/Dean-Stark (SDS) extractor. The extract is concentrated for cleanup.

2.1.3 Fish and other tissue – A 20-g aliquot of 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 a Soxhlet extractor. The extract is evaporated to dryness, and the lipid content is determined.

2.2 After extraction, a labeled cleanup standard is spiked into the extract which is then cleaned up using back-extraction with sulfuric acid and/or base, and gel permeation, silica gel, or Florisil chromatography. Activated carbon and high-performance liquid chromatography (HPLC) can be used for further isolation of specific congener groups. Prior to the cleanup procedures cited above, tissue extracts are cleaned up using an anthropogenic isolation column.

2.3 After cleanup, the extract is concentrated to 20 µL. Immediately prior to injection, labeled injection internal standards are added to each extract and an aliquot of the extract is injected into the gas chromatograph (GC). The analytes are separated by the GC and detected by a high-resolution (≥10,000) mass spectrometer. Two exact m/z's are monitored at each level of chlorination (LOC) throughout a pre-determined retention time window.

2.4 An individual CB congener is identified by comparing the GC retention time and ion-abundance ratio of two exact m/z's with the corresponding retention time of an authentic standard and the theoretical or acquired ion-abundance ratio of the two exact m/z's. Isomer specificity for certain of the CB congeners is achieved using GC columns that resolve these congeners.

2.5 Quantitative analysis is performed in one of two ways using selected ion current profile (SICP) areas:

2.5.1 For the Toxics and the LOC CBs, the GC/MS is multi-point calibrated and the concentration is determined using the isotope dilution technique.

2.5.2 For all congeners other than the Toxics and LOC CBs, the GC/MS is calibrated at a single concentration and the concentrations are determined using the internal standard technique. The internal standards are the labeled congeners spiked into the sample, thus affording recovery correction for all congeners.

2.5.3 For the labeled Toxics, labeled LOC CBs, and the cleanup standards, the GC/MS is calibrated using replicates at a single concentration and the concentrations of these labeled compounds are determined using the internal standard technique. The labeled injection internal standards are determined using the internal standard technique.

2.6 The quality of the analysis is assured through reproducible calibration and testing of the extraction, cleanup, and HRGC/HRMS systems.

3.0 Definitions

Definitions are in the glossary at the end of this Method.

4.0 Contamination and interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts, elevated baselines, and/or lock-mass suppression causing misinterpretation of chromatograms. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, reagents are cleaned by extraction or solvent rinse. Environmentally abundant CBs have been shown to be very difficult to completely eliminate from the laboratory at levels lower than the MDLs in this Method (Table 2), and baking of glassware in a kiln or furnace at 450 - 500 ºC may be necessary to remove these and other contaminants.

4.2 Proper cleaning of glassware is extremely important, because glassware may not only contaminate the samples but may also remove the analytes of interest by adsorption on the glass surface.

4.2.1 Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with fluoropolymer stopcocks, must be disassembled prior to detergent washing.

4.2.2 After detergent washing, glassware should be rinsed immediately, first with methanol, then with hot tap water. The tap water rinse is followed by another methanol rinse, then acetone, and then methylene chloride.

4.2.3 Baking of glassware in a kiln or other high temperature furnace (300 - 500 ºC) may be warranted after particularly dirty samples are encountered. The kiln or furnace should be vented to prevent laboratory contamination by CB vapors. Baking should be minimized, as repeated baking of glassware may cause active sites on the glass surface that may irreversibly adsorb CBs.

4.2.4 Immediately prior to use, the Soxhlet apparatus should be pre-extracted with toluene for approximately 3 hours (see Sections 12.3.1-12.3.3). The extraction apparatus (Section 6.4) should be rinsed with methylene chloride/toluene (80/20 mixture).

4.2.5 A separate set of glassware may to necessary to effectively preclude contamination when low-level samples are analyzed.

4.2.6 Concentration of extracts by Kuderna-Danish (K-D) concentrator and/or final concentration using nitrogen evaporation may help reduce levels of background PCBs in samples.

4.3 All materials used in the analysis must be demonstrated to be free from interferences by running reference matrix Method blanks (Section 9.5) initially and with each sample batch (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples).

4.3.1 The reference matrix must simulate, as closely as possible, the sample matrix under test. Ideally, the reference matrix should not contain the CBs in detectable amounts, but should contain potential interferents in the concentrations expected to be found in the samples to be analyzed.

4.3.2 When a reference matrix that simulates the sample matrix under test is not available, reagent water (Section 7.6.1) can be used to simulate water samples; playground sand (Section 7.6.2) or white quartz sand (Section 7.3.2) can be used to simulate soils; filter paper (Section 7.6.3) can be used to simulate papers and similar materials; and corn oil (Section 7.6.4) can be used to simulate tissues.

4.4 Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the CBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxydiphenyl ethers, benzylphenyl ethers, brominated diphenyl ethers, polynuclear aromatics, polychlorinated naphthalenes, and pesticides. Because very low levels of CBs are measured by this Method, elimination of interferences is essential. The cleanup steps given in Section 13 can be used to reduce or eliminate these interferences and thereby permit reliable determination of the CBs at the levels shown in Table 2.

4.5 Each piece of reusable glassware should be numbered to associate that glassware with the processing of a particular sample. This will assist the laboratory in tracking possible sources of contamination for individual samples, identifying glassware associated with highly contaminated samples that may require extra cleaning, and determining when glassware should be discarded.

4.6 Contamination of calibration solutions – The MDLs and MLs in Table 2 are the levels that can be achieved in the absence of laboratory backgrounds. Many of the MLs are greater than the equivalent concentrations of the calibration solutions. To prevent contamination, calibration solutions must be prepared in an area free from CB contamination using glassware free from contamination. If these requirements cannot be met or are difficult to meet in the laboratory, the

laboratory should prepare the calibration solutions in a contamination-free facility or have a vendor prepare the calibration standards and guarantee freedom from contamination.

4.7 Cleanup of tissue – The natural lipid content of tissue can interfere in the analysis of tissue samples for the CBs. The lipid contents of different species and portions of tissue can vary widely. Lipids are soluble to varying degrees in various organic solvents and may be present in sufficient quantity to overwhelm the column chromatographic cleanup procedures used for cleanup of sample extracts. Lipids must be removed by the anthropogenic isolation column procedure in Section 13.6, followed by the gel permeation chromatography procedure in Section 13.2. Florisil (Section 13.7) is recommended as an additional cleanup step.

4.8 If the laboratory air is a potential source of CB contamination, samples, reagents, glassware, and other materials should be dried in a glove box or other area free from contamination.

5.0 Safety

5.1 The toxicity or carcinogenicity of each chemical used in this Method has not been precisely determined; however, each compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.1.1 PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. On the basis of the available toxicological and physical properties of the CBs, pure standards should be handled only by highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks.

5.1.2 It is recommended that the laboratory purchase dilute standard solutions of the analytes in this Method. However, if primary solutions are prepared, they must be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator must be worn when high concentrations are handled.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this Method. A reference file of material safety data sheets (MSDSs) should also be made available to all personnel involved in these analyses. It is also suggested that the laboratory perform personal hygiene monitoring of each analyst who uses this Method and that the results of this monitoring be made available to the analyst. Additional information on laboratory safety can be found in References 5-8. The references and bibliography at the end of Reference 7 are particularly comprehensive in dealing with the general subject of laboratory safety.

5.3 The pure CBs and samples suspected to contain these compounds are handled using essentially the same techniques employed in handling radioactive or infectious materials. Well-ventilated, controlled access laboratories are required. Assistance in evaluating the health hazards of particular laboratory conditions may be obtained from certain consulting laboratories and from State Departments of Health or Labor, many of which have an industrial health service. Each laboratory must develop a strict safety program for handling these compounds. The practices in Reference 9 for handling chlorinated dibenzo-*p*-dioxins and dibenzofurans (CDDs/CDFs) are also recommended for handling the CBs.

5.3.1 Facility – When finely divided samples (dusts, soils, dry chemicals) are handled, all operations (including removal of samples from sample containers, weighing, transferring, and mixing) should be performed in a glove box demonstrated to be leak tight or in a fume

hood demonstrated to have adequate air flow. Gross losses to the laboratory ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in the case of an accident.

5.3.2 Protective equipment – Disposable plastic gloves, apron or lab coat, safety glasses or mask, and a glove box or fume hood adequate for radioactive work should be used. During analytical operations that may give rise to aerosols or dusts, personnel should wear respirators equipped with activated carbon filters. Eye protection (preferably full face shields) must be worn while working with exposed samples or pure analytical standards. Latex gloves are commonly used to reduce exposure of the hands. When handling samples suspected or known to contain high concentrations of the CBs, an additional set of gloves can also be worn beneath the latex gloves.

5.3.3 Training – Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

5.3.4 Personal hygiene – Hands and forearms should be washed thoroughly after each manipulation and before breaks (coffee, lunch, and shift).

5.3.5 Confinement – Isolated work areas posted with signs, segregated glassware and tools, and plastic absorbent paper on bench tops will aid in confining contamination.

5.3.6 Effluent vapors – The effluent of the sample splitter from the gas chromatograph (GC) and from roughing pumps on the mass spectrometer (MS) should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols to condense CB vapors.

5.3.7 Waste Handling – Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors and other personnel should be trained in the safe handling of waste.

5.3.8 Decontamination

5.3.8.1 Decontamination of personnel – Use any mild soap with plenty of scrubbing action.

5.3.8.2 Glassware, tools, and surfaces – Chlorothene NU Solvent is a less toxic solvent that should be effective in removing CBs. Satisfactory cleaning may be accomplished by rinsing with Chlorothene, then washing with any detergent and water. If glassware is first rinsed with solvent, the wash water may be disposed of in the sewer. Given the cost of disposal, it is prudent to minimize solvent wastes.

5.3.9 Laundry – Clothing known to be contaminated should be collected in plastic bags. Persons that convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows of the potential problem. The washer should be run through a cycle before being used again for other clothing.

5.3.10 Wipe tests – A useful method of determining cleanliness of work surfaces and tools is to perform a wipe test of the surface suspected of being contaminated.

5.3.10.1 Using a piece of filter paper moistened with Chlorothene or other solvent, wipe an area approximately 10 x 10 cm.

5.3.10.2 Extract and analyze the wipe by GC with an electron capture detector (ECD) or by this Method.

5.3.10.3 Using the area wiped (e.g., 10 x 10 cm = 0.01 m^2), calculate the concentration in µg/m2. A concentration less than 1 µg/m2 indicates acceptable cleanliness; anything higher warrants further cleaning. More than 100 $\mu g/m^2$ constitutes an acute hazard and requires prompt cleaning before further use of the equipment or work space, and indicates that unacceptable work practices have been employed.

5.4 Biosolids samples may contain high concentrations of biohazards, and must be handled with gloves and opened in a hood or biological safety cabinet to prevent exposure. Laboratory staff should know and observe the safety procedures required in a microbiology laboratory that handles pathogenic organisms when handling biosolids samples.

6.0 Apparatus and materials

Note: *Brand names, suppliers, and part numbers are for illustration purposes only and no endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here. Meeting the performance requirements of this Method is the responsibility of the laboratory.*

6.1 Sampling equipment for discrete or composite sampling

6.1.1 Sample bottles and caps

6.1.1.1 Liquid samples (waters, sludges and similar materials containing 5 percent solids or less) – Sample bottle, amber glass, 1.1-L minimum, with screw cap.

6.1.1.2 Solid samples (soils, sediments, sludges, paper pulps, filter cake, compost, and similar materials that contain more than 5 percent solids) – Sample bottle, wide mouth, amber glass, 500-mL minimum.

6.1.1.3 If amber bottles are not available, samples must be protected from light.

6.1.1.4 Bottle caps – Threaded to fit sample bottles. Caps must be lined with fluoropolymer.

6.1.1.5 Cleaning

6.1.1.5.1 Bottles are detergent water washed, then solvent rinsed before use.

6.1.1.5.2 Liners are detergent water washed and rinsed with reagent water (Section 7.6.1).

6.1.2 Compositing equipment – Automatic or manual compositing system incorporating glass containers cleaned per bottle cleaning procedure above. Only glass or fluoropolymer tub-

ing must be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing must be thoroughly rinsed with methanol, followed by repeated rinsing with reagent water to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

6.2 Equipment for glassware cleaning

Note: *If blanks from bottles or other glassware or with fewer cleaning steps than required above show no detectable CB contamination, unnecessary cleaning steps and equipment may be eliminated.*

6.2.1 Laboratory sink with overhead fume hood

6.2.2 Kiln – Capable of reaching 450 ºC within 2 hours and maintaining 450 - 500 ºC within ±10 ºC, with temperature controller and safety switch (Cress Manufacturing Co., Santa Fe Springs, CA, B31H, X31TS, or equivalent). See the precautions in Section 4.2.3.

6.3 Equipment for sample preparation

6.3.1 Laboratory fume hood of sufficient size to contain the sample preparation equipment listed below.

6.3.2 Glove box (optional)

6.3.3 Tissue homogenizer – VirTis Model 45 Macro homogenizer (American Scientific Products H-3515, or equivalent) with stainless steel Macro-shaft and Turbo-shear blade.

6.3.4 Meat grinder – Hobart, or equivalent, with 3- to 5-mm holes in inner plate.

6.3.5 Equipment for determining percent moisture

6.3.5.1 Oven – Capable of maintaining a temperature of 110 ±5 ºC

6.3.5.2 Desiccator

6.3.6 Balances

6.3.6.1 Analytical – Capable of weighing 0.1 mg

6.3.6.2 Top loading – Capable of weighing 10 mg

6.4 Extraction apparatus

6.4.1 Water samples

6.4.1.1 pH meter, with combination glass electrode

6.4.1.2 pH paper, wide range (Hydrion Papers, or equivalent)

6.4.1.3 Graduated cylinder, 1-L capacity

6.4.1.4 Liquid/liquid extraction – Separatory funnels, 250-, 500-, and 2000-mL, with fluoropolymer stopcocks

6.4.1.5 Solid-phase extraction

6.4.1.5.1 1-L filtration apparatus, including glass funnel, frit support, clamp, adapter, stopper, filtration flask, and vacuum tubing (Figure 4). For wastewater samples, the apparatus should accept 90 or 144 mm disks. For drinking water or other samples containing low solids, smaller disks may be used.

6.4.1.5.2 Vacuum source – Capable of maintaining 25 in. Hg, equipped with shutoff valve and vacuum gauge

6.4.1.5.3 Glass-fiber filter – Whatman GMF 150 (or equivalent), 1 micron pore size, to fit filtration apparatus in Section 6.4.1.5.1

6.4.1.5.4 Solid-phase extraction disk containing octadecyl (C_{18}) bonded silica uniformly enmeshed in an inert matrix – Fisher Scientific 14-378F (or equivalent), to fit filtration apparatus in Section 6.4.1.5.1

6.4.1.6 Continuous liquid/liquid extraction (CLLE) – Fluoropolymer or glass connecting joints and stopcocks without lubrication, 1.5-2 L capacity (Hershberg-Wolf Extractor, Cal-Glass, Costa Mesa, California, 1000 mL or 2000 mL, or equivalent).

6.4.2 Soxhlet/Dean-Stark (SDS) extractor (Figure 5 and Reference 10) for filters and solid/sludge samples

6.4.2.1 Soxhlet – 50-mm ID, 200-mL capacity with 500-mL flask (Cal-Glass LG-6900, or equivalent, except substitute 500-mL round-bottom flask for 300-mL flat-bottom flask)

6.4.2.2 Thimble – 43 × 123 to fit Soxhlet (Cal-Glass LG-6901-122, or equivalent)

6.4.2.3 Moisture trap – Dean Stark or Barret with fluoropolymer stopcock, to fit Soxhlet

6.4.2.4 Heating mantle – Hemispherical, to fit 500-mL round-bottom flask (Cal-Glass LG-8801-112, or equivalent)

6.4.2.5 Variable transformer – Powerstat (or equivalent), 110-volt, 10-amp

6.4.3 Beakers – 400- to 500-mL

6.4.4 Spatulas – Stainless steel

6.5 Filtration apparatus

6.5.1 Pyrex glass wool – Solvent-extracted using a Soxhlet or SDS extractor for 3 hours minimum

6.5.2 Glass funnel – 125- to 250-mL

6.5.3 Glass-fiber filter paper – Whatman GF/D (or equivalent), to fit glass funnel in Section 6.5.2.

6.5.4 Drying column – 15- to 20-mm ID Pyrex chromatographic column equipped with coarse-glass frit or glass-wool plug

6.5.5 Buchner funnel – 15-cm

6.5.6 Glass-fiber filter paper for Buchner funnel above

6.5.7 Filtration flasks – 1.5- to 2.0-L, with side arm

6.5.8 Pressure filtration apparatus – Millipore YT30 142 HW, or equivalent

6.6 Centrifuge apparatus

6.6.1 Centrifuge – Capable of rotating 500-mL centrifuge bottles or 15-mL centrifuge tubes at 5,000 rpm minimum

6.6.2 Centrifuge bottles – 500-mL, with screw-caps, to fit centrifuge

6.6.3 Centrifuge tubes – 12- to 15-mL, with screw-caps, to fit centrifuge

6.7 Cleanup apparatus

6.7.1 Automated gel permeation chromatograph (Analytical Biochemical Labs, Inc, Columbia, MO, Model GPC Autoprep 1002, or equivalent)

6.7.1.1 Column – 600-700 mm long × 25 mm ID glass, packed with 70 g of 200-400 mesh SX-3 Bio-beads (Bio-Rad Laboratories, Richmond, CA, or equivalent)

6.7.1.2 Syringe – 10-mL, with Luer fitting

6.7.1.3 Syringe filter holder – stainless steel, and glass-fiber or fluoropolymer filters (Gelman 4310, or equivalent)

6.7.1.4 UV detectors – 254-nm, preparative or semi-preparative flow cell (Isco, Inc., Type 6; Schmadzu, 5-mm path length; Beckman-Altex 152W, 8-µL micro-prep flow cell, 2-mm path; Pharmacia UV-1, 3-mm flow cell; LDC Milton-Roy UV-3, monitor #1203; or equivalent).

6.7.2 Reverse-phase high-performance liquid chromatograph (Reference 4)

6.7.2.1 Pump – Perkin-Elmer Series 410, or equivalent

6.7.2.2 Injector – Perkin-Elmer ISS-100 Autosampler, or equivalent

6.7.2.3 6-Port switching valve – Valco N60, or equivalent

6.7.2.4 Column – Hypercarb, 100 x 4.6 mm, 5 µm particle size, Keystone Scientific, or equivalent

6.7.2.5 Detector – Altex 110A (or equivalent) operated at 0.02 AUFS at 235 nm

6.7.2.6 Fraction collector – Isco Foxy II, or equivalent

6.7.3 Pipets

6.7.3.1 Disposable, Pasteur, 150-mm long x 5-mm ID (Fisher Scientific 13-678-6A, or equivalent)

6.7.3.2 Disposable, serological, 50-mL (8- to 10- mm ID)

6.7.4 Glass chromatographic columns

6.7.4.1 150-mm long x 8-mm ID, (Kontes K-420155, or equivalent) with coarse-glass frit or glass-wool plug and 250-mL reservoir

6.7.4.2 200-mm long x 15-mm ID, with coarse-glass frit or glass-wool plug and 250-mL reservoir

6.7.4.3 300-mm long x 22-mm ID, with coarse-glass frit, 300-mL reservoir, and glass or fluoropolymer stopcock

6.7.5 Oven – For baking and storage of adsorbents, capable of maintaining a constant temperature (± 5 ºC) in the range of 105-250 ºC

6.8 Concentration apparatus

6.8.1 Rotary evaporator – Buchi/Brinkman-American Scientific No. E5045-10 or equivalent, equipped with a variable temperature water bath

6.8.1.1 Vacuum source for rotary evaporator equipped with shutoff valve at the evaporator and vacuum gauge

6.8.1.2 A recirculating water pump and chiller are recommended, as use of tap water for cooling the evaporator wastes large volumes of water and can lead to inconsistent performance as water temperatures and pressures vary.

6.8.1.3 Round-bottom flask – 100-mL and 500-mL or larger, with ground-glass fitting compatible with the rotary evaporator

6.8.2 Kuderna-Danish (K-D) concentrator

6.8.2.1 Concentrator tube – 10-mL, graduated (Kontes K-570050-1025, or equivalent) with calibration verified. Ground-glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

6.8.2.2 Evaporation flask – 500-mL (Kontes K-570001-0500, or equivalent), attached to concentrator tube with springs (Kontes K-662750-0012 or equivalent)

6.8.2.3 Snyder column – Three-ball macro (Kontes K-503000-0232, or equivalent)

6.8.2.4 Boiling chips

6.8.2.4.1 Glass or silicon carbide – Approximately 10/40 mesh, extracted with methylene chloride and baked at 450 ºC for one hour minimum

6.8.2.4.2 Fluoropolymer (optional) – Extracted with methylene chloride

6.8.2.5 Water bath – Heated, with concentric ring cover, capable of maintaining a temperature within ± 2 ºC, installed in a fume hood

6.8.3 Nitrogen evaporation apparatus – Equipped with water bath controlled in the range of 30 - 60 ºC (N-Evap, Organomation Associates, Inc., South Berlin, MA, or equivalent), installed in a fume hood

6.8.4 Sample vials

6.8.4.1 Amber glass, 2- to 5-mL with fluoropolymer-lined screw-cap

6.8.4.2 Glass, 0.3-mL, conical, with fluoropolymer-lined screw or crimp cap

6.9 Gas chromatograph – Must have splitless or on-column injection port for capillary column, temperature program with isothermal hold, and must meet all of the performance specifications in Section 10.

6.9.1 GC column – Any GC column or column system (2 or more columns) that provides unique resolution and identification of the Toxics for determination of a TEQ_{PCB} using TEFs (Reference 1). Isomers may be unresolved so long as they have the same TEF and response factor and so long as these unresolved isomers are uniquely resolved from all other congeners. For example, the SPB-octyl column (Section 6.9.1.3) achieves unique GC resolution of all Toxics except congeners with congener numbers 156 and 157. This isomeric pair is uniquely resolved from all other congeners and these congeners have the same TEF and response factor.

6.9.1.1 If an SPB-octyl column is used, it must meet the specification in Section 6.9.1 and the following additional specifications:

6.9.1.1.1 The retention time for decachlorobiphenyl (DeCB; PCB 209) must be greater than 55 minutes.

6.9.1.1.2 The column must uniquely resolve congeners 34 from 23 and 187 from 182, and congeners 156 and 157 must co-elute within 2 seconds at the peak maximum. Unique resolution means a valley height less than 40 percent of the shorter of the two peaks that result when the Diluted combined 209 congener solution (Section 7.10.2.2) is analyzed (see Figures 6 and 7).

6.9.1.1.3 The column must be replaced when any of the criteria in Sections 6.9.1 - 6.9.1.1.2 are not met.

6.9.1.2 If a column or column system alternate to the SPB-octyl column is used, specifications similar to those for the SPB-octyl column (Sections 6.9.1 -

6.9.1.1.2) must be developed and be functionally equivalent to those specifications.

6.9.1.3 Suggested column – 30 ± 5-m long x 0.25 ± 0.02-mm ID; 0.25-µm film SPB-octyl (Supelco 2-4218, or equivalent). This column is capable of meeting the requirements in Sections 6.9.1 - 6.9.1.1.2.

Note: *The SPB-octyl column is subject to rapid degradation when exposed to oxygen. The analyst should exclude oxygen from the carrier gas, should eliminate air leaks, and should cool the injector, column, and transfer line before opening the column to the atmosphere. For further information on precluding oxidation, contact the column manufacturer.*

6.9.1.4 Column for resolution of additional congeners – See Appendix A for details on the DB-1 column. The DB-1 column is optional and is capable of uniquely resolving the congener pair with congener numbers 156 and 157. When used in combination with the SPB-octyl column (Section 6.9.1.3), the two-column system is capable of resolving a total of approximately 180 CB congeners.

6.10 Mass spectrometer – 28- to 40-eV electron impact ionization, must be capable of selectively monitoring a minimum of 22 exact m/z's minimum at high resolution (≥10,000) during a period less than 1.5 seconds, and must meet all of the performance specifications in Section 10.

6.11 GC/MS interface – The mass spectrometer (MS) must be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beams.

6.12 Data system – Capable of collecting, recording, storing, and processing MS data

6.12.1 Data acquisition – The signal at each exact m/z must be collected repetitively throughout the monitoring period and stored on a mass storage device.

6.12.2 Response factors and multipoint calibrations – The data system must record and maintain lists of response factors (response ratios for isotope dilution) and multipoint calibrations. Computations of relative standard deviation (RSD) are be used to test calibration linearity. Statistics on initial (Section 9.4) and ongoing (Section 15.5.4) performance should be computed and maintained, either on the instrument data system, or on a separate computer system.

7.0 Reagents and standards

7.1 pH adjustment and back-extraction

7.1.1 Potassium hydroxide – Dissolve 20 g reagent grade KOH in 100 mL reagent water.

7.1.2 Sulfuric acid – Reagent grade (specific gravity 1.84)

7.1.3 Hydrochloric acid – Reagent grade, 6N

7.1.4 Sodium chloride – Reagent grade, prepare at 5% (w/v) solution in reagent water

7.2 Solution drying and evaporation

7.2.1 Solution drying – Sodium sulfate, reagent grade, granular, anhydrous (Baker 3375, or equivalent), rinsed with methylene chloride (20 mL/g), baked at 400 ºC for 1 hour minimum, cooled in a desiccator, and stored in a pre-cleaned glass bottle with screw-cap that prevents moisture from entering. If, after heating, the sodium sulfate develops a noticeable grayish cast (due to the presence of carbon in the crystal matrix), that batch of reagent is not suitable for use and should be discarded. Extraction with methylene chloride (as opposed to simple rinsing) and baking at a lower temperature may produce sodium sulfate that is suitable for use.

7.2.2 Tissue drying – Sodium sulfate, reagent grade, powdered, treated and stored as in Section 7.2.1

7.2.3 Prepurified nitrogen

7.3 Extraction

7.3.1 Solvents – Acetone, toluene, cyclohexane, hexane, methanol, methylene chloride, isooctane, and nonane; distilled in glass, pesticide quality, lot-certified to be free of interferences

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Note: *Some solvents; e.g., isooctane and nonane, may need to be re-distilled to eliminate CB backgrounds.*

7.3.2 White quartz sand, 60/70 mesh – For Soxhlet/Dean-Stark extraction (Aldrich Chemical, Cat. No. 27-437-9, or equivalent). Bake at 450 ºC for 4 hour minimum.

7.4 GPC calibration solution – Prepare a solution containing 2.5 mg/mL corn oil, 0.05 mg/mL bis(2-ethylhexyl) phthalate (BEHP), 0.01 mg/mL methoxychlor, 0.002 mg/mL perylene, and 0.008 mg/mL sulfur, or at concentrations appropriate to the response of the detector.

7.5 Adsorbents for sample cleanup

7.5.1 Silica gel

7.5.1.1 Activated silica gel – 100-200 mesh, Supelco 1-3651 (or equivalent), 100-200 mesh, rinsed with methylene chloride, baked at 180 ºC for a minimum of 1 hour, cooled in a desiccator, and stored in a precleaned glass bottle with screw-cap that prevents moisture from entering.

7.5.1.2 Acid silica gel (30% w/w) – Thoroughly mix 44 g of concentrated sulfuric acid with 100 g of activated silica gel in a clean container. Break up aggregates with a stirring rod until a uniform mixture is obtained. Store in a screw-capped bottle with fluoropolymer-lined cap.

7.5.1.3 Basic silica gel – Thoroughly mix 30 g of 1N sodium hydroxide with 100 g of activated silica gel in a clean container. Break up aggregates with a stirring rod until a uniform mixture is obtained. Store in a screw-capped bottle with fluoropolymer-lined cap.

7.5.1.4 Potassium silicate

7.5.1.4.1 Dissolve 56 g of high purity potassium hydroxide (Aldrich, or equivalent) in 300 mL of methanol in a 750- to 1000-mL flat-bottom flask.

7.5.1.4.2 Add 100 g of activated silica gel (Section 7.5.1.1) and a stirring bar, and stir on an explosion-proof hot plate at 60-70 ºC for 1-2 hours.

7.5.1.4.3 Decant the liquid and rinse the potassium silicate twice with 100-mL portions of methanol, followed by a single rinse with 100 mL of methylene chloride.

7.5.1.4.4 Spread the potassium silicate on solvent-rinsed aluminum foil and dry for 2-4 hours in a hood. Observe the precaution in Section 4.8.

7.5.1.4.5 Activate overnight at 200-250 ºC prior to use.

7.5.2 Carbon

7.5.2.1 Carbopak C – (Supelco 1-0258, or equivalent)

7.5.2.2 Celite 545 – (Supelco 2-0199, or equivalent)

7.5.2.3 Thoroughly mix 18.0 g Carbopak C and 18.0 g Celite 545 to produce a 50% w/w mixture. Activate the mixture at 130 ºC for a minimum of 6 hours. Store in a desiccator.

Note: *The carbon column has been included in this Method to allow separation of co-planar congeners 77, 126, and 169 from other congeners and interferences, should such separation be desired.*

7.5.3 Anthropogenic isolation column – Pack the column in Section 6.7.4.3 from bottom to top with the following:

7.5.3.1 2 g silica gel (Section 7.5.1.1)

7.5.3.2 2 g potassium silicate (Section 7.5.1.4)

7.5.3.3 2 g granular anhydrous sodium sulfate (Section 7.2.1)

7.5.3.4 10 g acid silica gel (Section 7.5.1.2)

7.5.3.5 2 g granular anhydrous sodium sulfate

7.5.4 Florisil column

7.5.4.1 Florisil – PR grade, 60-100 mesh (U.S. Silica Corp, Berkeley Springs, WV, or equivalent). Alternatively, prepacked Florisil columns may be used. Use the following procedure for Florisil activation and column packing.

7.5.4.1.1 Fill a clean 1- to 2-L bottle ½ to 2/3 full with Florisil and place in an oven at 130-150 ºC for a minimum of three days to activate the Florisil.

7.5.4.1.2 Immediately prior to use, dry pack a 300-mm x 22-mm ID glass column (Section 6.7.4.3) bottom to top with 0.5-1.0 cm of warm to hot anhydrous sodium sulfate (Section 7.2.1), 10-10.5 cm of warm to hot activated Florisil (Section 7.5.4.1.1), and 1-2 cm of warm to hot anhydrous sodium sulfate. Allow the column to cool and wet immediately with 100 mL of n-hexane to prevent water from entering.

7.5.4.2 Using the procedure in Section 13.7.3, establish the elution pattern for each carton of Florisil or each lot of Florisil columns received.

7.6 Reference matrices – Matrices in which the CBs and interfering compounds are not detected by this Method

7.6.1 Reagent water – Bottled water purchased locally, or prepared by passage through activated carbon

7.6.2 High-solids reference matrix – Playground sand or similar material. Prepared by extraction with methylene chloride and/or baking at 450 ºC for a minimum of 4 hours.

7.6.3 Paper reference matrix – Glass-fiber filter, Gelman type A, or equivalent. Cut paper to simulate the surface area of the paper sample being tested.

7.6.4 Tissue reference matrix – Corn or other vegetable oil.

7.6.5 Other matrices – This Method may be verified on any reference matrix by performing the tests in Section 9.2. Ideally, the matrix should be free of the CBs, but in no case must the background level of the CBs in the reference matrix exceed the minimum levels in Table 2. If low background levels of the CBs are present in the reference matrix, the spike level of the analytes used in Section 9.2 should be increased to provide a spike-to-background ratio of approximately 5 (Reference 11).

7.7 Standard solutions – Prepare from materials of known purity and composition or purchase as solutions or mixtures with certification to their purity, concentration, and authenticity. If the chemical purity is 98 % or greater, the weight may be used without correction to calculate the concentration of the standard. Observe the safety precautions in Section 5 and the recommendation in Section 5.1.2.

Note: *Native PCB standards are available from several suppliers.* $^{13}C_{12}$*-labeled congeners are available from Cambridge Isotope Laboratories and Wellington Laboratories, and may be available from other suppliers. Listing of these suppliers does not constitute a recommendation or endorsement for use. Part numbers are for reference only.*

7.7.1 For preparation of stock solutions from neat materials, dissolve an appropriate amount of assayed reference material in solvent. For example, weigh 10 to 20 mg of PCB 126 to three significant figures in a 10-mL ground-glass-stoppered volumetric flask and fill to the mark with nonane. After the compound is completely dissolved, transfer the solution to a clean 15-mL vial with fluoropolymer-lined cap.

7.7.2 When not being used, store standard solutions in the dark at room temperature in screw-capped vials with fluoropolymer-lined caps. Place a mark on the vial at the level of the solution so that solvent loss by evaporation can be detected. Replace the solution if solvent loss has occurred.

7.8 Native (unlabeled) stock solutions

Note: *Some of the part numbers for solutions described below contain the identifier "1668A." These part numbers remain valid for Method 1668C.*

7.8.1 Native Toxics/LOC stock solution – Prepare to contain the native Toxics and LOC CBs at the concentrations shown in Table 3, or purchase Accu-Standard M1668A-C-NT-LOC-WD-GCPC, or equivalent. If additional CBs are to be determined by isotope dilution (e.g., 170 and 180), include the additional native compounds in this stock solution.

7.8.2 Native 209 CB congener stock solutions – Solutions containing CB congeners to calibrate the SPB-octyl column.

Note: *If a column other than the SPB-octyl column is used, solutions that will allow separation of all 209 congeners on that column must be prepared.*

7.8.2.1 Native congener mix stock solutions for separation of individual congeners on the SPB-octyl column – Prepare the five solutions with the congeners listed in Table 4 at the concentrations shown in Table 3 or purchase Accu-Standard M-1668A-1, M-1668A-2, M-1668A-3, M-1668-4, and M-1668-5, or equivalent.

7.8.2.2 Combined 209 congener stock solution – Combine equal volumes of the standards in Section 7.8.2.1 to form a stock solution containing all CB congeners. This solution will be at 1/5 the concentration of the 5 individual solutions.

7.8.3 Stock solutions should be checked for signs of degradation prior to preparation of calibration or performance test standards. Reference standards that can be used to determine the accuracy of standard solutions are available from several vendors.

7.9 Labeled compound stock solutions (Table 3)

7.9.1 Labeled Toxics/LOC/window-defining stock solution – Prepare in isooctane or nonane at the concentrations in Table 3 or purchase Cambridge Isotope Laboratories (CIL) EC-4977, or equivalent. If additional CBs are to be determined by isotope dilution (e.g., 170 and 180), include the additional labeled compounds in this stock solution.

7.9.2 Labeled cleanup standard stock solution – Prepare labeled CBs 28, 111, and 178 in iso-octane or nonane at the concentration shown in Table 3 or purchase CIL EC-4978, or equivalent.

7.9.3 Labeled injection internal standard stock solution – Prepare labeled CBs 9, 52, 101, 138, and 194 in nonane or isooctane at the concentrations shown in Table 3, or purchase CIL EC-4979, or equivalent.

7.10 Calibration standards

7.10.1 Calibration standards – Combine and dilute the solutions in Sections 7.8.1 and 7.9 to produce the calibration solutions in Table 5 or purchase CIL EC-4976, or equivalent, for the CS-1 to CS-5 set of calibration solutions. If a 6-point calibration is used, prepare the CS-0.2 solution or purchase CIL EC-4976-0.2, or equivalent. These solutions permit the relative response (labeled to native) and response factor to be measured as a function of concentration. The CS-3 standard (CIL EC-4976-3, or equivalent) is used for calibration verification (VER).

7.10.2 Solutions of congener mixes

7.10.2.1 Diluted individual solutions

7.10.2.1.1 The 5 individual solutions, when analyzed individually, allow resolution of all 209 congeners on the SPB-octyl column, and are used for establishing retention time and other data for each congener. The elution order of the congeners present in each of the 5 solutions (Section 7.8.2.1) is given in Table 4.

7.10.2.1.2 Individually combine an aliquot of each individual mix stock solution (Section 7.8.2.1) with an aliquot of the Labeled Toxics/LOC/window-defining stock solution (Section 7.9.1), the Labeled cleanup standard stock solution (Section 7.9.2), and the Labeled injection internal standard stock solution (7.9.3) to produce concentrations of 100 ng/mL for the labeled compounds and 25, 50, and 75 ng/mL for the MoCB-TrCB, TeCB-HpCB, and OcCB-DeCB congeners, respectively, as shown in Table 3.

7.10.2.2 Diluted combined 209 congener solution

7.10.2.2.1 This solution combines the 5 individual mixes with the labeled compounds to allow single-point calibration of the congeners not included in the multi-point calibration, and establishes an average response factor for the co-eluting isomeric congeners.

7.10.2.2.2 Combine an aliquot of the combined 209 congener solution (Section 7.8.2.2) with an aliquot of the Labeled Toxics/LOC/window-defining stock solution (Section 7.9.1), the Labeled cleanup standard stock solution (Section 7.9.2), and the Labeled injection internal standard stock solution (7.9.3) to produce the same concentrations as in the diluted individual mix solutions (Section 7.10.2.1.2 and Table 3).

7.11 Native Toxics/LOC standard spiking solution – Used for determining initial precision and recovery (IPR; Section 9.2) and ongoing precision and recovery (OPR; Section 15.5). Dilute the Native Toxics/LOC stock solution (Section 7.8.1) with acetone to produce a concentration of the Toxics at 1 ng/mL, as shown in Table 3. When 1 mL of this solution spiked into the IPR (Section 9.2.1) or OPR (Section 15.5) and concentrated to a final volume of 20 µL, the concentration in the final volume will be 50 ng/mL (50 pg/µL). Prepare only the amount necessary for each reference matrix with each sample batch.

7.12 Labeled Toxics/LOC/window-defining standard spiking solution – This solution is spiked into each sample (Section 9.3) and into the IPR (Section 9.2.1), OPR (Section 15.5), and blank (Section 9.5) to measure recovery. Dilute the Labeled Toxics/LOC/window-defining stock solution (Section 7.9.1) with acetone to produce a concentration of the labeled compounds at 2 ng/mL, as shown in Table 3. When 1 mL of this solution is spiked into an IPR, OPR, blank, or sample and concentrated to a final extract volume of 20 µL, the concentration in the final extract volume will be 100 ng/mL (100 pg/µL). Prepare only the amount necessary for each reference matrix with each sample batch.

7.13 Labeled cleanup standard spiking solution – This solution is spiked into each extract prior to cleanup to measure the efficiency of the cleanup process. Dilute the Labeled cleanup standard stock solution (Section 7.9.2) in methylene chloride to produce a concentration of the cleanup standards at 2 ng/mL, as shown in Table 3. When 1 mL of this solution is spiked into a sample extract and concentrated to a final volume of 20 µL, the concentration in the final volume will be 100 ng/mL (100 pg/µL).

7.14 Labeled injection internal standard spiking solution – This solution is added to each concentrated extract prior to injection into the HRGC/HRMS. Dilute the Labeled injection internal standard stock solution (Section 7.9.3) in nonane to produce a concentration of the injection internal standards at 1000 ng/mL, as shown in Table 3. When 2 µL of this solution is spiked into a 20 µL extract, the concentration of each injection internal standard will be nominally 100 ng/mL (100 pg/µL).

Note: *The addition of 2 µL of the Labeled injection internal standard spiking solution to a 20-µL final extract has the effect of diluting the concentration of the components in the extract by 10%. Provided all calibration solutions and all extracts undergo this dilution as a result of adding the Labeled injection internal standard spiking solution, the effect of the 10% solution is compensated, and correction for this dilution should not be made.*

7.15 QC Check Sample – A QC Check Sample should be obtained from a source independent of the calibration standards. Ideally, this check sample would be a certified Standard Reference Material (SRM) containing the CBs in known concentrations in a sample matrix similar to the matrix under test. The National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland has SRMs, and the Institute for National Measurement Standards of the National Research Council of Canada in Ottawa has certified reference materials (CRMs) for CBs in various matrices.

7.16 Stability of solutions – Standard solutions used for quantitative purposes (Sections 7.9 through 7.14) should be assayed periodically (e.g., every 6 months) against SRMs from NIST (if available), or certified reference materials from a source that will attest to the authenticity and concentration, to assure that the composition and concentrations have not changed.

8.0 Sample collection, preservation, storage, and holding times

8.1 Collect samples in amber glass containers following conventional sampling practices (Reference 12). Other sample collection techniques, or sample volumes may be used, if documented.

8.2 Aqueous samples

8.2.1 Samples that flow freely are collected as grab samples or in refrigerated bottles using automatic sampling equipment. Collect one liter (or a larger or smaller volume) of sample sufficient to meet project needs.

8.2.2 If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water. EPA Methods 330.4 and 330.5 may be used to measure residual chlorine (Reference 13).

8.2.3 Maintain aqueous samples in the dark at less than 6 ºC from the time of collection until receipt at the laboratory. If the sample will be frozen, allow room for expansion. Store in the dark at less than 6 ºC.

8.3 Solid, mixed-phase, semi-solid, and oily samples, excluding tissue.

8.3.1 Collect samples as grab samples using wide-mouth jars.

8.3.2 Maintain solid, semi-solid, oily, and mixed-phase samples in the dark at less than 6 ºC from the time of collection until receipt at the laboratory. Store solid, semi-solid, oily, and mixed-phase samples in the dark at less than -10 ºC.

8.4 Fish and other tissue samples

8.4.1 Fish may be cleaned, filleted, or processed in other ways in the field, such that the laboratory may expect to receive whole fish, fish fillets, or other tissues for analysis.

8.4.2 Collect fish, wrap in aluminum foil, and maintain at less than 6 ºC from the time of collection until receipt at the laboratory, to a maximum time of 24 hours. If a longer transport time is necessary, freeze the sample. Ideally, fish should be frozen upon collection and shipped to the laboratory on dry ice.

8.4.3 Freeze tissue samples upon receipt at the laboratory and maintain them in the dark at less than -10 ºC until prepared. Maintain unused sample in the dark at less than -10 ºC.

8.5 Holding times

8.5.1 There are no demonstrated maximum holding times associated with the CBs in aqueous, solid, semi-solid, tissue, or other sample matrices. If stored in the dark at less than 6 ºC, aqueous samples may be stored for up to one year. Similarly, if stored in the dark at less than -10 ºC, solid, semi-solid, multi-phase, and tissue samples may be stored for up to one year.

8.5.2 Store sample extracts in the dark at less than -10 ºC until analyzed. If stored in the dark at less than -10 ºC, sample extracts may be stored for one year.

9.0 Quality assurance/quality control

9.1 Each laboratory that uses this Method is required to operate a formal quality assurance program (Reference 14). The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the Method.

If the Method is to be applied to sample matrix other than water (e.g., soils, filter cake, compost, tissue) the most appropriate alternate reference matrix (Sections 7.6.2 - 7.6.5 and 7.15) is substituted for the reagent water matrix (Section 7.6.1) in all performance tests.

9.1.1 The laboratory must make an initial demonstration of the ability to generate acceptable precision and recovery with this Method. This demonstration is given in Section 9.2.

9.1.2 In recognition of advances that are occurring in analytical technology, and to overcome matrix interferences, the laboratory is permitted certain options to improve separations or lower the costs of measurements. These options include alternate extraction, concentration, and cleanup procedures, and changes in sample volumes, columns and detectors. Alternate determinative techniques, such as substitution of spectroscopic or immunoassay techniques for HRGC/HRMS technology, and changes that degrade Method performance, are not allowed without prior review and approval. If an analytical technique other than the techniques specified in this Method is used, that technique must have a specificity equal to or greater than the specificity of the techniques in this Method for the analytes of interest. (Note: For additional flexibility to make modifications without prior EPA review see 40 CFR Part 136.6.)

9.1.2.1 Each time a modification is made to this Method, the laboratory is required to repeat the procedure in Section 9.2. If MDLs would be affected by the change, the laboratory is required to demonstrate that the MDLs (40 CFR Part 136, Appendix B) are lower than one-third the regulatory compliance level or lower than five times the MDLs in this Method, whichever are greater. If calibration will be affected by the change, the instrument must be recalibrated per Section 10. Once the modification is demonstrated to produce results equivalent or superior to results produced by this Method as written, that modification may be used routinely thereafter, so long as the other requirements in this Method are met (e.g., labeled compound recovery).

9.1.2.2 The laboratory is required to maintain records of modifications made to this Method. These records include the following, at a minimum:

9.1.2.2.1 The names, titles, addresses, and telephone numbers of the analyst(s) that performed the analyses and modification, and of the quality control officer that witnessed and will verify the analyses and modifications.

9.1.2.2.2 A listing of pollutant(s) measured, by name and CAS Registry number.

9.1.2.2.3 A narrative stating reason(s) for the modifications (see Section 1.5).

9.1.2.2.4 Results from all quality control (QC) tests comparing the modified method to this Method, including:

a) Calibration (Section 10).
b) Calibration verification (Section 15.3).
c) Initial precision and recovery (Section 9.2).
d) Labeled compound recovery (Section 9.3).
e) Analysis of blanks (Section 9.5).
f) Accuracy assessment (Section 9.4).

9.1.2.2.5 Data that will allow an independent reviewer to validate each determination by tracing the instrument output (peak height, area, or other signal) to the final result. These data are to include:

a) Sample numbers and other identifiers.
b) Extraction dates.
c) Analysis dates and times.
d) Analysis sequence/run chronology.
e) Sample weight or volume (Section 11).
f) Extract volume prior to each cleanup step (Section 13).
g) Extract volume after each cleanup step (Section 13).
h) Final extract volume prior to injection (Section 14).
i) Injection volume (Section 14.3).
j) Dilution data, differentiating between dilution of a sample or extract (Section 17.5).
k) Instrument and operating conditions.
l) Column (dimensions, liquid phase, solid support, film thickness, etc).
m) Operating conditions (temperatures, temperature program, flow rates).
n) Detector (type, operating conditions, etc).
o) Chromatograms, printer tapes, and other recordings of raw data.
p) Quantitation reports, data system outputs, and other data to link the raw data to the results reported.

9.1.2.3 Alternate HRGC columns and column systems – See Sections 6.9.1. If a column or column system alternate to those specified in this Method is used, that column or column system must meet the requirements in Section 6.9.1 - 6.9.1.1.3.

9.1.3 Analyses of Method blanks are required to demonstrate freedom from contamination (Section 4.3). The procedures and criteria for analysis of a Method blank are described in Sections 9.5 and 15.6.

9.1.4 The laboratory must spike all samples with labeled compounds to monitor Method performance. This test is described in Section 9.3. When results of these spikes indicate atypical Method performance for samples, the samples are diluted to bring Method performance within acceptable limits. Procedures for dilution are given in Section 17.5.

9.1.5 The laboratory must, on an ongoing basis, demonstrate through calibration verification and the analysis of the ongoing precision and recovery standard (OPR) and blanks that the analytical system is in control. These procedures are given in Sections 15.1 through 15.6.

9.1.6 The laboratory should maintain records to define the quality of data generated. Development of accuracy statements is described in Section 9.4.

9.2 Initial precision and recovery (IPR) – To establish the ability to generate acceptable precision and recovery, the laboratory must perform the following operations.

9.2.1 For low solids (aqueous) samples, extract, concentrate, and analyze four 1-L aliquots of reagent water spiked with 1 mL each of the Native Toxics/LOC spiking solution (Section 7.11), the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12),

and the Labeled cleanup standard spiking solution (Section 7.13), according to the procedures in Sections 11 through 18. For an alternative sample matrix, four aliquots of the alternative reference matrix (Section 7.6) are used. All sample processing steps that are to be used for processing samples, including preparation (Section 11), extraction (Section 12), and cleanup (Section 13), must be included in this test.

9.2.2 Using results of the set of four analyses, compute the average percent recovery (X) of the extracts and the relative standard deviation (RSD) of the concentration for each compound, by isotope dilution for CBs with a labeled analog, and by internal standard for CBs without a labeled analog and for the labeled compounds.

9.2.3 For each CB and labeled compound, compare RSD and X with the corresponding limits for initial precision and recovery in Table 6. If RSD and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual RSD exceeds the precision limit or any individual X falls outside the range for recovery, system performance is unacceptable for that compound. Correct the problem and repeat the test (Section 9.2).

9.3 To assess Method performance on the sample matrix, the laboratory must spike all samples with the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12) and all sample extracts with the Labeled cleanup standard spiking solution (Section 7.13).

9.3.1 Analyze each sample according to the procedures in Sections 11 through 18.

9.3.2 Compute the percent recovery of the labeled Toxics/LOC/window-defining congeners and the labeled cleanup congeners using the internal standard method (Section 17.2).

9.3.3 The recovery of each labeled compound must be within the limits in Table 6. If the recovery of any compound falls outside of these limits, Method performance is unacceptable for that compound in that sample. Additional cleanup procedures must then be employed to attempt to bring the recovery within the normal range. If the recovery cannot be brought within the normal range after all cleanup procedures have been employed, water samples are diluted and smaller amounts of soils, sludges, sediments, and other matrices are analyzed per Section 18.

9.4 It is suggested, but not required, that recovery of labeled compounds from samples be assessed and records maintained.

9.4.1 After the analysis of 30 samples of a given matrix type (water, soil, sludge, pulp, etc.) for which the labeled compounds pass the tests in Section 9.3, compute the average percent recovery (R) and the standard deviation of the percent recovery (S_R) for the labeled compounds only. Express the assessment as a percent recovery interval from $R - 2S_R$ to $R + 2S_R$ for each matrix. For example, if R = 90% and S_R = 10% for five analyses of pulp, the recovery interval is expressed as 70 to 110%.

9.4.2 Update the accuracy assessment for each labeled compound in each matrix on a regular basis (e.g., after each five to ten new measurements).

9.5 Method blanks – A reference matrix Method blank is analyzed with each sample batch (Section 4.3) to demonstrate freedom from contamination. The matrix for the Method blank must be similar to the sample matrix for the batch, e.g., a 1-L reagent water blank (Section 7.6.1), high-solids

reference matrix blank (Section 7.6.2), paper matrix blank (Section 7.6.3); tissue blank (Section 7.6.4), or alternative reference matrix blank (Section 7.6.5).

9.5.1 Spike 1.0 mL each of the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12), and the Labeled cleanup standard spiking solution (Section 7.13) into the Method blank, according to the procedures in Sections 11 through 18. Prepare, extract, clean up, and concentrate the Method blank. Analyze the blank immediately after analysis of the OPR (Section 15.5) to demonstrate freedom from contamination.

9.5.2 If any CB (Table 1) is found in the blank at greater than two times the minimum level (Table 2) or one-third the regulatory compliance limit, whichever is greater; or if any potentially interfering compound is found in the blank at the minimum level for each CB given in Table 2 (assuming a response factor of 1 relative to the quantitation reference in Table 2 at that level of chlorination for a potentially interfering compound; i.e., a compound not listed in this Method), analysis of samples must be halted until the sample batch is re-extracted and the extracts re-analyzed, and the blank associated with the sample batch shows no evidence of contamination at these levels. All samples must be associated with an uncontaminated Method blank before the results for those samples may be reported or used for permitting or regulatory compliance purposes.

9.6 QC Check Sample – Analyze the QC Check Sample (Section 7.15) periodically to assure the accuracy of calibration standards and the overall reliability of the analytical process. It is suggested that the QC Check Sample be analyzed at least quarterly.

9.7 The specifications contained in this Method can be met if the apparatus used is calibrated properly and then maintained in a calibrated state. The standards used for calibration (Section 10), calibration verification (Section 15.3), and for initial (Section 9.2) and ongoing (Section 15.5) precision and recovery should be identical, so that the most precise results will be obtained. A GC/MS instrument will provide the most reproducible results if dedicated to the settings and conditions required for determination of CBs by this Method.

9.8 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when the internal standard method is used.

10.0 Calibration

10.1 Establish the operating conditions necessary to meet the retention times (RTs) and relative retention times (RRTs) for the CBs in Table 2.

Note: *RTs, RRTs, and RRT limits may differ slightly from those in Table 2.*

10.1.1 Suggested GC operating conditions:

Injector temperature:	270 ºC
Interface temperature:	290 ºC
Initial temperature:	75 ºC
Initial time:	2 minutes
Temperature program:	75-150 ºC at 15 ºC/minute
	150-290 ºC at 2.5 ºC/minute
Final time:	1 minute

Note: *All portions of the column that connect the GC to the ion source should remain at or above the interface temperature specified above during analysis to preclude condensation of less volatile compounds.*

The GC conditions may be optimized for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, blanks, IPR and OPR standards, and samples.

10.1.2 Retention time calibration for the CB congeners

10.1.2.1 Separately inject each of the diluted individual congener solutions (Section 7.10.2.1.2). Establish the beginning and ending retention times for the scan descriptors in Table 7. Scan descriptors other than those listed in Table 7 may be used provided the MLs in Table 2 are met. Store the retention time (RT) and relative retention time (RRT) for each congener in the data system.

10.1.2.2 The absolute retention time of CB 209 must exceed 55 minutes on the SPB-octyl column; otherwise, the GC temperature program must be adjusted and this test repeated until the minimum retention time criterion is met. If a GC column or column system alternate to the SPB-octyl column is used, a similar minimum retention time specification must be established for the alternate column or column systems so that interferences that may be encountered in environmental samples will be resolved from the analytes of interest. This specification is deemed to be met if the retention time of CB 209 is greater than 55 minutes on such alternate column.

10.1.2.3 Inject the Diluted combined 209 congener solution (Section 7.10.2.2). Adjust the chromatographic conditions and scan descriptors until the RT and RRT for all congeners are approximately within the windows in Table 2 and the column performance specifications in Sections 6.9.1 - 6.9.1.2 are met. If an alternate column is used, adjust the conditions for that column. If column performance is unacceptable, optimize the analysis conditions or replace the column and repeat the performance tests. Confirm that the scan descriptor changes at times when CBs do not elute.

10.1.2.4 After the column performance tests are passed (Section 10.1.2.2 - 10.1.2.3), calculate and store the RT and RRT for the resolved congeners and the RT and RRT for the isomeric congeners that co-elute. The windows in Table 2 were developed based on the GC conditions given in Section 10.1.1.

10.2 Mass spectrometer (MS) resolution

10.2.1 Using perfluorokerosene (PFK) (or other reference substance) and a molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10% valley) at m/z 330.9792 or any other significant PFK fragment in the range of 300 to 350. For each descriptor (Table 7), monitor and record the resolution and exact m/z's of three to five reference peaks covering the mass range of the descriptor. The level of PFK (or other reference substance) metered into the HRMS during analyses should be adjusted so that the amplitude of the most intense selected lock-mass m/z signal (regardless of the descriptor number) does not exceed 10% of the full-scale deflection for a given set of detector

parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

Note: *Different lots and types of PFK can contain varying levels of contamination, and excessive PFK (or other reference substance) may cause noise problems and contamination of the ion source necessitating increased frequency of source cleaning. A minor PFK mass (223.9872) is known to interfere with dichlorobiphenyl secondary quantitation ion (M+2). Careful selection of the grade and purity of PFK and minimization of the amount of PFK bled into the HRMS has been shown to correct this problem.*

10.2.2 The analysis time for CBs may exceed the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, mass-drift correction is mandatory and a lock-mass m/z from PFK or other reference substance is used for drift correction. The lock-mass m/z is dependent on the exact m/z's monitored within each descriptor, as shown in Table 7. The deviation between each monitored exact m/z and the theoretical m/z (Table 7) must be less than 5 ppm.

10.2.3 Obtain a selected ion current profile (SICP) at the two exact m/z's specified in Table 7 and at ≥10,000 resolving power at each LOC for the native congeners and congener groups and for the labeled congeners. Because of the extensive mass range covered in each function, it may not be possible to maintain 10,000 resolution throughout the mass range during the function. Therefore, resolution must be ≥8,000 throughout the mass range and must be ≥10,000 in the center of the mass range for each function.

10.2.4 If the HRMS has the capability to monitor resolution during the analysis, it is acceptable to terminate the analysis when the resolution falls below the minimum (Section 10.2.3) to save re-analysis time.

10.3 Ion abundance ratios, minimum levels, and signal-to-noise ratios. Choose an injection volume of either 1 or 2 µL, consistent with the capability of the HRGC/HRMS instrument. Inject a 1 or 2 µL aliquot of the CS-1 calibration solution (Table 5) using the GC conditions in Section 10.1.1.

10.3.1 Measure the SICP areas for each congener or congener group, and compute the ion abundance ratios at the exact m/z's specified in Table 7. Compare the computed ratio to the theoretical ratio given in Table 8.

10.3.1.1 The exact m/z's to be monitored in each descriptor are shown in Table 7. Each group or descriptor must be monitored in succession as a function of GC retention time to ensure that the CBs of interest are detected. Additional m/z's may be monitored in each descriptor, and the m/z's may be divided among more than the descriptors listed in Table 7, provided that the laboratory is able to monitor the m/z's of all CBs that may elute from the GC in a given LOC window. The laboratory must also monitor exact m/z's for congeners at higher levels of chlorination to determine if fragments will compromise measurement of congeners at lower levels of chlorination.

10.3.1.2 The mass spectrometer must be operated in a mass-drift correction mode, using PFK (or other reference substance) to provide lock m/z's. The lock mass for each group of m/z's is shown in Table 7. Each lock mass must be monitored and must not vary by more than ± 20% throughout its respective retention time

window. Variations of lock mass by more than 20% indicate the presence of co-eluting interferences that raise the source pressure and may significantly reduce the sensitivity of the mass spectrometer. Re-injection of another aliquot of the sample extract may not resolve the problem and additional cleanup of the extract may be required to remove the interference. A lock mass interference or suppression in a retention time region in which CBs and labeled compounds do not elute may be ignored.

10.3.2 All CBs and labeled compounds in the CS-1 standard must be within the QC limits in Table 8 for their respective ion abundance ratios; otherwise, the mass spectrometer must be adjusted and this test repeated until the m/z ratios fall within the limits specified. If the adjustment alters the resolution of the mass spectrometer, resolution must be verified (Section 10.2.3) prior to repeat of the test.

10.3.3 Verify that the HRGC/HRMS instrument achieves a minimum level (ML) for each congener no greater than 2 times the MLs in Table 2. The peaks representing the CBs and labeled compounds in the CS-1 calibration standard must have signal-to-noise ratios (S/N) ≥ 10; otherwise, the mass spectrometer must be adjusted and this test repeated until the minimum levels in Table 2 are met.

Note: *The MDLs and MLs in Table 2 are based on the levels of contamination normally found in laboratories. Lower levels may be readily achievable if segregation and extensive cleaning of glassware are employed. If lower levels are achievable, these lower levels must be established as described in Section 17.6.1.4.1.*

10.4 Calibration by isotope dilution – Isotope dilution is used for calibration of the Toxics/LOC CBs. The reference compound for each native compound its labeled analog, as listed in Table 2. A 5- or 6-point calibration encompassing the concentration range is prepared for each native congener.

10.4.1 For the Toxics/LOC CBs determined by isotope dilution, the relative response (RR) (labeled to native) vs. concentration in the calibration solutions (Table 5) is computed over the calibration range according to the procedures described below. Five calibration points are employed for less-sensitive HRMS instruments (e.g., VG 70); five or six points may be employed for more-sensitive instruments (e.g., Micromass Autospec Ultima).

10.4.2 The response of each Toxics/LOC CB relative to its labeled analog is determined using the area responses of both the primary and secondary exact m/z's specified in Table 7, for each calibration standard, as follows:

$$RR = \frac{(A1_n + A2_n)\, C_l}{(A1_l + A2_l)\, C_n}$$

where:

$A1_n$ and $A2_n$ = The measured areas at the primary and secondary m/z's for the PCB

$A1_l$ and $A2_l$ = The measured areas at the primary and secondary m/z's for the labeled compound

C_l = The concentration of the labeled compound in the calibration standard (Table 4)

C_n = The concentration of the native compound in the calibration standard (Table 4)

10.4.3 To calibrate the analytical system by isotope dilution, inject calibration standards CS-1 through CS-5 (Section 7.10 and Table 5) for a less sensitive instrument or CS-0.2 through CS-5 for a more sensitive instrument. Use a volume identical to the volume chosen in Section 10.3, the procedure in Section 14, and the conditions in Section 10.1.1. Compute and store the relative response (RR) for each Native Toxics/LOC CB at each concentration. Compute the average (mean) RR and the RSD of the 5 (or 6) RRs.

10.4.4 Linearity – If the RR for any Native Toxics/LOC CB is constant (less than 20% RSD), the average RR may be used for that congener; otherwise, the complete calibration curve for that congener must be used over the calibration range.

10.5 Calibration by internal standard – Internal standard calibration is applied to determination of the native CBs for which a labeled compound is not available, determination of the Labeled Toxics/LOC/window-defining congeners and Labeled cleanup congeners for performance tests and intra-laboratory statistics (Sections 9.4 and 15.5.4), and determination of the Labeled injection internal standards except for CB 178. The reference compound for each compound is listed in Table 2. For the native congeners (other than the Native Toxics/LOC CBs), calibration is performed at a single point using the Diluted combined 209 congener solution (Section 7.10.2.2 and Table 5). For the labeled compounds, calibration is performed using data from the 5 (or 6) points in the calibration for the Native Toxics/LOC CBs (Section 10.4).

10.5.1 Response factors – Internal standard calibration requires the determination of response factors (RF) defined by the following equation:

$$RF = \frac{(A1_s + A2_s)\, C_{is}}{(A1_{is} + A2_{is})\, C_s}$$

where:

$A1_s$ and $A2_s$ = The measured areas at the primary and secondary m/z's for the PCB

$A1_{is}$ and $A2_{is}$ = The measured areas at the primary and secondary m/z's for the internal standard

C_{is} = The concentration of the internal standard (Table 5)

C_s = The concentration of the compound in the calibration standard (Table 5)

10.5.2 To single-concentration calibrate the analytical system for native CBs other than the Native Toxics/LOC CBs by internal standard, inject the Diluted combined 209 congener solution (Section 7.10.2.2 and Table 3). Use a volume identical to the volume chosen in Section 10.3, the procedure in Section 14, and the conditions in Section 10.1.1.

10.5.3 Compute and store the response factor (RF) for all native CBs except the Native Toxics/LOC CBs. Use the average (mean) response of the labeled compounds at each level of chlorination (LOC) as the quantitation reference, to a maximum of 5 labeled congeners, as shown in Table 2. For the combinations of isomeric congeners that co-elute, compute a combined RF for the co-eluted group. For example, for congener 122, the areas at the two exact m/z's for 104L, 105L, 114L, 118L, and 123L are summed and the total area is divided by 5 (because there are 5 congeners in the quantitation reference).

Note: *All labeled congeners at each LOC are used as reference to reduce the effect of an interference if a single congener is used as reference. Other quantitation references and procedures may be used provided that the results produced are as accurate as results produced by the quantitation references and procedures described in this Section.*

10.5.4 Compute and store the response factor (RF) for the labeled compounds, except CB 138. For the Labeled Toxics/LOC/window-defining compounds and the Labeled cleanup standards, use the nearest eluted Labeled injection internal standard as the quantitation reference, as given in Table 2. The Labeled injection internal standards are referenced to CB 138, as shown in Table 2.

11.0 Sample preparation

11.1 Sample preparation involves modifying the physical form of the sample so that the CBs can be extracted efficiently. In general, the samples must be in a liquid form or in the form of finely divided solids in order for efficient extraction to take place. Table 9 lists the phases and suggested quantities for extraction of various sample matrices.

For samples known or expected to contain high levels of the CBs, the smallest sample size representative of the entire sample should be used (see Section 18). For all samples, the blank and IPR/OPR aliquots must be processed through the same steps as the sample to check for contamination and losses in the preparation processes.

11.1.1 For samples that contain particles, percent solids and particle size are determined using the procedures in Sections 11.2 and 11.3, respectively.

11.1.2 Aqueous samples – Because CBs may be bound to suspended particles, the preparation of aqueous samples is dependent on the solids content of the sample.

11.1.2.1 Aqueous samples containing one percent solids or less are prepared per Section 11.4 and extracted directly using one of the extraction techniques in Section 12.2.

11.1.2.2 For aqueous samples containing greater than one percent solids, a sample aliquot sufficient to provide 10 g of dry solids is used, as described in Section 11.5.

11.1.3 Solid samples are prepared using the procedure described in Section 11.5 followed by extraction using the SDS procedure in Section 12.3.

11.1.4 Multi-phase samples – The phase(s) containing the CBs is separated from the non-CB phase using pressure filtration and centrifugation, as described in Section 11.6. The CBs will be in the organic phase in a multi-phase sample in which an organic phase exists.

11.1.5 Procedures for grinding, homogenization, and blending of various sample phases are given in Section 11.7.

11.1.6 Tissue samples – Preparation procedures for fish and other tissues are given in Section 11.8.

11.2 Determination of percent suspended solids

Note: *This aliquot is used for determining solids content of the sample, not for determination of CBs.*

11.2.1 Aqueous liquids and multi-phase samples consisting of mainly an aqueous phase

11.2.1.1 Desiccate and weigh a GF/D filter (Section 6.5.3) to three significant figures.

11.2.1.2 Filter 10.0 ± 0.02 mL of well-mixed sample through the filter.

11.2.1.3 Dry the filter a minimum of 12 hours at 110 ±5 ºC and cool in a desiccator.

11.2.1.4 Calculate percent solids as follows:

$$\% \text{ solids} = \frac{\text{weight of sample aliquot after drying (g) - weight of filter (g)}}{10 \text{ g}} \text{ x } 100$$

11.2.2 Non-aqueous liquids, solids, semi-solid samples, and multi-phase samples in which the main phase is not aqueous; but not tissues

11.2.2.1 Weigh 5 to 10 g of sample to three significant figures in a tared beaker.

11.2.2.2 Dry a minimum of 12 hours at 110 ± 5 ºC, and cool in a desiccator.

11.2.2.3 Calculate percent solids as follows:

$$\% \text{ solids} = \frac{\text{weight of sample aliquot after drying (g)}}{\text{weight of sample aliquot before drying (g)}} \text{ x } 100$$

11.3 Estimation of particle size

11.3.1 Spread the dried sample from Section 11.2.2.2 on a piece of filter paper or aluminum foil in a fume hood or glove box.

11.3.2 Estimate the size of the particles in the sample. If the size of the largest particles is greater than 1 mm, the particle size must be reduced to 1 mm or less prior to extraction using the procedures in Section 11.7.

11.4 Preparation of aqueous samples containing one percent suspended solids or less

11.4.1 Aqueous samples containing one percent suspended solids or less are prepared using the procedure below and extracted using the one of the extraction techniques in Section 12.2.

11.4.2 Preparation of sample and QC aliquots

11.4.2.1 Mark the original level of the sample on the sample bottle for reference. Weigh the sample plus bottle to ± 1 g. After extraction (Section 12.2), re-weigh the sample bottle and convert the weight to volume assuming a density of 1.00 g/mL.

11.4.2.2 Spike 1.0 mL of the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12) into the sample bottle. Cap the bottle and mix the sample by careful shaking. Allow the sample to equilibrate for 1 to 2 hours, with occasional shaking.

11.4.2.3 For each sample or sample batch (to a maximum of 20 samples) to be extracted during the same 12-hour shift, place two 1.0-L aliquots of reagent water in clean sample bottles or flasks.

11.4.2.4 Spike 1.0 mL of the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12) into both reagent water aliquots. One of these aliquots will serve as the Method blank.

11.4.2.5 Spike 1.0 mL of the Native Toxics/LOC standard spiking solution (Section 7.11) into the remaining reagent water aliquot. This aliquot will serve as the OPR (Section 15.5).

11.4.2.6 For extraction using SPE, add 5 mL of methanol to the sample and QC aliquots. Cap and shake the sample and QC aliquots to mix thoroughly, and proceed to Section 12.2 for extraction.

11.5 Preparation of samples containing greater than one percent solids

11.5.1 Weigh a well-mixed aliquot of each sample (of the same matrix type) sufficient to provide 10 g of dry solids (based on the solids determination in Section 11.2) into a clean beaker or glass jar.

11.5.2 Spike 1.0 mL of the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12) into the sample.

11.5.3 For each sample or sample batch (to a maximum of 20 samples) to be extracted during the same 12 hour shift, weigh two 10-g aliquots of the appropriate reference matrix (Section 7.6) into clean beakers or glass jars.

11.5.4 Spike 1.0 mL of the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12) into both reference matrix aliquots. Spike 1.0 mL of the Native Toxics/LOC standard spiking solution (Section 7.11) into one reference matrix aliquot. This aliquot will serve as the OPR (Section 15.5). The other aliquot will serve as the Method blank.

11.5.5 Stir or tumble and equilibrate the aliquots for 1 to 2 hours.

11.5.6 Decant excess water. If necessary to remove water, filter the sample through a glass-fiber filter and discard the aqueous liquid.

11.5.7 If particles >1 mm are present in the sample (as determined in Section 11.3.2), spread the sample on clean aluminum foil in a hood. After the sample is dry, grind to reduce the particle size (Section 11.7).

11.5.8 Extract the sample and QC aliquots using the SDS procedure in Section 12.3.

11.6 Multi-phase samples

11.6.1 Using the percent solids determined in Section 11.2.1 or 11.2.2, determine the volume of sample that will provide 10 g of solids, up to 1 L of sample.

11.6.2 Spike 1.0 mL of the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12) into the amount of sample determined in Section 11.6.1, and into the OPR

and blank. Spike 1.0 mL of the Native Toxics/LOC standard spiking solution (Section 7.11) into the OPR. Pressure filter the sample, blank, and OPR through Whatman GF/D glass-fiber filter paper (Section 6.5.3). If necessary to separate the phases and/or settle the solids, centrifuge these aliquots prior to filtration.

11.6.3 Discard any aqueous phase (if present). Remove any non-aqueous liquid present and reserve the maximum amount filtered from the sample (Section 11.6.1) or 10 g, whichever is less, for combination with the solid phase (Section 12.3.5).

11.6.4 If particles >1 mm are present in the sample (as determined in Section 11.3.2) and the sample is capable of being dried, spread the sample and QC aliquots on clean aluminum foil in a hood. Observe the precaution in Section 4.8.

11.6.5 After the aliquots are dry or if the sample cannot be dried, reduce the particle size using the procedures in Section 11.7 and extract the reduced-size particles using the SDS procedure in Section 12.3. If particles >1 mm are not present, extract the particles and filter in the sample and QC aliquots directly using the SDS procedure in Section 12.3.

11.7 Sample grinding, homogenization, or blending – Samples with particle sizes greater than 1 mm (as determined in Section 11.3.2) are subjected to grinding, homogenization, or blending. The method of reducing particle size to less than 1 mm is matrix-dependent. In general, hard particles can be reduced by grinding with a mortar and pestle. Softer particles can be reduced by grinding in a Wiley mill or meat grinder, by homogenization, or in a blender.

11.7.1 Each size-reducing preparation procedure on each matrix must be verified by running the tests in Section 9.2 before the procedure is employed routinely.

11.7.2 The grinding, homogenization, or blending procedures must be carried out in a glove box or fume hood to prevent particles from contaminating the work environment.

11.7.3 Grinding – Certain papers and pulps, slurries, and amorphous solids can be ground in a Wiley mill or heavy duty meat grinder. In some cases, reducing the temperature of the sample to freezing or to dry ice or liquid nitrogen temperatures can aid in the grinding process. Grind the sample aliquots from Sections 11.5.7 or 11.6.5 in a clean grinder. Do not allow the sample temperature to exceed 50 ºC. Grind the blank and reference matrix aliquots using a clean grinder.

11.7.4 Homogenization or blending – Particles that are not ground effectively, or particles greater than 1 mm in size after grinding, can often be reduced in size by high speed homogenization or blending. Homogenize and/or blend the particles or filter from Sections 11.5.7 or 11.6.5 for the sample, blank, and OPR aliquots.

11.7.5 Extract the aliquots using the SDS procedure in Section 12.3.

11.8 Fish and other tissues – Prior to processing tissue samples, the laboratory must determine the exact tissue to be analyzed. Common requests for analysis of fish tissue include whole fish-skin on, whole fish-skin removed, edible fish fillets (filleted in the field or by the laboratory), specific organs, and other portions. Once the appropriate tissue has been determined, the sample must be homogenized.

11.8.1 Homogenization

11.8.1.1 Samples are homogenized while still frozen, where practical. If the laboratory must dissect the whole fish to obtain the appropriate tissue for analysis, the unused tissues may be rapidly refrozen and stored in a clean glass jar for subsequent use.

11.8.1.2 Each analysis requires 10 g of tissue (wet weight). Therefore, the laboratory should homogenize at least 20 g of tissue to allow for re-extraction of a second aliquot of the same homogenized sample, if re-analysis is required. When whole fish analysis is necessary, the entire fish is homogenized.

11.8.1.3 Homogenize the sample in a tissue homogenizer (Section 6.3.3) or grind in a meat grinder (Section 6.3.4). Cut tissue that is too large to feed into the grinder into smaller pieces. To assure homogeneity, grind three times.

11.8.1.4 Transfer approximately 10 g (wet weight) of homogenized tissue to a clean, tared, 400- to 500-mL beaker.

11.8.1.5 Transfer the remaining homogenized tissue to a clean jar with a fluoropolymer-lined lid. Seal the jar and store the tissue at less than -10 ºC. Return any tissue that was not homogenized to its original container and store at less than -10 ºC.

11.8.2 QC aliquots

11.8.2.1 Prepare a Method blank by adding approximately 1-2 g of the oily liquid reference matrix (Section 7.6.4) to a 400- to 500-mL beaker.

11.8.2.2 Prepare a precision and recovery aliquot by adding 1-2 g of the oily liquid reference matrix (Section 7.6.4) to a separate 400- to 500-mL beaker. Record the weight to the nearest 10 mg. If the initial precision and recovery test is to be performed, use four aliquots; if the ongoing precision and recovery test is to be performed, use a single aliquot.

11.8.3 Spiking

11.8.3.1 Spike 1.0 mL of the Labeled Toxics/LOC/window-defining standard spiking solution (Section 7.12) into the sample, blank, and OPR aliquot.

11.8.3.2 Spike 1.0 mL of the Native Toxics/LOC standard spiking solution (Section 7.11) into the OPR aliquot.

11.8.4 Extract the aliquots using the procedures in Section 12.4.

12.0 Extraction and concentration

12.1 Extraction procedures include: solid-phase (Section 12.2.1), separatory funnel (Section 12.2.2), and continuous liquid/liquid (Section 12.2.3) for aqueous liquids; Soxhlet/Dean-Stark (Section 12.3) for solids and filters; and Soxhlet extraction (Section 12.4) for tissues. Acid/base back-extraction (Section 12.5) is used for initial cleanup of extracts.

Macro-concentration procedures include: rotary evaporation (Section 12.6.1), heating mantle (Section 12.6.2), and Kuderna-Danish (K-D) evaporation (Section 12.6.3). Micro-concentration uses nitrogen evaporation (Section 12.7).

12.2 Extraction of aqueous liquids

12.2.1 Solid-phase extraction of samples containing less than one percent solids

12.2.1.1 Disk preparation

12.2.1.1.1 Remove the test tube from the suction flask (Figure 4). Place an SPE disk on the base of the filter holder and wet with methylene chloride. While holding a GMF 150 filter above the SPE disk with tweezers, wet the filter with methylene chloride and lay the filter on the SPE disk, making sure that air is not trapped between the filter and disk. Clamp the filter and SPE disk between the 1-L glass reservoir and the vacuum filtration flask.

12.2.1.1.2 Rinse the sides of the reservoir with approx 15 mL of methylene chloride using a squeeze bottle or pipet. Apply vacuum momentarily until a few drops appear at the drip tip. Release the vacuum and allow the filter/disk to soak for approx one minute. Apply vacuum and draw all of the methylene chloride through the filter/disk. Repeat the wash step with approx 15 mL of acetone and allow the filter/disk to air dry.

12.2.1.2 Sample extraction

12.2.1.2.1 Pre-wet the disk by adding approx 20 mL of methanol to the reservoir. Pull most of the methanol through the filter/disk, retaining a layer of methanol approx 2 mm thick on the filter. Do not allow the filter/disk to go dry from this point until the extraction is completed.

12.2.1.2.2 Add approx 20 mL of reagent water to the reservoir and pull most through, leaving a layer approx 2 mm thick on the filter/disk.

12.2.1.2.3 Allow the sample (Section 11.4.2.6) to stand for 1-2 hours, if necessary, to settle the suspended particles. Decant the clear layer of the sample, the blank (Section 11.4.2.4), or IPR/OPR aliquot (Section 11.4.2.5) into its respective reservoir and turn on the vacuum to begin the extraction. Adjust the vacuum to complete the extraction in no less than 10 minutes. For samples containing a high concentration of particles (suspended solids), the extraction time may be an hour or longer.

12.2.1.2.4 Before all of the sample has been pulled through the filter/disk, add approx 50 mL of reagent water to the sample bottle, swirl to suspend the solids (if present), and pour into the reservoir. Pull through the filter/disk. Use additional reagent water rinses until all solids are removed.

12.2.1.2.5 Before all of the sample and rinses have been pulled through the filter/disk, rinse the sides of the reservoir with small portions of reagent water.

12.2.1.2.6 Partially dry the filter/disk under vacuum for approx 3 minutes.

12.2.1.3 Elution of the filter/disk

12.2.1.3.1 Release the vacuum, remove the entire filter/disk/reservoir assembly from the vacuum flask, and empty the flask. Insert a test tube for eluant collection into the flask. The test tube should have sufficient capacity to contain the total volume of the elution solvent (approx 50 mL) and should fit around the drip tip. The drip tip should protrude into the test tube to preclude loss of sample from spattering when vacuum is applied. Reassemble the filter/disk/reservoir assembly on the vacuum flask.

12.2.1.3.2 Wet the filter/disk with 4-5 mL of acetone. Allow the acetone to spread evenly across the disk and soak for 15-20 seconds. Pull the acetone through the disk, releasing the vacuum when approx 1 mm thickness remains on the filter.

12.2.1.3.3 Rinse the sample bottle with approx 20 mL of methylene chloride and transfer to the reservoir. Pull approx half of the solvent through the filter/disk and release the vacuum. Allow the filter/disk to soak for approx 1 minute. Pull all of the solvent through the disk. Repeat the bottle rinsing and elution step with another 20 mL of methylene chloride. Pull all of the solvent through the disk.

12.2.1.3.4 Release the vacuum, remove the filter/disk/reservoir assembly, and remove the test tube containing the sample solution. Quantitatively transfer the solution to a 250-mL separatory funnel and proceed to Section 12.5 for back-extraction.

12.2.2 Separatory funnel extraction

12.2.2.1 Pour the spiked sample (Section 11.4.2.2) into a 2-L separatory funnel. Rinse the bottle or flask twice with 5 mL of reagent water and add these rinses to the separatory funnel.

12.2.2.2 Add 60 mL methylene chloride to the empty sample bottle. Seal the bottle and shake 60 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel, and extract the sample by shaking the funnel for 2 minutes with periodic venting. Allow the organic layer to separate from the aqueous phase for a minimum of 10 minutes. If an emulsion forms and is more than one-third the volume of the solvent layer, employ mechanical techniques to complete the phase separation (see note below). Drain the methylene chloride extract through a solvent-rinsed glass funnel approximately one-half full of granular anhydrous sodium sulfate (Section 7.2.1) supported on clean glass-fiber paper into a solvent-rinsed concentration device (Section 12.6).

Note: *If an emulsion forms, the laboratory must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, use of phase separation paper, centrifugation, use of an ultrasonic bath with ice, addition of NaCl, or other physical methods. Alternatively, solid-phase (Section 12.2.1), CLLE (Section 12.2.3), or other extraction techniques may be used to prevent emulsion formation. Any alternative technique is acceptable so long as the requirements in Section 9.2 are met.*

12.2.2.3 Extract the water sample two more times with 60-mL portions of methylene chloride. Drain each portion through the sodium sulfate into the concentrator. After the third extraction, rinse the separatory funnel with at least 20 mL of methylene chloride, and drain this rinse through the sodium sulfate into the concentrator. Repeat this rinse at least twice.

12.2.2.4 Concentrate the extract using one of the macro-concentration procedures in Section 12.6 and proceed to back extraction in Section 12.5. Set aside the concentration device for use after back extraction or other cleanup.

12.2.3 Continuous liquid/liquid extraction

12.2.3.1 Place 100-150 mL methylene chloride in each continuous extractor and 200-300 mL in each distilling flask.

12.2.3.2 Pour the sample(s), blank, and QC aliquots into the extractors. Rinse the sample containers with 50-100 mL methylene chloride and add to the respective extractors. Include all solids in the extraction process.

12.2.3.3 Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, 1-2 drops of methylene chloride per second will fall from the condenser tip into the water. Extract for 16-24 hours.

12.2.3.4 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7 to 10 cm of granular anhydrous sodium sulfate into a 500-mL K-D evaporator flask equipped with a 10-mL concentrator tube. Rinse the distilling flask with 30-50 mL of methylene chloride and pour through the drying column. Concentrate and exchange to hexane per Section 12.6 and back extract per Section 12.5. Set aside the concentration device for use after back extraction or other cleanup.

12.3 SDS extraction of samples containing particles

Note: *SDS extraction with toluene may cause loss of some of the mono- through tri- CB congeners. If this loss is excessive, use Soxhlet extraction with methylene chloride (Section 12.4) and increase the amount of powdered, anhydrous sodium sulfate as necessary to provide a free-flowing mixture.*

12.3.1 Charge a clean extraction thimble (Section 6.4.2.2) with 5.0 g of 100/200 mesh silica (Section 7.5.1.1) topped with 100 g of quartz sand (Section 7.3.2).

Note: *Do not disturb the silica layer throughout the extraction process.*

12.3.2 Place the thimble in a clean extractor. Place 30 to 40 mL of toluene in the receiver and 200 to 250 mL of toluene in the flask.

12.3.3 Pre-extract the glassware by heating the flask until the toluene is boiling. When properly adjusted, 1 to 2 drops of toluene will fall per second from the condenser tip into the receiver. Extract the apparatus for a minimum of 3 hours.

12.3.4 After pre-extraction, cool and disassemble the apparatus. Rinse the thimble with toluene and allow to air dry.

12.3.5 Load the wet sample and/or filter from Sections 11.5.8, 11.6.5, or 11.7.5 and any non-aqueous liquid from Section 11.6.3 into the thimble and manually mix into the sand layer with a clean metal spatula, carefully breaking up any large lumps of sample.

12.3.6 Reassemble the pre-extracted SDS apparatus, and add a fresh charge of toluene to the receiver and reflux flask. Apply power to the heating mantle to begin re-fluxing. Adjust the reflux rate to match the rate of percolation through the sand and silica beds until water removal lessens the restriction to toluene flow. Frequently check the apparatus for foaming during the first 2 hours of extraction. If foaming occurs, reduce the reflux rate until foaming subsides.

12.3.7 Drain the water from the receiver at 1-2 hours and 8-9 hours, or sooner if the receiver fills with water. Reflux the sample for a total of 16-24 hours. Cool and disassemble the apparatus. Record the total volume of water collected.

12.3.8 Remove the distilling flask. Drain the water from the Dean-Stark receiver and add any toluene in the receiver to the extract in the flask.

12.3.9 Concentrate the extracts from particles to approximately 10 mL using the rotary evaporator (Section 12.6.1) or heating mantle (Section 12.6.2), transfer to a 250-mL separatory funnel, and proceed with back-extraction (Section 12.5). Set aside the concentration device for use after back-extraction or other cleanup.

12.4 Soxhlet extraction of tissue (References 3 and 15)

Note: *This procedure includes determination of the lipid content of the sample (Sections 12.4.8 - 12.4.9), using the same sample extract that is analyzed by GC/MS. Alternatively, a separate sample aliquot may be used for the lipid determination. If a separate aliquot is used, use nitrogen to evaporate the main portion of the sample extract only to the extent necessary to effect the solvent exchange to n-hexane, so that loss of low molecular weight CBs is avoided, i.e., it is not necessary to dry the main portion of the sample to constant weight (Section 12.4.8).*

12.4.1 Add 30 to 40 g of powdered anhydrous sodium sulfate (Section 7.2.2) to each of the beakers (Section 11.8.4) and mix thoroughly. Cover the beakers with aluminum foil and dry until the mixture becomes a free-flowing powder (30 minutes minimum). Remix prior to extraction to prevent clumping.

12.4.2 Assemble and pre-extract the Soxhlet apparatus per Sections 12.3.1-12.3.4, except use methylene chloride for the pre-extraction and rinsing and omit the quartz sand.

12.4.3 Reassemble the pre-extracted Soxhlet apparatus and add a fresh charge of methylene chloride to the reflux flask.

12.4.4 Transfer the sample/sodium sulfate mixture (Section 12.4.1) to the Soxhlet thimble, and install the thimble in the Soxhlet apparatus.

12.4.5 Rinse the beaker with several portions of solvent and add to the thimble. Fill the thimble/receiver with solvent. Extract for 18-24 hours.

12.4.6 After extraction, cool and disassemble the apparatus.

12.4.7 Quantitatively transfer the extract to a macro-concentration device (Section 12.6), and concentrate to near dryness. Set aside the concentration apparatus for re-use.

12.4.8 Complete the removal of the solvent using the nitrogen blow evaporation procedure (Section 12.7) and a water bath temperature of 60 ºC. Weigh the receiver, record the weight, and return the receiver to the blowdown apparatus, concentrating the residue until a constant weight is obtained.

12.4.9 Percent lipid determination

12.4.9.1 Redissolve the residue in the receiver in hexane and spike 1.0 mL of the Labeled cleanup standard spiking solution (Section 7.13) into the solution.

12.4.9.2 Transfer the residue/hexane to the anthropogenic isolation column (Section 13.6), retaining the boiling chips in the concentration apparatus. Use several rinses to assure that all material is transferred. If necessary, sonicate or heat the receiver slightly to assure that all material is re-dissolved. Allow the receiver to dry. Weigh the receiver and boiling chips.

12.4.9.3 Calculate the lipid content to the nearest three significant figures as follows:

$$\% \text{ lipid} = \frac{\text{weight of residue (g)}}{\text{weight of tissue (g)}} \text{ x } 100$$

12.4.9.4 The laboratory should determine the lipid content of the blank, IPR, and OPR to assure that the extraction system is working effectively.

12.5 Back-extraction with base and acid

12.5.1 Back-extraction may not be necessary for some samples. For some samples, the presence of color in the extract may indicate that back-extraction is necessary. If back-extraction is not necessary, spike 1.0 mL of the Labeled cleanup standard spiking solution (Section 7.13) into the extract and concentrate the extract for cleanup or analysis (Sections 12.6 and 12.7). If back-extraction is necessary, spike 1.0 mL of the Labeled cleanup standard spiking solution (Section 7.13) into the separatory funnels containing the sample and QC extracts from Section 12.2.3.4 or 12.3.9.

12.5.2 Partition the extract against 50 mL of potassium hydroxide solution (Section 7.1.1). Shake for 2 minutes with periodic venting into a hood. Remove and discard the aqueous layer. Repeat the base washing until no color is visible in the aqueous layer, to a maximum of four washings. Minimize contact time between the extract and the base to prevent degradation of the CBs. Stronger potassium hydroxide solutions may be employed for back-extraction, provided that the laboratory meets the specifications for labeled compound recovery and demonstrates acceptable performance using the procedure in Section 9.2.

12.5.3 Partition the extract against 50 mL of sodium chloride solution (Section 7.1.4) in the same way as with base. Discard the aqueous layer.

12.5.4 Partition the extract against 50 mL of sulfuric acid (Section 7.1.2) in the same way as with base. Repeat the acid washing until no color is visible in the aqueous layer, to a maximum of four washings.

12.5.5 Repeat the partitioning against sodium chloride solution and discard the aqueous layer.

12.5.6 Pour each extract through a drying column containing 7 to 10 cm of granular anhydrous sodium sulfate (Section 7.2.1) into a macro-concentration device (Section 12.6). If a concentration device was set aside from extraction, that concentration device may be re-used. Rinse the separatory funnel with 30 to 50 mL of solvent, and pour through the drying column. Re-concentrate the sample and QC aliquots per Sections 12.6-12.7, and clean up the samples and QC aliquots per Section 13.

12.6 Macro-concentration – Extracts in toluene are concentrated using a rotary evaporator or a heating mantle; extracts in methylene chloride or hexane are concentrated using a rotary evaporator, heating mantle, or Kuderna-Danish apparatus.

Note: *In the concentration procedures below, the extract must not be allowed to concentrate to dryness because the mono- through tri-chlorobiphenyls may be totally or partially lost.*

12.6.1 Rotary evaporation – Concentrate the extracts in separate round-bottom flasks.

12.6.1.1 Assemble the rotary evaporator according to manufacturer's instructions, and warm the water bath to 45 ºC. On a daily basis, pre-clean the rotary evaporator by concentrating 100 mL of clean extraction solvent through the system. Archive both the concentrated solvent and the solvent in the catch flask for a contamination check if necessary. Between samples, three 2- to 3- mL aliquots of solvent should be rinsed down the feed tube into a waste beaker.

12.6.1.2 Attach the round-bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system, and begin rotating the sample flask.

12.6.1.3 Lower the flask into the water bath, and adjust the speed of rotation and the temperature as required to complete concentration in 15 to 20 minutes. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

Note: *If the rate of concentration is too fast, analyte loss may occur.*

12.6.1.4 When the liquid in the concentration flask has reached an apparent volume of approximately 2 mL, remove the flask from the water bath and stop the rotation. Slowly and carefully admit air into the system. Be sure not to open the valve so quickly that the sample is blown out of the flask. Rinse the feed tube with approximately 2 mL of solvent.

12.6.1.5 Proceed to Section 12.6.4 for preparation for back-extraction or micro-concentration and solvent exchange.

12.6.2 Heating mantle – Concentrate the extracts in separate round-bottom flasks.

12.6.2.1 Add one or two clean boiling chips to the round-bottom flask, and attach a three-ball macro Snyder column. Prewet the column by adding approximately 1 mL of solvent through the top. Place the round-bottom flask in a heating mantle, and apply heat as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood.

12.6.2.2 When the liquid has reached an apparent volume of approximately 10 mL, remove the round-bottom flask from the heating mantle and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the glass joint into the receiver with small portions of solvent.

12.6.2.3 Proceed to Section 12.6.4 for preparation for back-extraction or micro-concentration and solvent exchange.

12.6.3 Kuderna-Danish (K-D) – Concentrate the extracts in separate 500-mL K-D flasks equipped with 10-mL concentrator tubes. The K-D technique is used for solvents such as methylene chloride and hexane. Toluene is difficult to concentrate using the K-D technique unless a water bath fed by a steam generator is used.

12.6.3.1 Add 1 to 2 clean boiling chips to the receiver. Attach a three-ball macro Snyder column. Prewet the column by adding approximately 1 mL of solvent through the top. Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam.

12.6.3.2 Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood.

12.6.3.3 When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of solvent. A 5-mL syringe is recommended for this operation.

12.6.3.4 Remove the three-ball Snyder column, add a fresh boiling chip, and attach a two ball micro Snyder column to the concentrator tube. Prewet the column by adding approximately 0.5 mL of solvent through the top. Place the apparatus in the hot water bath.

12.6.3.5 Adjust the vertical position and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood.

12.6.3.6 When the liquid reaches an apparent volume of 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes.

12.6.3.7 Proceed to 12.6.4 for preparation for back-extraction or micro-concentration and solvent exchange.

12.6.4 Preparation for back-extraction or micro-concentration and solvent exchange

12.6.4.1 For back-extraction (Section 12.5), transfer the extract to a 250-mL separatory funnel. Rinse the concentration vessel with small portions of hexane, adjust the hexane volume in the separatory funnel to 10 to 20 mL, and proceed to back-extraction (Section 12.5).

12.6.4.2 For determination of the weight of residue in the extract, or for clean-up procedures other than back-extraction, transfer the extract to a blowdown vial using 2-3 rinses of solvent. Proceed with micro-concentration and solvent exchange (Section 12.7).

12.7 Micro-concentration and solvent exchange

12.7.1 Extracts to be subjected to GPC cleanup are exchanged into methylene chloride. Extracts to be cleaned up using silica gel, carbon, Florisil, and/or HPLC are exchanged into hexane.

12.7.2 Transfer the vial containing the sample extract to a nitrogen evaporation device. Adjust the flow of nitrogen so that the surface of the solvent is just visibly disturbed.

Note: *A large vortex in the solvent may cause analyte loss.*

12.7.3 Lower the vial into a 45 ºC water bath and continue concentrating.

12.7.3.1 If the extract or an aliquot of the extract is to be concentrated to dryness for weight determination (Sections 12.4.8 and 13.6.4), blow dry until a constant weight is obtained.

12.7.3.2 If the extract is to be concentrated for injection into the GC/MS or the solvent is to be exchanged for extract cleanup, proceed as follows:

12.7.4 When the volume of the liquid is approximately 100 µL, add 2 to 3 mL of the desired solvent (methylene chloride for GPC and HPLC, or hexane for the other cleanups) and continue concentration to approximately 100 µL. Repeat the addition of solvent and concentrate once more.

12.7.5 If the extract is to be cleaned up by GPC, adjust the volume of the extract to 5.0 mL with methylene chloride. If the extract is to be cleaned up by HPLC, concentrate the extract to 1.0 mL. Proceed with GPC or HPLC cleanup (Section 13.2 or 13.5, respectively).

12.7.6 If the extract is to be cleaned up by column chromatography (silica gel, Carbopak/Celite, or Florisil), bring the final volume to 1.0 mL with hexane. Proceed with column cleanup (Sections 13.3, 13.4, or 13.7).

12.7.7 If the extract is to be concentrated for injection into the GC/MS (Section 14), quantitatively transfer the extract to a 0.3-mL conical vial for final concentration, rinsing the larger vial with hexane and adding the rinse to the conical vial. Reduce the volume to approximately 100 µL. Add 20 µL of nonane to the vial, and evaporate the solvent to the level of the nonane. Seal the vial and label with the sample number. Store in the dark at room temperature until ready for GC/MS analysis. If GC/MS analysis will not be performed on the same day, store the vial at less than -10 ºC.

13.0 Extract cleanup

13.1 Cleanup may not be necessary for relatively clean samples (e.g., treated effluents, groundwater, drinking water). If particular circumstances require the use of a cleanup procedure, the laboratory may use any or all of the procedures below or any other appropriate procedure. Before using a cleanup procedure, the laboratory must demonstrate that the requirements of Section 9.2 can be met using the cleanup procedure.

13.1.1 Gel permeation chromatography (Section 13.2) removes high molecular weight interferences that cause GC column performance to degrade. It should be used for all soil and sediment extracts. It may be used for water extracts that are expected to contain high molecular weight organic compounds (e.g., polymeric materials, humic acids). It should also be used for tissue extracts after initial cleanup on the anthropogenic isolation column (Section 13.6).

13.1.2 Acid, neutral, and basic silica gel (Section 13.3) and Florisil (Section 13.7) are used to remove non-polar and polar interferences.

13.1.3 Carbopak/Celite (Section 13.4) can be used to separate CBs 77, 126, and 169 from the mono- and di- ortho-substituted CBs, if desired.

13.1.4 HPLC (Section 13.5) is used to provide specificity for certain congeners and congener groups.

13.1.5 The anthropogenic isolation column (Section 13.6) is used for removal of lipids from tissue samples.

13.2 Gel permeation chromatography (GPC)

13.2.1 Column packing

13.2.1.1 Place 70 to 75 g of SX-3 Bio-beads (Section 6.7.1.1) in a 400- to 500-mL beaker.

13.2.1.2 Cover the beads with methylene chloride and allow to swell overnight (a minimum of 12 hours).

13.2.1.3 Transfer the swelled beads to the column (Section 6.7.1.1) and pump solvent through the column, from bottom to top, at 4.5 to 5.5 mL/minute prior to connecting the column to the detector.

13.2.1.4 After purging the column with solvent for 1 to 2 hours, adjust the column head pressure to 7 to 10 psig and purge for 4 to 5 hours to remove air. Maintain a head pressure of 7 to 10 psig. Connect the column to the detector (Section 6.7.1.4).

13.2.2 Column calibration

13.2.2.1 Load 5 mL of the GPC calibration solution (Section 7.4) into the sample loop.

13.2.2.2 Inject the GPC calibration solution and record the signal from the detector. The elution pattern will be corn oil, BEHP, methoxychlor, perylene, and sulfur.

13.2.2.3 Set the "dump time" to allow >85% removal of BEHP and >85% collection of methoxychlor.

13.2.2.4 Set the "collect time" to the time of the sulfur peak maximum.

13.2.2.5 Verify calibration with the GPC calibration solution after every 20 extracts. Calibration is verified if the recovery of the methoxychlor is greater than 85%. If calibration is not verified, the system must be recalibrated using the GPC calibration solution, and the previous sample batch must be re-extracted and cleaned up using the calibrated GPC system.

13.2.3 Extract cleanup – GPC requires that the column not be overloaded. The column specified in this Method is designed to handle a maximum of 0.5 g of material from an aqueous, soil, or mixed-phase sample in a 5-mL extract, and has been shown to handle 1.5 g of lipid from a tissue sample in a 5-mL extract. If the extract is known or expected to contain more than these amounts, the extract is split into aliquots for GPC, and the aliquots are combined after elution from the column. The residue content of the extract may be obtained gravimetrically by evaporating the solvent from a 50-µL aliquot.

13.2.3.1 Filter the extract or load through the filter holder (Section 6.7.1.3) to remove particles. Load the 5.0-mL extract onto the column.

13.2.3.2 Elute the extract using the calibration data determined in Section 13.2.2. Collect the eluate in a clean 400- to 500-mL beaker. Allow the system to rinse for additional 10 minutes before injecting the next sample.

13.2.3.3 Rinse the sample loading tube thoroughly with methylene chloride between extracts to prepare for the next sample.

13.2.3.4 If an extract is encountered that could overload the GPC column to the extent that carry-over could occur, a 5.0-mL methylene chloride blank must be run through the system to check for carry-over.

13.2.3.5 Concentrate the eluate per Sections 12.6 and 12.7 for further cleanup or injection into the GC/MS.

13.3 Silica gel cleanup

13.3.1 Place a glass-wool plug in a 15-mm ID chromatography column (Section 6.7.4.2). Pack the column bottom to top with: 1 g silica gel (Section 7.5.1.1), 4 g basic silica gel (Section 7.5.1.3), 1 g silica gel, 8 g acid silica gel (Section 7.5.1.2), 2 g silica gel, and 4 g granular anhydrous sodium sulfate (Section 7.2.1). Tap the column to settle the adsorbents.

13.3.2 Pre-elute the column with 50 to 100 mL of hexane. Close the stopcock when the hexane is within 1 mm of the sodium sulfate. Discard the eluate. Check the column for channeling. If channeling is present, discard the column and prepare another.

13.3.3 Apply the concentrated extract to the column. Open the stopcock until the extract is within 1 mm of the sodium sulfate.

13.3.4 Rinse the receiver twice with 1-mL portions of hexane, and apply separately to the column. Elute the CBs with 25 mL of hexane and collect the eluate.

13.3.5 Concentrate the eluate per Section 12.6 and 12.7 for further cleanup or injection into the HPLC or GC/MS.

13.3.6 For extracts of samples known to contain large quantities of other organic compounds, it may be advisable to increase the capacity of the silica gel column. This may be accomplished by increasing the strengths of the acid and basic silica gels. The acid silica gel (Section 7.5.1.2) may be increased in strength to as much as 40% w/w (6.7 g sulfuric acid added to 10 g silica gel). The basic silica gel (Section 7.5.1.3) may be increased in strength to as much as 33% w/w (50 mL 1N NaOH added to 100 g silica gel), or the potassium silicate (Section 7.5.1.4) may be used.

Note: *The use of stronger acid silica gel (44% w/w) may lead to charring of organic compounds in some extracts. The charred material may retain some of the analytes and lead to lower recoveries of the CBs. Increasing the strengths of the acid and basic silica gel may also require different volumes of hexane than those specified above to elute the analytes from the column. The performance of the Method after such modifications must be verified by the procedure in Section 9.2.*

13.4 Carbon column (Reference 16)

13.4.1 Cut both ends from a 50-mL disposable serological pipet (Section 6.7.3.2) to produce a 20-cm column. Fire-polish both ends and flare both ends if desired. Insert a glass-wool plug at one end, and pack the column with 3.6 g of Carbopak/Celite (Section 7.5.2.3) to form an adsorbent bed 20 cm long. Insert a glass-wool plug on top of the bed to hold the adsorbent in place.

13.4.2 Pre-elute the column with 20 mL each in succession of toluene, methylene chloride, and hexane.

13.4.3 When the solvent is within 1 mm of the column packing, apply the n-hexane sample extract to the column. Rinse the sample container twice with 1-mL portions of hexane and apply separately to the column. Apply 2 mL of hexane to complete the transfer.

13.4.4 Elute the column with 25 mL of n-hexane and collect the eluate. This fraction will contain the mono- and di-ortho CBs. If carbon particles are present in the eluate, filter through glass-fiber filter paper.

13.4.5 Elute the column with 15 mL of methanol and discard the eluate. The fraction discarded will contain residual lipids and other potential interferents, if present.

13.4.6 Elute the column with 15 mL of toluene and collect the eluate. This fraction will contain CBs 77, 126, and 169. If carbon particles are present in the eluate, filter through glass-fiber filter paper.

13.4.7 Concentrate the fractions per Section 12.6 and 12.7 for further cleanup or injection into the HPLC or GC/MS.

13.5 HPLC (References 4 and 17)

13.5.1 Column calibration

13.5.1.1 Prepare a calibration standard containing the Toxics and other congeners of interest at the concentrations of the stock solution in Table 3, or at a concentration appropriate to the response of the detector.

13.5.1.2 Inject the calibration standard into the HPLC and record the signal from the detector. Collect the eluant for reuse. Elution will be in the order of the di-ortho, mono-ortho, and non-ortho congeners.

13.5.1.3 Establish the collection time for the congeners of interest. Following calibration, flush the injection system with solvent to ensure that residual CBs are removed from the system.

13.5.1.4 Verify the calibration with the calibration solution after every 20 extracts. Calibration is verified if the recovery of the CBs is 75 to 125% compared to the calibration (Section 13.5.1.1). If calibration is not verified, the system must be recalibrated using the calibration solution, and the previous 20 samples must be re-extracted and cleaned up using the calibrated system.

13.5.2 Extract cleanup – HPLC requires that the column not be overloaded. The column specified in this Method is designed to handle a maximum of 5-50 µg of a given CB, depending on the congener (Reference 17). If the amount of material in the extract will overload the column, split the extract into fractions and combine the fractions after elution from the column.

13.5.2.1 Rinse the sides of the vial containing the sample and adjust to the volume required for the sample loop for injection.

13.5.2.2 Inject the sample extract into the HPLC.

13.5.2.3 Elute the extract using the calibration data determined in Section 13.5.1. Collect the fraction(s) in clean 20-mL concentrator tubes.

13.5.2.4 If an extract containing greater than 500 µg of total CBs is encountered, a blank must be run through the system to check for carry-over.

13.5.2.5 Concentrate the eluate per Section 12.7 for injection into the GC/MS.

13.6 Anthropogenic isolation column (Reference 3) – Used for removal of lipids from tissue extracts

13.6.1 Prepare the column as given in Section 7.5.3.

13.6.2 Pre-elute the column with 100 mL of hexane. Drain the hexane layer to the top of the column, but do not expose the sodium sulfate.

13.6.3 Load the sample and rinses (Section 12.4.9.2) onto the column by draining each portion to the top of the bed. Elute the CBs from the column into the apparatus used for concentration (Section 12.4.7) using 200 mL of hexane.

13.6.4 Remove a small portion (e.g., 50 µL) of the extract for determination of residue content. Estimate the percent of the total that this portion represents. Concentrate the small portion to constant weight per Section 12.7.3.1. Calculate the total amount of residue in the extract. If more than 500 mg of material remains, repeat the cleanup using a fresh anthropogenic isolation column.

13.6.5 If necessary, exchange the extract to a solvent suitable for the additional cleanups to be used (Section 13.2-13.5 and 13.7).

13.6.6 Clean up the extract using the procedures in Sections 13.2-13.5 and 13.7. GPC (Section 13.2) and Florisil (Section 13.7) are recommended as minimum additional cleanup steps.

13.6.7 Following cleanup, concentrate the extract to 20 µL as described in Section 12.7 and proceed with the analysis in Section 14.

13.7 Florisil cleanup (Reference 18)

13.7.1 Begin to drain the n-hexane from the column (Section 7.5.4.1.2). Adjust the flow rate of eluant to 4.5-5.0 mL/min.

13.7.2 When the n-hexane is within 1 mm of the sodium sulfate, apply the sample extract (in hexane) to the column. Rinse the sample container twice with 1-mL portions of hexane and apply to the column.

13.7.3 Elute the mono-ortho and di-ortho CBs with approx 165 mL of n-hexane and collect the eluate. Elute the non-ortho co-planar CBs with approx 100 mL of 6% ether:hexane and collect the eluate. The exact volumes of solvents will need to be determined for each batch of Florisil. If the mono/di-ortho CBs are not to be separated from the non-ortho co-planar CBs, elute all CBs with 6% ether:hexane.

13.7.4 Concentrate the eluate(s) per Sections 12.6-12.7 for further cleanup or for injection into the HPLC or GC/MS.

14.0 HRGC/HRMS analysis

14.1 Establish the operating conditions given in Section 10.1.

14.2 Add 2 µL of the labeled injection internal standard spiking solution (Section 7.14) to the 20 µL sample extract immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction. If an extract is to be reanalyzed and evaporation has occurred, do not add more labeled injection internal standard spiking solution. Rather, bring the extract back to its previous volume (e.g., 19 µL) with pure nonane (18 µL if 2 µL injections are used).

14.3 Inject 1.0 or 2.0 µL of the concentrated extract containing the Labeled injection internal standards using on-column or splitless injection. The volume injected must be identical to the volume used for calibration (Section 10.3).

14.3.1 Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes.

14.3.2 Monitor the exact m/z's at each LOC throughout the LOC retention time window. Where warranted, monitor m/z's associated with congeners at higher levels of chlorination to assure that fragments are not interfering with the m/z's for congeners at lower levels of chlorination. Also where warranted, monitor m/z's associated with interferents expected to be present.

14.3.3 Stop data collection after $^{13}C_{12}$-DeCB has eluted. Return the column to the initial temperature for analysis of the next extract or standard.

15.0 System and laboratory performance

15.1 At the beginning of each 12-hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all native CBs and labeled compounds. For these tests, analyze the diluted combined 209 congener solution (Section 7.10.2.2) to verify all performance criteria. Adjustment and/or recalibration (Section 10) must be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, IPRs, and OPRs be analyzed.

15.2 MS resolution – Static resolving power checks must be performed at the beginning and at the end of each shift per Section 10.2.1. If analyses are performed on successive shifts, only the beginning of shift static resolving power check is required. If the requirement in Section 10.2.1 cannot be met, the problem must be corrected before analyses can proceed. If any of the samples in the previous shift may be affected by poor resolution, those samples must be re-analyzed.

15.3 Calibration verification

15.3.1 Inject and analyze the Diluted combined 209 congener solution (Section 7.10.2.2.2) using the procedure in Section 14.

15.3.2 The m/z abundance ratios for each native CB and labeled compound in the VER standard must be within the limits in Table 8; otherwise, the mass spectrometer must be adjusted until the m/z abundance ratios fall within the limits specified when the verification test is be repeated. If the adjustment alters the resolution of the mass spectrometer, resolution must be verified (Section 10.2.1) prior to repeat of the verification test.

15.3.3 The GC peak representing each native CB and labeled compound in the VER standard must be present with a S/N of at least 10; otherwise, the mass spectrometer must be adjusted and the verification test repeated.

15.3.4 Compute the recovery of the Toxics/LOC CBs by isotope dilution (Section 17.1) and the labeled compounds by internal standard (17.2). These recoveries are computed based on the calibration data in Section 10.

15.3.5 For each compound, compare the recovery with the calibration verification limit in Table 6. If all compounds meet the acceptance criteria, calibration has been verified and analysis of standards and sample extracts may proceed. If, however, any compound fails its respective limit, the measurement system is not performing properly. In this event, prepare a fresh calibration standard or correct the problem and repeat the resolution (Section 15.2) and verification (Section 15.3) tests, or recalibrate (Section 10). If recalibration is required, recalibration for the 209 congeners (Section 10.5) must also be performed.

15.4 Retention times and GC resolution

15.4.1 Retention times

15.4.1.1 Absolute – The absolute retention times of the Labeled Toxics/LOC/window defining standard congeners (Section 7.12) in the verification test (Section 15.3) must be within ± 15 seconds of the respective retention times in the calibration or, if an alternate column or column system is employed, within ± 15 seconds of the respective retention times in the calibration for the alternate column or column system (Section 6.9.1.2).

15.4.1.2 Relative – The relative retention times of native CBs and labeled compounds in the verification test (Section 15.3) must be within their respective RRT limits in Table 2 or, if an alternate column or column system is employed, within their respective RRT limits for the alternate column or column system (Section 6.9.1.2).

15.4.1.3 If the absolute or relative retention time of any compound is not within the limits specified, the GC is not performing properly. In this event, adjust the GC and repeat the verification test (Section 15.3) or recalibrate (Section 10), or replace the GC column and either verify calibration or recalibrate.

15.4.2 GC resolution and minimum analysis time

15.4.2.1 As a final step in calibration verification, GC resolution and minimum analysis time are verified and response factors for congeners other than the Toxics and LOC CBs are updated.

15.4.2.2 The resolution and minimum analysis time specifications in Sections 6.9.1.1.2 and 6.9.1.1.1, respectively, must be met for the SPB-octyl column or, if an alternate column or column system is employed, must be met as specified for the alternate column or column system (Section 6.9.1.2). If these specifications are not met, the GC analysis conditions must be adjusted until the specifications are met, or the column must be replaced and the calibration verification tests repeated Sections 15.4.1 through 15.4.2.2), or the system must be recalibrated (Section 10).

15.4.2.3 After the resolution and minimum analysis time specifications are met, update the retention times and relative retention times for all congeners, and response factors for all congeners except the Toxics and LOC CBs. For the Toxics and LOC CBs, the multi-point calibration data must be used (Section 10.4) and verified (Section 15.3.4).

15.5 Ongoing precision and recovery

15.5.1 Analyze the extract of the ongoing precision and recovery (OPR) aliquot (Section 11.4.2.5, 11.5.4, 11.6.2, or 11.8.3.2) prior to analysis of samples from the same batch.

15.5.2 Compute the percent recovery of the Toxics/LOC CBs by isotope dilution (Section 10.4). Compute the percent recovery of each labeled compound by the internal standard method (Section 10.5).

15.5.3 For the Toxics/LOC CBs and labeled compounds, compare the recovery to the OPR limits given in Table 6. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, the extraction/concentration processes are not being performed properly for that compound. In this event, correct the problem, re-prepare, extract, and clean up the sample batch and repeat the ongoing precision and recovery test (Section 15.5).

15.5.4 If desired, add results that pass the specifications in Section 15.5.3 to initial and previous ongoing data for each compound in each matrix. Update QC charts to form a graphic representation of continued laboratory performance. Develop a statement of laboratory accuracy for each congener in each matrix type by calculating the average percent recovery (R) and the standard deviation of percent recovery (S_R). Express the accuracy as a recovery interval from R - $2S_R$ to R + $2S_R$. For example, if R = 95% and S_R = 5%, the accuracy is 85 to 105%.

15.6 Blank – Analyze the Method blank extracted with each sample batch immediately following analysis of the OPR aliquot to demonstrate freedom from contamination and freedom from carryover from the OPR analysis. If CBs will be carried from the OPR into the Method blank, analyze one or more aliquots of solvent between the OPR and the Method blank. The results of the analysis of the blank must meet the specifications in Section 9.5.2 before sample analyses may proceed.

16.0 Qualitative determination

A CB or labeled compound is identified in a standard, blank, or sample when all of the criteria in Sections 16.1 through 16.4 are met.

16.1 The signals for the two exact m/z's in Table 7 must be present and must maximize within the same two scans.

16.2 The signal-to-noise ratio (S/N) for the GC peak at each exact m/z must be greater than or equal to 2.5 for each CB detected in a sample extract, and greater than or equal to 10 for all CBs in the calibration and verification standards (Sections 10.3.3 and 15.3.3).

Note: *An interference between DiCB m/z 223.9974 and PFK m/z 223.9872 may preclude meeting the S/N requirement for the DiCB congeners. If identification is ambiguous, an experienced spectrometrist (Section 1.4) must determine the presence or absence of the congener.*

16.3 The ratio of the integrated areas of the two exact m/z's specified in Table 7 must be within the limit in Table 8, or within ± 15 percent of the ratio in the midpoint (CS-3) calibration or calibration verification (VER), whichever is most recent.

16.4 The relative retention time of the peak for a CB must be within the RRT QC limits specified in Table 2 or within similar limits developed from calibration data (Section 10.1.2). If an alternate column or column system is employed, the RRT for the CB must be within its respective RRT QC limits for the alternate column or column system (Section 6.9.1.2).

Note: *For native CBs determined by internal standard quantitation, a given CB congener may fall within more than one RT window and be mis-identified unless the RRT windows are made very narrow, as in Table 2. Therefore, consistency of the RT and RRT with other congeners and the labeled compounds may be required for rigorous congener identification. Retention time regression analysis may aid in this identification.*

16.5 Because of congener overlap and the potential for interfering substances, it is possible that all of the identification criteria (Sections 16.1-16.4) may not be met. It is also possible that loss of one or more chlorines from a highly chlorinated congener may inflate or produce a false concentration for a less-chlorinated congener that elutes at the same retention time (see Section 18.5). If identification is ambiguous, an experienced spectrometrist (Section 1.4) must determine the presence or absence of the congener.

16.6 If the criteria for identification in Sections 16.1-16.5 are not met, the CB has not been identified and the result for that congener may not be reported or used for permitting or regulatory compliance purposes. If interferences preclude identification, a new aliquot of sample must be extracted, further cleaned up, and analyzed.

17.0 Quantitative determination

17.1 Isotope dilution quantitation

17.1.1 By adding a known amount of the Labeled Toxics/LOC/window-defining compounds to every sample prior to extraction, correction for recovery of the CBs can be made because the native compound and its labeled analog exhibit similar effects upon extraction, concentration, and gas chromatography. Relative responses (RRs) are used in conjunction with the calibration data in Section 10.4 to determine concentrations in the final extract, so long as labeled compound spiking levels are constant.

17.1.2 Compute the concentrations in the extract of the Native Toxics/LOC CBs using the RRs from the calibration data (Section 10.4) and following equation:

$$C_{ex}\ (\text{ng/mL}) = \frac{(A1_n + A2_n)\, C_l}{(A1_l + A2_l)\, RR}$$

where:

C_{ex} = concentration of the PCB in the extract (ng/mL) and the other terms are as defined in Section 10.5.1

17.2 Internal standard quantitation and labeled compound recovery

17.2.1 Compute the concentrations in the extract of the labeled compounds (except labeled CB 178) and of the native compounds other than those in the Native Toxics/LOC standard using the response factors determined from calibration (Section 10.5) or calibration verification (Section 15.4.2.3) and the following equation:

$$C_{ex}\ (ng/mL) = \frac{(A1_s + A2_s)\,C_{is}}{(A1_{is} + A2_{is})\,RF}$$

where:

C_{ex} = concentration of the native or labeled compound in the extract (ng/mL) and the other terms are as defined in Section 10.5.1

17.2.2 Using the concentration in the extract determined above, compute the percent recovery of the Labeled Toxics/LOC/window-defining CBs and the Labeled cleanup standard CBs using the following equation:

$$\text{Recovery}\,(\%) = \frac{\text{Concentration found}\,(ng/mL)}{\text{Concentration spiked}\,(ng/mL)}\ \text{x}\ 100$$

17.3 The concentration of a native CB in the solid phase of the sample is computed using the concentration of the compound in the extract and the weight of the solids (Section 11.2.2.3), as follows:

$$\text{Concentration in solid sample}\ (ng/kg) = \frac{C_{ex}\ V_{ex}}{W_s}$$

where:

C_{ex} = The concentration of the compound in the extract (ng/mL).
V_{ex} = The extract volume in mL.
W_s = The sample weight (dry weight) in kg.

17.4 The concentration of a native CB in the aqueous phase of the sample is computed using the concentration of the compound in the extract and the volume of water extracted (Section 11.4.2.1), as follows:

$$\text{Concentration in aqueous sample}\ (ng/L) = \frac{C_{ex}\ V_{ex}}{V_s}\ \text{x}\ 1000$$

where:

C_{ex} = The concentration of the compound in the extract (pg/mL).
V_{ex} = The extract volume in mL.
V_s = The sample volume in liters.

17.5 If the SICP area at either quantitation m/z for any congener exceeds the calibration range of the system, dilute the sample extract by the factor necessary to bring the concentration within the calibration range, adjust the concentration of the Labeled injection internal standard to 100 pg/µL in the extract, and analyze an aliquot of this diluted extract. If the CBs cannot be measured reliably by isotope dilution, dilute and analyze an aqueous sample or analyze a smaller portion of a soil, tissue, or mixed-phase sample. Adjust the CB congener concentrations, detection limits, and minimum levels to account for the dilution.

17.6 Reporting of results – Results are reported to three significant figures for the CBs and labeled compounds found in all standards, blanks, and samples.

17.6.1 Reporting units and levels

17.6.1.1 Aqueous samples – Report results in pg/L (parts-per-quadrillion).

17.6.1.2 Samples containing greater than 1% solids (soils, sediments, filter cake, compost) – Report results in ng/kg based on the dry weight of the sample. Report the percent solids so that the result may be converted to aqueous units.

17.6.1.3 Tissues – Report results in ng/kg of wet tissue, not on the basis of the lipid content of the tissue. Report the percent lipid content, so that the data user can calculate the concentration on a lipid basis if desired.

17.6.1.4 Reporting level

17.6.1.4.1 Report the result for each congener at or above the minimum level of quantitation (ML; Table 2) for analyses of blanks, standards, and samples. The MLs in Table 2 are the levels that can be achieved in the presence of common laboratory contamination. A laboratory may establish an ML for a CB congener lower than the MLs in Table 2. MLs may be established as low as the lowest calibration point (Table 5) provided that the concentration of the congener in a minimum of 10 blanks for a sample medium (e.g., water, soil, sludge, tissue) is significantly below the ML in Table 2. "Significant" means that the ML for the congener is no less than 2 standard deviations above the mean (average) level in the minimum of 10 blanks (Reference 19). The blanks must be analyzed during the same period that samples are analyzed, ideally over an approximately 1-month period.

17.6.1.4.2 Standards (VER, IPR, OPR) and samples – Report the result for each congener at or above the ML (Table 2) to 3 significant figures. Report results below the ML as <ML (where ML is the concentration at the ML) or as required by the regulatory authority or permit.

17.6.1.4.3 Blanks – Report the result for each congener above the ML to 3 significant figures. Report a result below the ML but above the MDL to 2 significant figures. Report a result below the MDL as <MDL (where MDL is the concentration at the MDL) or as required by the regulatory authority or permit.

17.6.1.4.4 Blank correction – Blank-corrected results may be reported in addition to reporting of separate results for samples (Section 17.6.1.4.1) and blanks (Section 17.6.1.4.2). The recommended procedure for blank correction (Reference 19) is that a result is significantly above the blank level, and the level in the blank may be subtracted, if the result is 2 standard deviations above the mean (average) of results of analyses of 10 or more blanks for a sample medium.

17.6.2 Results for a CB in a sample that has been diluted are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (Section 17.5).

17.6.3 For a CB having a labeled analog, report results at the least dilute level at which the area at the quantitation m/z is within the calibration range (Section 17.5) and the labeled compound recovery is within the normal range for the Method (Section 9.3 and Table 6).

17.6.4 If requested, the total concentration of all congeners at a given level of chlorination (homolog; i.e., total TrCB, total PeCB, total HxCB) may be reported by summing the concentrations of all congeners identified at that LOC, including both the Toxics and other congeners. Also if requested, total CBs may be reported by summing all congeners identified at all LOCs.

17.6.5 Reporting of coeluting PCB congeners–Optionally, Delaware River Basin Commission (DRBC) data qualifier flags and conventions for reporting coeluting congeners (see http://www.state.nj.us/drbc/PCB_info.htm), or other reporting convention agreed upon between the laboratory and the discharger/permittee or regulatory/control authority, may be used.

18.0 Analysis of complex samples

18.1 Some samples may contain high levels (>10 ng/L; >1000 ng/kg) of the compounds of interest, interfering compounds, and/or polymeric materials. Some extracts may not concentrate to 20 µL (Section 12.7.7); others may overload the GC column and/or mass spectrometer. Fragment ions from congeners at higher levels of chlorination may interfere with determination of congeners at lower levels of chlorination.

18.2 Analyze a smaller aliquot of the sample (Section 17.5) when the extract will not concentrate to 20 µL after all cleanup procedures have been exhausted. If a smaller aliquot of soils or mixed-phase samples is analyzed, attempt to assure that the sample is representative.

18.3 Perform integration of peak areas and calculate concentrations manually when interferences preclude computerized calculations.

18.4 Several laboratories have reported that backgrounds of many of the CB congeners are difficult to eliminate, and that these backgrounds can interfere with the determination of the CBs in environmental samples. Backgrounds of Toxics with congener numbers 105, 114, 118, 123, 156, 157, and 167 are common. The effects of contamination on results for these congeners should be understood in order to make a reliable determination.

18.5 Interferences may pose a problem in the determination of congeners 81, 123, 126, and 169 in some environmental samples. Loss of one or more chlorines from a highly chlorinated congener may inflate or produce a false concentration for a less-chlorinated congener that elutes at the same retention time. If, upon inspection of the chromatogram, the possibility of interferences is evident (e.g., high concentrations of fragments from loss of one or two chlorines from higher chlorinated closely eluting congeners), carbon column fractionation (Section 13.4) and analysis is recommended.

18.6 Recovery of labeled compounds – In most samples, recoveries of the labeled compounds will be similar to those from reagent water or from the alternate matrix (Section 7.6).

18.6.1 If the recovery of any of the labeled compounds is outside of the normal range (Table 6), a diluted sample must be analyzed (Section 17.5).

18.6.2 If the recovery of any of the labeled compounds in the diluted sample is outside of normal range, the Diluted combined 209 congener solution (Section 7.10.2.2.2) must be analyzed and calibration verified (Section 15.3).

18.6.3 If the calibration cannot be verified, a new calibration must be performed and the original sample extract reanalyzed.

18.6.4 If calibration is verified and the diluted sample does not meet the limits for labeled compound recovery, the Method does not apply to the sample being analyzed and the result may not be reported or used for permitting or regulatory compliance purposes. In this case, alternate extraction and cleanup procedures in this Method or an alternate GC column must be employed to resolve the interference. If all cleanup procedures in this Method and an alternate GC column have been employed and labeled compound recovery remains outside of the normal range, extraction and/or cleanup procedures that are beyond this scope of this Method will be required to analyze the sample.

19.0 Pollution prevention

19.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Many opportunities for pollution prevention exist in laboratory operation. EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address waste generation. When wastes cannot be reduced feasibly at the source, the Agency recommends recycling as the next best option.

19.2 The CBs in this Method are used in extremely small amounts and pose little threat to the environment when managed properly. Standards should be prepared in volumes consistent with laboratory use to minimize the disposal of excess volumes of expired standards.

19.3 For information about pollution prevention that may be applied to laboratories and research institutions, consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Governmental Relations and Science Policy, 1155 16th Street NW, Washington DC 20036, 202/872-4477.

20.0 Waste management

20.1 The laboratory is responsible for complying with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance is also required with any sewage discharge permits and regulations. An overview of requirements can be found in *Environmental Management Guide for Small Laboratories* (EPA 233-B-98-001).

20.2 Samples containing HCl or H_2SO_4 to pH <2 are hazardous and must be neutralized before being poured down a drain or must be handled as hazardous waste.

20.3 The CBs decompose above 800 ºC. Low-level waste such as absorbent paper, tissues, animal remains, and plastic gloves may be burned in an appropriate incinerator. Gross quantities (milligrams) should be packaged securely and disposed of through commercial or governmental channels that are capable of handling extremely toxic wastes.

20.4 Liquid or soluble waste should be dissolved in methanol or ethanol and irradiated with ultraviolet light with a wavelength shorter than 290 nm for several days. Use F40 BL or equivalent lamps. Analyze liquid wastes, and dispose of the solutions when the CBs can no longer be detected.

20.5 For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* and *Less is Better-Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington, DC 20036.

21.0 Method performance

The original version of Method 1668 was validated in single-laboratory studies at Pacific Analytical, Inc., Carlsbad, California and AXYS Analytical Services, Ltd., Sidney, British Columbia, Canada. The next version, Method 1668A, was validated and data were collected at AXYS Analytical (Reference 20). Method 1668A was subjected to peer review in 1999, and published in 2000. In 2003-2004, EPA conducted an interlaboratory method validation study of Method 1668A (Reference 21), subjected the study to a peer review, and subsequently published interlaboratory performance data in Method 1668B.

After release of Method 1668B, it was reported to EPA that some of the QC acceptance criteria in Method 1668B did not allow excursions above 100 percent. As a result, the QC acceptance criteria were re-developed using data from the interlaboratory study and data from AXYS Analytical and TestAmerica-Knoxville, Tennessee. The revised QC acceptance criteria were published in addendum to the Interlaboratory Study Report (Reference 22).

Subsequent to development of the revised QC acceptance criteria, AXYS Analytical, TestAmerica-Knoxville, and Battelle-Columbus provided method detection limit (MDL) data to EPA. These data were combined to produced pooled MDLs and MLs (Reference 23). Method 1668B was revised to Method 1668C to incorporate the revised QC acceptance criteria and revised MDLs and MLs.

Figure 8 is a chromatogram showing method performance at each level of chlorination.

22.0 References

1. Van den Berg, Martin, Linda S. Birnbaum, Michael Denison, Mike De Vito, William Farland, Mark Feeley, Heidelore Fiedler, Helen Hakansson, Annika Hanberg, Laurie Haws, Martin Rose, Stephen Safe, Dieter Schrenk, Chiharu Tohyama, Angelika Tritscher, Jouko Tuomisto, Mats Tysklind, Nigel Walker, and Richard E. Peterson, 2006, "The 2005 World Health Organization Reevaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-like Compounds," *Toxicological Sciences* **93**(2): 223-241.

2. "Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update," NOAA Technical Memorandum NOS ORCS 130, Coastal Monitoring and Bioeffects Assessment Division, Office of Ocean Resources Conservation and Assessment, National Ocean Service, National Oceanic and Atmospheric Administration, U.S. Department of Commerce, N/ORCA2, SSMC4, 1305 East-West Highway, Silver Spring, MD 20910, p. 3, 1998.

3. Kuehl, D.W., B.C. Butterworth, J. Libal, and P. Marquis, "An Isotope Dilution High Resolution Gas Chromatography-High Resolution Mass Spectrometric Method for the Determination of Coplanar

Polychlorinated Biphenyls: Application to Fish and Marine Mammals," *Chemosphere* 22:9-10, 849-858, 1991.

4. Echols, Kathy, Robert Gale, Donald E. Tillitt, Ted Schwartz, and Jerome O'Laughlin, "An Automated HPLC Method for the Fractionation of Polychlorinated Biphenyls, Polychlorinated Dibenzo-*p*-dioxins, and Polychlorinated Dibenzofurans in Fish Tissue on a Porous Graphitic Carbon Column," *Environmental Toxicology and Chemistry* 16:8 1590-1597, 1997.

5. "Working with Carcinogens," Department of Health, Education, & Welfare, Public Health Service, Centers for Disease Control, NIOSH, Publication 77-206, August 1977, NTIS PB-277256.

6. "OSHA Safety and Health Standards, General Industry," OSHA 2206, 29 *CFR* 1910.

7. "Safety in Academic Chemistry Laboratories," ACS Committee on Chemical Safety, 1979.

8. "Standard Methods for the Examination of Water and Wastewater," 18th edition and later revisions, American Public Health Association, 1015 15th St, NW, Washington, DC 20005, 1-35: Section 1090 (Safety), 1992.

9. "Method 613 – 2,3,7,8-Tetrachlorodibenzo-*p*-dioxin," 40 *CFR* 136, Appendix A, Section 4.1.

10. Lamparski, L.L., and Nestrick, T.J., "Novel Extraction Device for the Determination of Chlorinated Dibenzo-*p*-dioxins (PCDDs) and Dibenzofurans (PCDFs) in Matrices Containing Water," *Chemosphere*, 19:27-31, 1989.

11. Provost, L.P., and Elder, R.S., "Interpretation of Percent Recovery Data," *American Laboratory*, 15: 56-83, 1983.

12. "Standard Practice for Sampling Water," ASTM Annual Book of Standards, ASTM, 1916 Race Street, Philadelphia, PA 19103-1187, 1980.

13. "Methods 330.4 and 330.5 for Total Residual Chlorine," USEPA, EMSL, Cincinnati, OH 45268, EPA 600/4-70-020, April 1979.

14. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA EMSL, Cincinnati, OH 45268, EPA-600/4-79-019, April 1979.

15. "Analytical Procedures and Quality Assurance Plan for the Determination of PCDD/PCDF in Fish", U.S. Environmental Protection Agency, Environmental Research Laboratory, Duluth, MN 55804, EPA/600/3-90/022, April 1990.

16. Storr-Hansen, E. and T. Cederberg, "Determination of Coplanar Polychlorinated Biphenyl (CB) Congeners in Seal Tissues by Chromatography on Active Carbon, Dual-Column High Resolution GC/ECD and High Resolution GC/High Resolution MS," *Chemosphere* 24:9, 1181-1196, 1992.

17. Echols, Kathy R., Robert W. Gale, Kevin Feltz, Jerome O'Laughlin, Donald E. Tillitt, and Ted R. Schwartz, "Loading capacity and chromatographic behavior of a porous graphitic carbon column for polychlorinated biphenyls," *J. Chromatog. A* 811: 135-144, 1998.

18. Tessari, J.D., Personal communication with Dale Rushneck, available from U.S. Environmental Protection Agency, Engineering and Analysis Division (4303T), 1200 Pennsylvania Avenue NW, Washington, DC 20460.

19. Ferrario, J.C., C. Byrne, A.E. Dupuy, Jr., "Background Contamination by Coplanar Polychlorinated Biphenyls (PCBs) in Trace Level High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) Analytical Procedures" *Chemosphere* 34:11, 2451-2465, 1997.

20. "Development of a Full Congener Version of Method 1668 and Application to the Analysis of 209 PCB Congeners in Aroclors," AXYS Analytical Services, available from U.S. Environmental Protection Agency, Engineering and Analysis Division (4303T), 1200 Pennsylvania Avenue NW, Washington, DC 20460.

21. "Method 1668A Interlaboratory Validation Study Report," March 2010, EPA-820-R-10-004, U.S. Environmental Protection Agency, Engineering and Analysis Division (4303T), 1200 Pennsylvania Avenue NW, Washington, DC 20460.

22. "Method 1668A Interlaboratory Study Report Addendum," March 2010, EPA-820-R-10-003, U.S. Environmental Protection Agency, Engineering and Analysis Division (4303T), 1200 Pennsylvania Avenue NW, Washington, DC 20460.

23. "Development of Pooled Method Detection Limits (MDLs) and Minimum Levels of Quantitation (MLs) for EPA Method 1668C," Brian Englert, USEPA, May 18 2010; available from U.S. Environmental Protection Agency, Engineering and Analysis Division (4303T), 1200 Pennsylvania Avenue NW, Washington, DC 20460.

23.0 Tables and Figures

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native and Labeled Chlorinated Biphenyl (CB) Congeners Determined by Isotope Dilution and Internal Standard HRGC/HRMS

CB congener name[1]	Congener number	CAS Registry number	Labeled analog name	Labeled analog congener number	CAS Registry number
2-MoCB	1	2051-60-7	$^{13}C_{12}$-2-MoCB[2]	1L	234432-85-0
3-MoCB	2	2051-61-8			
4-MoCB	3	2051-62-9	$^{13}C_{12}$-4-MoCB[2]	3L	208263-77-8
2,2'-DiCB	4	13029-08-8	$^{13}C_{12}$-2,2'-DiCB[2]	4L	234432-86-1
2,3-DiCB	5	16605-91-7			
2,3'-DiCB	6	25569-80-6			
2,4-DiCB	7	33284-50-3			
2,4'-DiCB[3]	8	34883-43-7			
2,5-DiCB	9	34883-39-1	$^{13}C_{12}$-2,5-DiCB[4]	9L	250694-89-4
2,6-DiCB	10	33146-45-1			
3,3'-DiCB	11	2050-67-1			
3,4-DiCB	12	2974-92-7			
3,4'-DiCB	13	2974-90-5			
3,5-DiCB	14	34883-41-5			
4,4'-DiCB	15	2050-68-2	$^{13}C_{12}$-4,4'-DiCB[2]	15L	208263-67-6
2,2',3-TrCB	16	38444-78-9			
2,2',4-TrCB	17	37680-66-3			
2,2',5-TrCB[3]	18	37680-65-2			
2,2',6-TrCB	19	38444-73-4	$^{13}C_{12}$-2,2',6-TrCB[2]	19L	234432-87-2
2,3,3'-TrCB	20	38444-84-7			
2,3,4-TrCB	21	55702-46-0			
2,3,4'-TrCB	22	38444-85-8			
2,3,5-TrCB	23	55720-44-0			
2,3,6-TrCB	24	55702-45-9			
2,3',4-TrCB	25	55712-37-3			
2,3',5-TrCB	26	38444-81-4			
2,3',6-TrCB	27	38444-76-7			
2,4,4'-TrCB[3]	28	7012-37-5	$^{13}C_{12}$-2,4,4'-TriCB[5]	28L	208263-76-7
2,4,5-TrCB	29	15862-07-4			
2,4,6-TrCB	30	35693-92-6			
2,4',5-TrCB	31	16606-02-3			
2,4',6-TrCB	32	38444-77-8			
2',3,4-TrCB	33	38444-86-9			
2',3,5-TrCB	34	37680-68-5			
3,3',4-TrCB	35	37680-69-6			
3,3',5-TrCB	36	38444-87-0			
3,4,4'-TrCB	37	38444-90-5	$^{13}C_{12}$-3,4,4'-TrCB[2]	37L	208263-79-0
3,4,5-TrCB	38	53555-66-1			
3,4',5-TrCB	39	38444-88-1			
2,2',3,3'-TeCB	40	38444-93-8			
2,2',3,4-TeCB	41	52663-59-9			
2,2',3,4'-TeCB	42	36559-22-5			
2,2',3,5-TeCB	43	70362-46-8			
2,2',3,5'-TeCB[3]	44	41464-39-5			

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native and Labeled Chlorinated Biphenyl (CB) Congeners Determined by Isotope Dilution and Internal Standard HRGC/HRMS

CB congener name[1]	Congener number	CAS Registry number	Labeled analog name	Labeled analog congener number	CAS Registry number
2,2',3,6-TeCB	45	70362-45-7			
2,2',3,6'-TeCB	46	41464-47-5			
2,2',4,4'-TeCB	47	2437-79-8			
2,2',4,5-TeCB	48	70362-47-9			
2,2',4,5'-TeCB	49	41464-40-8			
2,2',4,6-TeCB	50	62796-65-0			
2,2',4,6'-TeCB	51	68194-04-7			
2,2',5,5'-TeCB[3]	52	35693-99-3	$^{13}C_{12}$-2,2',5,5'-TeCB[4]	52L	208263-80-3
2,2',5,6'-TeCB	53	41464-41-9			
2,2',6,6'-TeCB	54	15968-05-5	$^{13}C_{12}$-2,2',6,6'-TeCB[2]	54L	234432-88-3
2,3,3',4'-TeCB	55	74338-24-2			
2,3,3',4'-TeCB	56	41464-43-1			
2,3,3',5-TeCB	57	70424-67-8			
2,3,3',5'-TeCB	58	41464-49-7			
2,3,3',6-TeCB	59	74472-33-6			
2,3,4,4'-TeCB	60	33025-41-1			
2,3,4,5-TeCB	61	33284-53-6			
2,3,4,6-TeCB	62	54230-22-7			
2,3,4',5-TeCB	63	74472-34-7			
2,3,4',6-TeCB	64	52663-58-8			
2,3,5,6-TeCB	65	33284-54-7			
2,3',4,4'-TeCB[3]	66	32598-10-0			
2,3',4,5-TeCB	67	73575-53-8			
2,3',4,5'-TeCB	68	73575-52-7			
2,3',4,6-TeCB	69	60233-24-1			
2,3',4',5-TeCB	70	32598-11-1			
2,3',4',6-TeCB	71	41464-46-4			
2,3',5,5'-TeCB	72	41464-42-0			
2,3',5',6-TeCB	73	74338-23-1			
2,4,4',5-TeCB	74	32690-93-0			
2,4,4',6-TeCB	75	32598-12-2			
2',3,4,5-TeCB	76	70362-48-0			
3,3',4,4'-TeCB[3,6]	77	32598-13-3	$^{13}C_{12}$-3,3',4,4'-TeCB[2,7]	77L	105600-23-5
3,3',4,5-TeCB	78	70362-49-1			
3,3',4,5'-TeCB	79	41464-48-6			
3,3',5,5'-TeCB	80	33284-52-5			
3,4,4',5-TeCB[6]	81	70362-50-4	$^{13}C_{12}$-3,4,4',5-TeCB[7]	81L	208461-24-9
2,2',3,3',4-PeCB	82	52663-62-4			
2,2',3,3',5-PeCB	83	60145-20-2			
2,2',3,3',6-PeCB	84	52663-60-2			
2,2',3,4,4'-PeCB	85	65510-45-4			
2,2',3,4,5-PeCB	86	55312-69-1			
2,2',3,4,5'-PeCB	87	38380-02-8			
2,2',3,4,6-PeCB	88	55215-17-3			
2,2',3,4,6'-PeCB	89	73575-57-2			
2,2',3,4',5-PeCB	90	68194-07-0			

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native and Labeled Chlorinated Biphenyl (CB) Congeners Determined by Isotope Dilution and Internal Standard HRGC/HRMS

CB congener name[1]	Congener number	CAS Registry number	Labeled analog name	Labeled analog congener number	CAS Registry number
2,2',3,4',6-PeCB	91	68194-05-8			
2,2',3,5,5'-PeCB	92	52663-61-3			
2,2',3,5,6-PeCB	93	73575-56-1			
2,2',3,5,6'-PeCB	94	73575-55-0			
2,2',3,5',6-PeCB	95	38379-99-6			
2,2',3,6,6'-PeCB	96	73575-54-9			
2,2',3',4,5-PeCB	97	41464-51-1			
2,2',3',4,6-PeCB	98	60233-25-2			
2,2',4,4',5-PeCB	99	38380-01-7			
2,2',4,4',6-PeCB	100	39485-83-1			
2,2',4,5,5'-PeCB[3]	101	37680-73-2	$^{13}C_{12}$-2,2',4,5,5'-PeCB[4]	101L	104130-39-4
2,2',4,5,6'-PeCB	102	68194-06-9			
2,2',4,5,'6-PeCB	103	60145-21-3			
2,2',4,6,6'-PeCB	104	56558-16-8	$^{13}C_{12}$-2,2',4,6,6'-PeCB[2]	104L	234432-89-4
2,3,3',4,4'-PeCB[3,6]	105	32598-14-4	$^{13}C_{12}$-2,3,3',4,4'-PeCB[7]	105L	208263-62-1
2,3,3',4,5-PeCB	106	70424-69-0			
2,3,3',4',5-PeCB	107	70424-68-9			
2,3,3',4,5'-PeCB	108	70362-41-3			
2,3,3',4,6-PeCB	109	74472-35-8			
2,3,3',4',6-PeCB	110	38380-03-9			
2,3,3',5,5'-PeCB	111	39635-32-0	$^{13}C_{12}$-2,3,3',5,5'-PeCB[5]	111 L	235416-29-2
2,3,3',5,6-PeCB	112	74472-36-9			
2,3,3',5',6-PeCB	113	68194-10-5			
2,3,4,4',5-PeCB[6]	114	74472-37-0	$^{13}C_{12}$-2,3,4,4',5-PeCB[7]	114 L	208263-63-2
2,3,4,4',6-PeCB	115	74472-38-1			
2,3,4,5,6-PeCB	116	18259-05-7			
2,3,4',5,6-PeCB	117	68194-11-6			
2,3',4,4',5-PeCB[3,6]	118	31508-00-6	$^{13}C_{12}$-2,3',4,4',5-PeCB[7]	118 L	104130-40-7
2,3',4,4',6-PeCB	119	56558-17-9			
2,3',4,5,5'-PeCB	120	68194-12-7			
2,3',4,5,'6-PeCB	121	56558-18-0			
2',3,3',4,5-PeCB	122	76842-07-4			
2',3,4,4',5-PeCB[6]	123	65510-44-3	$^{13}C_{12}$-2',3,4,4',5-PeCB[7]	123L	208263-64-3
2',3,4,5,5'-PeCB	124	70424-70-3			
2',3,4,5,6'-PeCB	125	74472-39-2			
3,3',4,4',5-PeCB[3,6]	126	57465-28-8	$^{13}C_{12}$-3,3',4,4',5-PeCB[2,7]	126L	208263-65-4
3,3',4,5,5'-PeCB	127	39635-33-1			
2,2',3,3',4,4'-HxCB[3]	128	38380-07-3			
2,2',3,3',4,5-HxCB	129	55215-18-4			
2,2',3,3',4,5'-HxCB	130	52663-66-8			
2,2',3,3',4,6-HxCB	131	61798-70-7			
2,2',3,3',4,6'-HxCB	132	38380-05-1			
2,2',3,3',5,5'-HxCB	133	35694-04-3			
2,2',3,3',5,6-HxCB	134	52704-70-8			
2,2',3,3',5,6'-HxCB	135	52744-13-5			
2,2',3,3',6,6'-HxCB	136	38411-22-2			

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native and Labeled Chlorinated Biphenyl (CB) Congeners Determined by Isotope Dilution and Internal Standard HRGC/HRMS

CB congener name[1]	Congener number	CAS Registry number	Labeled analog name	Labeled analog congener number	CAS Registry number
2,2',3,4,4',5-HxCB	137	35694-06-5			
2,2',3,4,4',5'-HxCB[3]	138	35065-28-2	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[4]	138L	208263-66-5
2,2',3,4,4',6-HxCB	139	56030-56-9			
2,2',3,4,4',6'-HxCB	140	59291-64-4			
2,2',3,4,5,5'-HxCB	141	52712-04-6			
2,2',3,4,5,6-HxCB	142	41411-61-4			
2,2',3,4,5,6'-HxCB	143	68194-15-0			
2,2',3,4,5',6-HxCB	144	68194-14-9			
2,2',3,4,6,6'-HxCB	145	74472-40-5			
2,2',3,4',5,5'-HxCB	146	51908-16-8			
2,2',3,4',5,6-HxCB	147	68194-13-8			
2,2',3,4',5,6'-HxCB	148	74472-41-6			
2,2',3,4',5',6-HxCB	149	38380-04-0			
2,2',3,4',6,6'-HxCB	150	68194-08-1			
2,2',3,5,5',6-HxCB	151	52663-63-5			
2,2',3,5,6,6'-HxCB	152	68194-09-2			
2,2',4,4',5,5'-HxCB[3]	153	35065-27-1			
2,2',4,4',5',6-HxCB	154	60145-22-4			
2,2',4,4',6,6'-HxCB	155	33979-03-2	$^{13}C_{12}$-2,2',4,4',6,6'-HxCB[2]	155L	234432-90-7
2,3,3',4,4',5-HxCB[6]	156	38380-08-4	$^{13}C_{12}$-2,3,3',4,4',5-HxCB[7]	156L	208263-68-7
2,3,3',4,4',5'-HxCB[6]	157	69782-90-7	$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[7]	157L	235416-30-5
2,3,3',4,4',6-HxCB	158	74472-42-7			
2,3,3',4,5,5'-HxCB	159	39635-35-3			
2,3,3',4,5,6-HxCB	160	41411-62-5			
2,3,3',4,5',6-HxCB	161	74472-43-8			
2,3,3',4',5,5'-HxCB	162	39635-34-2			
2,3,3',4',5,6-HxCB	163	74472-44-9			
2,3,3',4',5',6-HxCB	164	74472-45-0			
2,3,3',5,5',6-HxCB	165	74472-46-1			
2,3,4,4',5,6-HxCB	166	41411-63-6			
2,3',4,4',5,5'-HxCB[6]	167	52663-72-6	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[7]	167L	208263-69-8
2,3',4,4',5',6-HxCB	168	59291-65-5			
3,3',4,4',5,5'-HxCB[3,6]	169	32774-16-6	$^{13}C_{12}$-3,3',4,4',5,5'-HxCB[2,7]	169L	208263-70-1
2,2',3,3',4,4',5-HpCB[3]	170	35065-30-6	$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	160901-80-4
2,2'3,3',4,4',6-HpCB	171	52663-71-5			
2,2',3,3',4,5,5'-HpCB	172	52663-74-8			
2,2',3,3',4,5,6-HpCB	173	68194-16-1			
2,2',3,3',4,5,6'-HpCB	174	38411-25-5			
2,2',3,3',4,5',6-HpCB	175	40186-70-7			
2,2',3,3',4,6,6'-HpCB	176	52663-65-7			
2,2',3,3',4',5,6-HpCB	177	52663-70-4			
2,2',3,3',5,5',6-HpCB	178	52663-67-9	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[5]	178L	232919-67-4
2,2',3,3',5,6,6'-HpCB	179	52663-64-6			
2,2',3,4,4',5,5'-HpCB[3]	180	35065-29-3	$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	160901-82-6
2,2',3,4,4',5,6-HpCB	181	74472-47-2			
2,2',3,4,4',5,6'-HpCB	182	60145-23-5			

Table 1. Names, Congener Numbers, and CAS Registry Numbers for Native and Labeled Chlorinated Biphenyl (CB) Congeners Determined by Isotope Dilution and Internal Standard HRGC/HRMS

CB congener name[1]	Congener number	CAS Registry number	Labeled analog name	Labeled analog congener number	CAS Registry number
2,2',3,4,4',5',6-HpCB	183	52663-69-1			
2,2',3,4,4',6,6'-HpCB	184	74472-48-3			
2,2',3,4,5,5',6-HpCB	185	52712-05-7			
2,2',3,4,5,6,6'-HpCB	186	74472-49-4			
2,2',3,4',5,5',6-HpCB[3]	187	52663-68-0			
2,2',3,4',5,6,6'-HpCB	188	74487-85-7	$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB[2]	188L	234432-91-8
2,3,3',4,4',5,5'-HpCB[6]	189	39635-31-9	$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB[2,7]	189L	208263-73-4
2,3,3',4,4',5,6-HpCB	190	41411-64-7			
2,3,3',4,4',5',6-HpCB	191	74472-50-7			
2,3,3',4,5,5',6-HpCB	192	74472-51-8			
2,3,3',4',5,5',6-HpCB	193	69782-91-8			
2,2',3,3',4,4',5,5'-OcCB	194	35694-08-7	$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB[4]	194L	208263-74-5
2,2',3,3',4,4',5,6-OcCB[3]	195	52663-78-2			
2,2',3,3',4,4',5,6'-OcCB	196	42740-50-1			
2,2',3,3',4,4',6,6'-OcCB	197	33091-17-7			
2,2',3,3',4,5,5',6-OcCB	198	68194-17-2			
2,2',3,3',4,5,5',6'-OcCB	199	52663-75-9			
2,2',3,3',4,5,6,6'-OcCB	200	52663-73-7			
2,2',3,3',4,5',6,6'-OcCB	201	40186-71-8			
2,2',3,3',5,5',6,6'-OcCB	202	2136-99-4	$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB[2]	202L	105600-26-8
2,2',3,4,4',5,5',6-OcCB	203	52663-76-0			
2,2',3,4,4',5,6,6'-OcCB	204	74472-52-9			
2,3,3',4,4',5,5',6-OcCB	205	74472-53-0	$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB[2]	205L	234446-64-1
2,2',3,3',4,4',5,5',6-NoCB[3]	206	40186-72-9	$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB[2]	206L	208263-75-6
2,2',3,3',4,4',5,6,6'-NoCB	207	52663-79-3			
2,2',3,3',4,5,5',6,6'-NoCB	208	52663-77-1	$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB[2]	208L	234432-92-9
DeCB[3]	209	2051-24-3	$^{13}C_{12}$-DeCB[2]	209L	105600-27-9

1. Abbreviations for chlorination levels

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

2. Labeled level of chlorination (LOC) window-defining congener
3. National Oceanic and Atmospheric Administration (NOAA) congener of interest
4. Labeled injection internal standard
5. Labeled clean-up standard
6. World Health Organization (WHO) toxic congener
7. Labeled analog of WHO toxic congener

Table 2. Retention times (RT), RT references, relative retention times (RRTs), method detection limits (MDLs), and minimum levels of quantitation (MLs) for the 209 CB congeners on SPB-octyl.

Cl No. [1]	Congener No. [2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec) [8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
Compounds using 9L ($^{13}C_{12}$-2,5-DiCB) as Labeled injection internal standard												
	CB congener											
	Monochlorobiphenyls											
1	1	1L	13:44	1.0012	0.9988-1.0036	-1+3	1L	10	20	1.0	2	1
1	2	3L	16:08	0.9878	0.9847-0.9908	6	1L/3L	7	20	0.7	2	1
1	3	3L	16:21	1.0010	0.9990-1.0031	-1+3	3L	11	50	1.1	5	2.5
	Dichlorobiphenyls											
2	4	4L	16:40	1.0010	0.9990-1.0030	-1+3	4L	13	50	1.3	5	2.5
2	10	4L	16:53	1.0140	1.0110-1.0170	6	4L/15L	13	50	1.3	5	2.5
2	9	4L	18:55	1.1361	1.1331-1.1391	6	4L/15L	7	20	0.7	2	1
2	7	4L	19:07	1.1481	1.1451-1.1512	6	4L/15L	8	20	0.8	2	1
2	6	4L	19:26	1.1672	1.1642-1.1702	6	4L/15L	7	20	0.7	2	1
2	5	4L	19:48	1.1892	1.1862-1.1922	6	4L/15L	8	20	0.8	2	1
2	8	4L	19:56	1.1972	1.1942-1.2002	6	4L/15L	15	50	1.5	5	2.5
2	14	15L	21:42	0.9267	0.9246-0.9288	6	4L/15L	8	20	0.8	2	1
2	11	15L	22:42	0.9694	0.9673-0.9715	6	4L/15L	34	100	3.4	10	5
2	13	15L	23:03	0.9843	0.9822-0.9865	6	4L/15L	19	50	1.9	5	2.5
2	12	15L	23:06	0.9865	0.9843-0.9886	6	4L/15L					
2	13/12	15L	23:04	0.9851	0.9829-0.9872	6	4L/15L					
2	15	15L	23:26	1.0007	0.9993-1.0021	-1+3	15L	16	50	1.6	5	2.5
	Trichlorobiphenyls											
3	19	19L	20:19	1.0008	0.9992-1.0025	-1+3	19L	8	20	0.8	2	1
3	30	19L	22:15	1.0961	1.0936-1.0985	6	19L/37L	16	50	1.6	5	2.5
3	18	19L	22:23	1.1026	1.1002-1.1051	6	19L/37L					

Cl No. [1]	Congener No. [2,3]	RT Ref [4]	RT [5]	RRT [6]	RRT limits [7]	Window (sec) [8]	Quantitation reference [9]	Detection limits and minimum levels - Matrix and concentration [10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
3	30/18	19L	22:19	1.0993	1.0969-1.1018	6	19L/37L					
3	17	19L	22:49	1.1240	1.1215-1.1264	6	19L/37L	9	20	0.9	2	1
3	27	19L	23:06	1.1379	1.1355-1.1404	6	19L/37L	8	20	0.8	2	1
3	24	19L	23:14	1.1445	1.1420-1.1470	6	19L/37L	10	20	1.0	2	1
3	16	19L	23:25	1.1535	1.1511-1.1560	6	19L/37L	9	20	0.9	2	1
3	32	19L	24:57	1.2291	1.2266-1.2315	6	19L/37L	8	20	0.8	2	1
3	34	19L	25:17	1.2455	1.2430-1.2479	6	19L/37L	7	20	0.7	2	1
3	23	19L	25:26	1.2529	1.2504-1.2553	6	19L/37L	7	20	0.7	2	1
3	29	19L	25:47	1.2701	1.2660-1.2742	10	19L/37L					
3	26	19L	25:48	1.2709	1.2668-1.2750	10	19L/37L	12	50	1.2	5	2.5
3	29/26	19L	25:48	1.2709	1.2668-1.2750	10	19L/37L					
3	25	37L	26:04	0.8364	0.8348-0.8380	6	19L/37L	8	20	0.8	2	1
3	31	37L	26:25	0.8476	0.8460-0.8492	6	19L/37L	18	50	1.8	5	2.5
3	28	37L	26:44	0.8578	0.8551-0.8604	10	19L/37L					
3	20	37L	26:49	0.8604	0.8578-0.8631	10	19L/37L	22	50	2.2	5	2.5
3	28/20	37L	26:47	0.8594	0.8567-0.8620	10	19L/37L					
3	21	37L	26:58	0.8652	0.8626-0.8679	10	19L/37L					
3	33	37L	27:01	0.8668	0.8642-0.8695	10	19L/37L	21	50	2.1	5	2.5
3	21/33	37L	26:59	0.8658	0.8631-0.8684	10	19L/37L					
3	22	37L	27:29	0.8818	0.8802-0.8834	6	19L/37L	9	20	0.9	2	1
3	36	37L	29:05	0.9332	0.9316-0.9348	6	19L/37L	8	20	0.8	2	1
3	39	37L	29:30	0.9465	0.9449-0.9481	6	19L/37L	8	20	0.8	2	1
3	38	37L	30:10	0.9679	0.9663-0.9695	6	19L/37L	7	20	0.7	2	1
3	35	37L	30:42	0.9850	0.9834-0.9866	6	19L/37L	9	20	0.9	2	1
3	37	37L	31:11	1.0005	0.9995-1.0011	-1+3	37L	10	20	1.0	2	1
	Labeled Compounds											
1	1L	9L	13:43	0.7257	0.7125-0.7390	30	9L					
1	3L	9L	16:20	0.8642	0.8510-0.8774	30	9L					
2	4L	9L	16:39	0.8810	0.8677-0.8942	30	9L					
2	15L	9L	23:25	1.2390	1.2302-1.2478	20	9L					
3	19L	9L	20:18	1.0741	1.0608-1.0873	30	9L					
3	37L	52L	31:10	1.0841	1.0754-1.0928	30	52L					

Cl No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
Compounds using 52L ($^{13}C_{12}$-2,2',5,5'-TeCB) as Labeled injection internal standard												
	CB congener											
	Tetrachlorobiphenyls											
4	54	54L	23:51	1.0007	0.9993-1.0021	-1+3	54L	14	50	1.4	5	2.5
4	50	54L	26:07	1.0958	1.0923-1.0993	10	54L/81L/77L					
4	53	54L	26:09	1.0972	1.0937-1.1007	10	54L/81L/77L	25	100	2.5	10	5
4	50/53	54L	26:08	1.0965	1.0930-1.1000	10	54L/81L/77L					
4	45	54L	26:55	1.1294	1.1259-1.1329	10	54L/81L/77L					
4	51	54L	26:58	1.1315	1.1280-1.1350	10	54L/81L/77L	22	50	2.2	5	2.5
4	45/51	54L	26:57	1.1308	1.1273-1.1343	10	54L/81L/77L					
4	46	54L	27:18	1.1455	1.1434-1.1476	6	54L/81L/77L	10	20	1.0	2	1
4	52	54L	28:45	1.2063	1.2042-1.2084	6	54L/81L/77L	15	50	1.5	5	2.5
4	73	54L	28:52	1.2112	1.2091-1.2133	6	54L/81L/77L	14	50	1.4	5	2.5
4	43	54L	28:58	1.2154	1.2133-1.2175	6	54L/81L/77L	14	50	1.4	5	2.5
4	69	54L	29:08	1.2224	1.2189-1.2259	10	54L/81L/77L					
4	49	54L	29:16	1.2280	1.2245-1.2315	10	54L/81L/77L	26	100	2.6	10	5
4	69/49	54L	29:12	1.2252	1.2217-1.2287	10	54L/81L/77L					
4	48	54L	29:33	1.2399	1.2378-1.2420	6	54L/81L/77L	14	50	1.4	5	2.5
4	65	54L	29:49	1.2510	1.2476-1.2545	10	54L/81L/77L					
4	47	54L	29:50	1.2517	1.2483-1.2552	10	54L/81L/77L	40	100	4.0	10	5
4	44	54L	29:53	1.2538	1.2503-1.2573	10	54L/81L/77L					
4	65/47/44	54L	29:50	1.2517	1.2483-1.2552	10	54L/81L/77L					
4	62	54L	30:06	1.2629	1.2594-1.2664	10	54L/81L/77L					
4	75	54L	30:08	1.2643	1.2608-1.2678	10	54L/81L/77L	37	100	3.7	10	5
4	59	54L	30:12	1.2671	1.2636-1.2706	10	54L/81L/77L					
4	62/75/59	54L	30:09	1.2650	1.2615-1.2685	10	54L/81L/77L					
4	42	54L	30:26	1.2769	1.2748-1.2790	6	54L/81L/77L	16	50	1.6	5	2.5
4	41	54L	30:52	1.2951	1.2916-1.2986	10	54L/81L/77L					
4	71	54L	30:58	1.2993	1.2958-1.3028	10	54L/81L/77L	42	100	4.2	10	5
4	40	54L	31:01	1.3014	1.2979-1.3049	10	54L/81L/77L					
4	41/71/40	54L	30:58	1.2993	1.2958-1.3028	10	54L/81L/77L					
4	64	54L	31:12	1.3091	1.3070-1.3112	6	54L/81L/77L	13	50	1.3	5	2.5

Cl No. [1]	Congener No. [2,3]	RT Ref [4]	RT [5]	RRT [6]	RRT limits [7]	Window (sec) [8]	Quantitation reference [9]	Detection limits and minimum levels - Matrix and concentration [10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
4	72	81L	31:59	0.8336	0.8323-0.8349	6	54L/81L/77L	13	50	1.3	5	2.5
4	68	81L	32:18	0.8419	0.8406-0.8432	6	54L/81L/77L	14	50	1.4	5	2.5
4	57	81L	32:46	0.8540	0.8527-0.8553	6	54L/81L/77L	11	50	1.1	5	2.5
4	58	81L	33:05	0.8623	0.8610-0.8636	6	54L/81L/77L	14	50	1.4	5	2.5
4	67	81L	33:13	0.8658	0.8645-0.8671	6	54L/81L/77L	12	50	1.2	5	2.5
4	63	81L	33:30	0.8732	0.8719-0.8745	6	54L/81L/77L	12	50	1.2	5	2.5
4	61	81L	33:46	0.8801	0.8775-0.8827	12	54L/81L/77L					
4	70	81L	33:53	0.8831	0.8805-0.8858	12	54L/81L/77L					
4	76	81L	33:55	0.8840	0.8814-0.8866	12	54L/81L/77L	59	200	5.9	20	10
4	74	81L	33:57	0.8849	0.8827-0.8871	10	54L/81L/77L					
4	61/70/76/74	81L	33:55	0.8840	0.8814-0.8866	12	54L/81L/77L					
4	66	81L	34:15	0.8927	0.8914-0.8940	6	54L/81L/77L	17	50	1.7	5	2.5
4	55	81L	34:28	0.8983	0.8970-0.8997	6	54L/81L/77L	12	50	1.2	5	2.5
4	56	81L	35:03	0.9136	0.9123-0.9149	6	54L/81L/77L	15	50	1.5	5	2.5
4	60	81L	35:16	0.9192	0.9179-0.9205	6	54L/81L/77L	14	50	1.4	5	2.5
4	80	81L	35:32	0.9262	0.9248-0.9275	6	54L/81L/77L	11	50	1.1	5	2.5
4	79	81L	37:16	0.9713	0.9700-0.9726	6	54L/81L/77L	13	50	1.3	5	2.5
4	78	81L	37:52	0.9870	0.9857-0.9883	6	54L/81L/77L	16	50	1.6	5	2.5
4	81	81L	38:23	1.0004	0.9996-1.0013	-1+3	81L	18	50	1.8	5	2.5
4	77	77L	39:02	1.0004	0.9996-1.0013	-1+3	77L	14	50	1.4	5	2.5
Labeled compounds												
4	54L	52L	23:50	0.8290	0.8232-0.8348	20	52L					
4	81L	52L	38:22	1.3345	1.3287-1.3403	20	52L					
4	77L	52L	39:01	1.3571	1.3513-1.3629	20	52L					
Compounds using 101L ($^{13}C_{12}$-2,2',4,5,5'-PeCB) as Labeled injection internal standard												
CB congener												
Pentachlorobiphenyls												
5	104	104L	29:46	1.0000	0.9994-1.0017	-1+3	104L	14	50	1.4	5	2.5
5	96	104L	30:17	1.0174	1.0146-1.0202	10	104L/123L/114L/118L/105L	15	50	1.5	5	2.5
5	103	104L	32:11	1.0812	1.0795-1.0829	6	104L/123L/114L/118L/105L	11	50	1.1	5	2.5
5	94	104L	32:29	1.0913	1.0896-1.0929	6	104L/123L/114L/118L/105L	13	50	1.3	5	2.5
5	95	104L	33:00	1.1086	1.1058-1.1114	10	104L/123L/114L/118L/105L	77	200	7.7	20	10

Cl No. [1]	Congener No. [2,3]	RT Ref [4]	RT [5]	RRT [6]	RRT limits [7]	Window (sec) [8]	Quantitation reference [9]	Detection limits and minimum levels - Matrix and concentration [10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
5	100	104L	33:06	1.1120	1.1092-1.1148	10	104L/123L/114L/118L/105L					
5	93	104L	33:14	1.1165	1.1137-1.1193	10	104L/123L/114L/118L/105L					
5	102	104L	33:21	1.1204	1.1176-1.1232	10	104L/123L/114L/118L/105L					
5	98	104L	33:26	1.1232	1.1204-1.1260	10	104L/123L/114L/118L/105L					
5	95/100/93/102/98	104L	33:13	1.1159	1.1131-1.1187	15	104L/123L/114L/118L/105L					
5	88	104L	33:48	1.1355	1.1321-1.1389	12	104L/123L/114L/118L/105L					
5	91	104L	33:55	1.1394	1.1366-1.1422	10	104L/123L/114L/118L/105L	22	50	2.2	5	2.5
5	88/91	104L	33:52	1.1377	1.1344-1.1411	12	104L/123L/114L/118L/105L					
5	84	104L	34:14	1.1501	1.1484-1.1517	6	104L/123L/114L/118L/105L	11	20	1.1	2	1
5	89	104L	34:44	1.1669	1.1652-1.1685	6	104L/123L/114L/118L/105L	13	50	1.3	5	2.5
5	121	104L	34:57	1.1741	1.1725-1.1758	6	104L/123L/114L/118L/105L	12	50	1.2	5	2.5
5	92	123L	35:26	0.8639	0.8627-0.8651	6	104L/123L/114L/118L/105L	13	50	1.3	5	2.5
5	113	123L	36:01	0.8781	0.8761-0.8801	10	104L/123L/114L/118L/105L					
5	90	123L	36:03	0.8789	0.8769-0.8809	10	104L/123L/114L/118L/105L	47	200	4.7	20	10
5	101	123L	36:04	0.8793	0.8773-0.8813	10	104L/123L/114L/118L/105L					
5	113/90/101	123L	36:03	0.8789	0.8769-0.8809	10	104L/123L/114L/118L/105L					
5	83	123L	36:39	0.8935	0.8911-0.8960	12	104L/123L/114L/118L/105L					
5	99	123L	36:41	0.8944	0.8923-0.8964	10	104L/123L/114L/118L/105L	29	100	2.9	10	5
5	83/99	123L	36:40	0.8939	0.8915-0.8964	12	104L/123L/114L/118L/105L					
5	112	123L	36:51	0.8984	0.8972-0.8996	6	104L/123L/114L/118L/105L	14	50	1.4	5	2.5
5	119	123L	37:12	0.9069	0.9037-0.9102	16	104L/123L/114L/118L/105L					
5	109	123L	37:12	0.9069	0.9037-0.9102	16	104L/123L/114L/118L/105L					
5	86	123L	37:17	0.9090	0.9057-0.9122	16	104L/123L/114L/118L/105L					
5	97	123L	37:17	0.9090	0.9057-0.9122	16	104L/123L/114L/118L/105L	74	200	7.4	20	10
5	125	123L	37:21	0.9106	0.9074-0.9139	16	104L/123L/114L/118L/105L					
5	87	123L	37:25	0.9122	0.9102-0.9143	10	104L/123L/114L/118L/105L					
5	119/109/86/97/125/87	123L	37:19	0.9098	0.9065-0.9130	16	104L/123L/114L/118L/105L					
5	117	123L	37:57	0.9252	0.9228-0.9277	12	104L/123L/114L/118L/105L					
5	116	123L	38:02	0.9273	0.9248-0.9297	12	104L/123L/114L/118L/105L					
5	85	123L	38:05	0.9285	0.9265-0.9305	10	104L/123L/114L/118L/105L	38	100	3.8	10	5
5	117/116/85	123L	38:00	0.9265	0.9240-0.9289	12	104L/123L/114L/118L/105L					
5	110	123L	38:16	0.9330	0.9309-0.9350	10	104L/123L/114L/118L/105L	39	100	3.9	10	5

Cl No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
5	115	123L	38:18	0.9338	0.9317-0.9358	10	104L/123L/114L/118L/105L					
5	110/115	123L	38:17	0.9334	0.9313-0.9354	10	104L/123L/114L/118L/105L					
5	82	123L	38:40	0.9427	0.9415-0.9439	6	104L/123L/114L/118L/105L	15	50	1.5	5	2.5
5	111	123L	38:52	0.9476	0.9464-0.9488	6	104L/123L/114L/118L/105L	14	50	1.4	5	2.5
5	120	123L	39:21	0.9594	0.9581-0.9606	6	104L/123L/114L/118L/105L	13	50	1.3	5	2.5
5	108	123L	40:39	0.9911	0.9890-0.9931	10	104L/123L/114L/118L/105L					
5	124	123L	40:40	0.9915	0.9894-0.9935	10	104L/123L/114L/118L/105L	29	100	2.9	10	5
5	108/124	123L	40:39	0.9911	0.9890-0.9931	10	104L/123L/114L/118L/105L					
5	107	123L	40:54	0.9972	0.9959-0.9984	6	104L/123L/114L/118L/105L	17	50	1.7	5	2.5
5	123	123L	41:02	1.0004	0.9996-1.0012	-1+3	123L	17	50	1.7	5	2.5
5	106	123L	41:10	1.0037	1.0024-1.0049	6	104L/123L/114L/118L/105L	17	50	1.7	5	2.5
5	118	118L	41:22	1.0004	0.9996-1.0012	-1+3	118L	30	100	3.0	10	5
5	122	118L	41:49	1.0113	1.0101-1.0125	6	104L/123L/114L/118L/105L	12	50	1.2	5	2.5
5	114	114L	41:58	1.0004	0.9999-1.0012	-1+3	114L	15	50	1.5	5	2.5
5	105	105L	42:43	0.9996	0.9996-1.0012	-2+3	105L	17	50	1.7	5	2.5
5	127	105L	44:09	1.0332	1.0320-1.0343	6	104L/123L/114L/118L/105L	14	50	1.4	5	2.5
5	126	126L	45:58	1.0004	0.9996-1.0011	-1+3	126L	16	50	1.6	5	2.5
	Labeled compounds											
5	104L	101L	29:46	0.8257	0.8211-0.8303	20	101L					
5	123L	101L	41:01	1.1378	1.1331-1.1424	20	101L					
5	118L	101L	41:21	1.1470	1.1424-1.1516	20	101L					
5	114L	101L	41:57	1.1637	1.1590-1.1683	20	101L					
5	105L	101L	42:44	1.1854	1.1808-1.1900	20	101L					
5	126L	101L	45:57	1.2746	1.2700-1.2792	20	101L					
Compounds using 138L ($^{13}C_{12}$-2,2',3,4,4',5'-HxCB) as Labeled injection internal standard												
	CB congener											
	Hexachlorobiphenyls											
6	155	155L	35:44	1.0000	0.9995-1.0014	-1+3	155L	14	50	1.4	5	2.5
6	152	155L	36:07	1.0107	1.0093-1.0121	6	155L/156L/157L/167L/169L	14	50	1.4	5	2.5
6	150	155L	36:15	1.0145	1.0131-1.0159	6	155L/156L/157L/167L/169L	15	50	1.5	5	2.5
6	136	155L	36:44	1.0280	1.0266-1.0294	6	155L/156L/157L/167L/169L	16	50	1.6	5	2.5
6	145	155L	37:00	1.0354	1.0340-1.0368	6	155L/156L/157L/167L/169L	16	50	1.6	5	2.5

Cl No. [1]	Congener No. [2,3]	RT Ref [4]	RT [5]	RRT [6]	RRT limits [7]	Window (sec) [8]	Quantitation reference [9]	Detection limits and minimum levels - Matrix and concentration [10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
6	148	155L	34:26	1.0756	1.0742-1.0770	6	155L/156L/157L/167L/169L	14	50	1.4	5	2.5
6	151	155L	39:10	1.0961	1.0938-1.0984	10	155L/156L/157L/167L/169L	46	100	4.6	10	5
6	135	155L	39:17	1.0993	1.0970-1.1017	10	155L/156L/157L/167L/169L					
6	154	155L	39:21	1.1012	1.0989-1.1035	10	155L/156L/157L/167L/169L					
6	151/135/154	155L	39:15	1.0984	1.0961-1.1007	10	155L/156L/157L/167L/169L					
6	144	155L	39:47	1.1133	1.1119-1.1147	6	155L/156L/157L/167L/169L	15	50	1.5	5	2.5
6	147	155L	40:09	1.1236	1.1213-1.1259	10	155L/156L/157L/167L/169L	35	100	3.5	10	5
6	149	155L	40:12	1.1250	1.1227-1.1273	10	155L/156L/157L/167L/169L					
6	147/149	155L	40:10	1.1241	1.1217-1.1264	10	155L/156L/157L/167L/169L					
6	134	155L	40:27	1.1320	1.1297-1.1343	10	155L/156L/157L/167L/169L	33	100	3.3	10	5
6	143	155L	40:30	1.1334	1.1311-1.1357	10	155L/156L/157L/167L/169L					
6	134/143	155L	40:29	1.1329	1.1306-1.1353	10	155L/156L/157L/167L/169L					
6	139	155L	40:47	1.1413	1.1390-1.1437	10	155L/156L/157L/167L/169L	29	100	2.9	10	5
6	140	155L	40:48	1.1418	1.1395-1.1441	10	155L/156L/157L/167L/169L					
6	139/140	155L	40:47	1.1413	1.1390-1.1437	10	155L/156L/157L/167L/169L					
6	131	155L	41:03	1.1488	1.1474-1.1502	6	155L/156L/157L/167L/169L	17	50	1.7	5	2.5
6	142	155L	41:13	1.1535	1.1521-1.1549	6	155L/156L/157L/167L/169L	17	50	1.7	5	2.5
6	132	155L	41:36	1.1642	1.1618-1.1665	10	155L/156L/157L/167L/169L	16	50	1.6	5	2.5
6	133	155L	41:57	1.1740	1.1726-1.1754	6	155L/156L/157L/167L/169L	12	50	1.2	5	2.5
6	165	167L	42:23	0.8864	0.8853-0.8874	6	155L/156L/157L/167L/169L	13	50	1.3	5	2.5
6	146	167L	42:38	0.8916	0.8906-0.8926	6	155L/156L/157L/167L/169L	14	50	1.4	5	2.5
6	161	167L	42:47	0.8947	0.8937-0.8958	6	155L/156L/157L/167L/169L	13	50	1.3	5	2.5
6	153	167L	43:17	0.9052	0.9035-0.9069	10	155L/156L/157L/167L/169L	30	100	3.0	10	5
6	168	167L	43:21	0.9066	0.9048-0.9083	10	155L/156L/157L/167L/169L					
6	153/168	167L	43:19	0.9059	0.9041-0.9076	10	155L/156L/157L/167L/169L					
6	141	167L	43:34	0.9111	0.9101-0.9122	6	155L/156L/157L/167L/169L	17	50	1.7	5	2.5
6	130	167L	44:01	0.9205	0.9195-0.9216	6	155L/156L/157L/167L/169L	13	50	1.3	5	2.5
6	137	167L	44:14	0.9251	0.9240-0.9261	6	155L/156L/157L/167L/169L	15	50	1.5	5	2.5
6	164	167L	44:22	0.9278	0.9268-0.9289	6	155L/156L/157L/167L/169L	15	50	1.5	5	2.5
6	138	167L	44:42	0.9348	0.9324-0.9373	14	155L/156L/157L/167L/169L	63	200	6.3	20	10
6	163	167L	44:42	0.9348	0.9324-0.9373	14	155L/156L/157L/167L/169L					
6	129	167L	44:47	0.9366	0.9341-0.9390	14	155L/156L/157L/167L/169L					

Cl No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
6	160	167L	44:53	0.9387	0.9369-0.9404	10	155L/156L/157L/167L/169L					
6	138/163/129/160	167L	44:47	0.9366	0.9341-0.9390	14	155L/156L/157L/167L/169L					
6	158	167L	45:05	0.9428	0.9418-0.9439	6	155L/156L/157L/167L/169L	16	50	1.6	5	2.5
6	166	167L	45:59	0.9617	0.9599-0.9634	10	155L/156L/157L/167L/169L					
6	128	167L	46:09	0.9651	0.9634-0.9669	10	155L/156L/157L/167L/169L	29	100	2.9	10	5
6	128/166	167L	46:04	0.9634	0.9617-0.9651	10	155L/156L/157L/167L/169L					
6	159	167L	46:59	0.9826	0.9815-0.9836	6	155L/156L/157L/167L/169L	14	50	1.4	5	2.5
6	162	167L	47:18	0.9892	0.9881-0.9902	6	155L/156L/157L/167L/169L	13	50	1.3	5	2.5
6	167	167L	47:49	1.0000	0.9997-1.0010	-1+3	167L	13	50	1.3	5	2.5
6	156	156L/157L	49:05	0.9993	0.9983-1.0003	6	156L/157L					
6	157	156L/157L	49:09	1.0007	0.9990-1.0024	10	156L/157L	23	100	2.3	10	5
6	156/157	156L/157L	49:07	1.0000	0.9990-1.1010	6	156L/157L					
6	169	169L	52:31	1.0003	0.9997-1.0010	-1+3	169L	15	50	1.5	5	2.5
	Labeled compounds											
6	155L	138L	35:44	0.7997	0.7960-0.8034	20	138L					
6	167L	138L	47:49	1.0701	1.0664-1.0739	20	138L					
6	156L	138L	49:05	1.0985	1.0974-1.0996	20	138L					
6	157L	138L	49:08	1.0996	1.0959-1.1033	20	138L					
6	156L/157L	138L	49:07	1.0992	1.0981-1.1003	20	138L					
6	169L	138L	52:30	1.1749	1.1738-1.1761	20	138L					
Compounds using 194L($^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB) as Labeled injection internal standard												
	CB congener											
	Heptachlorobiphenyls											
7	188	188L	41:51	1.0000	0.9996-1.0012	-1+3	188L	15	50	1.5	5	2.5
7	179	188L	42:19	1.0112	1.0100-1.0123	6	188L/189L	14	50	1.4	5	2.5
7	184	188L	42:45	1.0215	1.0203-1.0227	6	188L/189L	14	50	1.4	5	2.5
7	176	188L	43:15	1.0335	1.0323-1.0346	6	188L/189L	12	50	1.2	5	2.5
7	186	188L	43:45	1.0454	1.0442-1.0466	6	188L/189L	15	50	1.5	5	2.5
7	178	188L	45:06	1.0777	1.0765-1.0789	6	188L/189L	14	50	1.4	5	2.5
7	175	188L	45:46	1.0936	1.0924-1.0948	6	188L/189L	14	50	1.4	5	2.5
7	187	188L	46:02	1.1000	1.0988-1.1012	6	188L/189L	17	50	1.7	5	2.5
7	182	188L	46:14	1.1047	1.1035-1.1059	6	188L/189L	13	50	1.3	5	2.5

Cl No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
7	183	188L	46:42	1.1159	1.1147-1.1171	6	188L/189L					
7	185	188L	46:53	1.1203	1.1191-1.1215	6	188L/189L	28	100	2.8	10	5
7	183/185	188L	46:47	1.1179	1.1167-1.1191	6	188L/189L					
7	174	188L	47:02	1.1239	1.1227-1.1251	6	188L/189L	15	50	1.5	5	2.5
7	177	188L	47:30	1.1350	1.1338-1.1362	6	188L/189L	11	50	1.1	5	2.5
7	181	188L	47:52	1.1438	1.1426-1.1450	6	188L/189L	13	50	1.3	5	2.5
7	171	188L	48:10	1.1509	1.1489-1.1529	10	188L/189L					
7	173	188L	48:11	1.1513	1.1501-1.1525	6	188L/189L	30	100	3.0	10	5
7	171/173	188L	48:10	1.1509	1.1489-1.1529	6	188L/189L					
7	172	189L	49:47	0.9035	0.9026-0.9044	6	188L/189L	13	50	1.3	5	2.5
7	192	189L	50:06	0.9093	0.9083-0.9102	6	188L/189L	13	50	1.3	5	2.5
7	193	189L	50:26	0.9153	0.9144-0.9162	6	188L/189L					
7	180	189L[11]	50:27	0.9156	0.9147-0.9165	6	188L/189L[11]	30	100	3.0	10	5
7	193/180	189L	50:26	0.9153	0.9144-0.9162	6	188L/189L					
7	191	189L	50:51	0.9229	0.9220-0.9238	6	188L/189L	13	50	1.3	5	2.5
7	170	189L[11]	51:54	0.9419	0.9410-0.9428	6	188L/189L[11]	12	50	1.2	5	2.5
7	190	189L	52:26	0.9516	0.9507-0.9525	6	188L/189L	14	50	1.4	5	2.5
7	189	189L	55:07	1.0003	0.9997-1.0009	-1+3	189L	13	50	1.3	5	2.5
	Octachlorobiphenyls											
8	202	202L	47:32	1.0004	0.9996-1.0011	-1+3	202L	24	100	2.4	10	5
8	201	202L	48:31	1.0210	1.0193-1.0228	10	202L/205L	20	50	2.0	5	2.5
8	204	202L	49:11	1.0351	1.0340-1.0361	6	202L/205L	21	50	2.1	5	2.5
8	197	202L	49:27	1.0407	1.0396-1.0417	6	202L/205L					
8	200	202L	49:40	1.0452	1.0442-1.0463	6	202L/205L	43	100	4.3	10	5
8	197/200	202L	49:33	1.0428	1.0417-1.0438	6	202L/205L					
8	198	202L	52:30	1.1049	1.1031-1.1066	10	202L/205L					
8	199	202L	52:32	1.1056	1.1045-1.1066	6	202L/205L	37	100	3.7	10	5
8	198/199	202L	52:31	1.1052	1.1035-1.1070	10	202L/205L					
8	196	205L	53:13	0.9207	0.9198-0.9216	6	202L/205L	20	50	2.0	5	2.5
8	203	205L	53:26	0.9245	0.9236-0.9253	6	202L/205L	18	50	1.8	5	2.5
8	195	205L	54:55	0.9501	0.9493-0.9510	6	202L/205L	22	50	2.2	5	2.5
8	194	205L	57:19	0.9916	0.9908-0.9925	6	202L/205L	18	50	1.8	5	2.5

Cl No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								MDL	ML	MDL	ML	ML
8	205	205L	57:49	1.0003	0.9997-1.0009	-1+3	205L	15	50	1.5	5	2.5
Nonachlorobiphenyls												
9	208	208L	54:33	1.0003	0.9997-1.0009	-1+3	208L	16	50	1.6	5	2.5
9	207	208L	55:32	1.0183	1.0174-1.0193	6	208L/206L	19	50	1.9	5	2.5
9	206	206L	59:37	1.0003	0.9997-1.0008	-1+3	206L	16	50	1.6	5	2.5
Decachlorobiphenyl												
10	209	209L	61:15	1.0003	0.9997-1.0008	-1+3	209L	16	50	1.6	5	2.5
Labeled compounds												
7	188L	194L	41:51	0.7304	0.7275-0.7333	20	194L					
7	180L	194L	50:27	0.8805	0.8775-0.8834	20	194L					
7	170L	194L	51:53	0.9055	0.9026-0.9084	20	194L					
7	189L	194L	55:06	0.9616	0.9587-0.9645	20	194L					
8	202L	194L	47:31	0.8293	0.8264-0.8322	20	194L					
8	205L	194L	57:48	1.0087	1.0044-1.0131	30	194L					
9	208L	194L	54:32	0.9517	0.9488-0.9546	20	194L					
9	206L	194L	59:36	1.0401	1.0358-1.0445	30	194L					
10	209L	194L	61:14	1.0686	1.0643-1.0730	30	194L					
Labeled clean-up standards												
3	28L	52L	26:44	0.9266	0.9209-0.9324	20	52L					
5	111L	101L	38:51	1.0777	1.0730-1.0823	20	101L					
7	178L	138L	45:05	1.0090	1.0052-1.0127	20	138L					
Labeled injection internal standards												
2	9L	138L	18:54	0.4230	0.4183-0.4276	25	138L					
4	52L	138L	28:45	0.6434	0.6388-0.6481	25	138L					
5	101L	138L	36:03	0.8068	0.8021-0.8115	25	138L					
6	138L	138L	44:41	1.0000	0.9996-1.0011	100	138L					
8	194L	138L	57:18	1.2824	1.2777-1.2870	25	138L					

1. Number of chlorines on congener.
2. Suffix “L” indicates labeled compound.
3. Multiple congeners in a box indicates congeners that co-elute or may not be adequately resolved on a 30-m SPB-octyl column.
4. Retention time (RT) reference used to locate target congener.
5. Retention time of target congener.

6. Relative retention time (RRT) between the RT for the congener and RT for the reference.
7. RRT limits based on RT window. RTs, RRTs, and RRT limits may differ slightly from those in Table 2.
8. RT window width necessary to attempt to unambiguously identify the congener in the presence of other congeners.
9. Labeled congeners that form the quantitation reference. Areas from the exact m/z's of the congeners listed in the quantitation reference are summed, and divided by the number of congeners in the quantitation reference. For example, for congener 10, the areas at the exact m/z's for 4L and 15L are summed and the sum is divided by 2 (because there are 2 congeners in the quantitation reference).
10. MDLs for water pooled from data from AXYS Analytical, TestAmerica-Knoxville, and Battelle-Columbus (see Reference 24). MLs for water per ML procedure at 68 FR 11790. MDLs and MLs for "Other" and "Extract" calculated from sample amount and extract volume.
11. If congeners 170L and 180L are included in the calibration and spiking solutions, these congeners should be used as RT and quantitation references.

Table 3. Concentrations of Native and Labeled Chlorinated Biphenyls in Stock Solutions Spiking Solutions

CB Congener	Solution Concentrations		
	Stock (µg/mL)	Spiking (ng/mL)	Extract (ng/mL)
Native toxics/LOC[1]			
1	20	1.0	50
3	20	1.0	50
4	20	1.0	50
15	20	1.0	50
19	20	1.0	50
37	20	1.0	50
54	20	1.0	50
77	20	1.0	50
81	20	1.0	50
104	20	1.0	50
105	20	1.0	50
114	20	1.0	50
118	20	1.0	50
123	20	1.0	50
126	20	1.0	50
155	20	1.0	50
156	20	1.0	50
157	20	1.0	50
167	20	1.0	50
169	20	1.0	50
188	20	1.0	50
189	20	1.0	50
202	20	1.0	50
205	20	1.0	50
206	20	1.0	50
208	20	1.0	50
209	20	1.0	50
Native congener mix stock solutions[2]			
MoCB thru TrCB	2.5		
TeCB thru HpCB	5.0		
OcCB thru DeCB	7.5		
Labeled toxics/LOC/window-defining[3]			
1L	1.0	2.0	100
3L	1.0	2.0	100
4L	1.0	2.0	100
15L	1.0	2.0	100
19L	1.0	2.0	100
37L	1.0	2.0	100
54L	1.0	2.0	100
77L	1.0	2.0	100
81L	1.0	2.0	100
104L	1.0	2.0	100
105L	1.0	2.0	100

Table 3. Concentrations of Native and Labeled Chlorinated Biphenyls in Stock Solutions Spiking Solutions

CB Congener	Solution Concentrations		
	Stock (µg/mL)	Spiking (ng/mL)	Extract (ng/mL)
114L	1.0	2.0	100
118L	1.0	2.0	100
123L	1.0	2.0	100
126L	1.0	2.0	100
155L	1.0	2.0	100
156L	1.0	2.0	100
157L	1.0	2.0	100
167L	1.0	2.0	100
169L	1.0	2.0	100
188L	1.0	2.0	100
189L	1.0	2.0	100
202L	1.0	.2.0	100
205L	1.0	2.0	100
206L	1.0	2.0	100
208L	1.0	2.0	100
209L	1.0	2.0	100
Labeled clean-up[4]			
28L	1.0	2.0	100
111L	1.0	2.0	100
178L	1.0	2.0	100
Labeled injection internal[5]			
9L	5.0	1000	100
52L	5.0	1000	100
101L	5.0	1000	100
138L	5.0	1000	100
194L	5.0	1000	100

Diluted combined 209 congener[6]	**Solution Concentration (ng/mL)**	
Standard	**Native**	**Labeled**
Native congeners		
MoCB thru TrCB	25	
TeCB thru HpCB	50	
OcCB thru DeCB	75	
Labeled toxics/LOC/window-defining		100
Labeled cleanup		100
Labeled injection internal		100

1. Stock solution: Section 7.8.1; Spiking solution: Section 7.11
2. Section 7.8.2.1
3. Stock solution: Section 7.9.1; Spiking solution: Section 7.12
4. Stock solution: Section 7.9.2; Spiking solution: Section 7.13
5. Stock solution: Section 7.9.3; Spiking solution: Section 7.14
6. Section 7.10.2.2.2

Table 4. Composition of Individual Native CB Congener Solutions[1]

Solution Identifier				
A2	**B2**	**C2**	**D2**	**E2**
Accu-Standard part number				
M-1668A-1	**M-1668A-2**	**M-1668A-3**	**M-1668A-4**	**M-1668A-5**
2	7	13	25	1
10	5	17	21	3
9	12	29	69	4
6	18	20	47	15
8	24	46	42	19
14	23	65	a64	16
11	28	59	70	37
30	22	40	102	54
27	39	67	97	43
32	53	76	115	44
34	51	80	123	74
26	73	93	134	56
31	48	84	131	77
33	62	101	163	104
36	71	112	180	98
38	68	86		125
35	58	116		110
50	61	107		126
45	55	154		155
52	60	147		138
49	94	140		169
75	100	146		188
41	91	141		189
72	121	164		202
57	90	158		205
63	99	182		208
66	109	174		206
79	117	173		209
78	111	193		
81	108			
96	118			
103	114			
95	150			
88	145			
89	135			
92	149			
113	139			
83	132			
119	165			
87	168			
85	137			
82	160			
120	128			
124	162			
106	157			

Table 4. Composition of Individual Native CB Congener Solutions[1]

Solution Identifier				
A2	**B2**	**C2**	**D2**	**E2**
Accu-Standard part number				
M-1668A-1	**M-1668A-2**	**M-1668A-3**	**M-1668A-4**	**M-1668A-5**
122	184			
105	186			
127	187			
152	185			
136	181			
148	192			
151	197			
144	199/201			
143	203			
142				
133				
161				
153				
130				
129				
166				
159				
167				
156				
179				
176				
178				
175				
183				
177				
171				
172				
191				
170				
190				
201/200				
204				
200/199				
198				
196				
195				
194				
207				
Total number of congeners				
83	54	29	15	28

1. Congeners present in each standard solution are listed in elution order for each level of chlorination. Congener number (Table 1) listed first; BZ number listed second, where ambiguous. See Table 3 for concentrations of congeners in stock solutions and Table 5 for concentrations in calibration standards.

Table 5. Concentration of Congeners in Calibration and Calibration Verification Standards

Congener Name	Congener No.[1]	Solution Concentration (ng/mL)					
		CS-0.2 (Hi sens)[2]	CS-1	CS-2	CS-3 (VER)	CS-4	CS-5
Native toxics/LOC							
2-MoCB	1	0.20	1.0	5.0	50	400	2000
4-MoCB	3	0.20	1.0	5.0	50	400	2000
2,2'-DiCB	4	0.20	1.0	5.0	50	400	2000
4,4'-DiCB	15	0.20	1.0	5.0	50	400	2000
2,2',6'-TrCB	19	0.20	1.0	5.0	50	400	2000
3,4,4'-TrCB	37	0.20	1.0	5.0	50	400	2000
2,2',6,6'-TeCB	54	0.20	1.0	5.0	50	400	2000
3,3',4,4'-TeCB	77	0.20	1.0	5.0	50	400	2000
3,4,4',5-TeCB	81	0.20	1.0	5.0	50	400	2000
2,2',4,6,6'-PeCB	104	0.20	1.0	5.0	50	400	2000
2,3,3',4,4'-PeCB	105	0.20	1.0	5.0	50	400	2000
2,3,4,4',5-PeCB	114	0.20	1.0	5.0	50	400	2000
2,3',4,4',5-PeCB	118	0.20	1.0	5.0	50	400	2000
2',3,4,4',5-PeCB	123	0.20	1.0	5.0	50	400	2000
3,3',4,4',5-PeCB	126	0.20	1.0	5.0	50	400	2000
2,2',4,4',6,6'-HxCB	155	0.20	1.0	5.0	50	400	2000
2,3,3',4,4',5-HxCB	156	0.20	1.0	5.0	50	400	2000
2,3,3',4,4',5'-HxCB	157	0.20	1.0	5.0	50	400	2000
2,3',4,4',5,5'-HxCB	167	0.20	1.0	5.0	50	400	2000
3,3',4,4',5,5'-HxCB	169	0.20	1.0	5.0	50	400	2000
2,2',3,4',5,6,6'-HpCB	188	0.20	1.0	5.0	50	400	2000
2,3,3',4,4',5,5'-HpCB	189	0.20	1.0	5.0	50	400	2000
2,2',3,3',5,5',6,6'-OcCB	202	0.20	1.0	5.0	50	400	2000
2,3,3',4,4',5,5',6-OcCB	205	0.20	1.0	5.0	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB	206	0.20	1.0	5.0	50	400	2000
2,2',3,3',4',5,5',6,6'-NoCB	208	0.20	1.0	5.0	50	400	2000
DeCB	209	0.20	1.0	5.0	50	400	2000
Labeled toxics/LOC/window-defining							
$^{13}C_{12}$-2-MoCB	1L	100	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	3L	100	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	4L	100	100	100	100	100	100
$^{13}C_{12}$-4,4'-DiCB	15L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6'-TrCB	19L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100

Table 5. Concentration of Congeners in Calibration and Calibration Verification Standards

Congener Name	Congener No.[1]	Solution Concentration (ng/mL)					
		CS-0.2 (Hi sens)[2]	CS-1	CS-2	CS-3 (VER)	CS-4	CS-5
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4',5,5',6,6'-NoCB	208L	100	100	100	100	100	100
$^{13}C_{12}$-DeCB	209L	100	100	100	100	100	100
Labeled clean-up							
$^{13}C_{12}$-2,4,4'-TrCB	28L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	100	100	100	100	100
Labeled injection internal							
$^{13}C_{12}$-2,5-DiCB	9L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100

1. Suffix "L" indicates labeled compound.
2. Additional concentration used for calibration of high sensitivity HRGC/HRMS systems. If the ion abundance ratio (Table 8) cannot be achieved at this level (see Section 10.3.3), a calibration point at 0.4 or 0.5 ng/mL may be used.

Table 6. QC Acceptance Criteria for VER, IPR, OPR, and Labeled Compounds in Samples[1,2]

Congener Name	Congener No.[3]	Test Conc. (ng/mL)[4]	VER (%)[5]	IPR		OPR Recovery (%)	Labeled Compound Recovery in Samples (%)
				RSD (%)	Mean Recovery (%)		
2-MoCB	1	50	75 - 125	25	70 - 130	60 - 135	
4-MoCB	3	50	75 - 125	25	70 - 130	60 - 135	
2,2'-DiCB	4	50	75 - 125	25	70 - 130	60 - 135	
4,4'-DiCB	15	50	75 - 125	25	70 - 130	60 - 135	
2,2'6-TrCB	19	50	75 - 125	25	70 - 130	60 - 135	
3,4,4'-TrCB	37	50	75 - 125	25	70 - 130	60 - 135	
2,2'6,6'TeCB	54	50	75 - 125	25	70 - 130	60 - 135	
3,3',4,4'-TeCB	77	50	75 - 125	25	70 - 130	60 - 135	
3,4,4',5-TeCB	81	50	75 - 125	25	70 - 130	60 - 135	
2,2',4,6,6'-PeCB	104	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4'-PeCB	105	50	75 - 125	25	70 - 130	60 - 135	
2,3,4,4',5-PeCB	114	50	75 - 125	25	70 - 130	60 - 135	
2,3',4,4',5-PeCB	118	50	75 - 125	25	70 - 130	60 - 135	
2',3,4,4',5-PeCB	123	50	75 - 125	25	70 - 130	60 - 135	NA
3,3',4,4',5-PeCB	126	50	75 - 125	25	70 - 130	60 - 135	
2,2',4,4',6,6'-HxCB	155	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4',5-HxCB [6]	156	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4',5'-HxCB [6]	157	50	75 - 125	25	70 - 130	60 - 135	
2,3',4,4',5,5'-HxCB	167	50	75 - 125	25	70 - 130	60 - 135	
3,3',4,4',5,5'-HxCB	169	50	75 - 125	25	70 - 130	60 - 135	
2,2',3,4',5,6,6'-HpCB	188	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4',5,5'-HpCB	189	50	75 - 125	25	70 - 130	60 - 135	
2,2',3,3',5,5',6,6'-OcCB	202	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4',5,5',6-OcCB	205	50	75 - 125	25	70 - 130	60 - 135	
2,2',3,3',4,4',5,5',6-NoCB	206	50	75 - 125	25	70 - 130	60 - 135	
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	75 - 125	25	70 - 130	60 - 135	
DeCB	209	50	75 - 125	25	70 - 130	60 - 135	
$^{13}C_{12}$-2-MoCB	1L	100	50 - 145	70	20 - 135	15 - 145	5 - 145
$^{13}C_{12}$-4-MoCB	3L	100	50 - 145	70	20 - 135	15 - 145	5 - 145
$^{13}C_{12}$-2,2'-DiCB	4L	100	50 - 145	70	20 - 135	15 - 145	5 - 145
$^{13}C_{12}$-4,4'-DiCB	15L	100	50 - 145	70	20 - 135	15 - 145	5 - 145
$^{13}C_{12}$-2,2',6-TrCB	19L	100	50 - 145	70	20 - 135	15 - 145	5 - 145
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	50 - 145	70	20 - 135	15 - 145	5 - 145
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	50 - 145	70	20 - 135	15 - 145	5 - 145

Table 6. QC Acceptance Criteria for VER, IPR, OPR, and Labeled Compounds in Samples[1,2]

Congener Name	Congener No.[3]	Test Conc. (ng/mL)[4]	VER (%)[5]	IPR		OPR Recovery (%)	Labeled Compound Recovery in Samples (%)
				RSD (%)	Mean Recovery (%)		
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,3,3',4,4',5 -HxCB[6]	156L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[6]	157L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB	189L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	50 - 145	50	45 - 135	40 - 145	10 - 145
Cleanup standards							
$^{13}C_{12}$-2,4,4'-TrCB	28L	100	65 - 135	70	20 - 135	15 - 145	5 - 145
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	75 - 125	50	45 - 135	40 - 145	10 - 145
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	75 - 125	50	45 - 135	40 - 145	10 - 145

1. Reference 22 describes how interlaboratory results were pooled from analyses of wastewater, biosolids, and fish tissue samples.
2. QC acceptance criteria for IPR, OPR, and samples based on a 20-µL extract final volume
3. Suffix "L" indicates labeled compound.
4. See Table 5.
5. Section 15.3.
6. CBs 156/157 and 156L/157L are tested as the sum of the two congeners

NA = Not applicable

Table 7. Scan Descriptors, Levels of Chlorination, m/z Information, and Substances Monitored by HRGC/HRMS

Function and Chlorine Level	m/z[1]	m/z Type	m/z Formula	Substance
Fn-1; Cl-1	188.0393	M	$^{12}C_{12}H_9{}^{35}Cl$	Cl-1 CB
	190.0363	M+2	$^{12}C_{12}H_9{}^{37}Cl$	Cl-1 CB
	200.0795	M	$^{13}C_{12}H_9{}^{35}Cl$	$^{13}C_{12}$ Cl-1 CB
	202.0766	M+2	$^{13}C_{12}H_9{}^{37}Cl$	$^{13}C_{12}$ Cl-1 CB
	218.9856	lock	C_4F_9	PFK
Fn-2; Cl-2, 3	222.0003	M	$^{12}C_{12}H_8{}^{35}Cl_2$	Cl-2 PCB
	223.9974 (2)	M+2	$^{12}C_{12}H_8{}^{35}Cl{}^{37}Cl$	Cl-2 PCB
	225.9944	M+4	$^{12}C_{12}H_8{}^{37}Cl_2$	Cl-2 PCB
	234.0406	M	$^{13}C_{12}H_8{}^{35}Cl_2$	$^{13}C_{12}$ Cl-2 PCB
	236.0376	M+2	$^{13}C_{12}H_8{}^{35}Cl{}^{37}Cl$	$^{13}C_{12}$ Cl-2 PCB
	242.9856	lock	C_6F_9	PFK
	255.9613	M	$^{12}C_{12}H_7{}^{35}Cl_3$	Cl-3 PCB
	257.9584	M+2	$^{12}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Cl-3 PCB
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	$^{13}C_{12}$ Cl-3 PCB
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	$^{13}C_{12}$ Cl-3 PCB
Fn-3; Cl-3, 4, 5	255.9613	M	$^{12}C_{12}H_7{}^{35}Cl_3$	Cl-3 PCB
	257.9584	M+2	$^{12}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Cl-3 PCB
	259.9554	M+4	$^{12}C_{12}H_7{}^{35}Cl{}^{37}Cl_2$	Cl-3 PCB
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	$^{13}C_{12}$ Cl-3 PCB
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	$^{13}C_{12}$ Cl-3 PCB
	280.9825	lock	C_6F_{11}	PFK
	289.9224	M	$^{12}C_{12}H_6{}^{35}Cl_4$	Cl-4 PCB
	291.9194	M+2	$^{12}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	Cl-4 PCB
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_4$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{13}C_{12}H5{}^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}H5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}H5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
Fn-4; Cl-4, 5, 6	289.9224	M	$^{12}C_{12}H_6{}^{35}Cl_4$	Cl-4 PCB
	291.9194	M+2	$^{12}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	Cl-4 PCB
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}H_5{}^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	330.9792	lock	C_7F_{15}	PFK
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB

Table 7. Scan Descriptors, Levels of Chlorination, m/z Information, and Substances Monitored by HRGC/HRMS

Function and Chlorine Level	m/z[1]	m/z Type	m/z Formula	Substance
Fn-4; Cl-4, 5, 6	359.8415	M+2	$^{12}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Cl-6 PCB
	361.8385	M+4	$^{12}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{12}C_{12}H_4{}^{35}Cl_3{}^{37}Cl_3$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
Fn-5; Cl-5, 6, 7	323.8834	M	$^{12}C_{12}H_5{}^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	354.9792	lock	C_9F_{13}	PFK
	359.8415	M+2	$^{12}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Cl-6 PCB
	361.8385	M+4	$^{12}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{12}C_{12}H_4{}^{35}Cl_3{}^{37}Cl_3$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
	393.8025	M+2	$^{12}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Cl-7 PCB
	395.7995	M+4	$^{12}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Cl-7 PCB
	397.7966	M+6	$^{12}C_{12}H_3{}^{35}Cl_4{}^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	454.9728	QC	$C_{11}F_{17}$	PFK
Fn-6; Cl-7, 8, 9, 10	393.8025	M+2	$^{12}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Cl-7 PCB
	395.7995	M+4	$^{12}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Cl-7 PCB
	397.7966	M+6	$^{12}C_{12}H_3{}^{35}Cl_4{}^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	427.7635	M+2	$^{12}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Cl-8 PCB
	429.7606	M+4	$^{12}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Cl-8 PCB
	431.7576	M+6	$^{12}C_{12}H_2{}^{35}Cl_5{}^{37}Cl_3$	Cl-8 PCB
	439.8038	M+2	$^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	$^{13}C_{12}$ Cl-8 PCB
	441.8008	M+4	$^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	$^{13}C_{12}$ Cl-8 PCB
	442.9728	QC	$C_{10}F_{13}$	PFK
	454.9728	lock	$C_{11}F_{13}$	PFK
	461.7246	M+2	$^{12}C_{12}H_1{}^{35}Cl_8{}^{37}Cl$	Cl-9 PCB
	463.7216	M+4	$^{12}C_{12}H_1{}^{35}Cl_7{}^{37}Cl_2$	Cl-9 PCB
	465.7187	M+6	$^{12}C_{12}H_1{}^{35}Cl_6{}^{37}Cl_3$	Cl-9 PCB
	473.7648	M+2	$^{13}C_{12}H_1{}^{35}Cl_8{}^{37}Cl$	$^{13}C_{12}$ Cl-9 PCB
	475.7619	M+4	$^{13}C_{12}H_1{}^{35}Cl_7{}^{37}Cl_2$	$^{13}C_{12}$ Cl-9 PCB
	495.6856	M+2	$^{12}C_{12}H_4{}^{35}Cl_9{}^{37}Cl$	Cl-10 PCB
	497.6826	M+4	$^{12}C_{12}{}^{35}Cl_8{}^{37}Cl_2$	Cl-10 PCB
	499.6797	M+6	$^{12}C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Cl-10 PCB

Table 7. Scan Descriptors, Levels of Chlorination, m/z Information, and Substances Monitored by HRGC/HRMS

Function and Chlorine Level	m/z[1]	m/z Type	m/z Formula	Substance
Fn-6; Cl-7, 8, 9, 10	507.7258	M+2	$^{13}C_{12}\ ^{35}Cl_9\ ^{37}Cl$	$^{13}C_{12}$ Cl-10 PCB
	509.7229	M+4	$^{13}C_{12}\ ^{35}Cl_8\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-10 PCB
	511.7199	M+6	$^{13}C_{12}\ ^{35}Cl_7\ ^{37}Cl_3$	$^{13}C_{12}$ Cl-10 PCB

1. Isotopic masses used for accurate mass calculation

^{1}H	1.0078
^{12}C	12.0000
^{13}C	13.0034
^{35}Cl	34.9689
^{37}Cl	36.9659
^{19}F	18.9984

2. An interference with PFK m/z 223.9872 may preclude meeting 10:1 S/N for the DiCB congeners at the CS-0.2 and CS-1 calibration levels (Section 10.3.3 and Table 5). If this interferences occurs, 10:1 S/N must be met at the CS-2 level. See the note at Section 10.2.1 for information on how to minimize this interference.

Table 8. Theoretical Ion Abundance Ratios and QC Limits

Chlorine Atoms	m/z's Forming Ratio	Theoretical Ratio	Lower QC Limit	Upper QC Limit
1	M/(M+2)	3.13	2.66	3.60
2	M/(M+2)	1.56	1.33	1.79
3	M/(M+2)	1.04	0.88	1.20
4	M/(M+2)	0.77	0.65	0.89
5	(M+2)/(M+4)	1.55	1.32	1.78
6	(M+2)/(M+4)	1.24	1.05	1.43
7	(M+2)/(M+4)	1.05	0.89	1.21
8	(M+2)/(M+4)	0.89	0.76	1.02
9	(M+2)/(M+4)	0.77	0.65	0.89
10	(M+4)(M+6)	1.16	0.99	1.33

Table 9. Suggested Sample Quantities to be Extracted for Various Matrices[1]

Sample Matrix[2]	Example	Percent Solids	Phase	Quantity Extracted
Single-phase				
Aqueous	Drinking water	<1	– [3]	1000 mL
	Groundwater			
	Treated wastewater			
Solid	Dry soil	>20	Solid	10 g
	Compost			
	Ash			
Organic	Waste solvent	<1	Organic	10 g
	Waste oil			
	Organic polymer			
Tissue	Fish	–	Organic	10 g
	Human adipose			
Multi-phase - Liquid/Solid				
Aqueous/Solid	Wet soil	1-30	Solid	10 g
	Untreated effluent			
	Digested municipal sludge			
	Filter cake			
	Paper pulp			
Organic/solid	Industrial sludge	1-100	Both	10 g
	Oily waste			
Multi-phase - Liquid/Liquid				
Aqueous/organic	In-process effluent	<1	Organic	10 g
	Untreated effluent			
	Drum waste			
Multi-phase - Liquid/Liquid/Solid				
Aqueous/organic/solid	Untreated effluent	>1	Organic and solid	10 g
	Drum waste			

1. The quantity of sample to be extracted is adjusted to provide 10 g of solids (dry weight). One liter of aqueous samples containing one percent solids will contain 10 grams of solids. For aqueous samples containing greater than one percent solids, a lesser volume is used so that 10 grams of solids (dry weight) will be extracted. Other sample volumes may be used to meet project needs.

2. The sample matrix may be amorphous for some samples. In general, when the CBs are in contact with a multi-phase system in which one of the phases is water, they will be preferentially dispersed in or adsorbed on the alternate phase because of their low solubility in water.

3. Aqueous samples are filtered after spiking with the labeled compounds. The filtrate and the materials trapped on the filter are extracted separately, and the extracts are combined for cleanup and analysis.

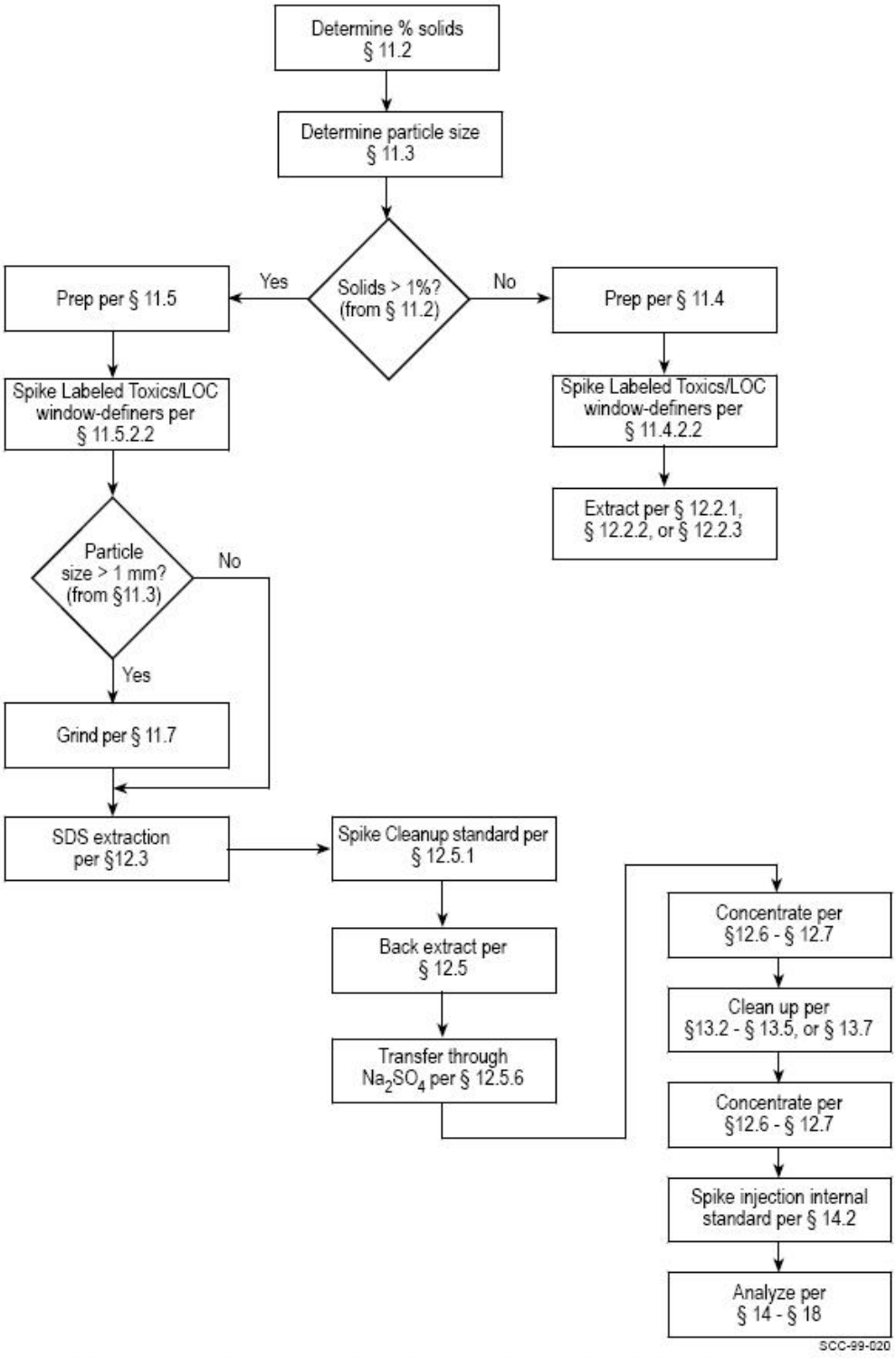

Figure 1 Flow Chart for Analysis of Aqueous and Solid Samples

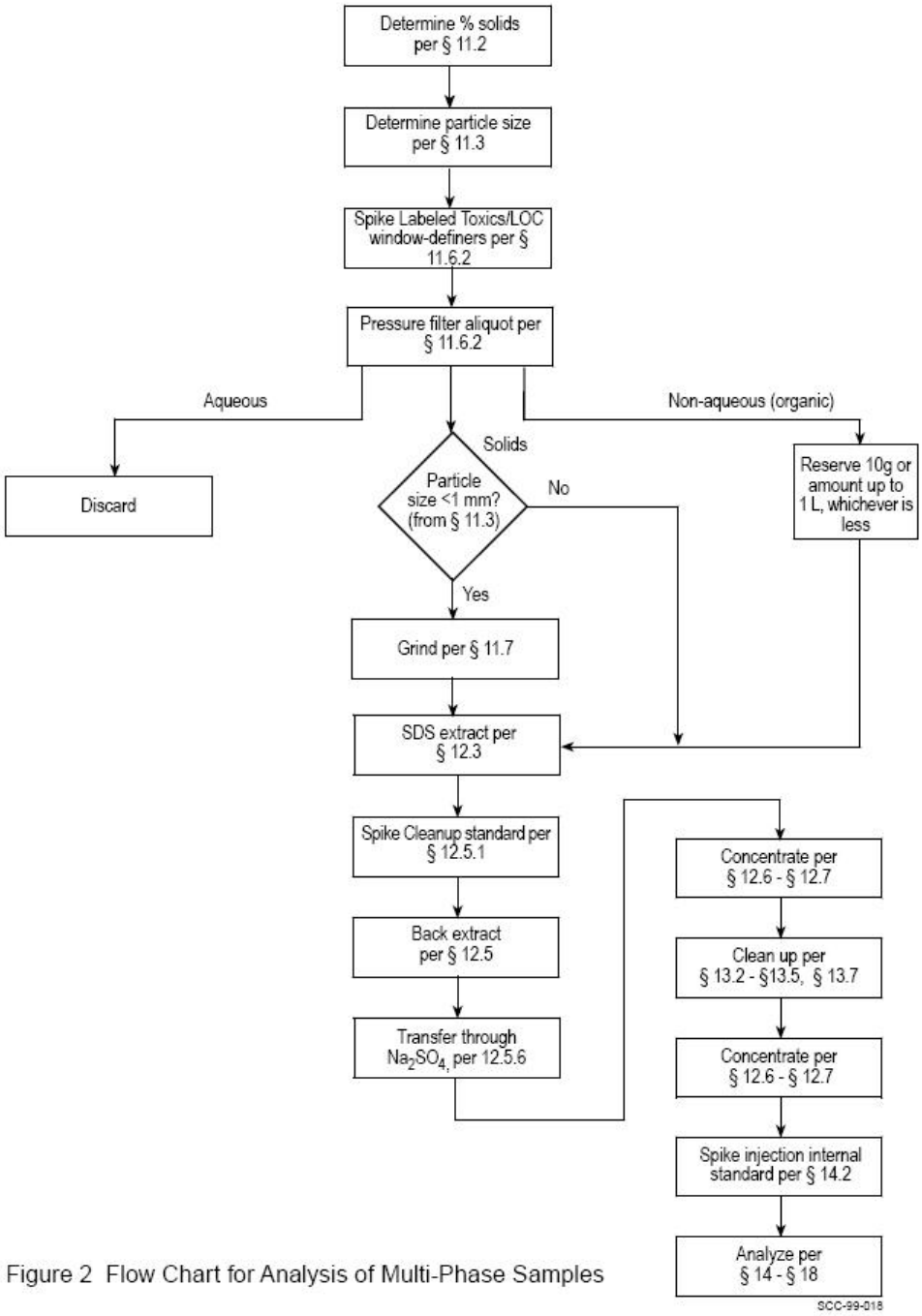

Figure 2 Flow Chart for Analysis of Multi-Phase Samples

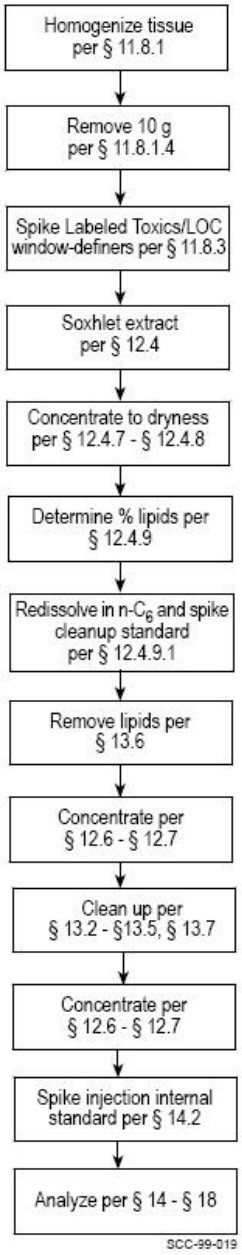

Figure 3 Flow Chart for Analysis of Tissue Samples

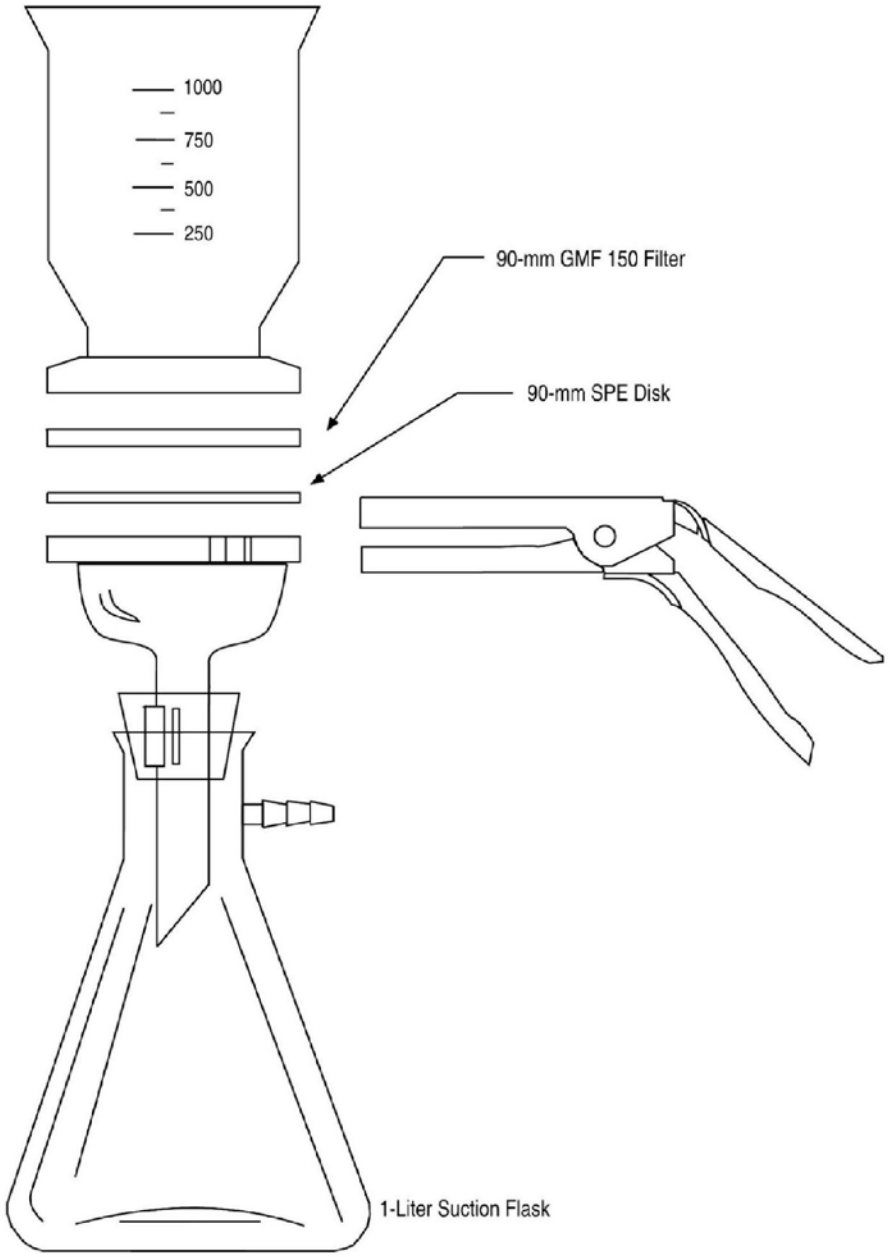

Figure 4. Solid-phase Extraction Apparatus

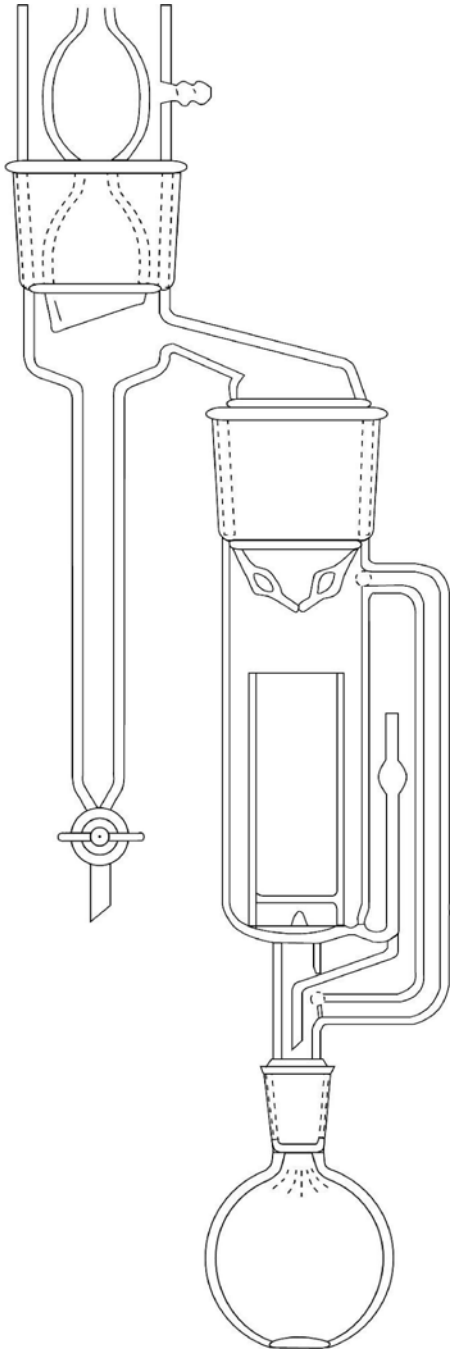

Figure 5. Soxhlet/Dean Stark Extractor

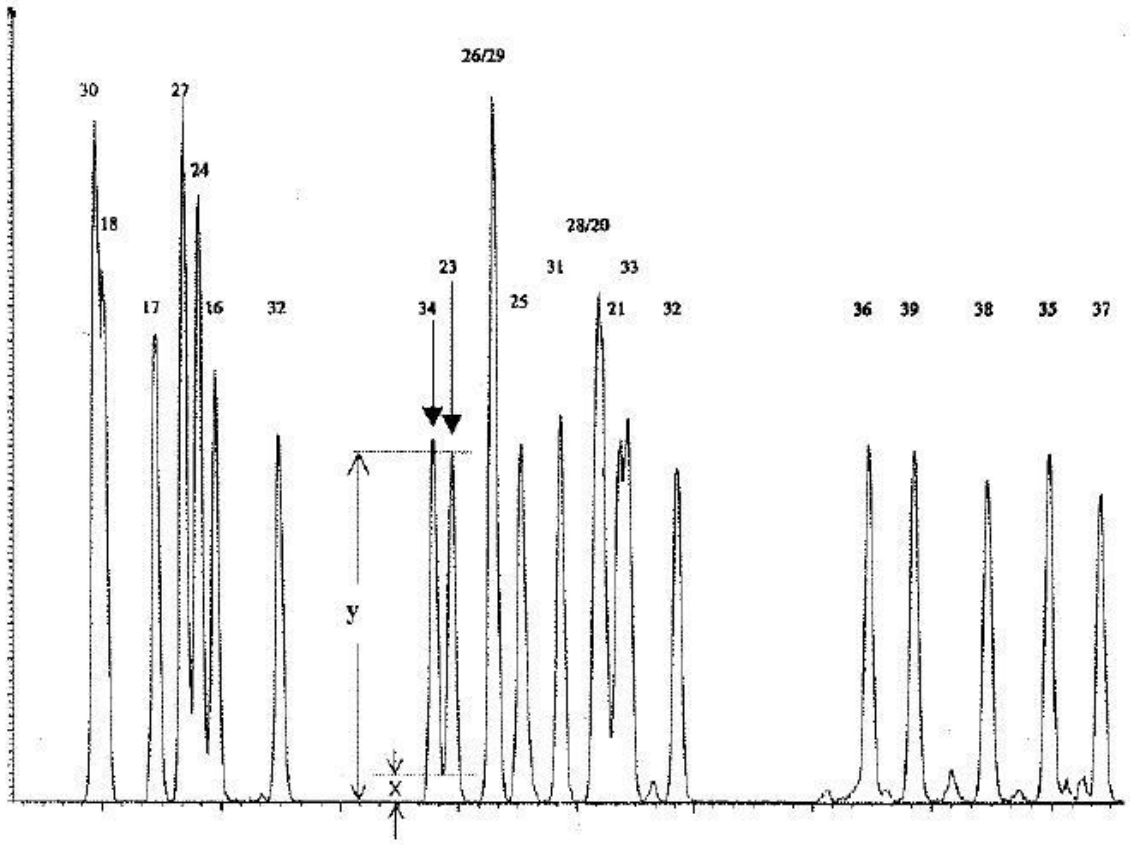

Figure 6 Octyl column resolution test #1: Separation of Cl-3 congeners 34 and 23 with valley <40% (i.e. 100x/y < 40%)

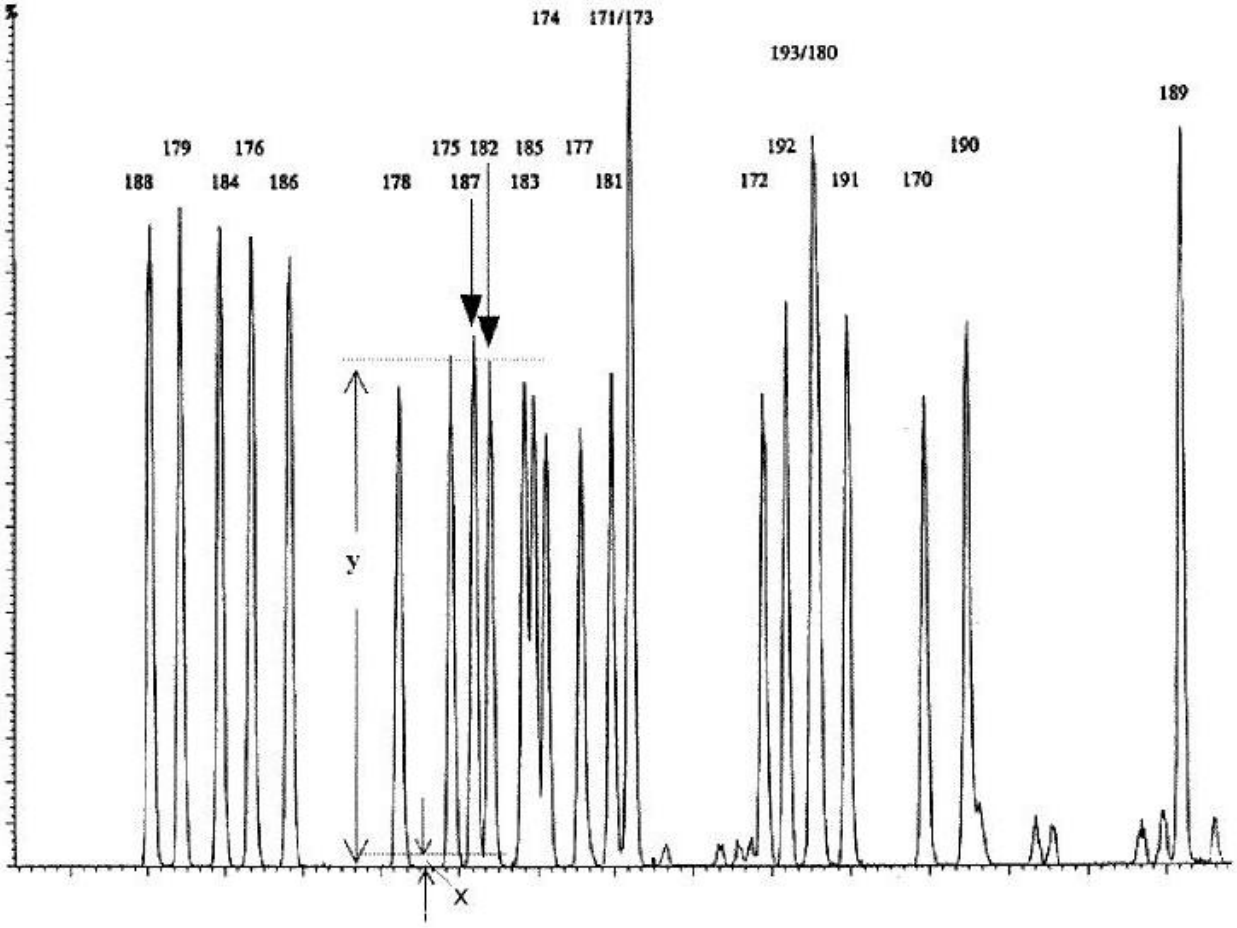

Figure 7 Octyl column resolution test #2: Separation of Cl-7 congeners 187 and 182 with valley < 40% (i.e. 100 x/y < 40%)

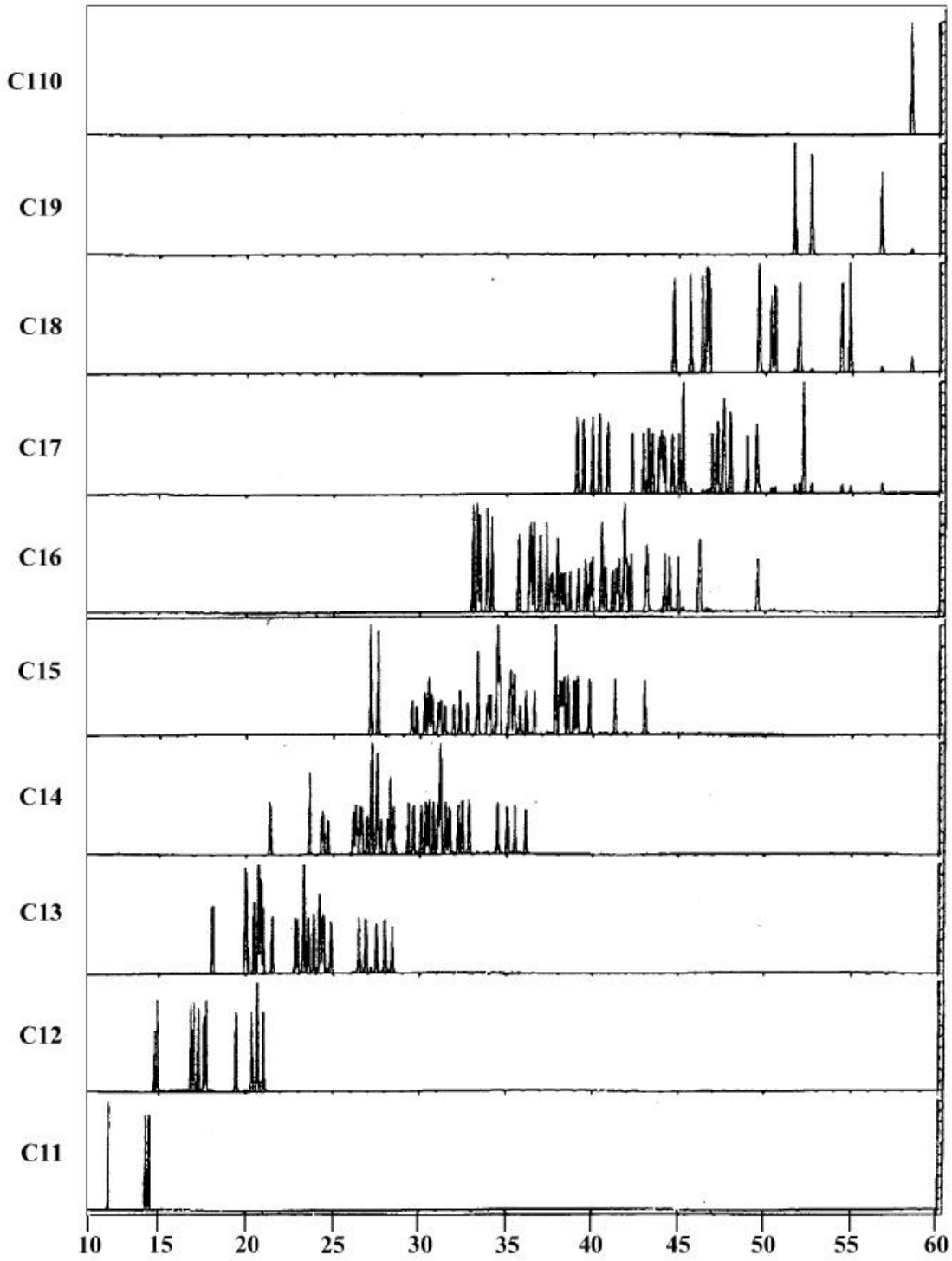

Figure 8. CB congeners at each level of chlorination on the SPB-octyl column

24.0 Glossary

These definitions and purposes are specific to this method, but have been conformed to common usage to the extent possible.

24.1 Units of weight and measure and their abbreviations

24.1.1 Symbols

°C	degrees Celsius
µL	microliter
µm	micrometer
<	less than
>	greater than
%	percent

24.1.2 Alphabetical abbreviations

cm	centimeter
g	gram
h	hour
ID	inside diameter
in.	inch
L	liter
M	molecular ion
m	meter
mg	milligram
min	minute
mL	milliliter
mm	millimeter
m/z	mass-to-charge ratio
N	normal; gram molecular weight of solute divided by hydrogen equivalent of solute, per liter of solution
OD	outside diameter
pg	picogram
ppb	part-per-billion
ppm	part-per-million
ppq	part-per-quadrillion
ppt	part-per-trillion
psig	pound-per-square-inch gauge
v/v	volume per unit volume
w/v	weight per unit volume

24.2 Definitions and acronyms (in alphabetical order)

Analyte – A CB tested for by this method. The analytes are listed in Table 1.

Calibration standard (CAL) – A solution prepared from a secondary standard and/or stock solutions and used to calibrate the response of the HRGC/HRMS instrument.

Calibration verification standard (VER) – The mid-point calibration standard (CS-3) that is used to verify calibration. See Table 5.

CB – Chlorinated biphenyl congener. One of the 209 individual chlorinated biphenyl congeners determined using this method. The 209 CBs are listed in Table 1.

CS-0.2, CS-1, CS-2, CS-3, CS-4, CS-5 – See Calibration standards and Table 5

DeCB – Decachlorobiphenyl (PCB 209)

DiCB – Dichlorobiphenyl

Field blank – An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratory or the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.

GC – Gas chromatograph or gas chromatography

GPC – Gel permeation chromatograph or gel permeation chromatography

HpCB – Heptachlorobiphenyl

HPLC – High performance liquid chromatograph or high performance liquid chromatography

HRGC – High resolution GC

HRMS – High resolution MS

HxCB – Hexachlorobiphenyl

Labeled injection internal standard – All five, or any one of the five, $^{13}C_{12}$-labeled CB congeners spiked into the concentrated extract immediately prior to injection of an aliquot of the extract into the HRGC/HRMS. The five Labeled injection internal standards in this method are CBs with congener numbers 9L, 52L, 101L, 138L, and 194L.

Internal standard – a labeled compound used as a reference for quantitation of other labeled compounds and for quantitation of native CB congeners other than the congener of which it is a labeled analog. See Internal standard quantitation.

Internal standard quantitation – A means of determining the concentration of (1) a naturally occurring (native) compound by reference to a compound other than its labeled analog and (2) a labeled compound by reference to another labeled compound

IPR – Initial precision and recovery; four aliquots of a reference matrix spiked with the analytes of interest and labeled compounds and analyzed to establish the ability of the laboratory to generate acceptable precision and recovery. An IPR is performed prior to the first time this method is used and any time the method or instrumentation is modified.

Isotope dilution quantitation – A means of determining a naturally occurring (native) compound by reference to the same compound in which one or more atoms has been isotopically enriched. In this method, all 12 carbon atoms in the biphenyl molecule are enriched with carbon-13 to produce $^{13}C_{12}$- labeled analogs of the chlorinated biphenyls. The $^{13}C_{12}$-labeled CBs are spiked into each

sample and allow identification and correction of the concentration of the native compounds in the analytical process.

K-D – Kuderna-Danish concentrator; a device used to concentrate the analytes in a solvent

Laboratory blank – See Method blank

Laboratory control sample (LCS) – See Ongoing precision and recovery standard (OPR)

Laboratory reagent blank – See Method blank

May – This action, activity, or procedural step is neither required nor prohibited.

May not – This action, activity, or procedural step is prohibited.

Method blank – An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

Method Detection Limit – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero (40 CFR 136, Appendix B)

Minimum level of quantitation (ML) – The lowest level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. The ML represents the lowest concentration at which an analyte can be measured with a known level of confidence. It may be equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed. The ML is calculated by multiplying the MDL (pooled or unpooled, as appropriate) by 3.18 and rounding the result to the number nearest to 1, 2, or 5 x 10^n, where n is zero or an integer (see 68 FR 11790).

MoCB – Monochlorobiphenyl

MS – Mass spectrometer or mass spectrometry

Must – This action, activity, or procedural step is required.

NoCB – Nonachlorobiphenyl

OcCB – Octachlorobiphenyl

OPR – Ongoing precision and recovery standard (OPR); a method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

Perfluorokerosene (PFK) – A mixture of compounds used to calibrate the exact m/z scale in the HRMS

Preparation blank – See Method blank

Quality control check sample (QCS) – A sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

PeCB – Pentachlorobiphenyl

PCB – Polychlorinated biphenyl

Reagent water – Water demonstrated to be free from the analytes of interest and potentially interfering substances at the method detection limit for the analyte.

Relative standard deviation (RSD) – The standard deviation times 100 divided by the mean. Also termed "coefficient of variation."

RF – Response factor. See Section 10.5.

RR – Relative response. See Section 10.4.

SDS – Soxhlet/Dean-Stark extractor; an extraction device applied to the extraction of solid and semi-solid materials (Reference 11 and Figure 5)

Signal-to-noise ratio (S/N) – The height of the signal as measured from the mean (average) of the noise to the peak maximum divided by the width of the noise

Should – This action, activity, or procedural step is suggested but not required.

SICP – Selected ion current profile; the line described by the signal at an exact m/z

SPE – Solid-phase extraction; an extraction technique in which an analyte is extracted from an aqueous sample by passage over or through a material capable of reversibly adsorbing the analyte. Also termed liquid-solid extraction.

Stock solution – A solution containing an analyte that is prepared using a reference material traceable to EPA, the National Institute of Science and Technology (NIST), or a source that will attest to the purity and authenticity of the reference material.

TeCB – Tetrachlorobiphenyl

TEF – Toxicity equivalency factor; an estimate of the toxicity of a specific congener relative to 2,3,7,8-tetrachlorodibenzo-*p*-dioxin

TEQ – The toxicity equivalent concentration in an environmental sample. It is the sum of the concentrations of each individual toxic PCB and each individual 2,3,7,8-substituted, tetra-through octa-chlorinated, dibenzo-*p*-dioxin and dibenzofuran multiplied by their respective TEFs (Reference 1).

TEQ_{PCB} – The portion of the TEQ attributable to the toxic PCBs

TrCB – Trichlorobiphenyl

Unique GC resolution or uniquely resolved – Two adjacent chromatographic peaks in which the height of the valley is less than 40 percent of the height of the shorter peak. See Section 6.9.1.1.2 and Figures 6 and 7 for unique resolution specific to the SPB-octyl column.

VER – See Calibration verification

Appendix A - Preliminary Information for Determination of 209 CBs on the DB-1 Column

1.0 Column and Conditions

1.1 Column – 30 ± 5-m long x 0.25 ± 0.02-mm ID; 0.25 µm film DB-1 (J&W, or equivalent).

1.2 Suggested GC operating conditions:

Injector temperature:	270 ºC
Interface temperature:	290 ºC
Initial temperature:	75 ºC
Initial time:	2 minutes
Temperature program:	75-150 ºC at 15 ºC/minute
	150-270 ºC at 2.5 ºC/minute
Final time:	7 minutes
Carrier gas velocity:	40 cm/sec at 200 ºC

Note: *The GC conditions may be optimized for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, blanks, IPR and OPR aliquots, and samples.*

2.0 Operating Information

2.1 Congener solutions – Mixes of individual congeners that will allow separation of all 209 congeners on the DB-1 column had not been developed when writing Method 1668C.

2.2 Elution order data – The congener mixes developed for the SPB-octyl column (Table 4 of Method 1668C) were run on the DB-1 column. Although some congeners in these mixes co-elute, the mixes allow determination of retention times for many congeners on the DB-1 column. These retention times are shown in Appendix Table A-1.

2.3 Window-defining congeners – The beginning and ending congeners at each level of chlorination are the same as for the SPB-octyl column. See Table 2 in Method 1668C.

2.4 Scan descriptors – The 6-function scan descriptors are shown in Appendix Table A-2.

Table A-1. Retention time (RT) References, Quantitation References, and Relative Retention Times (RRTs) for CB Congeners using a DB-1 Column

Labeled or Native CB[1]	Congener No.[2]	Retention Time and Quantitation References	Congener No.[2]	RT	RRT	RRT QC Limits[3]
$^{13}C_{12}$-2-MoCB[4]	1L	$^{13}C_{12}$-4-MoCB[4,5]	3L	09:17	0.8855	0.8776-0.8935
2-MoCB	1	$^{13}C_{12}$-2-MoCB[4]	1L	09:17	1.0000	0.9964-1.0072
3-MoCB	2	$^{13}C_{12}$-4-MoCB[4,5]	3L	10:22	0.9889	0.9809-0.9968
$^{13}C_{12}$-4-MoCB[4,5]	3L	$^{13}C_{12}$-2,2',5,5'-TeCB[7]	52L	10:29	0.5561	0.5473-0.5650
4-MoCB	3	$^{13}C_{12}$-4-MoCB[4,5]	3L	10:29	1.0000	0.9968-1.0064
$^{13}C_{12}$-2,2'-DiCB[4]	4L	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	11:08	0.7591	0.7477-0.7705
2,2'-DiCB	4	$^{13}C_{12}$-2,2'-DiCB[4]	4L	11:08	1.0000	0.9925-1.0075
2,6-DiCB	10	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	11:10	0.7614	0.7500-0.7727
2,5-DiCB	9	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	12:08	0.8273	0.8216-0.8330
2,4-DiCB	7	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	12:09	0.8284	0.8227-0.8341
2,3'-DiCB	6	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	12:31	0.8534	0.8477-0.8591
2,4'-DiCB[6]	8	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	12:43	0.8670	0.8614-0.8727
2,3-DiCB	5	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	12:46	0.8705	0.8648-0.8761
$^{13}C_{12}$-2,2',6-TrCB[4]	19L	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	13:31	0.7990	0.7892-0.8089
3,5-DiCB	14	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	13:36	0.9273	0.9216-0.9330
2,4,6-TrCB	30	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	14:06	0.8335	0.8286-0.8384
3,3'-DiCB	11	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	14:11	0.9670	0.9614-0.9727
3,4'-DiCB	13	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	14:26	0.9841	0.9784-0.9898
3,4-DiCB	12	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	14:27	0.9852	0.9795-0.9909
2,2',5-TrCB[6]	18	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	14:36	0.8631	0.8581-0.8680
$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	$^{13}C_{12}$-2,2',5,5'-TeCB[7]	52L	14:40	0.7781	0.7692-0.7869
4,4'-DiCB	15	$^{13}C_{12}$-4,4'-DiCB[4,5]	15L	14:40	1.0000	0.9977-1.0043
2,2',4-TrCB	17	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	14:43	0.8700	0.8650-0.8749
2,3',6-TrCB	27	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	15:06	0.8926	0.8877-0.8975
2,3,6-TrCB	24	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	15:06	0.8926	0.8877-0.8975
2,2',3-TrCB	16	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	15:26	0.9123	0.9074-0.9172
2,4',6-TrCB	32	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	15:29	0.9153	0.9103-0.9202
$^{13}C_{12}$-2,2',6,6'-TeCB[4]	54L	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	16:02	0.6139	0.6075-0.6203
2,2',6,6'-TeCB	54	$^{13}C_{12}$-2,2',6,6'-TeCB[4]	54L	16:02	1.0000	0.9979-1.0042
2',3,5-TrCB	34	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	16:03	0.9488	0.9438-0.9537
2,3,5-TrCB	23	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	16:07	0.9527	0.9478-0.9576
2,4,5-TrCB	29	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	16:18	0.9635	0.9586-0.9685

Table A-1. Retention time (RT) References, Quantitation References, and Relative Retention Times (RRTs) for CB Congeners using a DB-1 Column

Labeled or Native CB[1]	Congener No.[2]	Retention Time and Quantitation References	Congener No.[2]	RT	RRT	RRT QC Limits[3]
2,3',5-TrCB	26	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	16:29	0.9744	0.9695-0.9793
2,3',4-TrCB	25	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	16:36	0.9813	0.9764-0.9862
2,4',5-TrCB	31	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	16:52	0.9970	0.9921-1.0020
$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	$^{13}C_{12}$-2,2',5,5'-TeCB[7]	52L	16:55	0.8974	0.8930-0.9019
2,4,4'-TrCB[6]	28	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	16:55	1.0000	0.9980-1.0039
2,2',4,6-TeCB	50	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	16:55	0.6477	0.6414-0.6541
2,3,4-TrCB	21	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	17:21	1.0256	1.0207-1.0305
2,2',5,6'-TeCB	53	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	17:26	0.6675	0.6611-0.6739
2,3,3'-TrCB	20	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	17:22	1.0266	1.0217-1.0315
2',3,4-TrCB	33	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	17:24	1.0286	1.0236-1.0335
2,2',4,6'-TeCB	51	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	17:42	0.6777	0.6713-0.6841
2,3,4'-TrCB	22	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	17:43	1.0473	1.0424-1.0522
2,2',3,6-TeCB	45	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	18:00	0.6892	0.6828-0.6956
3,3',5-TrCB	36	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	18:16	1.0798	1.0749-1.0847
2,2',3,6'-TeCB	46	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	18:24	0.7045	0.6981-0.7109
3,4',5-TrCB	39	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	18:37	1.1005	1.0956-1.1054
$^{13}C_{12}$-2,2',5,5'-TeCB[7]	52L	$^{13}C_{12}$-2,2',5,5'-TeCB[7]	52L	18:51	1.0000	0.9956-1.0044
2,2',5,5'-TeCB[6]	52	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	18:51	0.7218	0.7154-0.7281
2,3',4,6-TeCB	69	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	18:52	0.7224	0.7160-0.7288
2,3',5',6-TeCB	73	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	18:57	0.7256	0.7192-0.7320
2,2',4,5'-TeCB	49	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	19:00	0.7275	0.7211-0.7339
2,2',3,5-TeCB	43	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	19:04	0.7301	0.7237-0.7364
3,4,5-TrCB	38	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	19:12	1.1350	1.1300-1.1399
2,2',4,4'-TeCB	47	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	19:15	0.7371	0.7307-0.7435
2,4,4',6-TeCB	75	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	19:20	0.7403	0.7339-0.7466
2,2',4,5-TeCB	48	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	19:20	0.7403	0.7339-0.7466
2,3,5,6-TeCB	65	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	19:31	0.7473	0.7409-0.7537
2,3,4,6-TeCB	62	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	19:36	0.7505	0.7441-0.7569
3,3',4-TrCB	35	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	19:41	1.1635	1.1586-1.1685
$^{13}C_{12}$-2,2',4,6,6'-PeCB[4]	104L	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	19:45	0.7037	0.6977-0.7096
2,2',4,6,6'-PeCB	104	$^{13}C_{12}$-2,2',4,6,6'-PeCB[4]	104L	19:45	1.0000	0.9983-1.0034
2,2',3,5'-TeCB[6]	44	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	19:55	0.7626	0.7562-0.7690

Table A-1. Retention time (RT) References, Quantitation References, and Relative Retention Times (RRTs) for CB Congeners using a DB-1 Column

Labeled or Native CB[1]	Congener No.[2]	Retention Time and Quantitation References	Congener No.[2]	RT	RRT	RRT QC Limits[3]
$^{13}C_{12}$-3,4,4'-TrCB[4]	37L	$^{13}C_{12}$-2,4,4'-TrCB[5]	28L	20:03	1.1852	1.1803-1.1901
3,4,4'-TrCB	37	$^{13}C_{12}$-3,4,4'-TrCB[4]	37L	20:03	1.0000	0.9983-1.0033
2,3,3',6-TeCB	59	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	20:05	0.7690	0.7626-0.7754
2,2',3,4'-TeCB	42	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	20:07	0.7703	0.7639-0.7766
2,3',5,5'-TeCB	72	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	20:36	0.7888	0.7824-0.7951
2,3',4',6-TeCB	71	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	20:36	0.7888	0.7824-0.7951
2,3,4',6-TeCB	64	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	20:37	0.7894	0.7830-0.7958
2,2',3,4-TeCB	41	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	20:39	0.7907	0.7843-0.7971
2,2',3,6,6'-PeCB	96	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	20:48	0.7411	0.7352-0.7470
2,3',4,5'-TeCB	68	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	20:52	0.7990	0.7926-0.8054
2,2',3,3'-TeCB	40	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	20:58	0.8028	0.7996-0.8060
2,3,3',5-TeCB	57	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	21:21	0.8175	0.8143-0.8207
2,2',4,5,'6-PeCB	103	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	21:22	0.7613	0.7553-0.7672
2,3',4,5-TeCB	67	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	21:38	0.8283	0.8251-0.8315
2,2',4,4',6-PeCB	100	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	21:41	0.7726	0.7666-0.7785
2,3,3',5'-TeCB	58	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	21:43	0.8315	0.8283-0.8347
2,3,4',5-TeCB	63	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	21:51	0.8366	0.8334-0.8398
2,2',3,5,6'-PeCB	94	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	22:05	0.7868	0.7809-0.7928
2,4,4',5-TeCB	74	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	22:07	0.8468	0.8437-0.8500
2,3,4,5-TeCB	61	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	22:11	0.8494	0.8462-0.8526
2,3',4',5-TeCB	70	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	22:20	0.8551	0.8519-0.8583
2 ',3,4,5-TeCB	76	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	22:25	0.8583	0.8551-0.8615
2,2',3',4,6-PeCB	98	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	22:28	0.8005	0.7975-0.8034
2,3',4,4'-TeCB[6]	66	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	22:29	0.8609	0.8577-0.8641
2,2',4,5,6'-PeCB	102	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	22:32	0.8029	0.7999-0.8058
2,2',3,5',6-PeCB	95	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	22:34	0.8040	0.8011-0.8070
2,2',3,5,6-PeCB	93	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	22:36	0.8052	0.8023-0.8082
3,3',5,5'-TeCB	80	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	22:45	0.8711	0.8679-0.8743
2,2',3,4,6-PeCB	88	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	22:49	0.8129	0.8100-0.8159
2,2',3,4',6-PeCB	91	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	22:55	0.8165	0.8135-0.8195
2,3,3',4'-TeCB	55	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	22:57	0.8787	0.8756-0.8819
2,3',4,5,'6-PeCB	121	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	23:04	0.8219	0.8189-0.8248

Table A-1. Retention time (RT) References, Quantitation References, and Relative Retention Times (RRTs) for CB Congeners using a DB-1 Column

Labeled or Native CB[1]	Congener No.[2]	Retention Time and Quantitation References	Congener No.[2]	RT	RRT	RRT QC Limits[3]
2,3,3',4'-TeCB	56	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	23:24	0.8960	0.8928-0.8992
2,3,4,4'-TeCB	60	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	23:24	0.8960	0.8928-0.8992
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB[4]	155L	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	23:43	0.7104	0.7054-0.7154
2,2',4,4',6,6'-HxCB	155	$^{13}C_{12}$-2,2',4,4',6,6'-HxCB[4]	155L	23:43	1.0000	0.9986-1.0028
2,2',3,3',6-PeCB	84	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	23:44	0.8456	0.8426-0.8486
2,2',3,5,5'-PeCB	92	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	23:50	0.8492	0.8462-0.8521
2,2',3,4,6'-PeCB	89	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	23:53	0.8510	0.8480-0.8539
2,2',3,4',5-PeCB	90	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	24:07	0.8593	0.8563-0.8622
$^{13}C_{12}$-2,2',4,5,5'-PeCB[7]	101L	$^{13}C_{12}$-2,2',4,5,5'-PeCB[7]	101L	24:11	1.0000	0.9966-1.0034
2,2',4,5,5'-PeCB[6]	101	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	24:11	0.8616	0.8587-0.8646
2,3,3',5',6-PeCB	113	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	24:23	0.8688	0.8658-0.8717
3,3',4,5'-TeCB	79	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	24:27	0.9362	0.9330-0.9394
2,2',4,4',5-PeCB	99	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	24:28	0.8717	0.8688-0.8747
2,2',3,4',6,6'-HxCB	150	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	24:52	0.7449	0.7399-0.7499
2,3',4,4',6-PeCB	119	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	24:54	0.8872	0.8842-0.8901
2,3,3',5,6-PeCB	112	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:00	0.8907	0.8878-0.8937
2,3,3',4,6-PeCB	109	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:09	0.8961	0.8931-0.8990
2,2',3,5,6,6'-HxCB	152	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	25:17	0.7574	0.7524-0.7624
2,2',3,3',5-PeCB	83	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:20	0.8919	0.8890-0.8949
2,2',3',4,5-PeCB	97	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:22	0.9038	0.9008-0.9068
2,2',3,4,5-PeCB	86	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:27	0.9068	0.9038-0.9097
$^{13}C_{12}$-3,4,4',5-TeCB[9]	81L	$^{13}C_{12}$-2,2',5,5'-TeCB[7]	52L	25:32	1.3546	1.3457-1.3634
3,4,4',5-TeCB[10]	81	$^{13}C_{12}$-3,4,4',5-TeCB[4,5,9]	77L	25:32	1.0000	0.9987-1.0026
2 ',3,4,5,6'-PeCB	125	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:36	0.9121	0.9091-0.9151
2,3,4',5,6-PeCB	117	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:37	0.9127	0.9097-0.9157
2,2',3,4,5'-PeCB	87	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:38	0.9133	0.9103-0.9163
3,3',4,5-TeCB	78	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	25:40	0.9598	0.9566-0.9630
2,2',3,4,6,6'-HxCB	145	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	25:42	0.7698	0.7649-0.7748
2,3,4,4',6-PeCB	115	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:44	0.9169	0.9139-0.9198
$^{13}C_{12}$-2,3,3',5,5'-PeCB[8]	111L	$^{13}C_{12}$-2,2',4,5,5'-PeCB[7]	101L	25:51	1.0689	1.0655-1.0724
2,3,3',5,5'-PeCB	111	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:51	0.9210	0.9181-0.9240
2,2',3,4,4'-PeCB	85	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:51	0.9210	0.9181-0.9240

Table A-1. Retention time (RT) References, Quantitation References, and Relative Retention Times (RRTs) for CB Congeners using a DB-1 Column

Labeled or Native CB[1]	Congener No.[2]	Retention Time and Quantitation References	Congener No.[2]	RT	RRT	RRT QC Limits[3]
2,3,4,5,6-PeCB	116	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	25:48	0.9192	0.9163-0.9222
$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	$^{13}C_{12}$-2,2',5,5'-TeCB[7]	52L	26:07	1.3855	1.3767-1.3943
3,3',4,4'-TeCB[6,10]	77	$^{13}C_{12}$-3,3',4,4'-TeCB[4,5,9]	77L	26:07	1.0000	0.9987-1.0026
2,2',3,3',6,6'-HxCB	136	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	26:10	0.7793	0.7743-0.7843
2,3',4,5,5'-PeCB	120	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	26:12	0.9335	0.9305-0.9365
2,2',3,4',5,6'-HxCB	148	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	26:14	0.7858	0.7808-0.7908
2,3,3',4',6-PeCB	110	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	26:16	0.9359	0.9329-0.9388
2,2',4,4',5,6'-HxCB	154	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	26:44	0.8008	0.7983-0.8033
2,2',3,3',4-PeCB	82	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	26:48	0.9549	0.9519-0.9578
2,2',3,5,5',6-HxCB	151	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	27:18	0.8178	0.8153-0.8203
2,2',3,3',5,6'-HxCB	135	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	27:31	0.8243	0.8218-0.8268
2 ',3,4,5,5'-PeCB	124	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	27:36	0.9834	0.9804-0.9863
2,2',3,4,5',6-HxCB	144	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	27:38	0.8278	0.8253-0.8303
2,3,3',4,5'-PeCB	108	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	27:40	0.9857	0.9828-0.9887
2,2',3,4',5,6-HxCB	147	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	27:44	0.8308	0.8283-0.8333
2,3,3',4',5-PeCB	107	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	27:45	0.9887	0.9857-0.9917
2,2',3,4',5',6-HxCB	149	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	28:01	0.8392	0.8367-0.8417
2,2',3,3',5,6-HxCB	134	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	28:35	0.8562	0.8537-0.8587
2,2',3,4,5,6'-HxCB	143	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	28:34	0.8557	0.8532-0.8582
$^{13}C_{12}$-2',3,4,4',5-PeCB[9]	123L	$^{13}C_{12}$-2,2',4,5,5'-PeCB[7]	101L	27:53	1.1530	1.1496-1.1564
2',3,4,4',5-PeCB[10]	123	$^{13}C_{12}$-2',3,4,4',5-PeCB[9]	123L	27:53	1.0000	0.9988-1.0024
2,2',3,4,4',6-HxCB	139	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	28:01	0.8392	0.8367-0.8417
2,3,3',4,5-PeCB	106	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	28:04	1.0000	0.9970-1.0030
$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	$^{13}C_{12}$-2,2',4,5,5'-PeCB[7]	101L	28:04	1.1606	1.1571-1.1640
2,3',4,4',5-PeCB[6,10]	118	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	28:04	1.0000	0.9988-1.0024
2,2',3,4,4',6'-HxCB	140	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	28:12	0.8447	0.8422-0.8472
$^{13}C_{12}$-2,3,4,4',5-PeCB[9]	114L	$^{13}C_{12}$-2,2',4,5,5'-PeCB[7]	101L	28:38	1.1840	1.1806-1.1875
2,3,4,4',5-PeCB[10]	114	$^{13}C_{12}$-2,3,4,4',5-PeCB[9]	114L	28:38	1.0000	0.9988-1.0023
2',3,3',4,5-PeCB	122	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	28:48	1.0261	1.0232-1.0291
2,2',3,3',4,6-HxCB	131	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	28:52	0.8647	0.8622-0.8672
2,2',3,4,5,6-HxCB	142	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	28:59	0.8682	0.8657-0.8707
2,2',3,3',5,5'-HxCB	133	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	28:59	0.8682	0.8657-0.8707

Table A-1. Retention time (RT) References, Quantitation References, and Relative Retention Times (RRTs) for CB Congeners using a DB-1 Column

Labeled or Native CB[1]	Congener No.[2]	Retention Time and Quantitation References	Congener No.[2]	RT	RRT	RRT QC Limits[3]
2,2',3,3',4,6'-HxCB	132	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	29:32	0.8847	0.8822-0.8872
2,3,3',5,5',6-HxCB	165	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	29:21	0.8792	0.8767-0.8817
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB[4]	188L	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	29:22	0.9511	0.7327-0.7411
2,2',3,4',5,6,6'-HpCB	188	$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB[4]	188L	29:22	1.0000	0.9989-1.0023
2,2',3,4',5,5'-HxCB	146	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	29:24	0.8807	0.8782-0.8832
$^{13}C_{12}$-2,3,3',4,4'-PeCB[9]	105L	$^{13}C_{12}$-2,2',4,5,5'-PeCB[7]	101L	29:30	1.2198	1.2130-1.2267
2,3,3',4,4'-PeCB[6,10]	105	$^{13}C_{12}$-2,3,3',4,4'-PeCB[9]	105L	29:30	1.0000	0.9989-1.0023
2,3,3',4,5',6-HxCB	161	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	29:32	0.8847	0.8822-0.8872
2,2',4,4',5,5'-HxCB[6]	153	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	29:48	0.8927	0.8902-0.8952
2,2',3,4,4',6,6'-HpCB	184	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	29:49	0.7482	0.7440-0.7524
3,3',4,5,5'-PeCB	127	$^{13}C_{12}$-2,3',4,4',5-PeCB[5,9]	118L	29:57	1.0671	1.0641-1.0701
2,3',4,4',5',6-HxCB	168	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	29:59	0.8982	0.8957-0.9006
2,2',3,4,5,5'-HxCB	141	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	30:31	0.9141	0.9116-0.9166
2,2',3,3',5,6,6'-HpCB	179	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	30:33	0.7666	0.7624-0.7708
2,2',3,4,4',5-HxCB	137	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	30:51	0.9241	0.9216-0.9266
2,2',3,3',4,5'-HxCB	130	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	30:57	0.9271	0.9246-0.9296
2,2',3,3',4,6,6'-HpCB	176	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	31:01	0.7783	0.7742-0.7825
$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[7]	138L	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[7]	138L	31:20	1.0000	0.9973-1.0027
2,2',3,4,4',5'-HxCB[6]	138	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	31:20	0.9386	0.9361-0.9411
2,3,3',4',5',6-HxCB	164	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	31:22	0.9396	0.9371-0.9421
2,3,3',4',5,6-HxCB	163	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	31:28	0.9426	0.9401-0.9451
2,3,3',4,5,6-HxCB	160	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	31:33	0.9451	0.9426-0.9476
2,3,3',4,4',6-HxCB	158	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	31:35	0.9461	0.9436-0.9486
2,2',3,4,5,6,6'-HpCB	186	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	31:36	0.7930	0.7888-0.7972
2,2',3,3',4,5-HxCB	129	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	31:48	0.9526	0.9501-0.9551
$^{13}C_{12}$-3,3',4,4',5-PeCB[4,9]	126L	$^{13}C_{12}$-2,2',4,5,5'-PeCB[7]	101L	31:49	1.3156	1.3088-1.3225
3,3',4,4',5-PeCB[6,10]	126	$^{13}C_{12}$-3,3',4,4',5-PeCB[4,9]	126L	31:49	1.0000	0.9990-1.0021
2,3,4,4',5,6-HxCB	166	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	32:13	0.9651	0.9626-0.9675
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[7]	178L	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[7]	178L	32:14	1.0000	0.9974-1.0026
2,2',3,3',5,5',6-HpCB	178	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	32:14	0.8089	0.8068-0.8110
2,2',3,3',4,5',6-HpCB	175	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	32:33	0.8168	0.8147-0.8189
2,3,3',4,5,5'-HxCB	159	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	32:43	0.9800	0.9775-0.9825

Table A-1. Retention time (RT) References, Quantitation References, and Relative Retention Times (RRTs) for CB Congeners using a DB-1 Column

Labeled or Native CB[1]	Congener No.[2]	Retention Time and Quantitation References	Congener No.[2]	RT	RRT	RRT QC Limits[3]
2,2',3,4',5,5',6-HpCB[6]	187	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	32:46	0.8223	0.8202-0.8243
2,2',3,4,4',5,6'-HpCB	182	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	32:47	0.8227	0.8206-0.8248
2,2',3,3',4,4'-HxCB[6]	128	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	32:52	0.9845	0.9820-0.9870
2,3,3',4',5,5'-HxCB	162	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	33:00	0.9885	0.9860-0.9910
2,2',3,4,4',5',6-HpCB	183	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	33:06	0.8306	0.8285-0.8327
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[7]	138L	33:23	1.0654	1.0628-1.0681
2,3',4,4',5,5'-HxCB[10]	167	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB[5,9]	167L	33:23	1.0000	0.9990-1.0020
2,2',3,4,5,5',6-HpCB	185	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	33:43	0.8461	0.8440-0.8482
2,2',3,3',4,5,6'-HpCB	174	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	34:07	0.8561	0.8540-0.8582
2,2',3,4,4',5,6-HpCB	181	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	34:11	0.8578	0.8557-0.8599
2,2',3,3',4',5,6-HpCB	177	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	34:22	0.8624	0.8603-0.8645
2,2'3,3',4,4',6-HpCB	171	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	34:40	0.8699	0.8678-0.8720
$^{13}C_{12}$-2,3,3',4,4',5 -HxCB[9]	156L	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[7]	138L	34:40	1.1064	1.1037-1.1090
2,3,3',4,4',5-HxCB[10]	156	$^{13}C_{12}$-2,3,3',4,4',5 -HxCB[9]	156L	34:40	1.0000	0.9990-1.0019
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB[4]	202L	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	34:56	0.8265	0.8245-0.8285
2,2',3,3',5,5',6,6'-OcCB	202	$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB[4]	202L	34:56	1.0000	0.9990-1.0019
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[9]	157L	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[7]	138L	34:57	1.1154	1.1128-1.1181
2,3,3',4,4',5'-HxCB[10]	157	$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[9]	157L	34:57	1.0000	0.9990-1.0019
2,2',3,3',4,5,6-HpCB	173	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	35:04	0.8800	0.8779-0.8821
2,2',3,3',4,5',6,6'-OcCB	201	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	35:25	0.8379	0.8360-0.8399
2,2',3,4,4',5,6,6'-OcCB	204	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	35:36	0.8423	0.8403-0.8442
2,2',3,3',4,5,5'-HpCB	172	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	35:41	0.8954	0.8934-0.8975
2,3,3',4,5,5',6-HpCB	192	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	35:51	0.8996	0.8975-0.9017
2,2',3,3',4,4',6,6'-OcCB	197	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	35:55	0.8498	0.8478-0.8517
2,2',3,4,4',5,5'-HpCB[6]	180	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	36:07	0.9063	0.9042-0.9084
2,3,3',4',5,5',6-HpCB	193	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	36:20	0.9118	0.9097-0.9138
2,3,3',4,4',5',6-HpCB	191	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	36:34	0.9176	0.9155-0.9197
2,2',3,3',4,5,6,6'-OcCB	200	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	36:49	0.8711	0.8691-0.8730
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB[4,9]	169L	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[7]	138L	37:19	1.1910	1.1883-1.1936
3,3',4,4',5,5'-HxCB[6,10]	169	$^{13}C_{12}$-3,3',4,4',5,5'-HxCB[4,9]	169L	37:19	1.0000	0.9991-1.0018
2,2',3,3',4,4',5-HpCB[6]	170	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	37:44	0.9469	0.9448-0.9490
2,3,3',4,4',5,6-HpCB	190	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	37:56	0.9519	0.9498-0.9540

Table A-1. Retention time (RT) References, Quantitation References, and Relative Retention Times (RRTs) for CB Congeners using a DB-1 Column

Labeled or Native CB[1]	Congener No.[2]	Retention Time and Quantitation References	Congener No.[2]	RT	RRT	RRT QC Limits[3]
2,2',3,3',4,5,5',6-OcCB	198	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	38:34	0.9125	0.9105-0.9144
2,2',3,3',4,5,5',6'-OcCB	199	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	38:43	0.9160	0.9140-0.9180
2,2',3,3',4,4',5,6'-OcCB	196	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	39:05	0.9247	0.9227-0.9267
2,2',3,4,4',5,5',6-OcCB	203	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	39:05	0.9247	0.9227-0.9267
$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[7]	178L	39:51	1.2363	1.2311-1.2415
2,3,3',4,4',5,5'-HpCB[10]	189	$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB[4,5,9]	189L	39:51	1.0000	0.9992-1.0017
2,2',3,3',4,4',5,6-OcCB[6]	195	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	40:45	0.9641	0.9621-0.9661
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB[4]	208L	$^{13}C_{12}$-Cl9-PCB-206[4,5]	206L	41:03	0.9149	0.9131-0.9168
2,2',3,3',4,5,5',6,6'-NoCB	208	$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB[4]	208L	41:03	1.0000	0.9992-1.0016
2,2',3,3',4,4',5,6,6'-NoCB	207	$^{13}C_{12}$-Cl9-PCB-206[4,5]	206L	41:32	0.9257	0.9238-0.9276
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB[5]	194L	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[7]	178L	42:16	1.3113	1.3061-1.3164
2,2',3,3',4,4',5,5'-OcCB	194	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	42:16	1.0000	0.9992-1.0016
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB[4]	205L	$^{13}C_{12}$-Cl8-PCB-194[5]	194L	42:44	1.0110	1.0091-1.0130
2,3,3',4,4',5,5',6-OcCB	205	$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB[4]	205L	42:44	1.0000	0.9992-1.0016
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB[4,5]	206L	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[7]	178L	44:52	1.3919	1.3868-1.3971
2,2',3,3',4,4',5,5',6-NoCB[6]	206	$^{13}C_{12}$-Cl9-PCB-206[4,5]	206L	44:52	1.0000	0.9993-1.0015
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB[4,5]	209L	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB[7]	178L	46:55	1.4555	1.4504-1.4607
2,2',3,3',4,4',5,5',6,6'-DeCB[6]	209	$^{13}C_{12}$-Cl10-PCB-209[4,5]	209L	46:55	1.0000	0.9993-1.0014

1. Abbreviations for chlorination levels

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

2. Suffix "L" indicates labeled compound
3. For native CBs determined by isotope dilution quantitation, RRT QC limits were constructed using -2 to +4 seconds around the retention time for the labeled analog. For native CBs determined by internal standard quantitation, RRT QC limits were constructed using a ± 2 percent window around the retention time for retention times in the range of 0.8-1.2 and a ± 4 percent window around the retention time for retention times <0.8 and >1.2. These windows may not be adequate for analyte identification (See the note in Section 16.4)
4. Labeled level of chlorination (LOC) window-defining congener
5. Labeled level of chlorination (LOC) quantitation congener
6. National Oceanic and Atmospheric Administration (NOAA) congener of interest
7. Instrument internal standard
8. Clean-up standard
9. Labeled internal standard for World Health Organization (WHO) toxic congener
10. WHO toxic congener

Table A-2. Scan Descriptors, Levels of Chlorination, m/z Information, and Substances Monitored by HRGC/HRMS

Function and Chlorine Level	m/z	m/z Type	m/z Formula	Substance
Fn-1 Cl-1	188.0393	M	$^{12}C_{12}H_9{}^{35}Cl$	Cl-1 PCB
	190.0363	M+2	$^{12}C_{12}H_9{}^{37}Cl$	Cl-1P CB
	200.0795	M	$^{13}C_{12}H_9{}^{35}Cl$	$^{13}C_{12}$ Cl-1 PCB
	202.0766	M+2	$^{13}C_{12}H_9{}^{37}Cl$	$^{13}C_{12}$ Cl-1 PCB
	218.9856	lock	C_4F_9	PFK
Fn-2 Cl-2,3	222.0003	M	$^{12}C_{12}H_8{}^{35}Cl_2$	Cl-2 PCB
	223.9974	M+2	$^{12}C_{12}H_8{}^{35}Cl\,{}^{37}Cl$	Cl-2 PCB
	225.9944	M+4	$^{12}C_{12}H_8{}^{37}Cl_2$	Cl-2 PCB
	234.0406	M	$^{13}C_{12}H_8{}^{35}Cl_2$	$^{13}C_{12}$ Cl-2 PCB
	236.0376	M+2	$^{13}C_{12}H_8{}^{35}Cl\,{}^{37}Cl$	$^{13}C_{12}$ Cl-2 PCB
	242.9856	lock	C_6F_9	PFK
	255.9613	M	$^{12}C_{12}H_7{}^{35}Cl_3$	Cl-3 PCB
	257.9584	M+2	$^{12}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Cl-3 PCB
Fn-3 Cl-3,4,5	255.9613	M	$^{12}C_{12}H_7{}^{35}Cl_3$	Cl-3 PCB
	257.9584	M+2	$^{12}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Cl-3 PCB
	259.9554	M+4	$^{12}C_{12}H_7{}^{35}Cl\,{}^{37}Cl_2$	Cl-3 PCB
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	$^{13}C_{12}$ Cl-3 PCB
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	$^{13}C_{12}$ Cl-3 PCB
	280.9825	lock	C_6F_{11}	PFK
	289.9224	M	$^{12}C_{12}H_6{}^{35}Cl_4$	Cl-4 PCB
	291.9194	M+2	$^{12}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	Cl-4 PCB
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_4$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}H_5{}^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
Fn-4 Cl-4,5,6	289.9224	M	$^{12}C_{12}H_6{}^{35}Cl_4$	Cl-4 PCB
	291.9194	M+2	$^{12}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	Cl-4 PCB
	301.9626	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+4	$^{13}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}H_5{}^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	330.9792	lock	C_7F_{15}	PFK
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	359.8415	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Cl-6 PCB
	361.8385	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}Cl_2$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB

Table A-2. Scan Descriptors, Levels of Chlorination, m/z Information, and Substances Monitored by HRGC/HRMS

Function and Chlorine Level	m/z	m/z Type	m/z Formula	Substance
Fn-5 Cl-5,6,7,8	323.8834	M	$^{12}C_{12}H_5{}^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	354.9792	lock	C_9F_{13}	PFK
	359.8415	M+2	$^{12}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Cl-6 PCB
	361.8385	M+4	$^{12}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{12}C_{12}H_4{}^{35}Cl_3{}^{37}Cl_3$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
	393.8025	M+2	$^{12}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Cl-7 PCB
	395.7995	M+4	$^{12}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Cl-7 PCB
	397.7966	M+6	$^{12}C_{12}H_3{}^{35}Cl_4{}^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	427.7635	M+2	$^{12}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Cl-8 PCB
	429.7606	M+4	$^{12}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Cl-8 PCB
	431.7576	M+6	$^{12}C_{12}H_2{}^{35}Cl_5{}^{37}Cl_3$	Cl-8 PCB
	439.8038	M+2	$^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	$^{13}C_{12}$ Cl-8 PCB
	441.8008	M+4	$^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	$^{13}C_{12}$ Cl-8 PCB
	454.9728	QC	$C_{11}F_{17}$	PFK
Fn-6 Cl-8,9,10	427.7635	M+2	$^{12}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Cl-8 PCB
	429.7606	M+4	$^{12}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Cl-8 PCB
	431.7576	M+6	$^{12}C_{12}H_2{}^{35}Cl_5{}^{37}Cl_3$	Cl-8 PCB
	439.8038	M+2	$^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	$^{13}C_{12}$ Cl-8 PCB
	441.8008	M+4	$^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	$^{13}C_{12}$ Cl-8 PCB
	442.9728	QC	$C_{10}F_{13}$	PFK
	454.9728	lock	$C_{11}F_{13}$	PFK
	461.7246	M+2	$^{12}C_{12}H_1{}^{35}Cl_8{}^{37}Cl$	Cl-9 PCB
	463.7216	M+4	$^{12}C_{12}H_1{}^{35}Cl_7{}^{37}Cl_2$	Cl-9 PCB
	465.7187	M+6	$^{12}C_{12}H_1{}^{35}Cl_6{}^{37}Cl_3$	Cl-9 PCB
	473.7648	M+2	$^{13}C_{12}H_1{}^{35}Cl_8{}^{37}Cl$	$^{13}C_{12}$ Cl-9 PCB
	475.7619	M+4	$^{13}C_{12}H_1{}^{35}Cl_7{}^{37}Cl_2$	$^{13}C_{12}$ Cl-9 PCB
	495.6856	M+2	$^{13}C_{12}H_4{}^{35}Cl_9{}^{37}Cl$	Cl-10 PCB
	499.6797	M+4	$^{12}C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Cl-10 PCB
	501.6767	M+6	$^{12}C_{12}{}^{35}Cl_6{}^{37}Cl_4$	Cl-10 PCB
	507.7258	M+2	$^{13}C_{12}H_4{}^{35}Cl_9{}^{37}Cl$	$^{13}C_{12}$ Cl-10 PCB
	509.7229	M+4	$^{13}C_{12}H_4{}^{35}Cl_8{}^{37}Cl_2$	$^{13}C_{12}$ Cl-10 PCB
	511.7199	M+6	$^{13}C_{12}H_4{}^{35}Cl_8{}^{37}Cl_4$	$^{13}C_{12}$ Cl-10 PCB

Isotopic masses used for accurate mass calculation

^{1}H	1.0078	^{37}Cl	36.9659
^{12}C	12.0000	^{19}F	18.9984
^{13}C	13.0034	^{35}Cl	34.9689

AMIGOS BRAVOS' EXHIBIT 21

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Environmental Scientist

AREAS OF EXPERTISE

- Quality Assurance Management
- Preparation and Review of Quality Assurance Program Plans and Project Plans
- Laboratory Protocol Development and Review
- Data Validation and Assessment
- Laboratory Audits
- Design and Development of Sampling and Monitoring Plans
- Regulatory Compliance/Permitting Assistance

EDUCATION

- M.S., Environmental Studies - University of Montana, 1976
- B.A., Biology - University of Oregon, 1972

QUALIFICATIONS

Ms. Bailey has 40 years of professional experience in environmental chemistry and quality assurance (QA). Ms. Bailey was formerly the technical director of a commercial environmental testing laboratory, where she gained extensive hands-on experience in a wide-range of organic, inorganic, and conventional analyses. Since then, she has been instrumental in ensuring laboratory performance has met Good Laboratory Practice (GLP) standards for a range of multi-disciplinary projects. She has been involved with numerous environmental investigations through the development of project QA and sampling plans; selection of protocols and coordination of laboratory services; performance of on-site field and laboratory audits; and QA review and interpretation of analytical data. Ms. Bailey founded EcoChem, Inc., an environmental consulting firm in 1983. At EcoChem she directed QA support services for nine separate natural resource damage assessment (NRDA) projects located throughout the United States. This NRDA support included review of data from more than 20 research and commercial laboratories for compliance to data quality objectives (DQOs) and litigation quality standards. On behalf of EcoChem, Ms. Bailey received two *Awards of Excellence* from the U.S. Small Business Administration. These awards were presented to EcoChem for outstanding service in data collection, data analysis, and data validation for a high-profile project involving close public and legal scrutiny.

SELECT PROJECT EXPERIENCE

Quality Assurance Oversight

- **Duwamish/Diagonal and Norfolk Sediment Remediation** - Project Director for a sediment remediation project of two Duwamish River sites of Metro/City of Seattle outfalls, as part of the Elliott Bay/Duwamish Restoration Program. The project resulted in the removal and/or isolation of sediment contaminants from aquatic life and human exposure and meet the

requirements of the 1991 NRDA Consent Decree and the Washington State Sediment Management Standards (SMS).

- **Deepwater Horizon Oil Spill NRDA** – Quality Assurance Coordinator for the Deepwater Horizon (BP) Oil Spill Natural Resource Damage Assessment. Coordinated and approved analytical methods for site assessment, oversaw sampling and analysis quality assurance. Audited laboratories performing analyses. Validated data for assessment use.

- **Los Alamos National Laboratory NRDA –** Drafted a Quality Assurance Management Plan for the LANL Trustee Council. The plan provides a blueprint for planning, implementing, and assessing the Trustee Council's quality systems for NRDA work performed, including the use of historical data.

- **Lower Fox River/Green Bay NRDA** - Directed development of a questionnaire to survey quality and usability of historical data sets for the Lower Fox River/Green Bay NRDA. Over fifty interviews were performed to assess the availability, nature, and extent of QA documentation associated with historical PCB data. The available information was summarized and data sets were recommended for further evaluation. Monitored and provided technical input for the chemical analysis of fish and bird tissue samples conducted by the laboratory and provided quality assurance oversight for data validation of the associated data packages.

- **Grand Calumet NRDA Data Validation** - Project Director for the validation of PCB, PAH, and metals sediment data for the natural resource damage assessment of the Grand Calumet River, Indiana Harbor Canal and Lake Michigan environments in northwest Indiana. Provided a detailed review of the *Sediment Characterization Study Report* and *Indiana Harbor and Canal Sediment Trap Investigation* for adequacy and defensibility in the context of a natural resource damage assessment.

- **Southern California Bight NRDA QA Support** - Directed chemistry QA support as a subcontractor to NOAA's NRDA Damage Assessment Center. The project involved over 2,000 sediment and tissue samples analyzed for polychlorinated byphenyl (PCB) congeners, DDT, and DDT metabolites. Involved with the initial performance evaluation process for laboratory selection, which involved reviewing laboratory qualifications for performing non-standard analytical procedures and evaluating results from the analyses of reference materials. Performed initial and mid-project QA audits of the two primary laboratories selected, and oversaw validation of the specialized analytical protocols. Throughout the project, worked with the laboratories and project scientists to assist with the development of a data set, which met data user requirements and was litigation quality.

- **Calcasieu Estuary NRDA Data Validation** - Project Director for data validation of existing sediment contamination data to develop draft concentration isopleths, and Quality Assurance Project Plans development for estuarine sediments and marshes in support of investigations at the Calcasieu Estuary, Lake Charles, Louisiana.

- **Salish/Kootenai Tribe Litigation Claim** - Consulted with technical experts to design, document, and implement a quality assurance plan for the development of an assessment of

the impact releases of hazardous materials in the form of mining wastes to harvestable fish biomass in the Upper Clark Fork River. As part of the QA/QC process, performed a quality control evaluation of electronic data set used as input to statistical and other data analysis processes for the Tribal demographic study. Conducted a quality assurance audit of data input, transfer, and manipulation conducted by another firm.

- **Coeur d'Alene Basin NRDA** - Responsible for providing overall QA management support for Phase I of the Coeur d'Alene Basin NRDA. Project planning involved working with the lead trustees, Coeur d'Alene Tribe and US Department of Fish and Wildlife for development of overall program QA policies. This support included reviewing work plans, assistance with the development of quality assurance project plans (QAPPs), selecting and coordinating laboratories, overseeing the performance of field and laboratory audits, and assessing data usability on both a scientific and litigation-quality basis.

- **Hylebos Waterway NRDA QA Support** - Performed on-site review of trace metals laboratory procedures, and directed validation of trace organic and metal sediment data. Evaluated comparability of results from two different sediment extraction techniques for trace metals. Compared results from PCB congener and PCB Aroclor analyses.

- **Hudson River Remedial Investigation/Feasibility Study** - Responsible for ensuring that data developed and analyses conducted were in accordance with the *Analytical Quality Assurance Plan*. Performed an on-site laboratory audit and worked with the laboratory in the development of project-specific DQOs. Managed the validation of the laboratory data, production of the validation report, and electronic data summary. Analyses involved determination of 109 PCB congeners in fish tissue.

- **Elliott Bay Natural Resource Damages Assessment Program** - Provides senior planning and coordination support to NOAA and other Elliott Bay Natural Resource Trustees for investigation and assessment of resource injuries in the Duwamish River and Elliott Bay due to PCBs and other contaminants.

- **Upper Pecos Site Ecological Risk Assessment (ERA) Data Quality Assessment (DQA)** - Provided QA support for an ERA associated with mining activities in New Mexico. Reviewed all site-related information on file at the state agency and selected investigations that were adequately documented for performing further data quality assessment. Documentation reviewed included QAPPs, sampling analysis plans (SAPs), and project reports that contained information relating to data collected from 1991 through 1994. The DQA report evaluated the data and associated documentation, as to its useability for the ERA.

- **Boeing-Everett Plant Historical Data Review and Database Management** - Project Director for the QA/QC review of more than 75,000 analytical results collected over a ten year period. Provided oversight for the design and input to an electronic database, which the client utilized to load to a Geographic Information System.

- **Kaiser Aluminum Industrial Laboratory QA** - Conducted a laboratory audit at Kaiser Aluminum in Tacoma, Washington. Evaluated regulatory compliance requirements and

procedures and revised the laboratory *Quality Assurance Program Manual* to meet laboratory certification requirements.

- **Vashon/Maury Island Child-Use Area Soil Sampling, Seattle-King County Department of Public Health/Washington Department of Ecology** - Provided quality assurance oversight of data validation and electronic manipulation of laboratory data in support of a human health risk investigation. This investigation involved arsenic and lead contamination caused by the operation of the former ASARCO smelter facility in Ruston, Washington.

- **Port of Tacoma Dredging Study** - Prepared a QAPP and finalized laboratory protocols for the analysis of sediments for a Port of Tacoma dredging study.

- **Puget Sound Dredged Disposal Analysis (PSDDA) Data Validation Guidance Manual** - Co-authored inorganic and conventional sections of the PSDDA *Data Validation Manual*.

- **QA/QC Guidance Manual Preparation** - Participated in the preparation of a QA/QC guidance manual for the USEPA. The manual outlined procedures to follow during the sampling and analysis of water, sediments, and tissues for priority pollutants.

- **Queen City Farms Superfund Remedial Investigation/Feasibility Study (RI/FS)** - Served as QA Project Manager for the Queen City Farms Superfund site. Prepared the QAPP, performed QA/QC oversight, and performed and directed Contract Laboratory Program (CLP) data validation.

- **Gas Works Park Soil Contamination Investigation** - Drafted QA/QC plan for sampling and analysis of soil for evaluation of polycyclic aromatic hydrocarbon (PAH) contamination at Gas Works Park in Seattle, Washington.

- **Commencement Bay Preliminary Survey** - Finalized QA/QC plan and coordinated sample handling for the Commencement Bay, Washington preliminary survey.

- **Northwest Transformer Site Treatability Study** - Managed the laboratory audit and QA/QC review of dioxin, dibenzofurans, and PCB laboratory analyses for an *in-situ* vitrification treatability study at the Northwest Transformer site in Everson, Washington.

Environmental Investigations

- **Seattle City Light PCB Research** - Project Manager for the design and performance of PCB cleanup research projects for Seattle City Light. This entailed the analysis of various solid matrices for PCBs.

- **Contaminant Fate and Transport Evaluation** - Evaluated and interpreted data from laboratory leachate procedures performed on soil from a hazardous waste site in Kent, Washington. Provided expert legal testimony regarding environmental fate of site contaminants.

- **Georgetown Steam Plant Site Remediation** - Managed the collection and PCB analyses of soil samples at the Georgetown Steam Plant excavation in Seattle, Washington.

- **Blakely Island Marina Dredge Material Disposal Permitting** - Managed sediment sampling and obtained a PSDDA open-water disposal permit for Blakely Island Marina.

Regulatory Compliance

- **Environmental Manager, Scott Paper Company** - Managed environmental compliance with all state, Federal and local regulations and permit requirements for this pulp and paper mill.

- **Scott Paper Company NPDES Permit Compliance** - Assisted Scott Paper Company with compliance issues and NPDES permit and EPA 308 order implementation requirements.

- **Wastewater Discharge Evaluations** - Evaluated toxicity data and monitoring requirements for modified marine sewage outfall discharge permits under the Clean Water Act Section 301(h).

Laboratory Coordination

- **Washington Department of Ecology (Ecology) Puget Sound Ambient Monitoring Program (PSAMP) Sediment Sample Validation** - Managed laboratory contracting for Ecology's 1992 PSAMP and managed the validation of sediment samples according to Puget Sound Estuarine Protocols.
- **Commencement Bay Feasibility Study** - Managed the sediment sample coordination between field and laboratory for the Commencement Bay Feasibility Study, implemented chain-of-custody procedures, and arranged sample transport. Responsible for QA/QC planning and CLP data review for over 500 samples from Commencement Bay.
- **Anaconda Smelter Site Investigation** - Wrote the QAPP and developed laboratory analytical protocol and reviewed QA/QC project plans for collection and analysis of soil, sediment, tailings, and water.
- **Scott Paper Company Bioassay Laboratory Supervision** - Supervised bioassay laboratory and developed toxicity screening procedures for chemical evaluation at Scott Paper Company in Everett, Washington. Provided support for National Pollutant Discharge Elimination System (NPDES) permitting.

Data Validation

- **South Tacoma Landfill RI/FS** - Managed data validation review of organic and inorganic laboratory analyses of over 700 soil, groundwater, storm and surface water, and sediment samples for metals, organic, and conventional analyses.

- **PSDDA Baseline Monitoring** - Managed the QA/QC review of trace metals and conventional laboratory analysis of 100 sediment samples.

FORMER REGISTRATIONS AND PROFESSIONAL AFFILIATIONS

Registered Quality Assurance Professional in Good Laboratory Practices (RQAP-GLP)

Member, American Chemical Society

Member, Society of Environmental Toxicology and Chemistry

Member, Society of Quality Assurance

TESTIMONY

Ms. Bailey has provided background litigation support on numerous projects. She was deposed regarding data collected at a NPL site in Kent, Washington. She provided expert testimony at a King County (Washington) hearing regarding the quality of data that supported an environmental impact statement for a proposed mine site. In 2000, she was deposed regarding the quality of organochlorine data that was collected to support the Southern California Bight NRDA (Montrose Chemical).

PRESENTATIONS AND PUBLICATIONS

Bailey, A.K. 2009. *Remedial Data Used for NRDA Habitat Equivalency Analysis—Analyte and Detection Limit Concerns.* Presentation at Fifth International Conference on Remediation of Contaminated Sediments. Jacksonville, Florida.

Bailey, A.K. 2006. *Reference Materials as Indicators of Analytical Data Quality for Human and Ecological Risk Assessments.* Presentation at Tenth International Symposium on Biological and Environmental Reference Materials. Charleston, South Carolina.

Bailey, A. K. 1997. *Sampling and Analytical Quality Assurance/Quality Control.* Presentation for the University of Washington Contaminated Sediment Conference, University of Washington, Seattle, Washington.

Bailey, A. K. and C. A. Manen. 1995. *Use of Standard Reference Materials as Indications of Analytical Data Quality*. ACS Waste Testing and Quality Assurance Symposium. Washington, D.C.

Bailey, A. K. 1989. *Interpretation of Laboratory QA/QC results,* Pacific Northwest American Water Works Association Annual Conference. Eugene, Oregon.

Bailey, A. K. 1987. *Use of Respirometers to Project BOD and Potential Toxicity of Process Chemicals,* West Coast Regional Meeting of the National Council of the Paper Industry for Air and Stream Improvement. Portland, Oregon.

Bailey, A. K. and T. J. Bechtel. 1987. *Effluent Toxicity Testing at Scott Paper Company,* Pacific Northwest Pollution Control Association Annual Conference. Spokane, Washington.

Bailey, A. K., K. Kreps, and W. G. Hansen. 1987. *Evaluation of Decontamination of Solid Surfaces Exposed to PCBs,* 1987 EPRI PCB Seminar. Kansas City, Missouri.

Bailey, A. K. 1985. *ICP Data Evaluation,* AOAC Pacific Northwest Regional Meeting. Olympia, Washington.

Bailey, A. K. 1976. *Concentrations of Heavy Metals in the Sediments of a Hydroelectric Impoundment*, Montana Academy of Sciences, 36:165-170.

EMPLOYMENT HISTORY

1983-2014	President	EcoChem, Inc.
1988-1989	Environmental Manager	Scott Paper Company
1985-1986	Environmental Chemist	Scott Paper Company
1984-1985	Environmental Chemist	Tetra Tech, Inc.
1978-1983	Technical Director	AM Test, Inc.
1977-1978	Project Chemist	Betz-Converse-Murdock, Inc.
1975-1977	Research Assistant	University of Montana

AMIGOS BRAVOS' EXHIBIT 22

REBUTTAL TESTIMONY OF ANN K. BAILEY

Q: Please state your name?

A: Ann Bailey.

Q: Ms. Bailey, what is your educational background?

A: I graduated with a B.A. in biology from University of Oregon in 1972, and in 1976 I graduated from University of Montana with a Master's degree in Environmental Studies, focusing on chemical contaminant measurements.

Q: Would you please describe your professional experience?

A: After working as a bench chemist performing a wide range of analyses, I worked for four years as technical director of a commercial testing laboratory in Seattle, Washington. There I managed a wide range of testing services for a number of municipalities and industrial clients. In 1983 I founded an environmental consulting firm, which included setting up a field polychlorinated biphenyls ("PCB") testing laboratory utilizing equipment and methods similar to U.S. Environmental Protection Agency ("EPA") Method 608 for PCBs. In the 1990s, I provided quality assurance oversight of laboratories performing both Aroclor and PCB congener analyses for a number of environmental investigations. The past 20 years I performed historical data review of analytical test results throughout the United States for a number of ecological assessments, including the Pecos Mine in New Mexico. I also was the Quality Assurance Coordinator for a number environmental investigations, including drafting a Quality Management Plan for the Los Alamos National Laboratory Natural Resource Damage Assessment.

Q: Is Amigos Bravos Exhibit 21 an accurate copy of your curriculum vitae?

A: Yes

Q: Ms. Bailey, would you please summarize the expert opinions you will provide in your testimony?

A: Yes. In my testimony, I will describe the methodology of two EPA methods for analyzing PCBs: EPA Method 608.3, which quantitates PCBs as Aroclors, and EPA Method 1668C, which quantitates PCBs as congeners.

I will testify that, in my opinion, EPA Method 608.3 is not sufficiently sensitive nor sufficiently specific to detect total PCBs as required for the New Mexico Water Quality Control Commission's ("Commission") numeric water quality standards for wildlife or for human health (when aquatic organisms are consumed from waters containing PCBs), and that EPA Method 1668C can detect PCBs at the State's numeric limits.

I will testify that, in my opinion, the State of New Mexico should not limit itself to use of analytical methods listed in 40 CFR Part 136 ("Part 136 Methods") for purposes of compliance with permits. Currently the Commission's regulations authorize use of other analytical methods from reliable sources. There are other methods, such as EPA Method 1668C, that are sensitive enough to reliably report total PCBs to the State's numeric limits, and are available to the State to monitor the discharge of pollutants into New Mexico's surface waters.

Q: Ms. Bailey, what are polychlorinated biphenyls or PCBs?

A: PCBs are a family of chlorinated organic compounds formed by two benzene rings linked by a single carbon-carbon bond. There are 209 possible arrangements of chlorine atoms on the biphenyl group. Each individual arrangement or compound is called a congener. The sum of all the congener concentrations in a medium is the true total PCB concentration in that medium. Aroclors are mixtures of congeners produced by chemical manufacturers for specific commercial or marketing purposes.

PCBs are man-made chemicals that were widely used in electric transformers, hydraulic fluids, paint additives, plasticizers, adhesives, and fire retardants prior to being banned in the late 1970s. They also bioaccumulate and biomagnify, which means they increase in concentration both in individual organisms and with each successive level of the food chain.

Q: What experience do you have sampling and analyzing PCBs in water media?

A: In the 1980s I set up a mobile PCB laboratory and analyzed various environmental samples for Seattle City Light. While working at Scott Paper Company, Everett, Washington, I oversaw the collection and analysis of wastewater and stormwater samples for PCBs. In the 1990s through the 2010s I developed and reviewed sampling and analysis for a number of remedial investigations involving PCBs, as well as natural resource damage assessments.

Q: What EPA approved analytical methods are there for PCBs in water that you are familiar with?

A: Common EPA approved methods for PCBs in aqueous matrices are EPA Method 608.3, EPA Method 625, EPA Method 8082A, and EPA Method 1668C.

Q: Would you explain how each of these methods works, and the differences between them?

A: Analytical Method 608 was developed in the 1970s when PCBs were initially being monitored for environmental purposes, and is a method that measures Aroclor concentrations. The PCBs are extracted from the water sample, then analyzed by gas chromatography with an electron capture detector ("ECD"). Based on the instrument printout of individual peaks representing concentrations of individual congeners, a pattern-recognition technique is used to qualitatively determine whether or not an Aroclor mixture is present; then a set of standards using that particular Aroclor is used for quantitation. For the quantitation, the method uses a small subset of peaks (generally 3 to 5 peaks out of 60 to 80 congeners present in each Aroclor)

to determine the concentration of the PCB mixture. This type of estimation is difficult if there are mixtures of PCB Aroclors present or if the medium and its Aroclors are weathered or otherwise degraded, because the patterns will not be distinct. In addition PCB congeners are present in some materials not as Aroclors, and thus would not be quantitated by this method.

EPA Method 625 utilizes detection by mass spectrometer rather than ECD, and therefore can more definitively identify a compound. However, its drawback is that it is less sensitive than EPA Method 608.3.

Analytical Method 8082A is very similar to Method 608.3. It uses similar equipment, but has different quality control requirements. Detection limits using this method are similar to those using Method 608.3.

EPA Method 1668C, developed in the 1990s, measures individual PCB congeners by isotope dilution and internal standard high resolution gas chromatography/high resolution mass spectrometry ("HRGC/HRMS"). Because HRMS is used as the detector, positive identification is provided for each compound. (The detector used for Method 608.3 cannot provide a positive identification of a compound.) Water samples are extracted in a similar manner as for Method 608.3; however, rather than measuring only a mixture of congeners in seven Aroclors, Method 1668C identifies and quantitates the concentration of each of the 209 PCB congeners in the sample. (Note that some congeners coelute, and therefore the number of congeners individually quantitated is fewer.)

Q: You state that EPA Method 608.3 quantitates PCBs as Aroclors and EPA Method 1668C quantitates PCBs as congeners, what difference does this make when calculating total PCBs to compare to the Commission's numeric water quality standards?

A: EPA Method 608.3 only quantitates any presence of PCBs if the instrument read-out presents a pattern of concentrations that matches a particular Aroclor. This type of estimation is difficult if there are mixtures of PCB Aroclors present or if Aroclors are weathered or otherwise degraded, as the patterns will not be distinct. If the patterns are not distinct, the laboratory most likely will not include PCBs within the total PCB result. In addition PCB congeners are present in some materials not as Aroclors (*e.g.*, some dyes contain an individual PCB congener), and thus these PCBs would not be quantitated by this method.

In contrast, EPA Method 1668C quantitates PCB concentrations based on the presence of individual PCB congeners, then summing these individual concentrations to obtain a total PCB concentration. This quantitation of individual congeners is critical for determining a total PCB value. As stated in by EPA:

> . . . EPA offered a different approach for expressing human health criteria for PCBs. Human health criteria would no longer be based on individual Aroclors, but rather on total PCBs concentrations. In the environment, PCBs occur as mixtures of congeners but these are different in composition than commercial mixtures (Aroclors). This is because PCB mixtures can change over time through partitioning among different environmental media (e.g., water, sediment), by

chemically transforming or preferentially bioaccumulating. Therefore, it can be imprecise and inappropriate to characterize environmental mixtures in terms of Aroclors. It is the Agency's view that expressing the criteria in terms of total PCBs rather than individual Aroclors better reflects current scientific thought.

64 Fed. Reg. 61,182, 61,184 (Nov. 9, 1999) (citations omitted)

Q: Are you familiar with the Commission's numeric water quality standards for PCB's?

A: Yes.

Q: What are those standards?

A: The standards are below, set forth in a chart, with a legend following:

Pollutant	CAS Number	DWS	WH	Aquatic Life			Type
				Acute	Chronic	HH-OO	
Polychlorinated Biphenyls (PCBs)	1336-36-3	0.50 µg/L	0.014 µg/L	2 µg/L	0.014 µg/L	0.00064 µg/L	C, P

DWS: domestic water supply
WH: wildlife habitat
HH-OO: human health organism only
C: cancer causing
P: persistent
µg/L: micrograms per liter

Q: These numeric criteria are set forth at 20.6.4.900.J NMAC of the Commission's water quality standards, correct?

A: Yes.

Q: Can EPA Method 608.3, testing for Aroclors, detect PCBs at the numeric levels of the Commission's standards?

A: No. EPA Method 608.3 is not be able to detect PCBs at the numeric limits for wildlife habitat, aquatic life chronic, or for aquatic life human health-organism only.

The method detection limit ("MDL") for Aroclor 1242 listed in EPA's protocol for Method 608.3 is 0.065 µg/L. No MDLs (nor any quantitation limits) are provided in the method for the other seven Aroclors listed in the method. However, the limits would be similar.

Q: Can EPA Method 1668C, testing for congeners, detect PCBs at the numeric levels of the Commission's standards?

A: Yes, Method 1668C can detect PCB congeners in water as low as 3 picogram per liter (0.000003 µg/L). Detection and quantitation limits can vary from congener to congener but are generally significantly less than 0.00064 µg/L, the lowest numeric limit in the Commission's standards for PCBs.

Q: Ms. Bailey, are you familiar with EPA's proposal in 2010 to adopt Method 1668C as an approved method under 40 CFR Part 136?

A: Yes.

Q: Do you know why that proposal was not promulgated as a final EPA regulation?

A: EPA deferred nationwide approval of Method 1668C in 2012 on the basis of industry comments concerning the documentation for the validation study and other reasons, and industry had concerns on costs of the method. EPA observed that "some states indicated that they are already requiring this method for use in permits and for other purposes," and that "this decision does not negate the merits of this method for the determination of PCB congeners in regulatory programs . . . ." 77 Fed. Reg. 29,758, 29,763 (May 18, 2012) [Amigos Bravos' Exhibit 23].

Q: Have you reviewed the testimony of John Toll, submitted on behalf of Triad National Security, LLC, and the U.S. Department of Energy (collectively, "DOE"), Exhibit 7 of DOE's Notice of Intent to Present Technical Testimony?

A: Yes.

Q: How does Mr. Toll characterize EPA's reasons for not adopting Method 1668C?

A: Mr. Toll, on page 8, lines 8-14, of his testimony, states:

> That draft rule was not finalized (See 77 Fed. 9 Reg. 29,758, 29,763), and the use of method 1668C was not approved under 40 CFR Part 136, because the method did not withstand the scrutiny of EPA's approval process. Noted shortcomings included a need for new detection and quantitation procedures as recommended by the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in the Clean Water Act Programs, and technical issues identified by laboratories and data users.

Q: In your view, is this a fair characterization of EPA's reasons for not adopting Method 1668C?

A: No, it is not. Mr. Toll implies that EPA found that Method 1668C had the "noted shortcomings," when in fact EPA only recited the criticisms of industry, but did not adopt them as EPA findings or conclusions. EPA stated:

> … industry and industry groups/associations were critical of the method for various reasons. Commenters opposing the method provided a detailed critique of the method, the inter-laboratory study, the peer reviews and the other supporting documentation. Among the criticisms of the inter-laboratory study, commenters argued that . . . [identifying seven concerns along with concerns about cost].

77 Fed. 9 Reg. 29,758, 29,763 [Ex. 23].

Mr. Toll omits important language from its decision, including that it found that Method 1668C "is being used in some states in their regulatory programs and by other groups for some projects with good success." *Id.* EPA found that the data from Method 1668C "shows that recoveries and precision for this method are within the performance achievable with other approved methods" and that "[s]ome states indicated that they are already requiring this method for use in permits and for other purposes." *Id.* And, critical to the deliberations of the Commission, EPA stated that:

> **This decision does not negate the merits of this method for the determination of PCB congeners in regulatory programs or for other purposes when analyses are performed by an experienced laboratory.**

Id. (emphasis added). EPA, therefore, expressly acknowledged the merits of use of Method 1668C for purposes of regulatory programs, like the State of New Mexico's.

Q: In his testimony, Mr. Toll refers to EPA developing an alternative method for PCBs. Immediately following his testimony quoted above, on page 8, beginning at line 14, Mr. Toll says that:

> **These issues are still unresolved, and EPA has, over the subsequent decade, developed an alternative polychlorinated biphenyl ("PCB") congener method specifically to overcome the problems with 1668C as a method for compliance monitoring. That alternative method is currently being evaluated in a multi-laboratory validation study, which is a step in EPA's 40 CFR Part 136 approval process.**

What do you know about the development of an alternative method?

A: I recently spoke with a colleague at SGS AXYS Analytical Services Ltd. (formerly AXYS Analytical Services, Ltd.), one of the laboratories involved with the development of 1668A-C, about the development of another method to detect PCBs. EPA has a validation study underway for a PCB Method using gas chromatography mass spectrometry. Axys was not aware of any method number assigned to it. This method does not replace EPA Method 1668C. It is simply another method that could possibly be used for PCB congener determination. It would be more sensitive than Method 608.3, but less sensitive than 1668C.

Q: Has EPA recognized Method 1668C?

A: Yes. In April 2010, the EPA Office of Water published Method 1668C, entitled Method 1668C Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, which is Amigos Bravos' Exhibit 20.

Q: What is your expert opinion regarding the validity, accuracy, and sensitivity of EPA Method 1668C?

A: Having reviewed results and associated quality control information from EPA Method 1668C for a number of environmental investigations, I find the method to be accurate and sensitive for the determination of PCB congeners. The method for quantitation and identification is more specific than the methods used by Method 608.3.

Q: Ms. Bailey, based on your knowledge and experience including your experience with states and local governments, do you believe that states should have the flexibility to use sampling and analysis methods in addition to Part 136 Methods?

A: Yes. I agree with EPA's position that if there are concerns about specific chemicals, there should be the ability for a regulator to use reliable methods to assess the concentration of those chemicals at the concentration of concern.

Q: Is the testimony you've provided accurate to the best of your knowledge?

A: Yes.

C Bailey

Ann Bailey

6/21/2021

Date

AMIGOS BRAVOS' EXHIBIT 23

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Parts 136, 260, 423, 430, and 435

[EPA–HQ–OW–2010–0192; FRL–9664–6]

RIN 2040–AF09

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: This rule modifies the testing procedures approved for analysis and sampling under the Clean Water Act. EPA proposed these changes for public comment on September 23, 2010. The changes adopted in this final rule fall into the following categories: New and revised EPA methods and new and revised methods published by voluntary consensus standard bodies (VCSB), such as ASTM International and the Standard Methods Committee; updated versions of currently approved methods; methods reviewed under the alternate test procedures (ATP) program; clarifications to the process for EPA approval for use of alternate procedures for nationwide and Regional use; minimum quality control requirements to improve consistency across method versions; corrections to previously approved methods; and revisions to sample collection, preservation, and holding time requirements. Finally, EPA makes changes to three effluent guideline regulations.

DATES: This regulation is effective on June 18, 2012. The incorporation by reference of these methods is approved by the Director of the Federal Register on June 18, 2012. For judicial review purposes, this final rule is promulgated as of 1:00 p.m. (Eastern time) on June 1, 2012 as provided at 40 CFR 23.2 and 23.7.

ADDRESSES: EPA has established a docket for this action under Docket ID No. EPA–HQ–OW–2010–0192. All documents in the docket are listed on the *http://www.regulations.gov* Web site. Although listed in the index, some information is not publically available, *e.g.*, CBI or other information whose disclosure is restricted by statute. Certain other materials, such as copyrighted material, are not placed on the Internet and will be publicly available only in hard copy form. Publicly available docket materials are available either electronically through *http://www.regulations.gov* or in hard copy at the HQ Water Docket Center, EPA/DC, EPA West, Room 3334, 1301 Constitution Ave. NW., Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is 202–566–1744, and the telephone number is 202–566–2426 for the HQ Water Docket.

FOR FURTHER INFORMATION CONTACT: For information regarding the changes to inorganic chemical methods, contact Lemuel Walker, Engineering and Analysis Division (4303T), USEPA Office of Science and Technology, 1200 Pennsylvania Ave. NW., Washington, DC 20460, 202–566–1077 (email: *walker.lemuel@epa.gov*). For information regarding the changes to organic chemical methods, contact Maria Gomez-Taylor, Engineering and Analysis Division (4303T), USEPA Office of Science and Technology, 1200 Pennsylvania Ave. NW., Washington, DC 20460, 202–566–1005 (email: *gomez-taylor.maria@epa.gov*). For information regarding the changes to microbiological and whole effluent toxicity methods, contact Robin Oshiro, Engineering and Analysis Division (4303T), USEPA Office of Science and Technology, 1200 Pennsylvania Ave. NW., Washington, DC 20460, 202–566–1075 (email: *oshiro.robin@epa.gov*).

SUPPLEMENTARY INFORMATION:

A. General Information

1. Does this action apply to me?

EPA Regions, as well as States, Territories and Tribes authorized to implement the National Pollutant Discharge Elimination System (NPDES) program, issue permits with conditions designed to ensure compliance with the technology-based and water quality-based requirements of the Clean Water Act (CWA). These permits may include restrictions on the quantity of pollutants that may be discharged as well as pollutant measurement and reporting requirements. If EPA has approved a test procedure for analysis of a specific pollutant, the NPDES permittee must use an approved test procedure (or an approved alternate test procedure if specified by the permitting authority) for the specific pollutant when measuring the required waste constituent. Similarly, if EPA has established sampling requirements, measurements taken under an NPDES permit must comply with these requirements. Therefore, entities with NPDES permits will potentially be affected by the actions in this rulemaking. Categories and entities that may potentially be affected by the requirements of today's rule include:

Category	Examples of potentially affected entities
State, Territorial, and Indian Tribal Governments.	States, Territories, and Tribes authorized to administer the NPDES permitting program; States, Territories, and Tribes providing certification under Clean Water Act section 401; State, Territorial, and Indian Tribal owned facilities that must conduct monitoring to comply with NPDES permits.
Industry	Facilities that must conduct monitoring to comply with NPDES permits.
Municipalities	POTWs or other municipality owned facilities that must conduct monitoring to comply with NPDES permits.

This table is not intended to be exhaustive, but rather provides a guide for readers regarding entities likely to be affected by this action. This table lists types of entities that EPA is now aware of that could potentially be affected by this action. Other types of entities not listed in the table could also be affected. To determine whether your facility is affected by this action, you should carefully examine the applicability language at 40 CFR 122.1 (NPDES purpose and scope), 40 CFR 136.1 (NPDES permits and CWA) and 40 CFR 403.1 (Pretreatment standards purpose and applicability). If you have questions regarding the applicability of this action to a particular entity, consult the appropriate person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

B. What process governs judicial review of this rule?

Under Section 509(b)(1) of the Clean Water Act (CWA), judicial review of today's CWA rule may be obtained by filing a petition for review in a United States Circuit Court of Appeals within 120 days from the date of promulgation of this rule. For judicial review purposes, this final rule is promulgated as of 1 p.m. (Eastern time) on June 1, 2012 as provided at 40 CFR 23.2. The

III. Changes Between the Proposed Rule and the Final Rule

Except as noted below, the content of the final rule is the same as that of the proposed rule.

A. EPA Is Not Adding EPA Method 1614A

The Agency proposed to add Method 1614A, "Brominated Diphenyl Ethers in Water, Soil, Sediment, and Tissue by HRGC/HRMS." EPA developed this method to determine 49 polybrominated diphenyl ether (PBDE) congeners in aqueous, solid, tissue, and multi-phase matrices. This method uses isotope dilution and internal standard high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The commenters were divided on whether EPA should approve this method. Two commenters stated that Method 1614A would be a valuable addition to the list of approved methods, while two other commenters stated that the method has not been sufficiently validated for use in Clean Water Act programs. Upon further evaluation of the data supporting the use of this test procedure and the peer review comments, EPA agrees with those commenters who stated that additional validation data are needed to fully characterize the performance of this method for various matrices and has decided not to include Method 1614A in today's final rule.

B. Deferral of Action on EPA Method 1668C

The Agency proposed to add EPA Method 1668C, "Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS." This method measures individual chlorinated biphenyl congeners in environmental samples by isotope dilution and internal standard high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). As discussed in the proposal, Part 136 methods for chlorinated biphenyls (PCBs) only measure a mixture of congeners in seven Aroclors—PCB–1016, PCB–1221, PCB–1232, PCB–1242, PCB–1248, PCB–1254, and PCB–1260, while Method 1668C can measure the 209 PCB congeners in these mixtures.

EPA began development of this method in 1995, initially covering 13 congeners labeled "toxic" by the World Health Organization. In 1999, EPA expanded the scope of the method to include all 209 PCB congeners. The method has been used to support several studies, including the 2001 National Sewage Sludge Survey and the National Lake Fish Tissue Survey. Since 1999, EPA has revised the method to incorporate additional information and data collected such as the results of an inter-laboratory validation study, peer reviews of the method and the validation study data, additional QC performance criteria and MDL data, and user experiences. In the development and subsequent multi-laboratory validation of this method, EPA evaluated method performance characteristics, such as selectivity, calibration, bias, precision, quantitation and detection limits. The Agency is aware that this method is being used in some states in their regulatory programs and by other groups for some projects with good success. For example, in a study of data comparability between two laboratories on samples collected from the Passaic River in New Jersey, in which 151 PCB congeners were identified and measured, accuracy, as measured by analysis of an NIST SRM, was 15% or better. Recoveries of the PCB congeners ranged from 90% to 124% and averaged 105%; precision ranged from 4.2 to 23% (Passaic River 2010). This type of data shows that recoveries and precision for this method are within the performance achievable with other approved methods.

EPA received comments from thirty-five individuals or organizations on this method. Of these commenters, five (three states, one laboratory, and one laboratory organization) supported the approval of this method. Some states indicated that they are already requiring this method for use in permits and for other purposes. On the other hand, industry and industry groups/associations were critical of the method for various reasons. Commenters opposing the method provided a detailed critique of the method, the inter-laboratory study, the peer reviews and the other supporting documentation. Among the criticisms of the inter-laboratory study, commenters argued that: (1) EPA did not produce documentation supporting changes to the method approved by EPA for the interlaboratory study, (2) the raw data for wastewater and biosolids was poor and is not fit for use in a comprehensive interlaboratory study, (3) EPA cited certain guidelines such as ASTM but deviated from those guidelines (*e.g.*, used only one Youden pair per matrix), (4) the peer reviewers' qualifications were questioned, (5) the addendum and the pooled MDLs/MLs were not subjected to peer review, (6) MDL/ML are flawed, the process to calculate MDLs/MLs for congeners that co-elute was flawed, the MDL/ML ignored the ubiquitous problem of background contamination, and (7) the validation study did not include all matrices in the method (soil and sediment excluded). In addition, some commenters also suggested that EPA should first promulgate new detection and quantitation procedures. Further, commenters raised questions about possible adverse effects of this new method on compliance monitoring as well as concerns about data reporting and costs.

EPA is still evaluating the large number of public comments and intends to make a determination on the approval of this method at a later date. In the meantime, the Agency has decided to go forward with the promulgation of the other proposed analytical methods to expedite their implementation by the regulated community and laboratories. This decision does not negate the merits of this method for the determination of PCB congeners in regulatory programs or for other purposes when analyses are performed by an experienced laboratory.

C. EPA Is Not Adding ASTM Methods D7574–09 and D7485–09

In today's rule, EPA is not adding two proposed ASTM methods, ASTM D7574–09 "Standard Test Method for Determination of Bisphenol A (BPA)," and ASTM D7485–09 "Standard Test Method for Determination of NP, OP, NP1EO, and NP2EO." These two methods involve liquid chromatography and tandem mass spectrometry (LC/MS/MS). The methods have been tested by a single laboratory in several environmental waters, and may be useful for many applications. However, EPA has decided to postpone approval of these two methods for general use until completion of a full inter-laboratory validation study designed to fully characterize the performance of these methods across multiple laboratories and matrices.

D. Revisions and Clarifications to EPA Method 200.7

EPA Method 200.5 "Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma—Atomic Emission Spectrometry" employs a plasma torch viewed in the axial orientation to measure chemical elements (metals). As stated earlier in today's rule, EPA is adding Method 200.5 for some metals in Table IB. Both Methods 200.5 and 200.7 are acceptable methods under Part 136 and both methods employ ICP/AES technology. However, Method 200.5 includes performance data for the axial configuration that is not in Method 200.7 because the axial technology torch