ARCADIS
Infrastructure, environment, buildings

Mr. Mike Boudloche, Trustee
555 N. Carancahua, Suite 600
Corpus Christi, TX 78478

ARCADIS U.S., Inc.
711 North Carancahua
Suite 1080
Corpus Christi
Texas 78401
Tel 361 883 1353
Fax 361 883 7565

Subject:
Lettered Bins Building Cleaning Work Plan
Former Encycle Facility
5500 Up River Road
Corpus Christi, Texas
TCEQ SWR No. 30003; EPA ID No. TXD008117186

Date:
May 9, 2012

Contact:
Ken Brandner

Phone:
(361) 883-1353

Dear Mr. Boudloche:

As you are aware, demolition activities are currently in progress at the former Encycle/Texas, Inc. site located at 5500 Up River Road in Corpus Christi, Texas, as approved by the U.S. Bankruptcy Court for the Southern District of Texas (Case No. 05-21304) on November 29, 2010. As shown on Figure 1, eight buildings are currently present at the site, including the Lettered Bins Building (Building 14). The Lettered Bins Building is not being demolished at this time because it will be utilized during the upcoming soil remediation project for temporary storage and stabilization of excavated soils prior to offsite disposal. Using this approach, the excavated soils will be placed inside the building for stabilization, reducing the potential for airborne and storm water dispersion of particulates relative to outdoor stabilization as was originally proposed in the May 27, 2005 report entitled "Corrective Measures Study and Corrective Measures Implementation Work Plan".

As requested the January 2012 letter from the United States Environmental Protection Agency (EPA), provided herein is a work plan to clean the Lettered Bins Building prior to use of the building for temporary storage and stabilization of excavated soils during the upcoming soil remediation project. A copy of the January 2012 EPA letter is provided in Attachment 1.

LETTERED BINS BUILDING CONSTRUCTION DETAILS

The Lettered Bins Building was constructed in the early 1940's and is approximately 50,000 square feet in size. The building has an approximate 8-inch-thick reinforced concrete floor with 16 interior concrete bins. The bins are enclosed on three sides with 18-inch-thick reinforced concrete walls. An interior concrete driveway is present directly north of the bins, which provides truck access for bulk loading and unloading of excavated soils (see Figure 2). The south exterior wall of the building is constructed of reinforced concrete, and west, north, and east exterior walls and roof are constructed of steel-framed corrugated metal panels with an approximate 1/8-inch-thick black galbestos coating.

A structural integrity assessment of the Lettered Bins Building was conducted by a Texas-registered professional engineer (Gary Jaster of Jaster-Quintanilla & Associates, Inc.) during March 2011, and the building was determined to be structurally sound. A copy of the structural integrity assessment is provided in Attachment 2. Although the building was determined to be structurally sound, the structural engineer recommended that no work be conducted in the building if the wind speed exceeds tropical storm force (≥39 miles per hour).

An asbestos inspection of the site buildings was previously conducted as detailed in the December 14, 2006 report entitled "Asbestos Inspection Results, Encycle Facility, 5500 Up River Road, Corpus Christi, Texas". The asbestos inspection results showed that the galbestos coating on the corrugated metal panels contains asbestos. Samples of the galbestos were also collected for analysis of polychlorinated biphenyls (PCBs) as described in the September 28, 2011 "Galbestos Sample Collection and Analysis Plan". The PCB concentrations in the galbestos samples from the Lettered Bins Building ranged from 39 to 23,000 milligrams per kilogram (mg/kg).

LETTERED BINS BUILDING CLEANING PROCEDURES

General

The galbestos coating on the corrugated metal panels on the Lettered Bins Building walls and roof is deteriorating. Pieces of galbestos have fallen onto the floor inside the building and onto the soils outside of the building. Therefore, the fallen galbestos will be removed, the building will be cleaned, and deteriorated galbestos wall panels will be sealed prior to use of the building for temporary storage and stabilization of excavated soils. The proposed scope of work will include the following Tasks:

- Task 1: Remove fallen galbestos.
- Task 2: Seal building exterior walls.
- Task 3: Clean building floor.
- Task 4: Collect verification samples.

Task 1: Remove Fallen Galbestos

The galbestos that has fallen onto the concrete floor of the Lettered Bins Building and onto the soils outside of the Lettered Bins Building generally ranges in size from less than one square inch to approximately four square feet. Several large intact galbestos panels (approximately 4-foot by 8-foot panels) are also present on the ground directly northwest of the Lettered Bins Building. The fallen galbestos will be wetted and manually removed by EPA-accredited asbestos abatement workers. The asbestos abatement workers will remove all of the visibly identified galbestos pieces within a 100-foot radius of the Lettered Bins Building. The asbestos abatement workers will wear appropriate personnel protective equipment (PPE) for

handling of asbestos and PCBs, including chemical resistant gloves and boots, safety glasses, hard hats, Tyvek or polyethylene coveralls, and respirators with high-efficiency particulate air purifying (HEPA) respirators.

The galbestos pieces will be wetted and placed into plastic bags, except pieces of galbestos that are larger than the plastic bags, which will be wetted and wrapped in 6-milliliter plastic (Visqueen) sheeting. The galbestos will then be placed into plastic-lined roll-off boxes that will be tarped at the end of each working day. The galbestos will be disposed of at the U.S. Ecology Texas Hazardous Waste Landfill in Robstown, Texas. This landfill is authorized to accept asbestos waste, and also PCB bulk product waste in accordance with 40 CFR Part 761.62. A Uniform Hazardous Waste Manifest will accompany each shipment to the landfill. The manifest will include the appropriate waste codes taken from the Notice of Registration for the Encycle site (Waste Code Nos. 00013111 and 00203951). Each manifest will be signed by the transporter, the Encycle trustee or the trustee's designated representative, and the disposal facility. Additional details regarding removal of the fallen galbestos is provided in the "Asbestos Abatement/Cleanup Project Design Specifications" (see Attachment 3).

Task 2 – Seal Lettered Bins Building Exterior Walls

As discussed above, the west, north and east exterior walls of the Lettered Bins Building contain corrugated metal panels with galbestos coating. Based on a visual examination of the exterior wall panels by a Texas Department of State Health Services (TDSHS)-licensed asbestos inspector (Kenneth Brandner, TDSHS License No. 20-5050), approximately 600 square feet of the panels contain galbestos coating that is in poor condition. The galbestos coating on these exterior panels has a potential to flake/fall onto the ground outside of the building. Therefore, to prevent the galbestos from these panels to affect soils, these deteriorated panels will be sealed by EPA-accredited asbestos abatement workers. The deteriorated panels will be sealed using asbestos-free galvanized or fiberglass wall panels. The asbestos-free panels will be bonded to the exterior of the deteriorated panels using glue and/or sheet metal screws. If sheet metal screws are used, asbestos-free caulk will be applied to the existing panels at the point where drilling is performed. Additional details regarding sealing of the building exterior walls is provided in the "Asbestos Abatement/Cleanup Project Design Specifications" (see Attachment 3).

Task 3 – Clean Lettered Bins Building Floor

All of the visible pieces of galbestos will be removed from the Lettered Bins Building Floor as described above in Task 1. The entire building floor, including all 16 bins, the west entrance area, and the driveway, will then be cleaned by EPA-accredited asbestos abatement workers. The floor will be cleaned by wet-wiping and HEPA vacuuming. The cleaning materials and debris will be bagged and placed into a lined roll-off box. The roll-off box will be tarped at the end of each working day, and the cleaning materials will be transported to the U.S. Ecology Texas landfill in

Robstown, Texas for disposal as described above in Task 1. Additional details regarding cleaning of the Lettered Bins Building Floor is provided in the "Asbestos Abatement/Cleanup Project Design Specifications" (see Attachment 3).

Task 4 – Collect Verification Samples

Following completion of Tasks 1 through 3 above, verification samples will be collected for analyses of both asbestos and PCBs to confirm that the Lettered Bins Building has been adequately cleaned. The asbestos verification samples will consist of three air samples collected from the western, central, and eastern portions of the Lettered Bins Building, respectively. The air samples will be collected by an EPA-accredited air monitoring technician, and analyzed for asbestos using Phase-Contrast Microscopy (PCM). Each sample will be collected using an air sample pump with a minimum sample volume of 1,200 liters. The air samples will be analyzed for asbestos using NIOSH Method 7400, with a method detection limit (MDL) of 0.01 fibers per cubic centimeter (f/cc) or lower. The asbestos target cleanup concentration for each verification sample is 0.01 f/cc. If the verification sample analytical results show that the target cleanup concentration has not been achieved in one or more verification samples, additional cleaning of the building will be conducted and additional verification samples will be collected until the target cleanup concentration has been met.

The PCB verification samples will consist of 18 wipe samples collected from the Lettered Bins Building floor. As shown on Figure 2, one wipe sample will be collected from each of the 16 concrete bins, the west entrance area, and the concrete driveway. Prior to removal of the galbestos fragments from the building floor as described above in Task 1, the 18 wipe sample locations will be marked (spray painted) in areas where fallen galbestos pieces are observed, to ensure the wipe samples are representative of areas where fallen galbestos fragments had been present on the floor.

Each wipe sample will be collected from a 100 cm^2 area. The wipe samples will be collected using a laboratory-proved gauze pad soaked in hexane, then placed into a laboratory-provided widemouth glass jar with a Teflon-lined lid. The sample containers will be securely capped, labeled, placed into a cooler with ice, and submitted with chain-of-custody documentation to an independent analytical laboratory. The wipe samples will be analyzed for PCBs using EPA Method 8082A, with toluene as the extraction solvent. The EPA Method 8082A laboratory methodology is provided in Attachment 4, and will include analysis of Aroclor 1268.

The method detection limit (MDL) for PCB wipe sample analysis will be 10 micrograms per 100 square centimeters or lower. The target cleanup concentration for the PCB wipe samples will be 10 micrograms per 100 square centimeters in accordance with 40 CFR Part 761.61(a)(4)(ii). If the PCB verification sample analytical results show that the target cleanup concentration has not been achieved in one or more verification samples, additional cleaning of those areas of the building

will be conducted and additional verification samples will be collected until the target cleanup concentration has been met.

SCHEDULE

Implementation of this Lettered Bins Building Cleaning Work Plan can be initiated within 30 days following EPA approval of this Work Plan, and completed within 60 days following EPA approval of this Work Plan. A TDSHS Asbestos/Demolition Notification Form will be submitted to the TDSHS ten working days prior to start of work. Following completion of work, and letter report will be submitted to the TCEQ and EPA that documents the work conducted, and provides the verification sample analytical results and manifests.

In addition to this work plan, another work plan (Additional COPC Assessment Work Plan) is being prepared as a separate document. The Additional COPC Assessment Work Plan will include procedures for collection and analyses of additional soil samples from the areas to be excavated during upcoming soil remediation activities. The additional soil samples will be analyzed for the following constituents of potential concern (COPCs) that were not previously included in the RCRA Facility Investigation: (1) asbestos, (2) PCBs, and (3) polycyclic aromatic hydrocarbons. The Lettered Bins Building Cleaning will be completed first, followed by implementation of the Additional COPC Assessment Work Plan. Following completion of the Additional COPC Assessment, soil remediation activities will be conducted. The soil remediation activities are anticipated to require up to 18 months to complete. After the soil remediation activities have been completed, pending available funding, a work plan will be prepared for demolition of the Lettered Bins Building.

Sincerely,

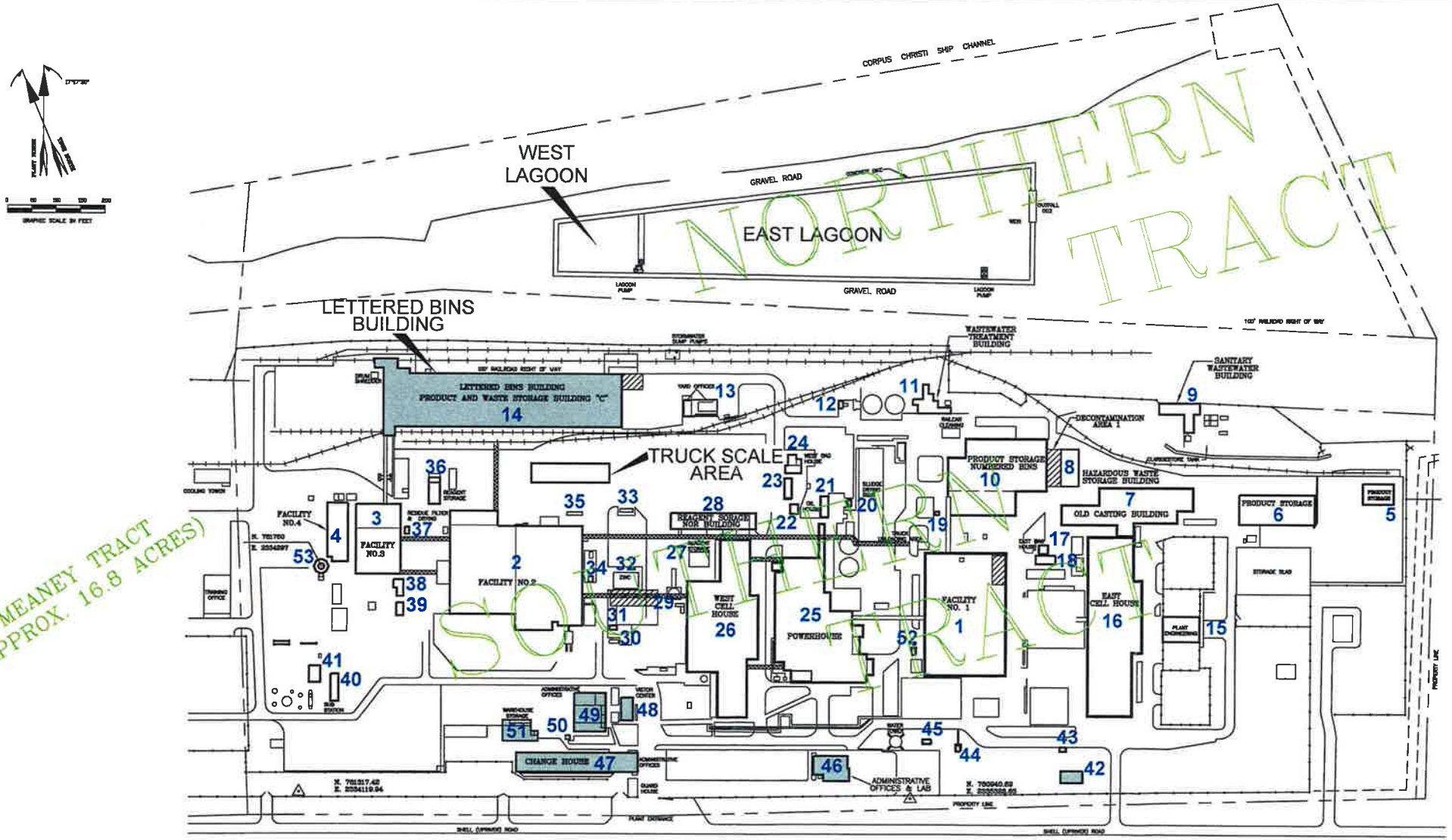
ARCADIS U.S., Inc

Kenneth Brandner

Kenneth Brandner, P.E., P.G.
Geological Engineer

Attachments

Figure 1:	Site Map
Figure 2:	Proposed PCB Verification Wipe Sample Locations
Attachment 1:	January 2012 EPA Letter
Attachment 2:	March 2011 Lettered Bins Building Structural Integrity Assessment
Attachment 3:	Asbestos Abatement/Cleanup Project Design Specifications
Attachment 4:	EPA Method 8082A



LEGEND

1 BUILDING ID NUMBER

DENOTES EXISTING BUILDING

ALL BUILDINGS NOT SHADED IN GRAY WERE DEMOLISHED DURING 2011 AND 2012

ARCADIS

SCALE VERIFICATION

THIS BAR REPRESENTS ONE INCH ON THE ORIGINAL DRAWING

USE TO VERIFY FIGURE REPRODUCTION SCALE

REV. NO.	DATE	DESCRIPTION	BY	APPR.

PROJECT NO.: [illegible] FILE NO.:

DRAWING: [illegible] PLOT SIZE: 11"x17"

DRAFTED BY: DPL DATE: 4/24/13

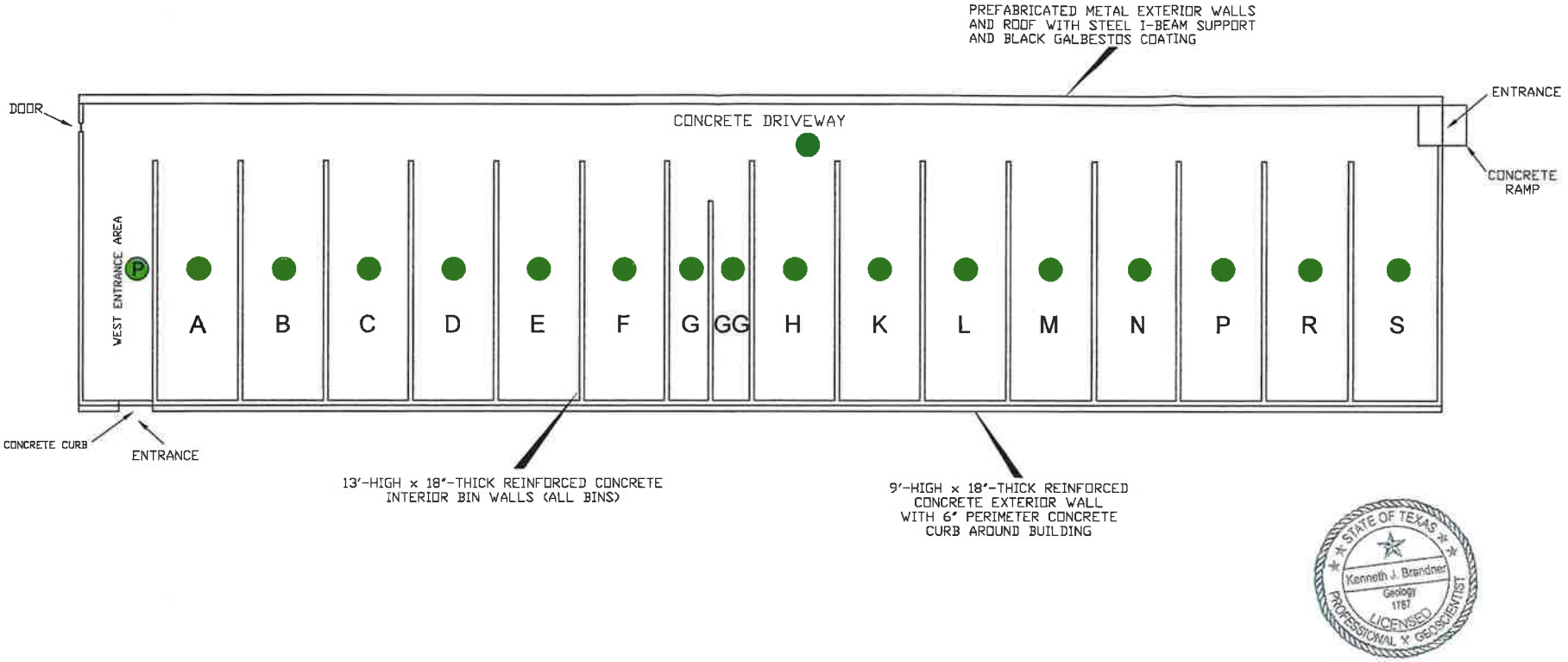
CHECKED BY: K. BRANDNER DATE:

APPROVED BY: K. BRANDNER DATE:

SITE MAP (UPDATED APRIL 2012)

CORPUS CHRISTI, TEXAS
ENCYCLE/TEXAS, INC.

FIGURE 1



LEGEND

CONTAINER STORAGE AREA

PROPOSED PCB WIPE SAMPLE LOCATION
(APPROXIMATE LOCATION, SAMPLE TO BE COLLECTED
DIRECTLY BELOW FALLEN GALBESTOS FRAGMENTS)

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PROPOSED
PCB VERIFICATION WIPE SAMPLE LOCATIONS
LETTERED BINS BUILDING
ENCYCLE/TEXAS, INC.
CORPUS CHRISTI, TEXAS

FIGURE
2

ATTACHMENT 1

JANUARY 2012 EPA LETTER

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 6
1445 ROSS AVENUE, SUITE 1200
DALLAS, TX 75202-2733

JAN 13 2012

JAN 23 2012

Mike Boudloche
Chapter 7 Bankruptcy Trustee for Encycle Texas
555 N. Carancahua, Suite 600
Corpus Christi, TX

RE: United States Environmental Protection Agency (EPA) Region 6 Response to the Analytical Report referring to Toxic Substances Control Act (TSCA) Polychlorinated Biphenyl (PCB) Regulations for the Encycle Facility located in Corpus Christi, Texas; EPA ID TXD008117186

Dear Mr. Boudloche:

The United States Environmental Protection Agency (EPA) Region 6 received an email November 16, 2011, from the office of your legal representative, Mr. Allan Kanner, which included a laboratory analytical report on Galbestos sampling at Encycle on October 11, 2011. The EPA appreciates your efforts in providing this report. The purpose of the sampling was to characterize the types of Galbestos waste identified at the Encycle facility to determine whether they are subject to the Toxic Substances Control Act polychlorinated biphenyl (PCB) regulations for bulk product waste (see 40 CFR 761.62). The laboratory analytical report showed that the PCB concentrations for all the Galbestos samples taken from the demolition area were less than 50 parts per million (ppm). Based on this finding, the Galbestos in the demolition area is not subject to the PCB regulations for bulk product waste.

However, EPA has other concerns related to Galbestos and PCBs at the site as follows:

1. Several of the samples of Galbestos from the Lettered Bins Building, which is not scheduled for demolition under the current demolition work, were reported to have PCB concentrations in excess of 50 ppm. If the Lettered Bins Building is demolished, the demolition, storage and disposal of the Galbestos on the building will need to be conducted in accordance with the PCB regulations referenced above.

2. Even if the Lettered Bins Building is not demolished, release of PCBs to the environment could potentially occur due to the age and weathered condition of the Galbestos on the building. Testing on site by EPA and the Texas Commission on Environmental Quality (TCEQ) has indicated that the Galbestos may be the source of the PCBs detected in soil and sediment on site.

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3. EPA understands that consideration is being given to temporarily storing soil in the Lettered Bins Building during the remediation phase of the work. If this is done, there may be a possibility that soil stored in the building could become contaminated from PCBs in the Galbestos siding/roofing. Any such use of the Lettered Bins Building for soil storage would require plans and procedures to be put in place to prevent contamination of the soil with PCBs while being stored.

4. As mentioned earlier, PCBs have been detected in the soil and sediment; therefore, PCBs must be included as a Constituent of Concern during site investigation/characterization and remediated as necessary in accordance with 40 CFR 761.61 (PCB remediation waste). Plans for characterization of the PCBs in the soil and sediment on site and for remediation, if necessary, will need to be reviewed and approved by EPA prior to implementation.

5. EPA understands that you have expressed an interest in resampling the Galbestos on the Lettered Bins Building. EPA and the TCEQ may want to take split samples in the event that you resample. Please provide EPA and TCEQ notice at least two weeks in advance of any such resampling, so that EPA and TCEQ will have adequate lead time to make necessary arrangements for split sampling.

If you have any questions regarding this matter, please contact me at (214) 665-8022 or Dr. Kishor Fruitwala, Chief of the Hazardous Waste Facility Assessment Section, at (214) 665-6669.

Sincerely,

Susan Spalding.
Associate Director for RCRA
Multimedia Planning and
Permitting Division

cc Robert E. Patton, Jr. TCEQ
Kenneth Bradner, Arcadis US, Inc.

ATTACHMENT 2

MARCH 2011 LETTERED BINS BUILDING STRUCTURAL INTEGRITY ASSESSMENT

JASTER-QUINTANILLA & ASSOCIATES, INC.
ENGINEERS

March 15, 2011

Mr. Mike Boudloche, Trustee
555 North Carancahua
Suite 600
Corpus Christi, TX 78478

Re: Structural Integrity Assessment
Former Encycle/Texas, Inc. /ASARCO Facility (Encycle)
5500 Up River Road
Corpus Christi, Nueces County, Texas

Dear Mr. Boudloche:

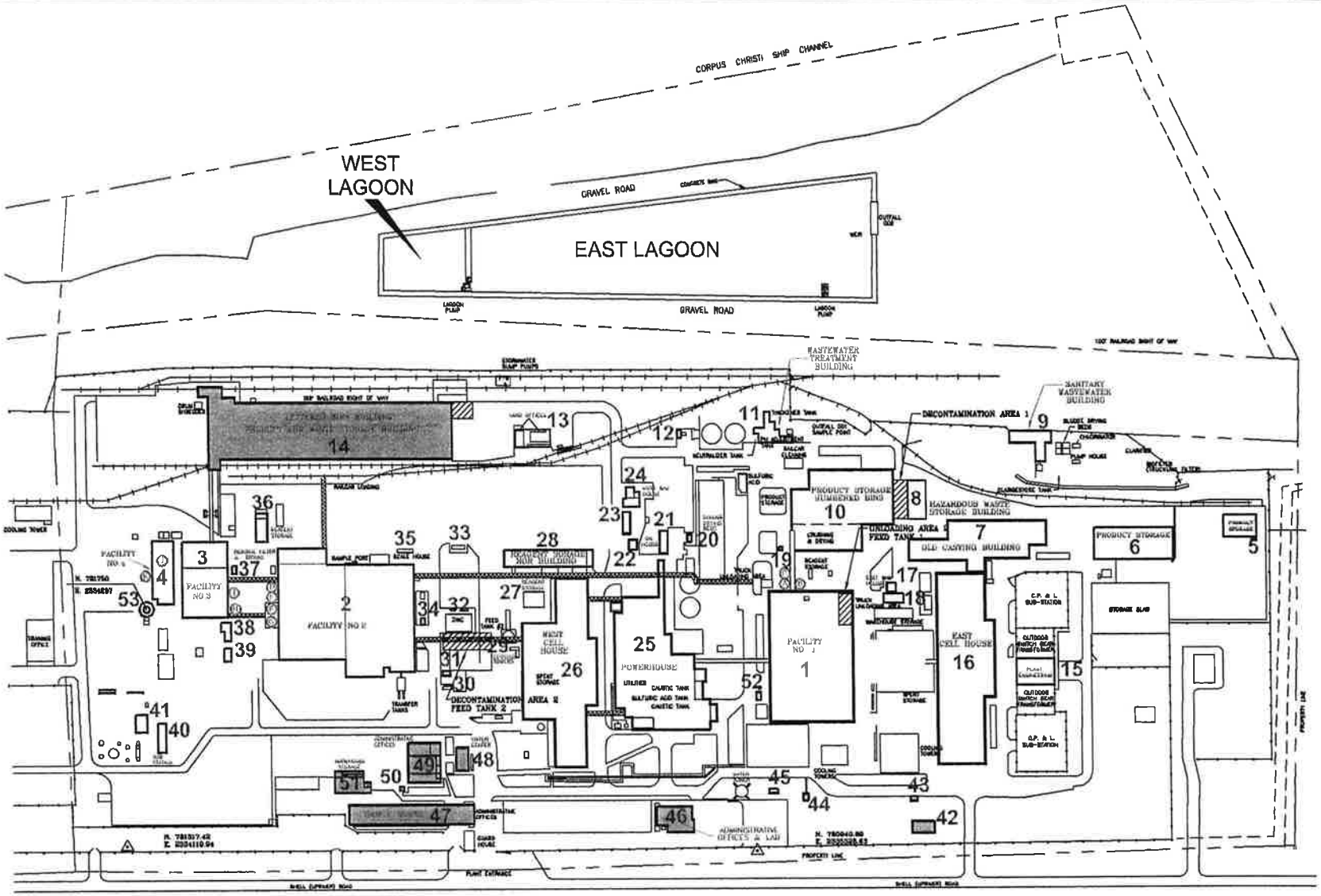
As you requested, I performed a visual observation and structural integrity assessment of Building Number 14 (Lettered Bins Building) at the former Encycle/ASARCO site on Wednesday, March 2, 2011 (Figure 1 is attached for building reference purposes).

The purpose of my observation and assessment was to identify portions of the building which are structurally unsound and should not be used for support during the asbestos abatement process. Mr. Armando G. Avalos, the trustee representative of the United States Bankruptcy Court, Southern District of Texas provided access to the site and Mr. Kenneth Brandner, P.E., of ARCADIS identified the building to be structurally assessed prior to initiation of asbestos abatement. My observation and opinion of the structural integrity of the building are based on visual assessment only. No drawings or design calculations of any of the structural elements of any part of the building framing or foundation were provided. No material testing of structural components was performed and no structural element was removed, uncovered or taken apart. My assessment of this building was made while standing on the ground surface and without benefit of any mechanical lifts or hoists. The assessment that follows is based on reasonable and prudent observations made during my visit to the site.

BUILDING 14 (Lettered Bins Building)

This building is a large predominantly conventional steel framed building with large three-sided concrete bins. In general this building appears to have sufficient structural integrity to allow for planned abatement, with abatement likely performed with ground level supported equipment.

The building has two elevated, interior catwalks near the center of the building spanning in the east-west direction. These catwalks are steel framed with a combination of checkered plate and wood decking serving as the deck surface. The decking appears to have adequate strength at the present time to support abatement personnel. There are areas at wood decking where gaps exist between



LEGEND

1 BUILDING ID NUMBER

A METAL SILOS

DENOTES BUILDING NOT TO BE DEMOLISHED

ARCADIS

SCALE VERIFICATION	REV. NO.	DATE	DESCRIPTION	BY	APPR.
THIS BAR REPRESENTS ONE INCH ON THE ORIGINAL DRAWING. USE TO VERIFY FIGURE REPRODUCTION SCALE					

BUILDING LOCATION MAP

CORPUS CHRISTI, TEXAS
ENCYCLE/TEXAS, INC.

FIGURE 1

ATTACHMENT 3

ASBESTOS ABATEMENT/CLEANUP PROJECT DESIGN SPECIFICATIONS

TURNSTONE EH&S, Inc.

April 23, 2012

Project Asbestos Consultant: Tim Chaney

1. Project Description:

The project referred to as the Lettered Bins Building at the Encycle Recycling Site located at 5500 Up River Road, Corpus Christi, Texas will consist of the clean-up and disposal of Galbestos debris on the interior and exterior of the building, to extend 100 feet from the building.

This material has fallen to the floor inside the building and onto the ground outside of the building from the walls and roof. The Lettered Bins Building is constructed with an 8-inch-thick reinforced concrete floor. The exterior south wall of the building is constructed of reinforced concrete. The west, north, and east exterior walls and the roof of the building are constructed with a metal frame containing corrugated metal panels with an approximate 1/8-inch-thick exterior black galbestos coating. 16 concrete bins are located inside the building, and each bin has reinforced concrete walls on three sides (east, south, and west sides). An interior driveway is located on the north side of the building interior, which serves as a truck loading/unloading area for each of the 16 bins.

The building interior scope of work will include removal of the fallen galbestos that is currently lying on the concrete floor of the building (in pieces ranging from less than 1 square inch to approximately 4 square feet). After removal of the fallen galbestos, the building floor shall be wet cleaned/vacuumed to remove any remaining galbestos pieces from the floor. The galbetsos pieces that can fit into a plastic bag shall be double bagged. The galbestos pieces that are too large for a plastic bag shall be wrapped in plastic. All of the removed galbestos shall be placed into a plastic lined roll off box that is tarped, then transported to the landfill (U.S. Ecology, Robstown, Texas).

The building interior can be subdivided into 18 areas for cleaning purposes: the 16 individual bins, the driveway north of the bins, and the concrete storage area west of Bin A (see attached map).

GENERAL INFORMATION

All work will be performed under the supervision of an accredited Asbestos Supervisor/Competent Person. This person will have current training, having taken a 40 hr. Contractor/Supervisor initial or 8 hr. refresher within the last 12 months. For handling of friable materials, workers will have accreditation as Asbestos workers, having taken a 32 hr. Asbestos Worker initial or 8 hr. refresher in the last 12 months. Intact materials may be handled by persons with a Class IV training, Awareness Training. These workers may not disturb the material or handle material that becomes friable. All abatement activities shall be implemented in accordance with this Work Plan and all applicable federal, state and local regulations, where appropriate.

Tim Chaney: Asbestos Consultants License No.: 105670; Expiration Date: April 30, 2014

Project Specific Scope of Work

The asbestos containing material (ACM) abatement project consists of the clean-up and disposal of Galbestos siding and roofing debris. This material is inside the building and on the ground outside the building, having fallen from the exterior of the building. The abatement contractor will also put non-ACM panels over existing panels to weatherproof the building.

Work Practices:

- Protective clothing: disposable full body coveralls with hood, rubber boots.
- Respiratory protection: at least ½ face with P-100 filtration.
- Electrical Safety: All power tools in the regulated area to be protected with Ground Fault Circuit Interrupters.
- Critical Barriers: Work inside the building. Areas free of contamination will be sealed from the Regulated Area through the use of critical barriers consisting of at least one layer of 6 mil thick polyethylene sheeting. Entrances to the building will have air-lock doors applied. Air conditioning ducts will be sealed with 2 layers of at least 6 mil thick polyethylene sheeting. Negative pressure is not required for this project.
- For outside debris cleanup, workers will thoroughly wet Galbestos debris prior to handling. Pieces will be immediately placed into a waste bag or container. Once materials are lowered to the ground, they must be spray with water before being containerized.
- Plastic sheeting will be placed under work on the building exterior walls.
- Worker Decontamination: At the edge of the work location a Decontamination area will be established utilizing 6 mil (.006 inch) plastic sheeting to cover the ground. This area will be large enough to allow staging of waste, cleaning of equipment, and decontamination of workers/clothing. Workers will clean work clothing/disposable suits with a HEPA vacuum before removing in the Decontamination Area. Entry to Regulated Area to be through Decontamination Area.
- Regulation of work area: signs meeting the requirements of OSHA 29 CFR1926.1101 (k)(7) must be placed at all approaches to the work area. Asbestos barricade tape must be placed across all access points to work area. Signage must be in a language that can be understood by all persons in and adjacent to the work area (English and Spanish).
- Waste Load Out: bagged or wrapped waste will be staged in the Decontamination Area. Bags of waste are to be cleaned of visible dust/debris before placement in waste truck/dumpster. Larger pieces of debris may be component wrapped in 2 layers of minimum 6 mil polyethylene.
- A working HEPA vacuum is to be on site for cleaning of worker clothing and cleaning up debris.
- Amended water will be applied to friable and non-friable material before and during handling. Broken pieces will be placed in labeled 6 mil bags (drums may be used) or wrapped in 6 mil sheeting and sealed. All friable materials are to be double-bagged or double-wrapped.
- Areas inside the building where Galbestos debris is found will be cleaned by wet wiping and HEPA vacuuming. Areas inside the building where debris cleanup is performed are to be free of visible dust and debris prior to clearance sampling.
- Disposal: Disposal will be according to all federal, state, and local requirements. If the total amount of friable material exceeds 260 linear feet, 160 square feet, or 35 cubic feet, the material will be disposed of at a Class I landfill and notification is required (to the Texas Department of State Health Services) 10 working days prior to work commencement. Material to be measured in units normally used for that type of material (e.g., linear feet for pipe). Individual landfills may have more restrictive requirements.

- Application of non-ACM panels over existing Galbestos panels - No disturbance of existing panels is allowed during panel installation. This includes sanding, grinding, abrading, or saw cutting (the asbestos NESHAP does allow slicing, shearing, or punching using sharp edged instruments). If panels are to be removed, nails or screws should be removed with a chisel, or removed with a wrench where possible. The contractor should avoid drilling through the existing panels where possible. If drilling is necessary, a caulk should be applied to the Galbestos at the point where drilling is performed.

Air Monitoring:

- Personnel – The abatement contractor is responsible for OSHA compliance monitoring of workers. The contractor may rely on a Negative Exposure Assessment if one has been developed.
- Clearance Sampling– The Air Monitoring Technician will determine the number and location(s) of Clearance Air Samples to be collected. The Air Monitoring Technician will verify that the areas that were cleaned are free of visible dust and debris.
- Analysis by Phase-Contrast Microscopy (PCM) is acceptable. Samples are to be collected and analyzed by NIOSH method 7400. A minimum of 1200 liters per sample is required.

Waste Management:

- ACM waste must be wetted before it is picked up and placed in a minimum 6 mil bag and sealed with duct tape. Bag is to be cleaned before double-bagging. Drums or other suitable waste containers are acceptable, but drums are to have a waste bag inside. Drums will be sealed and have OSHA labels and generator labels. Larger pieces may be component wrapped in two layers of sheeting of minimum 6 mil thickness, sealed with duct tape.
- Amended water to be used, mixed according to manufacturers' instruction. Airless sprayer or hand pumps to be used for application.
- Prior to bag out to roll-off box, all ACM waste must be double bagged or wrapped.
- If double bagged, exterior waste bags must also be 6 mil minimum and tied "goose neck" fashion.
- All containerized ACM waste must be identified with asbestos labels per OSHA 29 CFR 1926.1101. Generator labels will be applied before waste leaves the Regulated Area.
- All containerized ACM waste must be transferred to a roll-off or similar transport lined with 2 layers of 6 mil minimum thickness polyethylene sheet and covered or enclosed.
- All Transportation and Disposal must be conducted in compliance with all applicable state, federal, and local regulations.

Project Map - The on-site project manager will develop the site map for Clearance Sampling

Tim Chaney: Asbestos Consultants License No.: 105670; Expiration Date: April 30, 2014

ATTACHMENT 4

EPA METHOD 8082A

METHOD 8082A

POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be followed by individuals formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method may be used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors or as individual PCB congeners in extracts from solid, tissue, and aqueous matrices, using open-tubular, capillary columns with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). The Aroclors and PCB congeners listed below have been determined by this method, using either a single- or dual column analysis system, and this method may be appropriate for additional congeners and Aroclors (see Sec. 1.4). The method also may be applied to other matrices such as oils and wipe samples, if appropriate sample extraction procedures are employed.

Compound	CAS Registry No.[a]	IUPAC #
Aroclor 1016	12674-11-2	-
Aroclor 1221	11104-28-2	-
Aroclor 1232	11141-16-5	-
Aroclor 1242	53469-21-9	-
Aroclor 1248	12672-29-6	-
Aroclor 1254	11097-69-1	-
Aroclor 1260	11096-82-5	-
2-Chlorobiphenyl	2051-60-7	1
2,3-Dichlorobiphenyl	16605-91-7	5
2,2',5-Trichlorobiphenyl	37680-65-2	18
2,4',5-Trichlorobiphenyl	16606-02-3	31
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	44
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	52
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	87
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	110
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	141

Compound	CAS Registry No.[a]	IUPAC #
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	151
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	153
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	170
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	180
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	183
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	187
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206

[a]Chemical Abstract Service Registry No.

1.2 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns compared to those of Aroclor standards.

1.3 The seven Aroclors listed in Sec. 1.1 are those that are commonly specified in EPA regulations. The quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment. Therefore, this method provides procedures for the determination of a selected group of the 209 possible PCB congeners, as another means to measure the concentrations of weathered Aroclors. The 19 PCB congeners listed above have been tested by this method and were chosen for testing because many of them represent congeners specific to the common Aroclor formulations (see Table 6). These 19 PCB congeners do not represent the co-planar PCBs or the other PCBs of greatest toxicological significance. **The analytical procedures for these 19 congeners may be appropriate for the analysis of other congeners not specifically included in this method and may be used as a template for the development of such a procedure.** However, all 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either document the resolution of the congeners in question, or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

1.4 The PCB congener approach potentially affords greater quantitative accuracy when PCBs are known to be present. As a result, this method may be used to determine Aroclors, some PCB congeners, or "total PCBs," depending on regulatory requirements and project needs. The congener method is of particular value in determining weathered Aroclors. However, analysts should use caution when using the congener method when regulatory requirements are based on Aroclor concentrations. Also, this method is not appropriate as currently written for the determination of the co-planar PCB congeners at the very low (sub part per trillion) concentrations sometimes needed for risk assessment purposes.

1.5 Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS (e.g., Method 8270) is also recommended as a confirmation technique, if sensitivity permits (also see Sec. 11.11 of this method). GC/AED may also be used as a confirmation technique, if sensitivity permits (see Method 8085).

1.6 This method includes a dual-column option that describes a hardware configuration in which two GC columns are connected to a single injection port and to two separate detectors. The option allows one injection to be used for dual-column simultaneous analysis.

1.7 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.). Example chromatograms and GC conditions are provided as guidance.

1.8 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, and 8000) for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.9 Use of this method is restricted to use by, or under the supervision of, personnel appropriately experienced and trained in the use of gas chromatographs (GCs) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using the appropriate matrix-specific sample extraction technique.

2.1.1 Aqueous samples may be extracted at neutral pH with methylene chloride using either Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), Method 3535 (solid-phase extraction), or other appropriate technique or solvents.

2.1.2 Solid samples may be extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3540 (Soxhlet), Method 3541 (automated Soxhlet), Method 3545 (pressurized fluid extraction), Method 3546 (microwave extraction), Method 3550 (ultrasonic extraction), Method 3562 (supercritical fluid extraction), or other appropriate technique or solvents.

2.1.3 Tissue samples may be extracted using Method 3562 (supercritical fluid extraction), or other appropriate technique. The extraction techniques for other solid matrices (see Sec. 2.1.2) may be appropriate for tissue samples.

2.2 Extracts for PCB analysis may be subjected to a sequential sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, this method is not applicable to the analysis of those compounds. Instead, use Method 8081.

2.3 After cleanup, the extract is analyzed by injecting a measured aliquot into a gas chromatograph equipped with either a narrow- or wide-bore fused-silica capillary column and either an electron capture detector (GC/ECD) or an electrolytic conductivity detector (GC/ELCD).

2.4 The chromatographic data may be used to determine the seven Aroclors in Sec. 1.1, selected individual PCB congeners, or total PCBs (see Secs. 11.8 and 11.9).

3.0 DEFINITIONS

Refer to Chapter One and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Four for general guidance on the cleaning of glassware. Also refer to Methods 3500, 3600, and 8000 for a discussion of interferences.

4.2 Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories, as follows:

4.2.1 Contaminated solvents, reagents, or sample processing hardware.

4.2.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

4.2.3 Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD).

NOTE: A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples.

4.2.4 Coelution of related analytes -- All 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either

document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

4.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

4.3.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

4.3.2 Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.3.3 These materials can be removed prior to analysis using Method 3665 (sulfuric acid/permanganate cleanup).

4.4 Cross-contamination of clean glassware can routinely occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned.

4.4.1 Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware, and dry it in an oven at 130 °C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

CAUTION: Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. Therefore, exercise caution, and do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

4.4.2 Other appropriate glassware cleaning procedures may be employed, such as using a muffle furnace at 430 °C for at least 30 min. However, analysts are advised not to place volumetric glassware in a muffle furnace, since the heat will burn off the markings on the glassware and may warp the glassware, changing its volume.

4.5 Sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur contamination should be expected with sediment samples. Sulfur can be removed through the use of Method 3660.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 Gas chromatograph -- An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all necessary accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and recorder/integrator or data system. Electrolytic conductivity detectors (ELCDs) may also be employed if appropriate for project needs. If the dual-column option is employed, the gas chromatograph must be equipped with two separate detectors.

6.2 GC columns

This method describes procedures for both single-column and dual-column analyses. The single-column approach involves one analysis to determine that a compound is present, followed by a second analysis to confirm the identity of the compound (Sec. 11.11 describes how GC/MS confirmation techniques may be employed). The single-column approach may employ either narrow-bore ($\leq$ 0.32-mm ID) columns or wide-bore (0.53-mm ID) columns. The dual-column approach generally employs a single injection that is split between two columns that are mounted in a single gas chromatograph. The dual-column approach generally employs wide-bore (0.53-mm ID) columns, but columns of other diameters may be employed if the analyst can demonstrate and document acceptable performance for the intended application. A third alternative is to employ dual columns mounted in a single GC, but with each column connected to a separate injector and a separate detector.

The columns listed in this section were the columns used in developing the method. The listing of these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Laboratories may use these columns or other columns provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.2.1 Narrow-bore columns for single-column analysis (use both columns to confirm compound identifications unless another confirmation technique such as GC/MS is employed). Narrow-bore columns should be installed in split/splitless (Grob-type) injectors.

6.2.1.1 30-m x 0.25-mm or 0.32-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1-µm film thickness.

6.2.1.2 30-m x 0.25-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, or equivalent), 2.5 µm coating thickness, 1-µm film thickness.

6.2.2 Wide-bore columns for single-column analysis (use two of the three columns listed to confirm compound identifications unless another confirmation technique

such as GC/MS is employed). Wide-bore columns should be installed in 1/4-inch injectors, with deactivated liners designed specifically for use with these columns.

6.2.2.1 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5-µm or 0.83-µm film thickness.

6.2.2.2 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

6.2.2.3 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5-µm film thickness.

6.2.3 Wide-bore columns for dual-column analysis -- The three pairs of recommended columns are listed below.

6.2.3.1 Column pair 1

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5-µm film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

Column pair 1 is mounted in a press-fit Y-shaped glass 3-way union splitter (J&W Scientific, Catalog No. 705-0733) or a Y-shaped fused-silica connector (Restek, Catalog No. 20405), or equivalent.

NOTE: When connecting columns to a press-fit Y-shaped connector, a better seal may be achieved by first soaking the ends of the capillary columns in alcohol for about 10 sec to soften the polyimide coating.

6.2.3.2 Column pair 2

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 0.83-µm film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

Column pair 2 is mounted in an 8-in. deactivated glass injection tee (Supelco, Catalog No. 2-3665M), or equivalent.

6.2.3.3 Column pair 3

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5-µm film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (HP-608, DB-608, SPB-608, RTx-35, or equivalent), 0.5-µm film thickness.

Column pair 3 is mounted in separate injectors and separate detectors.

6.3 Column rinsing kit -- Bonded-phase column rinse kit (J&W Scientific, Catalog No. 430-3000), or equivalent.

6.4 Volumetric flasks -- 10-mL and 25-mL, for preparation of standards.

6.5 Analytical balance, capable of weighing to 0.0001 g.

7.0 REAGENTS AND STANDARDS.

7.1 Reagent-grade or pesticide-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at ≤6 °C in polytetrafluoroethylene (PTFE)-sealed containers in the dark. When a lot of standards is prepared, aliquots of that lot should be stored in individual small vials. All stock standard solutions must be replaced after one year, or sooner if routine QC (see Sec. 9.0) indicates a problem. All other standard solutions must be replaced after six months, or sooner if routine QC (see Sec. 9.0) indicates a problem.

7.2 Solvents used in the extraction and cleanup procedures (appropriate 3500 and 3600 series methods) include *n*-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane) and the solvents must be exchanged to *n*-hexane or isooctane prior to analysis. Therefore, *n*-hexane and isooctane will be required in this procedure. All solvents should be pesticide grade in quality or equivalent, and each lot of solvent should be determined to be free of phthalates.

7.3 The following solvents may be necessary for the preparation of standards. All solvent lots must be pesticide grade in quality or equivalent and should be determined to be free of phthalates.

7.3.1 Acetone, $(CH_3)_2CO$

7.3.2 Toluene, $C_6H_5CH_3$

7.4 Organic-free reagent water -- All references to water in this method refer to organic-free reagent water as defined in Chapter One.

7.5 Standard solutions

The following sections describe the preparation of stock, intermediate, and working standards for the compounds of interest. This discussion is provided as an example, and other approaches and concentrations of the target compounds may be used, as appropriate for the intended application. See Method 8000 for additional information on the preparation of calibration standards.

7.6 Stock standard solutions (1000 mg/L) -- May be prepared from pure standard materials or can be purchased as certified solutions.

7.6.1 Prepare stock standard solutions by accurately weighing 0.0100 g of pure compound. Dissolve the compound in isooctane or hexane and dilute to volume in a 10-mL volumetric flask. If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution.

7.6.2 Commercially-prepared stock standard solutions may be used at any concentration if they are certified by the manufacturer or by an independent source.

7.7 Calibration standards for Aroclors

7.7.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

Prepare a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector. See Method 8000 for additional information regarding the preparation of calibration standards.

7.7.2 Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described in Sec. 7.7.1 have been used to demonstrate the linearity of the detector, these single standards of the remaining five Aroclors also may be used to determine the calibration factor for each Aroclor when a linear calibration model through the origin is chosen (see Sec. 11.4). Prepare a standard for each of the other Aroclors. The concentrations should generally correspond to the mid-point of the linear range of the detector, but lower concentrations may be employed at the discretion of the analyst based on project requirements.

7.7.3 Other standards (e.g., other Aroclors) and other calibration approaches (e.g., non-linear calibration for individual Aroclors) may be employed to meet project needs. When the nature of the PCB contamination is already known, use standards of those particular Aroclors. See Method 8000 for information on non-linear calibration approaches.

7.8 Calibration standards for PCB congeners

7.8.1 If results are to be determined for individual PCB congeners, then standards for the pure congeners must be prepared. The table in Sec. 1.1 lists 19 PCB congeners that have been tested by this method along with the IUPAC numbers designating these congeners. This procedure may be appropriate for other congeners as well, but the analyst must either document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

7.8.2 Stock standards may be prepared in a fashion similar to that described for the Aroclor standards, or may be purchased as commercially-prepared solutions. Stock standards should be used to prepare a minimum of five concentrations by dilution of the stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector.

7.9 Internal standard

7.9.1 When PCB congeners are to be determined, the use of an internal standard is highly recommended. Decachlorobiphenyl may be used as an internal standard, added to each sample extract prior to analysis, and included in each of the initial calibration standards.

7.9.2 When PCBs are to be determined as Aroclors, an internal standard is typically not used, and decachlorobiphenyl is employed as a surrogate (see Sec. 7.10).

7.9.3 When decachlorobiphenyl is an analyte of interest, as in some PCB congener analyses, see Sec. 7.10.3.

7.10 Surrogate standards

The performance of the method should be monitored using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards. The choice of surrogate compounds will depend on analysis mode chosen, e.g., Aroclors or congeners. The following compounds are recommended as surrogates. Other surrogates may be used, provided that the analyst can demonstrate and document performance appropriate for the data quality needs of the particular application.

7.10.1 When PCBs are to be determined as Aroclors, decachlorobiphenyl may be used as a surrogate, and is added to each sample prior to extraction. Prepare a solution of decachlorobiphenyl in acetone. The recommended spiking solution concentration is 5 mg/L. Tetrachloro-*m*-xylene also may be used as a surrogate for Aroclor analysis. If used, the recommended spiking solution concentration is 5 mg/L in acetone. (Other surrogate concentrations may be used, as appropriate for the intended application.)

7.10.2 When PCB congeners are to be determined, decachlorobiphenyl is recommended for use as an internal standard, and therefore it cannot also be used as a surrogate. Tetrachloro-*m*-xylene may be used as a surrogate for PCB congener analysis. The recommended spiking solution concentration is 5 mg/L in acetone. (Other surrogate concentrations may be used, as appropriate for the intended application.)

7.10.3 If decachlorobiphenyl is a target congener for the analysis, 2,2',4,4',5,5'-hexabromobiphenyl may be used as an internal standard or a surrogate.

7.11 DDT analog standard -- Used to determine if the commonly found DDT analogs (DDT, DDE, and DDD) elute at the same retention times as any of the target analytes (congeners or Aroclors). A single standard containing all three compounds should be sufficient. The concentration of the standard is left to the judgement of the analyst.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Four, "Organic Analytes."

8.2 Extracts should be stored under refrigeration in the dark and should be analyzed within 40 days of extraction.

NOTE: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 for QC procedures to ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to Method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 8000, 3500, or 3600.

9.3 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples.

9.3.1 Include a calibration standard after each group of 20 samples (it is *recommended* that a calibration standard be included after every 10 samples to minimize the number of repeat injections) in the analysis sequence as a calibration check. Thus, injections of method blank extracts, matrix spike samples, and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total. The response factors for the calibration should be within ±20 percent of the initial calibration (see Sec. 11.6.2). When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

9.3.2 Whenever quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different from the average area calculated during initial calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criteria must be reanalyzed. The retention times of the internal standards must also be evaluated. A retention time shift of >30 sec necessitates reanalysis of the affected sample.

9.4 Initial demonstration of proficiency

9.4.1 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 for information on how to accomplish a demonstration of proficiency.

9.4.2 It is suggested that the QC reference sample concentrate (as discussed in Methods 8000 and Method 3500) contain PCBs as Aroclors at 10-50 mg/L in the concentrate for water samples, or PCBs as congeners at the same concentrations. A 1-mL volume of this concentrate spiked into 1 L of reagent water will result in a sample concentration of 10-50 µg/L. If Aroclors are not expected in samples from a particular source, then prepare the QC reference samples with a mixture of Aroclors 1016 and 1260. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for the QC reference sample. See Method 8000 for additional information on how to accomplish this demonstration. Other concentrations may be used, as appropriate for the intended application.

9.4.3 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples. Refer to Method 8000 for procedures for evaluating method performance.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the laboratory should monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

9.6 Sample quality control for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, method sensitivity). At a minimum, this should include the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample when surrogates are used. Any method blanks, matrix spike samples, and replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike

duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair, spiked with the Aroclor 1016/1260 mixture. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for spiking. Consult Method 8000 for information on developing acceptance criteria for the MS/MSD.

9.6.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Consult Method 8000 for information on developing acceptance criteria for the LCS.

9.6.3 Also see Method 8000 for the details on carrying out sample quality control procedures for preparation and analysis. In-house acceptance criteria for evaluating method performance should be developed using the guidance found in Method 8000.

9.7 Surrogate recoveries

If surrogates are used, the laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000 for information on evaluating surrogate data and developing and updating surrogate limits. Procedures for evaluating the recoveries of multiple surrogates and the associated corrective actions should be defined in an approved project plan.

9.8 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

10.0 CALIBRATION AND STANDARDIZATION

See Sec. 11.0 for information on calibration and standardization.

11.0 PROCEDURE

11.1 Sample extraction

11.1.1 Refer to Chapter Two and Method 3500 for guidance in choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510), a continuous liquid-liquid extractor (Method 3520), solid-phase extraction (Method 3535), or other appropriate technique. Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using one of the Soxhlet extraction methods (Method 3540 or 3541), pressurized fluid extraction (Method 3545), microwave extraction (Method 3546),

ultrasonic extraction (Method 3550), supercritical fluid extraction (Method 3562), or other appropriate technique or solvents. Tissue samples are extracted using supercritical fluid extraction (Method 3562) or other appropriate technique.

NOTE: The use of hexane-acetone generally reduces the amount of interferences that are extracted and improves signal-to-noise.

The choice of extraction solvent and procedure will depend on the analytes of interest. No single solvent or extraction procedure is universally applicable to all analyte groups and sample matrices. The analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest, for any solvent system and extraction procedure employed, *including* those specifically listed in this method. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Each new sample type must be spiked with the compounds of interest to determine the percent recovery. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

11.1.2 Reference materials, field-contaminated samples, or spiked samples should be used to verify the applicability of the selected extraction technique to each new sample type. Such samples should contain or be spiked with the compounds of interest in order to determine the percent recovery and the limit of detection for that sample type (see Chapter One). When other materials are not available and spiked samples are used, they should be spiked with the analytes of interest, either specific Aroclors or PCB congeners. When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture may be an appropriate choice for spiking. See Methods 3500 and 8000 for guidance on demonstration of initial method proficiency as well as guidance on matrix spikes for routine sample analysis.

11.1.3 The extraction techniques for solids may be applicable to wipe samples and other sample matrices not addressed in Sec. 11.1.1. The analysis of oil samples may need special sample preparation procedures that are not described here. Analysts should follow the steps described in Sec. 11.1.2 to verify the applicability of the sample preparation and extraction techniques for matrices such as wipes and oils.

11.2 Extract cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. Refer to Methods 3600, 3660 and 3665 for general guidance on extract cleanup.

11.3 GC conditions

This method allows the analyst to choose between a single-column or a dual-column configuration in the injector port. The columns listed in this section were the columns used to develop the method performance data. Listing these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Wide-bore or narrow-bore columns may be used with either option. Laboratories may use either the columns listed in this method or other capillary columns or columns of other dimensions, provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

11.3.1 Single-column analysis

This capillary GC/ECD method allows the analyst the option of using 0.25-mm or 0.32-mm ID capillary columns (narrow-bore) or 0.53-mm ID capillary columns (wide-bore). Narrow-bore columns generally provide greater chromatographic resolution than wide-bore columns, although narrow-bore columns have a lower sample capacity. As a result, narrow-bore columns may be more suitable for relatively clean samples or for extracts that have been prepared with one or more of the clean-up options referenced in the method. Wide-bore columns (0.53-mm ID) may be more suitable for more complex environmental and waste matrices. However, the choice of the appropriate column diameter is left to the professional judgement of the analyst.

11.3.2 Dual-column analysis

The dual-column/dual-detector approach recommends the use of two 30-m x 0.53-mm ID fused-silica open-tubular columns of different polarities, thus, different selectivities towards the target analytes. The columns may be connected to an injection tee and separate electron capture detectors, or to both separate injectors and separate detectors. However, the choice of the appropriate column dimensions is left to the professional judgement of the analyst.

11.3.3 GC temperature programs and flow rates

11.3.3.1 Table 1 lists suggested GC operating conditions for the analysis of PCBs as Aroclors for single-column analysis, using either narrow-bore or wide-bore capillary columns. Table 2 lists suggested GC operating conditions for the dual-column analysis. Use the conditions in these tables as guidance and establish the GC temperature program and flow rate necessary to separate the analytes of interest.

11.3.3.2 When determining PCBs as congeners, difficulties may be encountered with coelution of congener 153 and other sample components. When determining PCBs as Aroclors, chromatographic conditions should be adjusted to give adequate separation of the characteristic peaks in each Aroclor (see Sec. 11.4.6).

11.3.3.3 Tables 3 and 4 summarize example retention times of up to 73 Aroclor peaks determined during dual-column analysis using the operating conditions listed in Table 2. These retention times are provided as guidance as to what may be achieved using the GC columns, temperature programs, and flow rates described in this method. Each laboratory must determine retention times and retention time windows for their specific application of the method. Note that the peak numbers used in these tables are *not* the IUPAC congener numbers, but represent the elution order of the peaks on these GC columns.

11.3.3.4 Once established, the same operating conditions must be used for the analysis of samples and standards.

11.4 Calibration

11.4.1 Prepare calibration standards using the procedures in Sec. 7.0. Refer to Method 8000 and Sec. 9.3 for proper calibration techniques for both initial calibration and calibration verification. When PCBs are to be determined as congeners, the use of internal standard calibration is highly recommended. Therefore, the calibration standards

must contain the internal standard (see Sec. 7.9) at the same concentration as the sample extracts. When PCBs are to be determined as Aroclors, external standard calibration is generally used.

NOTE: Because of the sensitivity of the electron capture detector, always clean the injection port and column prior to performing the initial calibration.

11.4.2 When PCBs are to be quantitatively determined as congeners, an initial multi-point calibration must be performed that includes standards for all the target analytes (congeners). See Method 8000 for details on calibration options.

11.4.3 When PCBs are to be quantitatively determined as Aroclors, the initial calibration consists of two parts, described below.

11.4.3.1 As noted in Sec. 7.7.1, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. Therefore, an initial multi-point calibration is performed using the mixture of Aroclors 1016 and 1260 described in Sec. 7.7.1. See Method 8000 for guidance on the use of linear and non-linear calibrations.

11.4.3.2 Standards of the other five Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration through the origin, these standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture in Sec. 11.4.3.1 has been used to describe the detector response. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards in Sec. 11.4.3.1. For non-linear calibrations, see Sec. 11.4.3.3.

11.4.3.3 In situations where only a few Aroclors are of interest for a specific project, the analyst may employ a multi-point initial calibration of each of the Aroclors of interest (e.g., five standards of Aroclor 1232 if this Aroclor is of concern and linear calibration is employed) and not use the 1016/1260 mixture described in Sec. 11.4.3.1 or the pattern recognition standards described in 11.4.3.2. When non-linear calibration models are employed, more than five standards of each Aroclor of interest will be needed to adequately describe the detector response (see Method 8000).

11.4.4 Establish the GC operating conditions appropriate for the configuration (single-column or dual column, Sec. 11.3), using Tables 1 or 2 as guidance. Optimize the instrumental conditions for resolution of the target compounds and sensitivity. A final temperature of between 240 °C and 275 °C may be needed to elute decachlorobiphenyl. The use of injector pressure programming will improve the chromatography of late eluting peaks.

NOTE: Once established, the same operating conditions must be used for both calibrations and sample analyses.

11.4.5 A 2-µL injection of each calibration standard is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest.

11.4.6 Record the peak area (or height) for each congener or each characteristic Aroclor peak to be used for quantitation.

11.4.6.1 A minimum of 3 peaks must be chosen for each Aroclor, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors.

11.4.6.2 Late-eluting Aroclor peaks are generally the most stable in the environment. Table 5 lists diagnostic peaks in each Aroclor, along with example retention times on two GC columns suitable for single-column analysis. Table 6 lists 13 specific PCB congeners found in Aroclor mixtures. Table 7 lists PCB congeners with example retention times on a DB-5 wide-bore GC column. Use these tables as guidance in choosing the appropriate peaks. Each laboratory must determine retention times and retention time windows for their specific application of the method.

11.4.7 When determining PCB congeners by the internal standard procedure, calculate the response factor (RF) for each congener in the calibration standards relative to the internal standard, decachlorobiphenyl, using the equation that follows.

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

11.4.8 When determining PCBs as Aroclors by the external standard technique, calculate the calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards (from either Sec. 11.4.3.1 or 11.4.3.2) using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in nanograms)}}$$

Using the equation above, a calibration factor will be determined for each characteristic peak, using the total mass of the Aroclor injected. These individual calibration factors are used to quantitate sample results by applying the factor for each individual peak to the area of that peak, as described in Sec. 11.9.

For a five-point calibration, five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture, e.g., there will be at least 25 separate calibration factors for the mixture. The single standard for each of the other Aroclors (see Sec. 11.4.3.1) will generate at least three calibration factors, one for each selected peak.

If a non-linear calibration model is employed, as described in Method 8000, then additional standards containing each Aroclor of interest will be employed, with a corresponding increase in the total number of calibration factors.

11.4.9 The response factors or calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration, if a linear calibration model is used. This involves the calculation of the mean response or calibration factor, the standard deviation, and the relative standard deviation (RSD) for each congener or Aroclor peak.

When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the linear calibration models must be applied to the other five Aroclors for which only single standards are analyzed. If multi-point calibration is performed for individual Aroclors (see Sec. 11.4.3.3), use the calibration factors from those standards to evaluate linearity.

See Method 8000 for the specifics of the evaluation of the linearity of the calibration and guidance on performing non-linear calibrations. In general, non-linear calibrations also will consider each characteristic Aroclor peak separately.

11.5 Retention time windows

Absolute retention times are generally used for compound identification. When absolute retention times are used, retention time windows are crucial to the identification of target compounds, and should be established by one of the approaches described in Method 8000. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis. Analysts should consult Method 8000 for the details of establishing retention time windows. Other approaches to compound identification may be employed, provided that the analyst can demonstrate and document that the approaches are appropriate for the intended application. When PCBs are determined as congeners by an internal standard technique, absolute retention times may be used in conjunction with relative retention times (relative to the internal standard).

When conducting either Aroclor or congener analysis, it is important to determine that common single-component pesticides such as DDT, DDD, and DDE do not elute at the same retention times as the target congeners. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples. Therefore, in conjunction with determining the retention time windows of the congeners, the analyst should analyze a standard containing the DDT analogs. This standard need only be analyzed when the retention time

windows are determined. It is not considered part of the routine initial calibration or calibration verification steps in the method, nor are there any performance criteria associated with the analysis of this standard.

If Aroclor analysis is performed and any of the DDT analogs elute at the same retention time as an Aroclor peak that was chosen for use in quantitation (see Sec. 11.4.6), then the analyst must either adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Aroclor and does not correspond to a peak from a DDT analog. If PCB congener analysis is performed and any of the DDT analogs elute at the same retention time as a PCB congener of interest, then the analyst must adjust the GC conditions to achieve better resolution.

11.6 Gas chromatographic analysis of sample extracts

11.6.1 The same GC operating conditions used for the initial calibration must be employed for the analysis of samples.

11.6.2 Verify calibration at least once each 12-hr shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is recommended to minimize the number of samples requiring reinjection when QC limits are exceeded) and at the end of the analysis sequence. For Aroclor analyses, the calibration verification standard should be a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not *require* analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

11.6.2.1 The calibration factor for each analyte calculated from the calibration verification standard (CF_v) should not exceed a difference of more than ±20 percent when compared to the mean calibration factor from the initial calibration curve. If a calibration approach other than the RSD method has been employed for the initial calibration (e.g., a linear model not through the origin, a non-linear calibration model, etc.), consult Method 8000 for the specifics of calibration verification.

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 100$$

11.6.2.2 When internal standard calibration is used for PCB congeners, the response factor calculated from the calibration verification standard (RF_v) should not exceed a ±20 percent difference when compared to the mean response factor from the initial calibration. If a calibration approach other than the RSD method has been employed for the initial calibration (e.g., a linear model not through the origin, a non-linear calibration model, etc.), consult Method 8000 for the specifics of calibration verification.

above the QC limits was detected in a sample extract, then reinjection is necessary to ensure accurate quantitation.

If an analyte was not detected in the sample and the standard response is more than 20% below the initial calibration response, then reinjection is necessary. The purpose of this reinjection is to ensure that the analyte could be detected, if present, despite the change in the detector response, e.g., to protect against a false negative result.

11.6.7 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is *recommended* that standards be analyzed after every 10 samples (*required* after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative or quantitative QC criteria are exceeded.

11.6.8 The use of internal standard calibration techniques does not require that all sample results be bracketed with calibration verification standards. However, when internal standard calibration is used, the retention times of the internal standards and the area responses of the internal standards should be checked for each analysis. Retention time shifts of more than 30 sec from the retention time of the most recent calibration standard and/or changes in internal standard areas of more than -50 to +100% are cause for concern and must be investigated.

11.6.9 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

11.6.10 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it.

11.6.11 If compound identification or quantitation is precluded due to interferences (e.g., broad, rounded peaks or ill-defined baselines are present), corrective action is warranted. Cleanup of the extract or replacement of the capillary column or detector may be necessary. The analyst may begin by rerunning the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to Method 3600 for the procedures to be followed in sample cleanup.

11.7 Qualitative identification

The identification of PCBs as either Aroclors or congeners using this method with an electron capture detector is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. See Method 8000 for information on the establishment of retention time windows.

Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Confirmation is necessary when the sample composition is not well characterized. See Method 8000 for information on confirmation of tentative identifications. See Sec. 11.11 of this procedure for information on the use of GC/MS as a confirmation technique.

When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. See Method 8000 for a discussion of such a comparison and appropriate data reporting approaches.

11.7.1 When simultaneous analyses are performed from a single injection (the dual-column GC configuration described in Sec. 11.3), it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

11.7.2 The results of a single column/single injection analysis may be confirmed, if necessary, on a second, dissimilar, GC column. In order to be used for confirmation, retention time windows must have been established for the second GC column. In addition, the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis. That standard may be either the individual congeners, individual Aroclor or the Aroclor 1016/1260 mixture.

11.7.3 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern. This approach should not be attempted for samples from unknown or unfamiliar sources or for samples that appear to contain mixtures of Aroclors. In order to employ this approach, the analyst must document:

- The peaks that were evaluated when comparing the sample chromatogram and the Aroclor standard.
- The absence of major peaks representing any other Aroclor.
- The source-specific information indicating that Aroclors are anticipated in the sample (e.g., historical data, generator knowledge, etc.).

This information should either be provided to the data user or maintained by the laboratory.

11.7.4 See Sec. 11.11 for information on GC/MS confirmation.

11.8 Quantitation of PCBs as congeners

11.8.1 The quantitation of PCB congeners is accomplished by the comparison of the sample chromatogram to those of the PCB congener standards, using the internal standard technique (see Method 8000). Calculate the concentration of each congener.

11.8.2 Depending on project requirements, the PCB congener results may be reported as congeners, or may be summed and reported as total PCBs. The analyst should use caution when using the congener method for quantitation when regulatory requirements are based on Aroclor concentrations. See Sec. 11.9.3.

11.8.3 The analytical procedures for these 19 congeners may be appropriate for the analysis of other congeners not specifically included in this method and may be used

as a template for the development of such a procedure. However, all 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

11.9 Quantitation of PCBs as Aroclors

The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

11.9.1 Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, and 1254. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards.

11.9.2 Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen in Sec. 11.4.6.1. and the calibration model (linear or non-linear) established from the multi-point calibration of the 1016/1260 mixture. Non-linear calibration may result in different models for each selected peak. A concentration is determined using each of the characteristic peaks, using the individual calibration factor calculated for that peak in Sec. 11.4.8, and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor.

11.9.3 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. Samples containing more than one Aroclor present similar problems. If the purpose of the analysis is <u>not</u> regulatory compliance monitoring on the basis of Aroclor concentrations, then it may be more appropriate to perform the analyses using the PCB congener approach described in this method. If results in terms of Aroclors <u>are</u> required, then the quantitation as Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

11.10 Confirmation

Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation is necessary when the sample composition is not well characterized. Confirmatory techniques such as gas chromatography with a dissimilar column or a mass spectrometer should be used. See Method 8000 for information on confirmation of tentative identifications.

When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. See Method 8000 for a discussion of such a comparison and appropriate data reporting approaches.

When the dual-column approach is employed, the target phenols are identified and confirmed when they meet the identification criteria on both columns.

11.11 GC/MS confirmation

GC/MS confirmation may be used in conjunction with either single-or dual-column analysis if the concentration is sufficient for detection by GC/MS.

11.11.1 Full-scan quadrupole GC/MS will normally require a higher concentration of the analyte of interest than full-scan ion trap or selected ion monitoring techniques. The concentrations will be instrument-dependent, but values for full-scan quadrupole GC/MS may be as high as 10 ng/µL in the final extract, while ion trap or SIM may only be a concentration of 1 ng/µL.

11.11.2 The GC/MS must be calibrated for the target analytes when it is used for quantitative analysis. If GC/MS is used only for confirmation of the identification of the target analytes, then the analyst must demonstrate that those PCBs identified by GC/ECD can be confirmed by GC/MS. This demonstration may be accomplished by analyzing a single-point standard containing the analytes of interest at or below the concentrations reported in the GC/ECD analysis. When using SIM techniques, the ions and retention times should be characteristic of the Aroclors to be confirmed.

11.11.3 GC/MS confirmation should be accomplished by analyzing the same extract used for GC/ECD analysis and the extract of the associated blank.

11.12 GC/AED confirmation by Method 8085 may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/AED.

11.13 Chromatographic system maintenance as corrective action

When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

11.13.1 Splitter connections

For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few centimeters (up to 30 cm) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

11.13.2 Metal injector body

Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

11.13.2.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, rinse the entire inside of the injector port with acetone and then rinse it with toluene, catching the rinsate in the beaker.

13.4.1 Two field-contaminated certified reference materials were extracted by a single laboratory. One of the materials (EC-5) was a lake sediment from Environment Canada. The other material (EC-1) was soil from a dump site and was provided by the National Science and Engineering Research Council of Canada. The average recoveries for EC-5 are based on the certified value for that sample. The average recoveries for EC-1 are based on the certified value of the samples or a Soxhlet value, if a certified value was unavailable for a specific analyte. These data are provided for guidance purposes only.

13.4.2 Four certified reference materials were extracted by two independent laboratories. The materials included a marine sediment from NIST (SRM 1941), a fish tissue from NIST (SRM 2974), a sewage sludge from BCR European Union (CRM 392), and a soil sample from BCR European Union (CRM 481). The average recoveries were based on the certified value of the samples or a Soxhlet value, if a certified value was unavailable for a specific analyte. These data are provided for guidance purposes only.

13.4.3 A weathered sediment sample from Michigan (Saginaw Bay) was extracted by a single laboratory. Soxhlet extractions were carried out on this sample and the SFE recovery is relative to that for each congener. The average recoveries were based on the certified value of the samples. Additional data are shown in the tables for some congeners for which no certified values were available. These data are provided for guidance purposes only.

13.5 Tables 12 through 14 contain single laboratory recovery data for Aroclor 1254 using solid-phase extraction (Method 3535). Recovery data at 2, 10, and 100 µg/L are presented. Results represent three replicate solid-phase extractions of spiked wastewaters. Two different wastewaters from each wastewater type were spiked. All of the extractions were performed using 90-mm C_{18} disks. These data are provided for guidance purposes only.

13.6 Single-laboratory data were developed for PCBs extracted by pressurized fluid extraction (Method 3545) from sewage sludge, a river sediment standard reference material (SRM 1939), and a certified soil reference material (CRM911-050). Certified values were available for five PCB congeners for the sewage sludge and for four congeners in SRM 1939. The soil reference material was certified for Aroclor 1254. All pressurized fluid extractions were conducted using hexane:acetone (1:1), at 100 °C, 1300-1500 psi, and a 5-min static extraction. Extracts were analyzed by GC/ECD. The data are presented in Tables 15 through 17 and are reported in detail in Reference 13. These data are provided for guidance purposes only.

13.7 Single-laboratory accuracy data were obtained for PCBs extracted by microwave extraction (Method 3546) from three reference materials, EC-1, EC-2, and EC-3, from Environment Canada. Natural soils, glass fiber, and sand samples were also used as matrices that were spiked with PCBs. Concentrations varied between 0.2 and 10 µg/g (total PCBs). All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by GC/ECD. Method blanks, spikes and spike duplicates were included for the low concentration spikes; matrix spikes were included for all other concentrations. The data are presented in Tables 18 through 20 and are reported in detail in Reference 14. These data are provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The 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 their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society, Department of Government Relations and Science Policy, 1155 16th Street, NW, Washington, DC, 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

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17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.