

**SITE INSPECTION
SEVEN OUT LLC TANK
901 Francis Street
Waycross, Ware County, GA 31501
CERCLIS ID NO. GAN000407811
Vol. 1 of 3**



10508639

EPA ID: GAN000407811 Site Name: SEVEN OUT LLC TANK

State ID:

Alias Site Names:

City: WAYCROSS

County or Parish: WARE

State: GA

Refer to Report Dated: 10/20/2006

Report Type: SITE INSPECTION 001

Report Developed by: STATE

DECISION:

☒ 1. Further Remedial Site Assessment under CERCLA (Superfund) is not required because:

☒ 1a. Site does not qualify for further remedial site assessment under CERCLA (No Further Remedial Action Planned - NFRAP)

☐ 1b. Site may qualify for action, but is deferred to:

☐ 2. Further Assessment Needed Under CERCLA:

2a. Priority: ☐ Higher ☐ Lower

2b. Other: (recommended action) NFRAP (No Further Remedial Action Planned)

DISCUSSION/RATIONALE:

Site failed to score. Removal action eliminated large quantity of source materials. Groundwater pathway received a low score due to the extensive depth to the drinking water aquifer (300 ft and greater) and two clay layers above. No known nearby targets exist for the shallower aquifer. One shallow residential well exists within 1/4 mile radius, but that well is not used for drinking water. Barium was detected in the shallow aquifer (observed release), but levels are considerably below MCL. No known release to SW pathway and minimal targets exist in that pathway. Metals, benzene and some semi-vol organics, primarily PAHs, have been documented in soil, however minimal targets exist for that pathway.

Site Decision Made by: CAROLYN CALLIHAN

Signature: _____

Carolyn Callihan

Date: 11/16/2006

Georgia Department of Natural Resources

2 Martin Luther King, Jr. Drive, SE, Suite 1154, Atlanta, Georgia 30334

Noel Holcomb, Commissioner

Environmental Protection Division

Carol A. Couch, Ph.D., Director

404/656-2833

October 20, 2006

VIA MAIL COURIER

Ms. Carolyn Callihan
Superfund Site Assessment Manager
U.S. Environmental Protection Agency
Waste Management Division
Superfund Site Evaluation Section
61 Forsyth Street, SW
Atlanta, Georgia 30303-3104

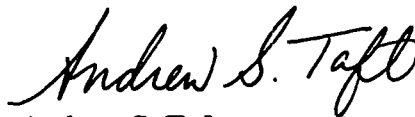
SITE: Seven Out LLC
BREAK: 1.9
OTHER: v. 3

RE: Seven Out LLC Tank Site
901 Francis Street
Waycross, Ware County, Georgia
Site Inspection Report

Dear Ms. Callihan:

Enclosed you will find a Site Inspection (SI) Report that has been completed for the above referenced site. Should you have any questions or comments regarding the SI Report, please contact Mr. Eddie Williams (Project Manager) at (404) 657-8660.

Sincerely,



Andrew S. Taft
CERCLA Pre-Remedial Coordinator
Hazardous Waste Management Branch

cc: Bruce Khaleghi, EPD (w/o enclosure)
Eddie Williams, EPD (w/o enclosure)

File: CERCLA Pre-Remedial (FY-2006)
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Georgia Department of Natural Resources

2 Martin Luther King, Jr. Drive, SE, Suite 1154, Atlanta, Georgia 30334

Noel Holcomb, Commissioner

Environmental Protection Division

Carol A. Couch, Ph.D., Director

404/656-2833

November 6, 2006

VIA MAIL COURIER

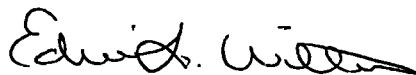
Ms. Carolyn Callihan
Superfund Site Assessment Manager
U.S. Environmental Protection Agency
Waste Management Division
Superfund Site Evaluation Section
61 Forsyth Street, SW
Atlanta, Georgia 30303-3104

RE: Seven Out LLC Tank Site
901 Francis Street
Waycross, Ware County, Georgia
Site Inspection Report Revisions

Dear Ms. Callihan:

Enclosed you will find three (3) replacement pages for the scoresheet portion of the Site Inspection (SI) Report. The narrative has been revised to include the text about the clay layers that exist above the primary residential and municipal groundwater aquifers. A complete copy of the narrative has been included with a highlighted page showing the revised portion. As suspected, the groundwater pathway score dropped significantly, thus also lowering the overall site score. Should you have any questions or comments regarding the SI Report or its revisions, please contact me at. (404) 657-8660.

Sincerely,



Edwin L. Williams
Advanced Geologist
Hazardous Waste Management Branch

c: Bruce Khaleghi, EPD (w/o enclosure)
Andy Taft, EPD (w/o enclosure)

File: CERCLA Pre-Remedial (FY-2006)
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SITE INSPECTION REPORT

**SEVEN OUT LLC TANK
901 FRANCIS STREET
WAYCROSS, WARE COUNTY, GEORGIA 31501
CERCLIS ID.NO. GAN000407811**

PREPARED FOR:

**U.S. ENVIRONMENTAL PROTECTION AGENCY REGION IV
ATLANTA FEDERAL BUILDING
61 FORSYTH STREET, S.W.
ATLANTA, GEORGIA 30303-3415**

PREPARED BY:

**EDWIN WILLIAMS
GEORGIA ENVIRONMENTAL PROTECTION DIVISION
2 MARTIN LUTHER KING JR. DRIVE, SE
SUITE 1154 EAST
ATLANTA, GEORGIA 30334**

REVIEWED BY:

**BRUCE KHALEGHI
GEORGIA ENVIRONMENTAL PROTECTION DIVISION**

OCTOBER 2006

CONTENTS

1.0	INTRODUCTION	4
2.0	SITE DESCRIPTION AND HISTORY	4
2.1	Location	4
2.2	Site Description	4
2.3	Operational History	5
2.4	Waste Characterization	7
3.0	WASTE/SOURCE SAMPLING	8
3.1	Sample Locations	8
3.2	Analytical Results	8
4.0	GROUNDWATER PATHWAY	9
4.1	Hydrogeologic Setting	9
4.2	Groundwater Targets	10
4.3	Sample Locations	11
4.4	Analytical Results	11
4.5	Groundwater Pathway Conclusions	11
5.0	SURFACE WATER PATHWAY	12
5.1	Hydrologic Setting	12
5.2	Surface Water Targets	13
5.3	Sample Locations	13
5.4	Analytical Results	14
5.5	Surface Water Pathway Conclusions	14
6.0	SOIL EXPOSURE	14
6.1	Physical Conditions	14
6.2	Soil Targets	15
6.3	Sample Locations	16
6.4	Analytical Results	16
6.5	Soil Exposure Conclusions	16
7.0	AIR PATHWAY	16
7.1	Physical Conditions	16
7.2	Air Targets	16
7.3	Sample Locations	17
7.4	Analytical Results	17
7.5	Air Pathway Conclusions	17
8.0	SUMMARY AND CONCLUSIONS	17
9.0	REFERENCES	18

TABLES (following narrative)

Table 1	Parcels Associated with Seven Out at 901 Francis Street
Table 2	Constituents Detected in EPA Samples Taken from On-Site Containers at Seven Out
Table 3	Qualified Releases to Soils Based on EPA Samples Taken from On-site Surface Soils.
Table 4	Identified Source Areas at Seven Out
Table 5	Qualified Releases to Surface Soils Based on SI Sampling
Table 6	Qualified Releases to Subsurface Soils Based on SI Sampling
Table 7	Proposed and Actual Source Soil Sampling Locations
Table 8	Population of Groundwater Users Within 4-Mile Radius of Seven Out
Table 9	Proposed and Actual Groundwater Sampling Locations
Table 10	Qualified Releases to Groundwater Based on SI Sampling
Table 11	Detected Constituents in Off-Site Wells
Table 12	Nearby Resident Population to Seven Out
Table 13	Population Within 4-Mile Radius of Seven Out

FIGURES (following tables)

Figure 1	Seven Out LLC Tank Site Location Map
Figure 2	Seven Out LLC Tank Site Layout
Figure 3	Detailed Site Map of Seven Out LLC Tank Site Showing Soil and Groundwater Sample Locations
Figure 4	1:24,000 Topographic Map Showing 4-Mile Radius of Groundwater Users
Figure 5	Surface Water Pathway Overland Runoff Route from Seven Out LLC Tank Site
Figure 6	1:24,000 Topographic Map Showing 15-Mile Surface Water Migration Pathway Target Distance Limit (TDL)
Figure 7	1:24,000 Wetland Maps for the 15-Mile Surface Water Target Distance Limit (TDL)

APPENDICES (in back)

Appendix A	List of Analytes
Appendix B	EPD Laboratory - Quality Assurance/Quality Control
Appendix C	Safety and Health Plan
Appendix D	Access Authorizations
Appendix E	Chain of Custody Documentation
Appendix F	Soil Analytical Data
Appendix G	Groundwater Analytical Data
Appendix H	References

PHOTOGRAPHS (see Reference 38)

1.0 INTRODUCTION

Under the authority of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), the Hazardous Waste Management Branch of the Georgia Environmental Protection Division (GA EPD) completed a Site Inspection (SI) Report for the site known as Seven Out LLC Tank located in Waycross, Ware County, Georgia (Reference 1). The purpose of this investigation was to collect information concerning conditions at and surrounding the Seven Out LLC Tank site sufficiently to assess the threat posed to human health and the environment as identified in the Preliminary Assessment (PA) dated September 2005 and to determine the need for additional CERCLA/SARA or other appropriate action. The scope of the investigation included: 1) review of available file information, 2) a comprehensive target survey, 3) on and off-site reconnaissance, and 4) soil and groundwater sample collection.

2.0 SITE DESCRIPTION AND HISTORY

2.1 Location

Waycross is the county seat for Ware County, which is located in the southeastern corner of the state of Georgia. The Seven Out LLC Tank site is located in Waycross, Georgia at 901 Francis Street and 0/3 Folks Street (Reference 2)(Figure 1). Topographic map locations provided in the Removal Assessment Sampling Plan were incorrect. Those maps showed the site location approximately 1.5 miles due west of its actual location (Reference 3). The 901 Francis Street location is the location of the main offices and storage areas and is south of the intersection where McDonald Street comes to a dead end into Francis Street (Reference 4). The 0/3 Folks Street location is the location of the tank farm and treatment plant and is south of the intersection where Folks street crosses Francis Street (Figure 2)(References 2 and 4). The geographic coordinates are latitude 31 degrees 12 minutes 26.9 seconds, North and longitude 82 degrees 21 minutes 51.5 seconds West. The Location was verified using a Global Positioning System (GPS) unit during the onsite reconnaissance (References 4 and 5). The GPS coordinates were taken at the back gate entrance to the property at 901 Francis Street, closest to the warehouse storage facility (Reference 4).

Ware County experience summers that are long and hot. The highest average daily temperatures occur in July and average 82 degrees F. Winters are mild and open with the coolest daily average temperatures of 50 degrees F in January. Precipitation occurs as rain, although a rare snowfall can occur. Year-round temperatures average 67 degrees F (Reference 6). Average annual precipitation is 52 inches (Reference 7).

2.2 Site Description

Upon inspection of the site on June 15-16, 2006 it was apparent that the site is no longer in operation (Reference 4). Property records obtained from the Ware County Assessors Office website indicate the site consists of four (4) parcels as shown in Table 1 (Reference 8).

An additional parcel of property that was the storage area for the frac/Baker tanks was on leased CSX railway property behind the subject property on Glenmore Avenue (Reference 4).

The site includes an office and storage building, the waste treatment area or tank farm [which includes more than thirty (30) tanks for waste or product storage], and paved parking/loading areas. Located at 901 Francis Street is the building Seven Out LLC formerly used for office and warehouse storage and a separate multi-vehicle bay building. Part of the storage building is just a shell of a brick building without a roof. At the time of the inspection these buildings were deserted and locked, where possible (Reference 4). The facility was surrounded by a secured and locked fencing. The tank farm (at the Folks Street address) is approximately 180 feet by 100 feet and has inadequate secondary containment in place (Reference 4). The tank farm is accessible to the public via Folks Street, Francis Street, or McDonald Street. There is no fencing around the tank farm. At 801 Francis Street is The Sports Shop, Inc., which is located between Francis Street and the Seven Out LLC tank farm (References 4 and 8). The property south of the site on Glenmore Avenue is owned by CSX Railroad. This area originally was a staging area for four frac/Baker tanks that were full of chemical wastes. The tanks had been removed at the time of the inspection (References 3 and 4). The area surrounding the Seven Out LLC facility is a mixed-use area including commercial, industrial, and residential property. The nearest residential property is located at 103 Folks Street approximately 220 feet from the tank farm area (Reference 4). Figure 2 depicts the site and surrounding area (Reference 4). Photographs taken during the onsite reconnaissance are included in the trip report (Reference 4).

2.3 Operational History

Sanborn Fire Insurance Maps (SFIM) indicate that the 901 Francis Street site was previously occupied by the Coca Cola Bottling Company and the Waycross Cooperative Laundry. The Laundry and Bottling Company are shown on the SFIM dated 1913 (Reference 9). The SFIM dated 1922 (Reference 10) shows the Bottling Company, but the Laundry is no longer shown as an active laundry, but only as storage. SFIM indicate that the 0/3 Folks Street site was previously occupied by a lime and cement warehouse in 1897 (Reference 9), a hay storage warehouse in 1908 (Reference 10), the W.N. Gramling Wholesale Grocerer Warehouse in 1913 (Reference 11) and the John D. Hopkins Hay and Grain facility in 1922 (Reference 12). Some of these buildings were enlarged prior to 1922 to form the Hopkins Hay and Grain facility (References 11 and 12). In 1930, a grocery was added to the John D. Hopkins Hay and Grain (Reference 13). The 1930/1947 maps indicate occupation of the site by a wholesale hay, grain, and grocery (Reference 14). Maps covering the time after 1947 could not be located. Aerial photographs as recent as 1999 show the buildings as shown on the 1922 SFIM still in place (Reference 15). These buildings no longer exist and have been partially replaced by the tank farm/treatment plant (Reference 4). It should be noted that the parcel of land located between the 901 Francis Street, 801 Francis Street, and 0/3 Folks Street locations is now vacant (Reference 4). SFIM indicate that the J.H. Gillon and Companies Machine Shop and Foundry occupied this area between 1897 and 1922 (References 11 and 12). No further documentation could be located on this facility.

The Seven Out LLC Tank site was constructed to be an industrial wastewater processing facility (Reference 16). A request for a GAEPD/USEPA identification number for a used oil processor was received by GAEPD, on behalf of BCX Waycross, on December 11, 2001, and number GAR000030007 was assigned (Reference 17). The facility was permitted by the City of Waycross to discharge non-domestic pre-treated wastewater to the sewer for processing by the POTW (Reference 18). A representative of EPD performed a Used Oil processor compliance evaluation inspection on April 22, 2003. At the time of this inspection the facility had not received any used oil for processing (Reference 19). The facility apparently began operation shortly after the above inspection because the City of Waycross issued eight (8) formal Notices of Violation for the months of May 2003 through January 2004 (Reference 20). These violations of the facility's discharge permit resulted in a Consent Order issued by the City on January 27, 2004 (Reference 21). This Consent Order was not signed by BCX, Inc., however a letter to the City dated March 3, 2004 stated that the facility was ceasing discharge to the City POTW (Reference 22). EPD conducted another inspection on April 6, 2004. At this time it was observed that the facility had ceased accepting industrial wastewater and stopped discharging to the Waycross POTW (Reference 23). On April 23, 2004, EPD issued a Notice of Violation to the facility for failing to determine the contents of 27 tanks located in the facility's tank farm and in the four flocculation box tanks (frac tanks)(Reference 24). On June 2, 2004 a release of approximately 4000-5000 gallons of unknown liquid from a 10,000-gallon frac tank belonging to the facility occurred on the CSX property (Reference 25). EPD inspected the site and took soil samples from the spill area on June 23, 2004 (Reference 26). EPD sent a proposed Consent Order to the facility on July 20, 2004 for violations observed during the inspections conducted on April 6 and June 23, 2004, namely storing hazardous waste and unidentified wastewaters (Reference 27). EPD received responses from representatives of the facility, however the Consent Order was never signed by the facility (Reference 28). EPD requested assistance from EPA with sampling at the site. Sampling of the tanks and containers was performed on August 23-26, 2004 by EPA contractor Tetra Tech EM, Inc. Constituents detected in the tanks included metals, volatile organics, and semi-volatile organics. Complete sampling results are included in the Removal Assessment Report prepared by Tetra Tech (References 16 and 29).

EPD requested assistance from EPA with the remediation of the facility by letter dated January 21, 2005 (Reference 30). The EPA initiated removal operations at the facility on January 28, 2005 (Reference 31) and removal activities continued until EPA left the site on June 10, 2005 (References 32 and 33). A total of 338,250 gallons of pumpable liquid wastes were removed during the removal activities. Of those amounts, 1,650 gallons was manifested as hazardous waste (Reference 34).

CSX Transportation (CSXT) contracted with Shaw Environmental in October 2005 to complete a removal of the contents of the frac and Baker tanks and the tanks themselves from the property that had been leased to BCX (Seven Out LLC) on the north end of the CSX rail yard, adjacent to the south side of the Seven Out LLC property. During the restoration of the site, nine (9) tanks on the leased property were emptied, cleaned and prepared for disposal. Appreciable amounts of residual sludge were documented in the one remaining Baker tank and one other tank. A total of 6,350 gallons of non-hazardous residual sludge, wastewater and cleaning fluids

were disposed from the tank cleaning operations. The Baker tank was removed by the Baker company. Recyclable materials were taken to Waycross Recycling Company in Waycross and approximately fifty-six (56) tons of solid waste were taken to Waste Management Chesser Island Road Landfill. Soil sampling after the cleanup of the site did not record any constituents in the soils at levels above the Georgia Environmental Protection Division Hazardous Sites Response Act reportable quantities (Reference 35).

2.4 Waste Characterization

Wastes handled at the facility were diverse. Because the facility was designed to treat contaminated wastewater, wastes included metals, volatile organics, and semi-volatile organics (Reference 3). Those wastes identified in the containers on site are provided in Table 2. No pesticide or herbicide wastes were documented to have been processed at the facility. Other potential wastes were evaluated due to the former existence of a cooperative laundry and a foundry at this site (References 4 and 11). The timeframe for the operation of the cooperative laundry was prior to commercial use of most chlorinated solvents in the United States. Perchloroethylene/tetrachloroethylene (PCE) was introduced into the dry cleaning industry in the U.S. in the 1930s. Trichloroethylene (TCE) was first used in the dry cleaning industry in the U.S. in the late 1920s. 1,1,1-trichloroethane (TCA) was not commercially used in the U.S. until the 1950s. Methylene chloride's use in the U.S. did not become important to industry until after World War II. Carbon tetrachloride, however, was imported into the U.S. as early as 1898. Under the trade name Carbona, carbon tetrachloride was a popular dry cleaning and spot-removing agent. It was produced in the U.S. by the Dow Chemical and Warner Chemical companies in the early 1900s (Reference 36). None of these chemicals have been documented in the tanks or containers at the tank site (Reference 3).

The foundry was in existence on part of the property of the Seven Out LLC tank farm/treatment plant (References 11 and 12). Foundries operated during the time period of the Gillon Machine Shop and Foundry generally were cupola foundries (Reference 37). The SFIM indicated that the coke ovens for this foundry were located in two different sections of the foundry. One was located in the section nearest Francis Street and the other was located in the section most closely to the area now occupied by the tank farm/treatment plant (References 10, 11, and 12). Because this foundry generated slag wastes that could have been disposed on the property, wastes that could be attributed to the foundry could include lead, zinc, cadmium, and other metals (Reference 34). All of these chemicals have also been recorded in the tanks and containers at the tank site (Reference 3).

Table 3 provides analytical results from the EPA-contracted soil sampling activities at the site prior to removal actions (Reference 3). Figure 3 shows the EPA-contractor soil sampling locations. A separate, documented removal action was completed at the frac tank area by CSX Railroad. Documentation of the removal indicates that no constituents were recorded in soils above the Georgia Hazardous Site Response Action notification levels (Reference 35).

Several sources areas have already been identified from previous sampling and or removal activities at the site (Reference 3). Table 4 provides those sources that have been

identified prior to the development of this SI Sampling Plan. Figure 4 details the locations of these identified source areas (References 3 and 4).

Although over 300,00 gallons of waste liquids have been removed from the tanks and containers on site, over 100,000 gallons of unpumpable wastes remain. Table 2 provides analytical data on the contents of those tanks and containers.

3.0 WASTE/SOURCE SAMPLING

3.1 Sample Locations

Analytical data has been categorized by sampling locations into different source areas for the purpose of better representing the contamination that is specific to each area of the site. These source areas are only represented by contaminated soils for which analytical data have been recorded. The soil/source locations are presented in Figures 3 and complete analytical documentation is in Appendix E. The contaminants by source area are presented in the following discussions on the source areas. Table 1 provides EPA removal contractor records for constituents that were identified in the waste tanks of the tank farm (References 3 and 16). These constituents make up Source Area #1. At the time of the SI Reconnaissance, approximately 131,500 gallons of sludge waste remained in the tanks on-site (Reference 34). Table 5 presents the analytical data for the constituents that were 3 times background or greater than the Practical Quantitation Limit (PQL) for those non-detect (ND) values for constituents with no background values. The containment factor for all of the identified contaminated soil source areas are: groundwater pathway = 10; surface water pathway = 10, air pathway = 10. Table 7 lists the proposed soil sample locations at the potential source areas and the reasons for any deviations from the sampling plan for those samples (Reference 38).

3.2 Analytical Results

All soil/source samples were obtained by hand augering, using dedicated stainless steel auger buckets, stainless steel spoons, and glass sample mixing dishes and stainless steel mixing bowls for each sample. Sampling was conducted in accordance with the EPA Region IV SEDS Standard Operating Procedures and Quality Assurance Manual (EPASOPQAM) and volatile organic compound (VOC) samples were obtained using Encore devices. Volatile samples were taken first, prior to mixing of the samples. Analysis of the VOCs was completed using Method 8260 and analysis of the semi-volatiles was completed using Method 8270. Metals analyses were completed using Method 6010 and Pesticides were completed using Method 8081. Analyses for PCEs were completed using Method 8082. The following constituents were recorded as qualified releases to soils at the site: acetone, naphthalene, flouranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)flouranthene, arsenic, barium, cadmium, chromium, copper, manganese, nickel, lead, vanadium, zinc, mercury, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, dieldrin, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, methoxychlor, mirex, and gamma-chlordane. Although there were many qualified releases documented in the analytical results, it should be noted that five (5) samples had detections of a total of six (6) constituents whose concentrations exceeded the EPA Region 9 Residential PRG values. Of those five (5) samples, only five (5) constituents had

concentrations that exceeded the Georgia Environmental Protection Division Reportable Quantities values. Those exceedances were for benzo(a)anthracene, chrysene, benzo(b)fluoranthene, arsenic, lead, and mercury. Table 1 provides EPA removal contractor records for constituents that were identified in the waste tanks of the tank farm (References 3 and 16). Tables 5 and 6 provide the analytical results per source area. The complete analytical data package for soils can be found in Appendix E.

4.0 GROUNDWATER PATHWAY

4.1 Hydrogeologic Setting

The Seven Out LLC Tank site lies in the east-central part of Ware County along the boundary of the Bacon Terraces and Okefenokee Swamp districts of the Eastern Gulf Coastal Plain Section of the Coastal Plain Province. Topography in the Bacon Terraces District consists of several moderately dissected terraces that normally parallel the coastline. These terraces generally vary in elevation between 15 and 30 feet each, running from an elevation of 180 feet above sea level (ASL) to an elevation of 330 feet ASL (Reference 39). Drainage is primarily to the Satilla River Basin. The Satilla River Basin consists of: 37% forest cover, 24% wetlands, 2% urban land cover, and 18% in agriculture. Drainage from the northern and western portions of the district flows to the tributaries that comprise the Suwannee River basin, which eventually empty into the Atlantic Ocean (Reference 40).

The site is underlain by Pleistocene and Pliocene sands and gravels, primarily associated with the Brandywine, Coharie, and Sunderland formations. The Brandywine Formation consists of less than 50 feet of sand and gravel which resembles the coarser sands of the Hawthorn, unconformably stratigraphically below the Brandywine. Should any formations overlie the Brandywine, these would probably lie unconformably. The Coharie Formation may overlie the Brandywine Formation or the Hawthorn, both unconformably. The lithology of the Coharie includes less than 50 feet of sand, some coarse in nature. In some area, angular pebbles may be present, however, other areas may contain smooth flat pebbles of transparent quartz. The Sunderland Formation consists of fine white or light-gray sand. Where the Sunderland is present, it may be overlain by marshy, boggy environments, associated with wetlands. The unconformities within all of the mentioned formations result from continuous advances and retreats of the Atlantic Ocean (Reference 41).

The sources at the site were either relatively small (contaminated soils in the frac tank area and in the area behind the secondary containment berm) or were primarily contained in a concrete secondary containment system (References 4, 16, 29). As evidenced during rainfall during the VSI for the previous PA reconnaissance, infiltration rate is not high and the subsurface to a great depth does not appear to be highly permeable (Reference 42). Releases to the soils would most likely have migrated to the surface water pathway more readily than the groundwater pathway at the site. The site is not karst according to the City of Waycross Wellhead Protection Plan (Reference 43).

Municipal and industrial groundwater production in the Waycross area occurs primarily from the Ocala Limestone of the Principal Floridan Aquifer at depths exceeding 500 feet.

Residential wells that are installed in the area penetrate the Miocene/Pliocene aquifers (around 300 feet deep) or the shallower aquifers (20-40 feet deep). Current residential wells are installed in the Pliocene aquifers, however, the area does include some older wells that are completed in the shallow aquifers (References 44 and 45). The well logs for the City Well #3 indicate a 15-foot sandy clay and a 135-foot sandy clay below the shallow aquifer at 20 feet, separating possible site contamination from the primary residential and municipal groundwater aquifers (Reference 46). Previously operated wells in the Waycross area encountered groundwater in a fine to coarse-grained sand between 350 and 400 feet deep and in limestone below 500 feet deep (Reference 46). Within the Coastal Plain, groundwater is usually encountered in medium to coarse sands and limestone.

The primary public supplies of groundwater are provided by the City of Waycross, Waycross/Ware Industrial Park, and the Satilla Regional Water and Sewer Authority (References 47 and 48). Groundwater withdrawal in Ware County accounts for 84% of all water supplies in the county. Groundwater is the sole water supply used for public consumption. Roughly half of the groundwater withdrawn in Ware County is used for public supply (Reference 49).

The Seven Out LLC Tank site lies within an area determined to have a higher (Drastic Rating > 141) groundwater pollution susceptibility rating (Reference 50). The site is not located within a significant groundwater recharge area (Reference 51).

There have been no known complaints about the appearance, smell, or taste of well water from groundwater users anywhere within the 4-mile target distance of the site.

Because the area covered by the site is small and because the surrounding area is mostly residential or commercial, resources were not identified during the Visual Site Inspection (Reference 4).

Although groundwater contamination of the deep aquifer is not suspected at this site, the potential for shallow aquifer contamination exists. The large number of secondary targets and the potential for groundwater contamination is a cause for concern. Four deep wells that serve 19,114 people are within 0.25-0.50 mile from the site (Reference 47). At the time of the Visual Site Inspection, there was an estimated 131,500 gallons of potentially hazardous waste at the site. At least one shallow residential well exists within the ¼ mile radius of the site, however the water from that well is not used for drinking water. The residence is hooked up to the municipal supply (Reference 52). The Coca Cola Bottling Company well identified on the Well Survey Maps is no longer in service (References 53 and 57).

4.2 Ground Water Targets

Major suppliers of groundwater include the City of Waycross (3 wells), the Satilla Regional Water and Sewer Authority (5 wells), and the Waycross/Ware County Industrial Park (2 wells). Seven (7) of these wells exist within the 4-mile radius in the Floridan aquifer: City of Waycross has three (3) within the ¼-1/2 mile radius, Satilla Regional Water and Sewer Authority has three (3) wells, and the Waycross/Ware County Industrial Park has one (1) well. One of the City of Waycross wells (Well #1) has not been used for some time (Reference 38). The site does

not fall within a wellhead protection area of any of the City of Brunswick wells. Two (2) additional wells in the Floridan aquifer are at the Baptist Retirement Village, west of the site in the 2-3 mile radius (References 47, 48, and 54). Additional wells include numerous residential wells located within the four (4) mile radius (References 53 and 55). Table 8 provides a complete summary of groundwater wells and user population identified within the 4-mile radius. Figure 4 shows the 4-mile radius from the site with the major groundwater wells identified (References 47, 48, 53, 54, and 56).

4.3 Sample Locations

Proposed sample locations for groundwater and justification for modifications to those sampling locations are provided in Table 9. Each onsite groundwater sample was obtained by using a dedicated groundwater sampling equipment that was implaced using a Geoprobe truck-mounted rig. The groundwater was purged a minimum of three well volumes, or until dry. Turbidity was recorded below ten (10) NTUs before inorganic sampling was initiated. Sampling was conducted in accordance with the EPA Region IV SESD Standard Operating Procedures and Quality Assurance Manual (EPASOPQAM) using dedicated sampling equipment. Seven (7) samples from on-site locations were planned during the SI using a Geoprobe truck-mounted rig. Five (5) samples were retrieved using the GeoProbe rig. Three (3) supply wells were proposed for sampling during the SI, however, only two (2) wells were sampled. Figure 3 shows the on-site sampling locations. The supply wells that were sampled are shown in Figure 4 (Reference 38).

4.4 Analytical results

Analysis of the VOCs was completed using Method 8260 and analysis of the semi-volatiles was completed using Method 8270. Metals analyses were completed using Method 6010 and Pesticides were completed using Method 8081. Analyses for PCBs were completed using Method 8082. Table 4 provides the qualifying releases to groundwater based on practical quantitation limits (PQLs). The complete analytical reports can be found in Appendix F. One (1) contaminant, barium, was detected as a qualified release to groundwater at the site. The recorded detection of barium, however, was considerably below the Safe Drinking Water MCL. Selenium was also detected above background, however, it was not detected in on-site soil samples, nor in on-site tank samples. Therefore, it does not qualify as a qualified release. The selenium detections of 0.0084 and 0.0086 mg/l were above the non-detect PQL of the background sample of 0.005 mg/l, but well below the MCL of 0.050 mg/l. Table 10 provides the summary of the SI analytical results for qualified releases to groundwater on site. Table 11 provides the summary of the SI analytical results for the residential (non-drinking water well) and the City of Waycross Municipal Well #3. Lead was detected in the residential well at 22 ug/l and is suspected to be a result of the casing (probably galvanized steel) that is in the well.

4.5 Groundwater conclusions

Minor releases of contaminants have been recorded in the ground water on site. Barium and selenium have been documented in two downgradient shallow groundwater samples, however, the concentrations are considerably less than the Safe Drinking Water MCLs for those constituents.

Since no targets exist in this groundwater aquifer and since minimal contamination has been documented, the risk of groundwater contamination from the site appears negligible.

5.0 SURFACE WATER PATHWAY

5.1 Hydrologic Setting

The Seven Out LLC Tank site lies in an area of minimal flooding outside of both the 100-year and 500-year flood zones. It is designated as "Zone X" on the Flood Plain Insurance Map (Reference 58). Overland flow from the site flows into a drainage ditch south of the tank farm and north of the railroad tracks on the site. Figure 5 shows the overland runoff route. This drainage ditch continues west, roughly parallel to the railroad tracks, for approximately 1200 feet into an unnamed creek. Just south of the ditch is a petroleum facility, C & M Oil Company. Overland runoff from this facility would also enter the drainage ditch. The location where flow from the drainage ditch empties into the unnamed creek is the Probable Point of Entry (PPE). Immediately south of this intersection, on a hill overlooking the unnamed creek, is an abandoned former BP fuel tank farm. Overland runoff from this facility would migrate to the unnamed creek. This creek flows northeast for approximately 2000 feet before continuing underground by culvert. Water then flows underground in an east direction for approximately 3000 feet before emerging near the intersection of Lee Avenue and Memorial Drive (Hwy 23). Water then flows east for less than 1000 feet before joining the City Drainage Canal. The City Drainage Canal is approximately 1.0 mile from the PPE. The City Drainage Canal flows in a northeast direction for approximately 3 miles before joining the Satilla River (Figure 6). The canal was constructed to provide stormwater runoff protection for the city. As a result all stormwater runoff generated within the city proper is directed to the canal whereby, the stormwater and any contaminants that it carries with it are discharged to the Satilla River. Three creeks or streams enter the City Drainage Canal. Bailey Branch enters from the northwest approximately 1.8 miles from the PPE. Caney Branch enters from the northwest approximately 2.4 miles west of the PPE (Reference 4). The Satilla River is approximately 3.8 miles from the PPE. The 15-mile total distance limit (TDL) continues along the Satilla River (Reference 59). Figure 6 traces the 15-mile surface water migration pathway TDL from the PPE (Reference 59). The nearest wetland appears to be approximately 3.0 miles from the PPE along the City Drainage Canal. From this point downstream the remaining 12 miles of the TDL are bordered by wetlands that include palustrine forested and palustrine scrub types (Figure 7)(Reference 60). The United States Geological Survey (USGS) has a gauging station located on the Satilla River approximately 0.5 mile upstream of the confluence with the City Drainage Canal. Annual mean streamflow at this location ranges from a low of 228 cfs in 2002 to a high of 2,589 cfs in 1964 (Reference 61). Rainfall in the region averages 51 inches annually with average annual runoff at 11 inches (Reference 7).

The entire 3-mile length of the City Drainage Canal is listed on the Draft 2006 305(b) 303(d) Georgia Environmental Protection Division (GEPD) proposal document for rivers and streams that do not fully support their designated uses. Fecal coliform bacterial levels from urban runoff prohibit the Canal from fully supporting its designated uses. Additionally, the Satilla River is also listed on the Draft 2006 305(b) 303(d) proposed document for failing to fully

support its designated use. Low dissolved oxygen concentrations prohibit the Satilla River from meeting its designated uses from the point where the City Drainage Canal drains into it to the completion of its 15-mile downstream migration pathway (References 62 and 63). The Satilla River at the U.S. 84 bridge crossing is listed in the "Georgia Dept. of Natural Resources Guidelines for Eating Fish from Georgia Waters" for mercury in bass. The recommendation for eating bass from this location is 1 meal/month (Reference 64).

Overflowing wastes from the tanks at the facility have been removed, thereby significantly reducing any remaining contaminants from mobilizing into the surface water migration pathway. Additionally, numerous sources of attribution contaminants are located along the 3-mile migration pathway of the City of Waycross Drainage Canal to the Satilla River. One former bulk fuel facility is located just upgradient of the PPE. Another operating bulk fuel facility shares a common drainage ditch with the Seven Out site. Approximately 2000 feet from the PPE, the perennial stream flows underground through a series of drainage culverts and storm sewers that carry the downtown City of Waycross storm water runoff through town. These underground conduits resurface near the intersection of Plant Avenue (Highway 84) and Highway 82. For the remainder of the 3 miles, the City of Waycross Drainage Canal carries stormwater and overland runoff through Waycross to the Satilla River. Along the path, Bailey Branch and Caney Branch empty into the Drainage Canal. At the point of discharge into the Satilla River, the City's wastewater treatment plant discharges its effluent into the nearby wetland. No surface water or sediment sampling was proposed (References 4 and 59).

5.2 Surface Water Targets

There are no drinking water intakes on the surface water pathway at or within fifteen (15) miles downstream from the Seven Out LLC Tank site (References 4, 54, and 59). Therefore, no primary or secondary surface water drinking water intake targets were identified. Although the City Drainage Canal is recreationally fished, primarily by youngsters, no documentation of consumption could be verified. Residents that were interviewed indicated that the fish from the canal were not fit to eat (References 65 and 66). Although the Satilla River does not fully support its designated use, bucketsitters (those who fish and put their catch for consumption in the buckets on which they sit) do fish in the Satilla River (References 65 and 67). The nearest wetland appears to be approximately 3.0 miles from the PPE near the discharge of the City Drainage Canal into the Satilla River (References 4 and 41). From this point downstream the remaining 12 miles are bordered almost continuously by wetlands (Figure 7)(References 4 and 38).

5.3 Sample Locations

No surface water intakes exist along the 15-mile TDL of the surface water pathway. The nearest fishery, though small, is more than 3 miles downstream of the PPE. For a portion of the 3-mile trek to the Satilla River, the fishery, and the nearest wetland environments, the surface water pathway disappears underground and becomes a part of the City of Waycross stormwater runoff and control system. During this time contaminants from throughout the downtown portion of the city mingle with the incoming flow from the unnamed tributary. Because of this system, it would

be very difficult to eliminate other sources of contamination from the unnamed tributary. Additionally, the Waycross Drainage Canal empties into the Satilla River in wetlands that are adjacent to the water treatment plant on the Satilla River. Because of the probable inability to attribute contamination from the site to one of the targets, no sampling of the surface water pathway was proposed or completed.

5.4 Analytical Results

Since no samples were taken, no analyses were completed.

5.5 Surface Water Conclusions

The surface water pathway lacks drinking water intake targets. Fishery targets exist, however, they are at least three (3) miles downstream. Accessibility to the bulk of the fishery targets in the Satilla River is limited to boaters. The fishery in the Satilla River is not considered large. Sensitive environments in the form of wetlands exist along the Satilla River, however, these targets are more than three (3) miles from the site. Because the surface water pathway goes underground within the downtown district of Waycross into a series of drainage culverts which lead to the Waycross Drainage Canal, attribution from numerous other sources cannot be eliminated. Because of the lack of significant targets, the surface water pathway is not considered a critical concern.

6.0 SOIL EXPOSURE

6.1 Physical Conditions

The Seven Out LLC Tank site is underlain by soils associated with the Norfolk Sand. The Norfolk Sand is a loose, unconsolidated gray or brown, medium to coarse sand that extends for about 7 inches in depth. The subsoil below 7 inches to a depth of 36 inches is comprised of a loose incoherent yellow sand, usually coarser and lighter in texture than the soil. The subsoil at 30-36" may include soft iron concretions where mottled and exhibit a sticky texture as it becomes more clayey in texture. Fine quartz sand gravel and coarse sand may be interspersed throughout the soil and subsoil. The Norfolk Sand most commonly occurs as irregularly shaped bodies along the Satilla River and its tributaries. Topography along these tributaries tends to be level to slightly rolling (Reference 6). Within the site, soil staining has been observed next to the tank farm secondary containment structure. Soil staining has been observed in the frac tank area, which is located on the adjacent CSX Railroad property (References 3, 4, 38, and 42).

Soil contamination has been documented at the site. Table 3 summarizes constituents that were detected in soil sampling conducted by EPA contractors. Metals, benzene, and semi-volatile organics (primarily PAHs) have been documented at the site (Reference 16). The Seven Out LLC Tank site is no longer in operation, therefore no site workers are exposed to potential contamination. Removals at the CSX Transportation site have resulted in the removal of over 55 tons of waste materials from that area (Reference 35). There are no residences, schools, or daycare

facilities within 200 feet of the suspected areas of soil contamination. The adjacent business, The Sports Shop, Inc., has three employees, however there is no suspected soil contamination at that location (Reference 4).

According to information provided by the Georgia Department of Natural Resources, six (6) endangered, threatened or unusual species have been identified in the northwestern quadrant of the Waycross East 7.5' topographic map in Ware County. These species include the Spotted Turtle (*Clemmys guttata*), the Gopher Tortoise (*Copherus polyphemus*), the Chapman Oak (*Quercus chapmanii*), the Parrot Pitcherplant (*Sarracenia psittacina*), the Sandhill Skullcap (*Scutellaria arenicola*), and the Ohoopee Burnelia (*Sideroxylon sp.1*) (References 68 and 69). These species have not been observed in habitat at the site (References 4, 38, and 42).

6.2 Soil Targets

The Seven Out LLC Tank site is underlain by soils associated with the Norfolk Sand. The Norfolk Sand is a loose, unconsolidated gray or brown, medium to coarse sand that extends for about 7 inches in depth. The subsoil below 7 inches to a depth of 36 inches is comprised of a loose incoherent yellow sand, usually coarser and lighter in texture than the soil. Fine quartz sand gravel and coarse sand may be interspersed throughout the soil and subsoil. The Norfolk Sand most commonly occurs as irregularly shaped bodies along the Satilla River and its tributaries. Topography along these tributaries tends to be level to slightly rolling (Reference 6). Within the site, soil staining has been observed next to the tank farm secondary containment structure. Soil staining has been observed in the frac tank area, which is located on the adjacent CSX Railroad property (References 3, 4, and 42).

Soil contamination has been documented at the site. Table 3 summarizes constituents that were detected in soil sampling conducted by EPA contractors. Metals, benzene, and semi-volatile organics (primarily PAHs) have been documented at the site (Reference 16). The Seven Out LLC Tank site is no longer in operation, therefore no site workers are exposed to potential contamination. There are no residences, schools, or daycare facilities within 200 feet of the suspected areas of soil contamination. The adjacent business, The Sports Shop, Inc., has three employees, however there is no suspected soil contamination at that location, although soil contamination is adjacent to the business's property (Reference 4).

Nearby resident populations, as provided by the Census of Population and Housing, 2000 (Reference 53) are summarized in Table 12. Approximately 5,498 people live within one mile of the site (Reference 35).

No endangered or threatened species of plants or animals have been observed on site (References 4, 38, and 42).

Although the soil exposure pathway appears to pose a minimum threat due to the lack of a resident population and a lack of workers at the site, additional sampling was proposed to better define the areas of contamination on all parcels of property relevant to this SI.

6.3 Sample Locations

Soil sample locations and modifications to the proposed soil locations are identified in Table 7. Soil sample locations are shown in Figure 3 (Reference 38). Soil sampling was conducted onsite and on the adjacent CSX Transportation Property. Twelve (12) surface and eight (8) subsurface soil samples were taken in areas determined to be areas of suspected or known contamination. All samples were obtained using dedicated stainless steel auger buckets, stainless steel spoons, and glass sampling dishes or stainless steel sampling bowls. Sampling was conducted in accordance with the EPA Region IV SESD Standard Operating Procedures and Quality Assurance Manual (EPASOPQAM). Volatile organic (VOC) samples were taken first using Encore devices (Reference 38).

6.4 Analytical Results

Documentation on the soil sampling conducted during previous investigations can be reviewed in Section 2.3 of this report. Tables 5 and 6 summarize the qualified releases to soils at this site based on analytical results of the SI sampling. Qualifying releases to soils include VOCs, SVOCs, Metals, and Pesticides. Although there were many qualified releases documented in the analytical results, it should be noted that five (5) samples had detections of a total of six (6) constituents whose concentrations exceeded the EPA Region 9 Residential PRG values. Of those five (5) samples, only five (5) constituents had concentrations that exceeded the Georgia Environmental Protection Division Reportable Quantities concentrations.

6.5 Soil Exposure Conclusion

Releases to soil have been confirmed at the Seven Out LLC Tank Site. However, no actual soil exposure targets were identified in the SI. While contamination exists in the soil, there are currently no identified actual targets, only potential targets in the form of nearby residents and adjacent workers. The soil exposure route does not appear to be a significant concern.

7.0 AIR PATHWAY

7.1 Physical Conditions

The Seven Out LLC Tank site is located at approximately 135 feet above sea level. The Seven Out LLC Tank site is located in the city of Waycross in a highly developed area. The area surrounding the Seven Out LLC facility is a mixed-use area including commercial, industrial, and residential property (References 4 and 59).

7.2 Air Targets

The major targets within the 4-mile radius of the site include the residents of the City of Waycross and surrounding Ware and Pierce counties. Census of Population and Housing, 2000 data indicates that the population within the 4-mile radius of the site is 25,333 people as summarized in Table 13 (Reference 53). Sensitive environments in the form of wetlands exist

within the 4-mile radius. Approximately 80 acres are within the 1-2 mile radius, approximately 480 acres are within the 2-3 mile radius and over 3,100 acres are within the 3-4 mile radius. Additional wetland acreage exists outside of the 4-mile radius. No primary targets were identified. The surrounding residents constitute potential targets as well as the wetlands. No release into the air is suspected from the Seven Out LLC Tank site.

7.3 Sample Locations

No air sample locations were proposed in the SI Sampling Plan for this site.

7.4 Analytical Results

No air samples were taken.

7.5 Air Pathway Conclusion

The air pathway is not considered to be a significant threat to residents and employees that live and work in the area, to other sensitive environments, or to endangered or protected plant and animal species because the site is no longer in operation, and the risk for particulate release or a gas release is minimal, since EPA has removed much of the wastes that were stored on site.

8.0 SUMMARY AND CONCLUSIONS

The Seven Out LLC Tank site is no longer in operation. Sample results obtained during this SI and during a Removal Assessment performed by Tetra Tech EM, Inc. indicate some soil contamination and the presence of hazardous constituents in the tanks at the site. Tank removals have decreased the volume of wastes in the tanks, however, over 100,000 gallons of waste remain in the tanks as a significant source. Groundwater contamination at the site is minimal and is below all regulatory levels. However, the remaining waste quantity in the tanks coupled with the large potential target groundwater drinking population cause the potential for groundwater contamination to be a concern. A removal of the remaining tank wastes would considerably remove the remaining risks. Due to a lack of significant surface water targets, the surface water pathway is not a critical exposure route. The lack of on-site employees or residents also eliminates the soil exposure route from being a critical component of the site. The only significant potential targets to the air pathway were the 4-mile radius population and the wetlands within the same radius. Because no release to air was suspected or documented, those targets were not considered to be affected. The site poses a risk because significant amounts of waste remain on site and a relatively large potential target population within a ½ mile radius obtain their drinking water from groundwater wells in a deep aquifer. Shallow groundwater at the site, however, does not appear to have been significantly impacted.

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TABLES

Table 1. Parcels Associated with Seven Out at 901 Francis Street

Address	Owner	Date Acquired	Size
903 Francis Street	Seven Out LLC, A Florida LL Company	12/5/2002	0.28 Acres
901 Francis Street	Seven Out LLC, A Florida LL Company	12/5/2002	0.87 Acres
0 Francis Street	Seven Out LLC, A Florida LL Company	12/5/2002	0.46 Acres
0/3 Folks Street	Seven Out LLC, A Florida LL Company	4/3/2002	0.46 Acres

(Reference 8)

Table 2– Constituents Detected in Samples Taken from On-site Containers at Seven-Out

Constituent	Highest Concentration	Container ID
Antimony	0.0208	CT-1-S/CT-1
Arsenic	NA	NA
Barium	3.7/4.8J	CD-3-S/CD-3/SS-2-S/
Cadmium	0.0806	ST-1/ST-1
Chromium	6.38	ST-1/ST-1
Cobalt	0.0716	SH-2-S/SH-2
Copper	31.7	CT-1-S/CT-1
Iron	2200	ST-1/ST-1
Lead	14.0	SH-4-S/SH-4
Manganese	29.3	ST-1/ST-1
Nickel	3.43	ST-1/ST-1
Zinc	21	ST-1/ST-1
Mercury	0.0057	Sulfuric Acid/Sulfuric Acid
1,2-Dichlorobenzene	0.560	DP-1 layer A/DP-1
1,4-Dichlorobenzene	0.780	DP-1 layer B/ DP-1
2-Butanone	2.2	OP-4-S/OP-4
4-Methyl-2-pentanone	0.850	NAOH/NAOH
Acetone	270J	SH-3-S/SH-3
Benzene	2.6	SH-4-S/SH-4
Chloroform	0.093	OP-4-S/OP-4
Isopropylbenzene	0.770	DP-1 layer B/ DP-1
m, p-Xylene	0.240	DP-1 layer B/ DP-1
Methyl tert-butyl ether	0.089	OP-4-S/OP-4
o-Xylene	0.440	DP-1 layer B/ DP-1
Toluene	0.130	DP-1 layer B/ DP-1
1-1'-Biphenyl	0.490	CT-1-S/CT-1
2-Methylnaphthalene	4.0	CT-1-S/CT-1
4-Methylphenol	3.8	NAOH/NAOH
Bis (2-ethylhexyl)phthalate	2.40	CT-1-S/CT-1
Diethyl phthalate	5.70	DP-1 layer B/ DP-1
Dimethyl phthalate	0.510	DP-2-S/DP-2
Di-n-butyl phthalate	12.00	DP-1 layer B/ DP-1
Fluorene	1.2	CT-1-S/CT-1
Isophorone	0.150	F237/F237
Naphthalene	1.0	CD-2-S/CD-2, CT-1-S/CT-1
Phenanthrene	1.0	CT-1-S/CT-1
Phenol	180J	OP-4-S/OP-4
Pyrene	1.40	CT-1-S/CT-1

All samples units in mg/l

NA – Not analyzed

(Reference 10)

Table 3 – Qualifying Releases to Soil at Seven Out LLC Tank (based on EPA Sample Results)

EPA Sample SO-FRT (Frac Tank Spillage Area)					
Constituent	Detection	Background	PQL	Justification	
Barium	7.11	U	3.93	> PQL	
Copper	17.8	U	1.96	> PQL	
Iron	1080	596	NP	> 3X Background	
Lead	10.8	U	3.93	> PQL	
Magnesium	58.5	U	39.3	> PQL	
EPA Sample SO-SW (South Wall of Tank Farm)					
Constituent	Detection	Background	PQL	Justification	
Arsenic	151	U	3.93	> PQL	
Barium	75.2	U	3.93	> PQL	
Chromium	8.69	U	1.96	> PQL	
Cobalt	3.46	U	1.96	> PQL	
Copper	107	U	1.96	> PQL	
Iron	10800	596	NP	> 3X Background	
Lead	264	U	3.93	> PQL	
Manganese	169	4.26	NP	> 3X Background	
Nickel	4.62	U	3.93	> PQL	
Vanadium	8.58	U	3.93	> PQL	
Zinc	518	4.11	NP	> 3X Background	
Mercury	0.350	U	0.987	> PQL	
Acenaphthylene	1.3	U	0.330	> PQL	
Anthracene	1.0	U	0.330	> PQL	
Benzo(a)anthracene	2.4	U	0.330	> PQL	
Benzo(a)pyrene	2.8	U	0.330	> PQL	
Benzo(b)fluoranthene	1.8	U	0.330	> PQL	
EPA Sample SO-SW (South Wall of Tank Farm)(continued)					
Constituent	Detection	Background	PQL	Justification	
Benzo(g,h,i)perylene	2.4	U	0.330	> PQL	
Benzo(k)fluoranthene	3.2	U	0.330	> PQL	
Carbazole	0.370	U	0.330	> PQL	
Chrysene	3.1	U	0.330	> PQL	
Dibenz(a,h)anthracene	0.650	U	0.330	> PQL	
Fluoranthene	4.6	U	0.330	> PQL	
EPA Sample SO-SW (South Wall of Tank Farm)					
Constituent	Detection	Background	PQL	Justification	
Indeno(1,2,3-cd)pyrene	3.0	U	0.330	> PQL	
Phenanthrene	1.8	U	0.330	> PQL	
Pyrene	4.0	U	0.330	> PQL	
EPA Sample SO-DD (Drainage Ditch)					
Constituent	Detection	Background	PQL	Justification	
Barium	15.5	U	3.93	> PQL	
Chromium	7.93	U	1.96	> PQL	
Copper	59.2	U	1.96	> PQL	
Iron	4910	596	NP	> 3X Background	
Lead	17.7*	U	3.93	> PQL	
Magnesium	507	U	39.3	> PQL	
Manganese	74.7	4.26	NP	> 3X Background	
Vanadium	5.34	U	3.93	> PQL	

EPA	Sample SO-DD (Drainage Ditch)				
	Constituent	Detection	Background	PQL	Justification
	Zinc	32.3	4.11	NP	> 3X Background
	Benzene	0.032J	0.0066J	0.0066	> 3X Background
	2-Methylnaphthalene	0.610	U	0.330	> PQL
	Di-n-butyl phthalate	1.1	U	0.330	> PQL
	Phenanthrene	0.400	U	0.330	> PQL

All samples units in mg/kg

*A lead sample from the drainage ditch also failed a TCLP, registering 8.13 mg/l.

(Reference 10)

Table 4 –Source Areas at the Seven Out Site

Area	Description	Secondary Containment	Area/Volume	Verified as a Source Area	Source Area ID #
Soil at Former Foundry and Waste Storage Facility/Laundry	Contaminated Soil on Vacant Lot of Foundry and Waste Storage Facility	Not Present	0.5 acres	Yes	No. 1
Soil at Frac Tanks.	Contaminated Soil at Frac Tank Area on CSX Railroad property..	Not Present	60' X 150' Soil area is approximately 9,000 square feet	Yes	No. 2
Soil at Tank Farm	Contaminated Soil around Tank Farm.	Not Present	22' X 50' Soil area is approximately 1,100 square feet.	Yes	No. 3
Waste treatment area or Tank Farm.	37 tanks of different sizes containing unknown substances.	Yes, but inadequate	Area is approximately 18,000 square feet and volume of waste approximately 131,500 gallons.	Yes	No. 4

(References 3 and 4)

Table 5 – Qualified Releases to Surface Soils Associated with the Seven Out LLC Tank Site

Constituent	PQL	Background SS-010	Source #3					Source #2		Source #1			Source #3
			Wastewater Treatment Plant/Tank Farm					Frac Tank Areas		Foundry	Laundry	Storage Facility	Tank Farm
			SS-012	SS-014	SS-016	SS-016 Duplicate	SS-017	SS-018	SS-019	SS-020	SS-022	SS-024	SS-026
Acetone	0.120	ND	ND	ND	0.230	ND	ND	ND	0.140	ND	ND	ND	ND
Naphthalene	0.0061	ND	ND	ND	0.110	ND	ND	ND	ND	ND	ND	ND	ND
Flouranthene	5.9	ND	ND	ND	14	10	ND	ND	ND	ND	ND	ND	ND
Pyrene	5.9	ND	ND	ND	14	11	ND	ND	ND	ND	ND	ND	ND
Benzo(a)anthracene	5.9	ND	ND	ND	6.4	ND	ND	ND	ND	ND	ND	ND	ND
Chrysene	5.9	ND	ND	ND	6.4	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(b)fluoranthene	5.9	ND	ND	ND	7.5	ND	ND	ND	ND	ND	ND	ND	ND
Aluminum	20	3,100	1,100	4,200	3,800	3,500	3,900	1,600	6,900	8,700	2,800	6,200	5,100
Arsenic	8	ND	ND	ND	100	120	37	ND	66	ND	ND	ND	ND
Barium	20	25	ND	23	73	70	23	ND	61	45	ND	370	86
Calcium	500	ND	3,200	6,300	2,400	2,300	3,100	ND	37,000	960	980	5,300	4,800
Cadmium	0.5	ND	ND	ND	3.2	1.2	ND	ND	0.990	ND	ND	ND	0.620
Chromium	1	4.6	2.1	7.7	15	14	4.4	1.3	14	13	2.5	110	14
Copper	2.5	6.4	5.2	60	96	110	87	ND	41	420	5.9	150	140
Iron	10	1,600	790	3,900	11,000	22,000	4,000	370	6,800	7,900	1,000	35,000	8,600
Potassium	500	ND	ND	ND	ND	ND	ND	ND	560	ND	ND	ND	1,100
Magnesium	500	ND	ND	ND	ND	ND	ND	ND	770	ND	ND	ND	1,700
Manganese	1.5	32	6.9	53	120	190	38	1.6	65	64	15	240	130
Nickel	4	ND	ND	ND	5	6.5	ND	ND	5	ND	ND	ND	7.3
Lead	9	51	ND	57	260	230	67	ND	71	230	ND	540	150
Vanadium	5	ND	ND	9.7	9.8	9.5	ND	ND	17	15	ND	ND	35
Zinc	2	48	8.5	86	750	660	110	ND	140	350	16	360	340
Mercury	0.1	ND	ND	0.110	0.530	0.310	ND	ND	0.120	ND	ND	ND	ND
4,4'-DDD	0.045	ND	ND	ND	0.190	0.290	0.011	ND	ND	ND	ND	0.056	ND
4,4'-DDE	0.0035	ND	ND	0.0055	ND	ND	ND	ND	ND	ND	ND	0.120	ND
4,4'-DDT	0.039	ND	ND	ND	0.250	0.360	0.047	ND	ND	ND	ND	ND	ND
Dieldrin	0.0024	ND	ND	ND	ND	ND	0.090	ND	ND	ND	ND	ND	ND
Endosulfan Sulfate	0.0096	ND	ND	ND	ND	ND	0.059	ND	0.070	ND	ND	ND	ND
Endrin	0.0090	ND	ND	ND	ND	ND	0.020	ND	0.024	ND	ND	ND	ND
Endrin Aldehyde	0.0041	ND	ND	ND	ND	ND	ND	ND	0.087	ND	ND	ND	ND
Heptachlor	0.064	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.110
Methoxychlor	0.024	ND	ND	ND	ND	ND	0.270	ND	ND	ND	ND	0.033	ND
Mirex	0.0021	ND	ND	ND	0.096	ND	ND	ND	0.150	ND	ND	0.018	ND
gamma-Chlordane	0.0054	0.019	ND	0.007	ND	ND	ND	ND	0.087	ND	ND	ND	ND
alpha-Chlordane	0.0054	0.030	ND	ND	0.057	0.078	ND	ND	ND	ND	ND	ND	ND

Units in mg/kg

Bold and Shaded indicate releases above PQL or greater than 3X Background

Table 6 – Qualified Releases to Subsurface Soils at Associated Seven Out LLC Tank Site

Constituent	PQL	Background SS-011	Source #3		Source #1			
			WWTP/Tank Farm		Foundry	Laundry	Waste Storage Facility	
			SB-013	SB-015	SB-021	SB-023	SB-025	SB-025 Duplicate
Aluminum	40	16,000	16,000	2,400	8,900	12,000	11,000	11,000
Barium	20	ND	ND	ND	ND	ND	ND	ND
Cadmium	0.5	ND	ND	ND	ND	ND	0.580	ND
Chromium	1	14	11	2.7	8.8	10	10	12
Copper	2.5	ND	2.7	ND	8	ND	ND	ND
Iron	10	5,400	2,000	360	7,600	3,300	5,000	7,000
Manganese	1.5	5.1	5.1	1.9	7.7	4.1	3.3	7.1
Lead	9	ND	ND	ND	12	ND	ND	ND
Nickel	4	ND	5.7	ND	ND	ND	ND	ND
Vanadium	5	21	13	ND	17	14	16	18
Zinc	2	3.6	3.7	2.7	12.4	3.2	3.4	3.2
gamma-Chlordane	0.0056	ND	ND	ND	ND	ND	ND	ND
alpha-Chlordane	0.0056	ND	ND	ND	ND	ND	ND	ND

Units in mg/kg

Bold and Shaded represent greater than PQLs if Background is non-detect or 3 X Background

Table 7 – Proposed and Actual Source Soil Sample Locations

MEDIA	SAMPLE #	DEPTH OF SAMPLE	SAMPLING JUSTIFICATION
Surface Soil (0-2')	SO-SS-10	1' BGL	Background surface sample from area north of the facility
Subsurface Soil (3-5')	SO-SB-11	3-3.5' BGL	Background subsurface sample from area north of the facility
Surface Soil (0-2')	SO-SS-12	1' BGL	Surface sample taken from north of secondary containment where containment was breeched to identify a source area.
Subsurface Soil (3-5')	SO-SB-13	3-4' BGL (hit water at 3', sample taken above hydrated zone)	Subsurface sample taken from north of secondary containment where containment was breeched to identify a source area.
Surface Soil (0-2')	SO-SS-14	1' BGL	Surface sample taken in area of waste/wastewater pump station near railroad tracks to identify a source area.
Subsurface Soil (3-5')	SO-SB-15	3-3.5' BGL	Subsurface sample taken in area of waste/wastewater pump station near railroad tracks to identify a source area.
Surface Soil (0-2')	SO-SS-16 and SO-SS-16 DUP	1.5' Below Mulch in soil (very black soil)	Surface sample taken in drainage ditch in vicinity of previous EPA-contractor sample to identify a source area.
Surface Soil (0-2')	SO-SS-17	1' BGL	Surface sample taken in drainage ditch further west (downflow) of previous EPA-contractor sample to identify a source area.
Surface Soil (0-2')	SO-SS-18	1-2' Below BGL below 1' of gravel and commercial sand	Surface sample taken in area of frac tank spill to identify a source area.
Surface Soil (0-2')	SO-SS-19	1' BGL	Surface sample taken in area of frac tank east of SO-SS-18 to identify a source area.
Surface Soil (0-2')	SO-SS-20	1' BGL	Surface sample taken in area of former foundry to identify a source area.
Subsurface Soil (3-5')	SO-SB-21	3-3.5' BGL (sample contained coke/coal?)	Subsurface sample taken in area of former foundry to identify a source area.
Surface Soil (0-2')	SO-SS-22	1' below broken concrete pad (sample contained cut nail)	Surface sample taken inside of former laundry to identify a source area.

MEDIA	SAMPLE #	DEPTH OF SAMPLE	SAMPLING JUSTIFICATION
Subsurface Soil (3-5')	SO-SB-23	3-3.5' below broken concrete pad	Subsurface sample taken inside of former laundry to identify a source area.
Surface Soil (0-2')	SO-SS-24	1' BGL	Surface sample taken in area of waste storage facility to identify a source area.
Subsurface Soil (3-5')	SO-SB-25 and SO-SB-25 DUP	3-3.5' BGL (sample contained coal)	Subsurface sample taken in area of waste storage facility to identify a source area.
Surface Soil (0-2')	SO-SS-26	Accumulated sediment 0-1' deep in culvert entrance	Surface sample taken from facility side of culvert drainage pipe that discharges to ditch to identify a source area.
Surface Soil (0-2')	SO-SS-27	Not taken	Field decision was made to sample the sediment accumulated on the facility side of culvert drainage pipe instead of at discharge side, thus no attribution in ditch was required.

(Reference 38)

Table 8 – Groundwater Users Within 4-Mile Radius of Site

Distance	0-25 mile	25-50 mile	50-1 mile	1-2 mile	2-3 mile	3-4 mile	Totals
Wells	0	4	7	105	310	721	1,147
Population	0	19,114	20	592	5,492	2,405	27,623

(References 47, 48, 53, 54, and 56)

Table 9 - Proposed and Actual Groundwater Sample Locations

MEDIA	SAMPLE #	DEPTH OF SAMPLE	SAMPLING JUSTIFICATION
Shallow Groundwater	SO-GW-002	19.5' BGL	Background sample taken from on-site, in the far southeastern corner of site.
Shallow Groundwater	SO-GW-001	19.5' BGL	On-site sample taken from far northwestern corner of site, presumed to be downgradient of tank farm, laundry, and foundry.
Shallow Groundwater	SO-GW-003	Not taken	Field decision was made that GW-001 would satisfy the purpose of this sample.
Shallow Groundwater	SO-GW-004	19.5' BGL	On-site sample taken between facility storage area and former laundry to establish if a release to the shallow groundwater has occurred.
Shallow Groundwater	SO-GW-005 and SO-GW-005 DUP.	19.5' BGL	On-site sample taken at former foundry location to establish if a release to shallow groundwater has occurred
Shallow Groundwater	SO-GW-006	Not taken	Field decision was made that GW-001 and GW-004 would satisfy the purpose of this sample.
Shallow Groundwater	SO-GW-007	20-30' BGL	Residential well sample taken at nearby residence to establish if a target well has been impacted.
Shallow Groundwater	SO-GW-008	Not taken	No additional Residential well was located in the 20-30' aquifer to sample.
Deep Groundwater	SO-GW-009	600'	Municipal well sample taken at one of wells within 1/4-1/2 mile radius to establish if a potential target well has been impacted.

(Reference 38)

Table 10- Qualified Releases to Groundwater On Site

Constituent	PQL	Background (SO-GW-002)	Downgradient of 901 SO-GW-001	Between Laundry & Waste Storage Facility GW-004	Within the Foundry Footprint	
					GW-005	GW-005 DUP
Barium	0.200	<0.200	<0.200	<0.200	0.210	0.210

Units in mg/l

Bold and highlighted are greater than PQL where background = PQL.

Table 11 – Detected Constituents in Offsite Groundwater Wells

Constituent	Residential Well (810 Elizabeth Street) (non-drinking water well)	City of Waycross (Well #3)
Lead	0.022	<0.001

Units in mg/l

Table 12. Nearby Resident Population to Seven Out

Distance	0 - .25 mile	.25 - .50 mile	.50 – 1 mile	Total
Population	167	1241	4090	5498

(Reference 53)

Table 13 – Population Within 4-Mile Radius of Seven Out LLC Tank Site

Distance	0-.25 mile	.25-.50 mile	.50 - 1 mile	1-2 mile	2-3 miles	3-4 miles	Totals
Population	167	1241	4090	9644	7195	2995	25333

(Reference 53)

FIGURES

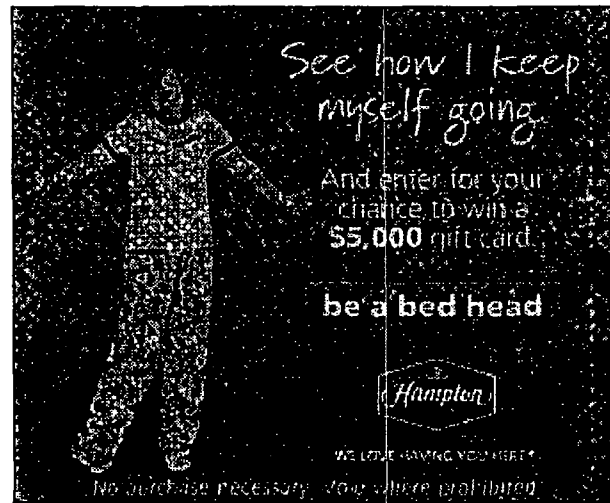


901 Francis St
Waycross GA
31503-2335 US

Notes:

Seven Out LLC (901 Francis Street)

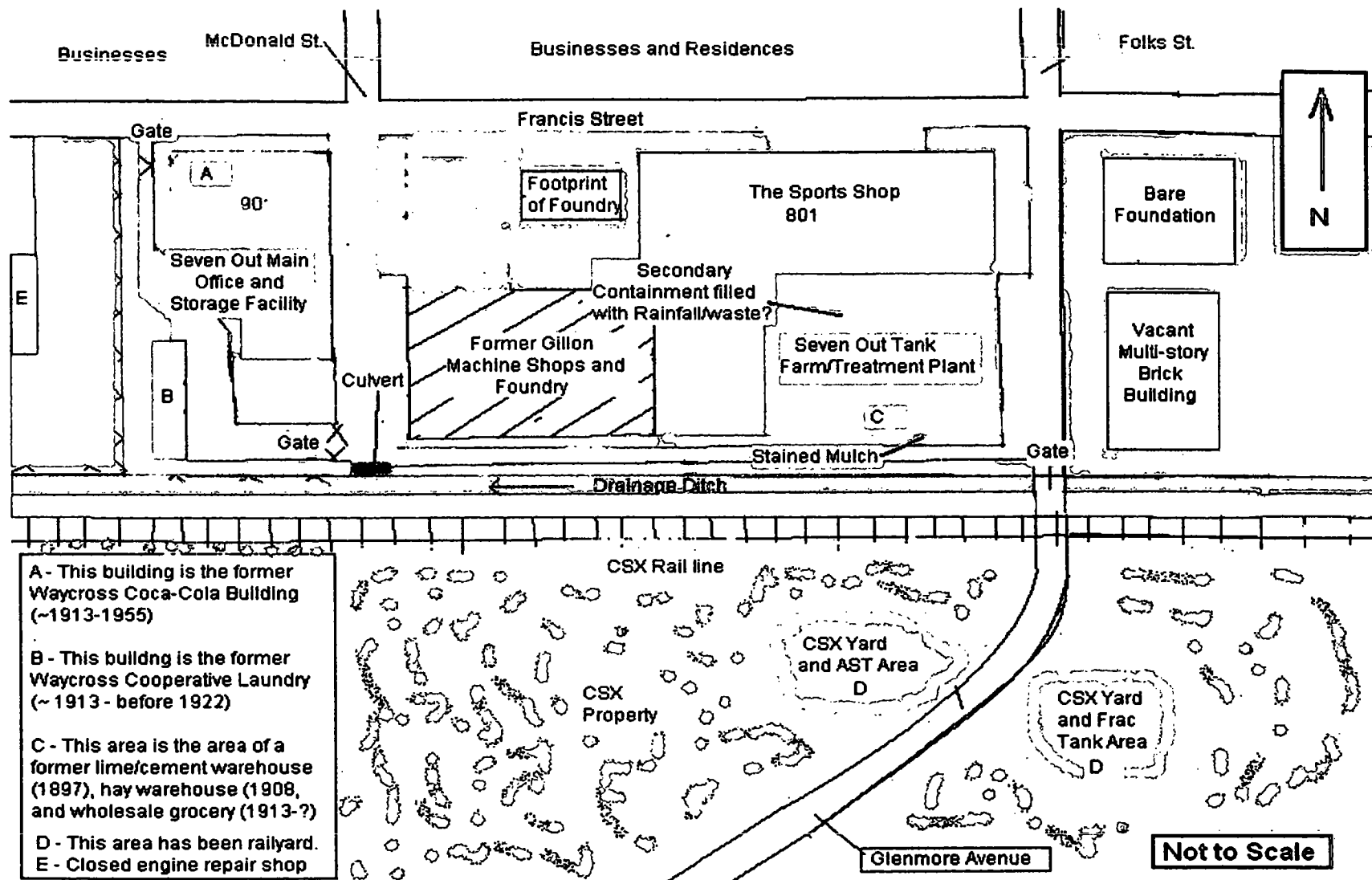
Seven Out Tank Site (3 Folks Street)



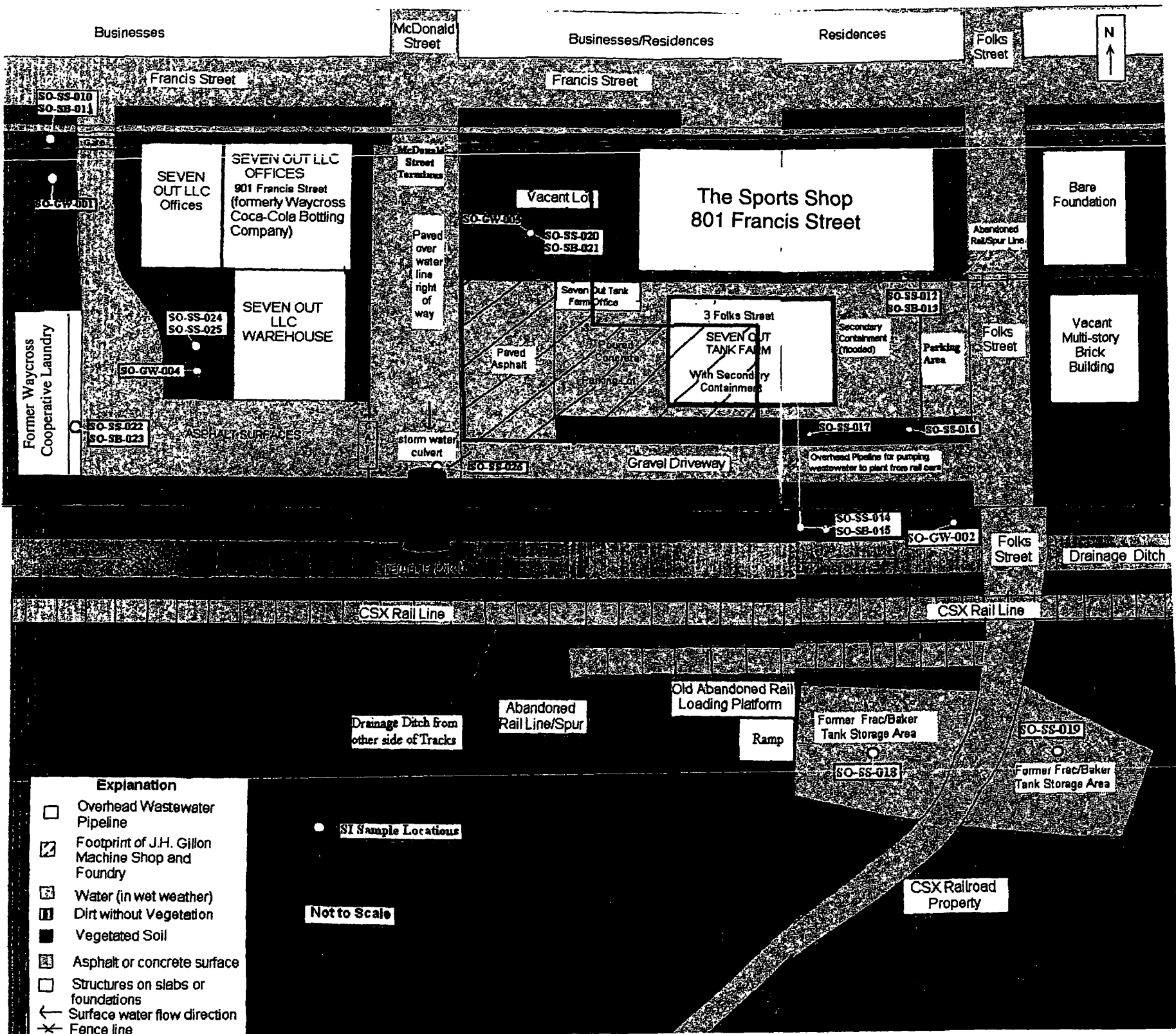
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Figure 1



- | | | | |
|--|-------------------------------------|----|-------------------------------|
| | Areas specific to Seven Out LLC Use | | Vegetated area |
| | Paved Areas (concrete or asphalt) | | Graveled area with vegetation |
| | Drainage from site | XX | Fenced |



U . S . E P A R E G I O N I V

SDMS

Unscannable Material Target Sheet

DocID: 10508639

Site ID: GAN000407811

Site Name:

Scrap Out LLC

Nature of Material:

Map:

✓

Computer Disks:

Photos:

CD-ROM:

Blueprints:

Oversized Report:

Slides:

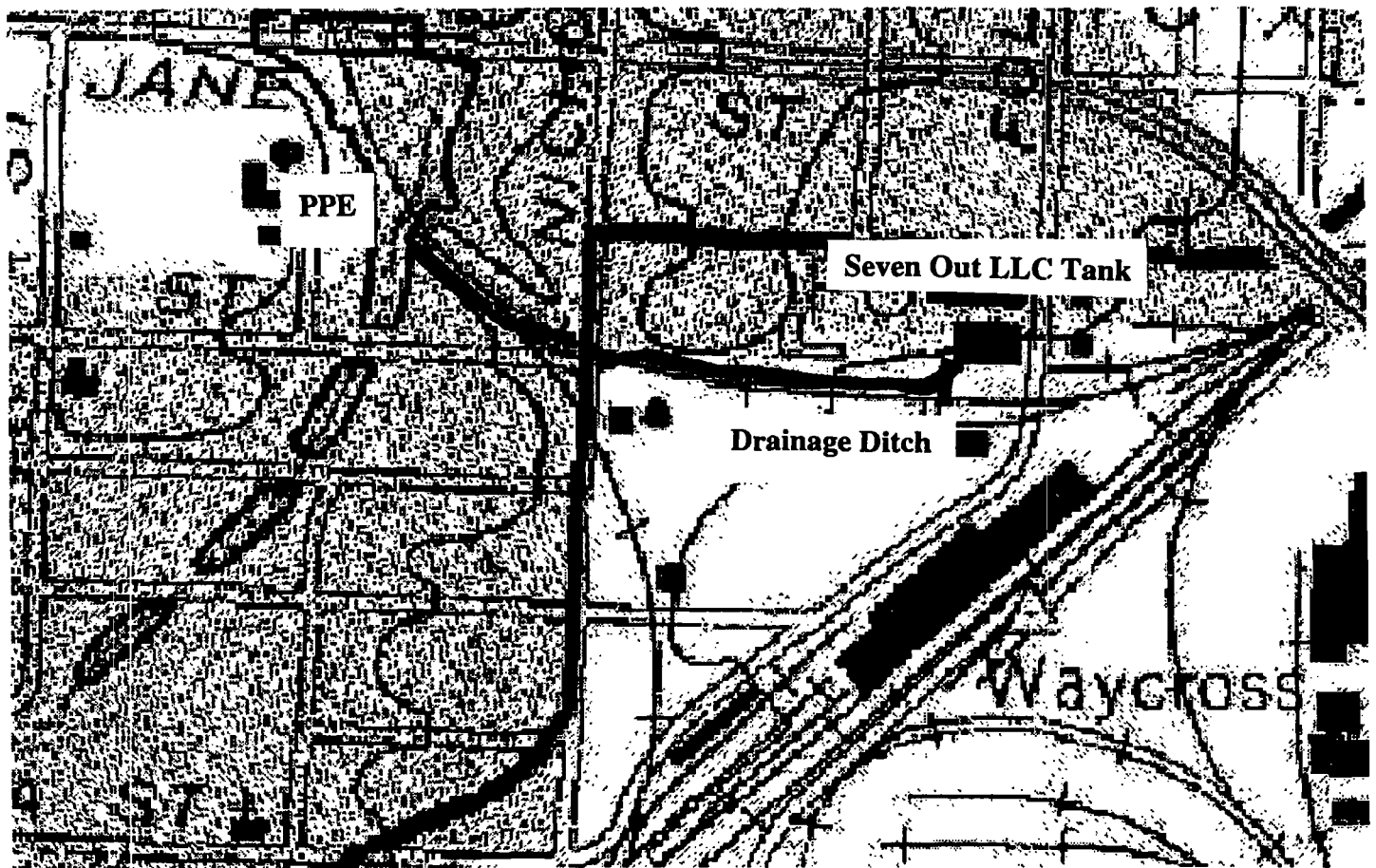
Log Book:

Other (describe):

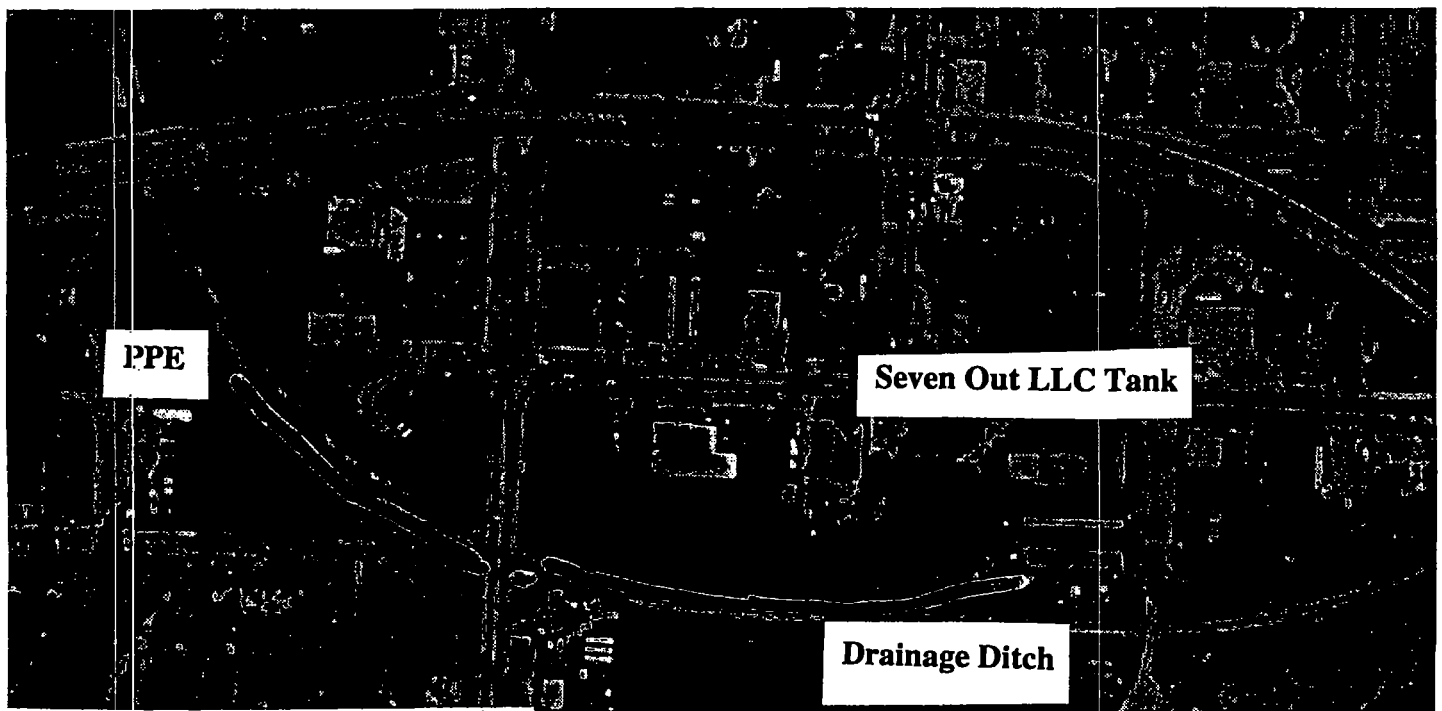
4 mile radius map

Amount of material:

Please contact the appropriate Records Center to view the material.



Seven Out LLC Tank Site Overland Runoff Route



(Modified from References 15 and 56)

Figure 5

U . S . E P A R E G I O N I V

SDMS

Unscannable Material Target Sheet

DocID: 10508639

Site ID: GAN000407811

Site Name: Series Out LLC

Nature of Material:

Map: ✓

Computer Disks:

Photos:

CD-ROM:

Blueprints:

Oversized Report:

Slides:

Log Book:

Other (describe): Topographic Map

Amount of material:

Please contact the appropriate Records Center to view the material.

U . S . E P A R E G I O N I V

SDMS

Unscannable Material Target Sheet

DocID: 105081039 Site ID: CAN000407811

Site Name: Seas Out LLC

Nature of Material:

Map: ✓

Computer Disks:

Photos:

CD-ROM:

Blueprints:

Oversized Report:

Slides:

Log Book:

Other (describe): Wetlands Inventory Map

Amount of material:

Please contact the appropriate Records Center to view the material.

APPENDIX A
LIST OF ANALYTES



U.S. Environmental Protection Agency

Superfund Analytical Services/Contract Laboratory Program (CLP)

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SOM01.1 Volatile Target Compound List and Corresponding CRQLs

COMPOUND	SOM01.1 CONTRACT REQUIRED QUANTITATION LIMITS				
	Trace Water by SIM (ug/L)	Trace Water (ug/L)	Low Water (ug/L)	Low Soil (ug/kg)	Med. Soil (ug/kg)
Dichlorodifluoromethane	--	0.50	5.0	5.0	250
Chloromethane EXIT Disclaimer	--	0.50	5.0	5.0	250
Vinyl chloride EXIT Disclaimer	--	0.50	5.0	5.0	250
Bromomethane	--	0.50	5.0	5.0	250
Chloroethane EXIT Disclaimer	--	0.50	5.0	5.0	250
Trichlorofluoromethane	--	0.50	5.0	5.0	250
1,1-Dichloroethene EXIT Disclaimer	--	0.50	5.0	5.0	250
1,1,2-Trichloro-1,2,2-trifluoroethane	--	0.50	5.0	5.0	250
Acetone EXIT Disclaimer	--	5.0	10	10	500
Carbon disulfide EXIT Disclaimer	--	0.50	5.0	5.0	250
Methyl acetate	--	0.50	5.0	5.0	250
Methylene chloride EXIT Disclaimer	--	0.50	5.0	5.0	250
trans-1,2-Dichloroethene	--	0.50	5.0	5.0	250
Methyl tert-butyl ether EXIT Disclaimer	--	0.50	5.0	5.0	250

1,1-Dichloroethane	EXIT Disclaimer	--	0.50	5.0	5.0	250
cis-1,2-Dichloroethene	EXIT Disclaimer	--	0.50	5.0	5.0	250
2-Butanone	EXIT Disclaimer	--	5.0	10	10	500
Bromochloromethane		--	0.50	5.0	5.0	250
Chloroform	EXIT Disclaimer	--	0.50	5.0	5.0	250
1,1,1-Trichloroethane	EXIT Disclaimer	--	0.50	5.0	5.0	250
Cyclohexane		--	0.50	5.0	5.0	250
Carbon tetrachloride	EXIT Disclaimer	--	0.50	5.0	5.0	250
Benzene		--	0.50	5.0	5.0	250
1,2-Dichloroethane	EXIT Disclaimer	--	0.50	5.0	5.0	250
1,4-Dioxane		2.0	20	100	100	5000
Trichloroethene	EXIT Disclaimer	--	0.50	5.0	5.0	250
Methylcyclohexane		--	0.50	5.0	5.0	250
1,2-Dichloropropane	EXIT Disclaimer	--	0.50	5.0	5.0	250
Bromodichloromethane	EXIT Disclaimer	--	0.50	5.0	5.0	250
cis-1,3-Dichloropropene	EXIT Disclaimer	--	0.50	5.0	5.0	250
4-Methyl-2-pentanone		--	5.0	10	10	500
Toluene	EXIT Disclaimer	--	0.50	5.0	5.0	250
trans-1,3-Dichloropropene		--	0.50	5.0	5.0	250
1,1,2-Trichloroethane	EXIT Disclaimer	--	0.50	5.0	5.0	250
Tetrachloroethene	EXIT Disclaimer	--	0.50	5.0	5.0	250
2-Hexanone	EXIT Disclaimer	--	5.0	10	10	500
Dibromochloromethane	EXIT Disclaimer	--	0.50	5.0	5.0	250
1,2-Dibromoethane	EXIT Disclaimer	0.050	0.50	5.0	5.0	250
Chlorobenzene	EXIT Disclaimer	--	0.50	5.0	5.0	250
Ethylbenzene	EXIT Disclaimer	--	0.50	5.0	5.0	250
o-Xylene	EXIT Disclaimer	--	0.50	5.0	5.0	250
m,p-Xylene	EXIT Disclaimer	--	0.50	5.0	5.0	250
Styrene	EXIT Disclaimer	--	0.50	5.0	5.0	250
Bromoform	EXIT Disclaimer	--	0.50	5.0	5.0	250
Isopropylbenzene		--	0.50	5.0	5.0	250

1,1,2,2-Tetrachloroethane EXIT Disclaimer	--	0.50	5.0	5.0	250
1,3-Dichlorobenzene EXIT Disclaimer	--	0.50	5.0	5.0	250
1,4-Dichlorobenzene EXIT Disclaimer	--	0.50	5.0	5.0	250
1,2-Dichlorobenzene EXIT Disclaimer	--	0.50	5.0	5.0	250
1,2-Dibromo-3-chloropropane EXIT Disclaimer	0.050	0.50	5.0	5.0	250
1,2,4-Trichlorobenzene	--	0.50	5.0	5.0	250
1,2,3-Trichlorobenzene	--	0.50	5.0	5.0	250

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SOM01.1 Semivolatile Target Compound List and Corresponding CRQLs

COMPOUND	SOM01.1 CONTRACT REQUIRED QUANTITATION LIMITS				
	Low Water by SIM (ug/L)	Low Water (ug/L)	Low Soil by SIM (ug/kg)	Low Soil (ug/kg)	Med. Soil (ug/kg)
Benzaldehyde	--	5.0	--	170	5,000
Phenol EXIT Disclaimer	--	5.0	--	170	5,000
Bis(2-chloroethyl) ether EXIT Disclaimer	--	5.0	--	170	5,000
2-Chlorophenol EXIT Disclaimer	--	5.0	--	170	5,000
2-Methylphenol	--	5.0	--	170	5,000
2,2'-Oxybis(1-chloropropane)	--	5.0	--	170	5,000
Acetophenone	--	5.0	--	170	5,000
4-Methylphenol	--	5.0	--	170	5,000
N-Nitroso-di-n propylamine	--	5.0	--	170	5,000
Hexachloroethane EXIT Disclaimer	--	5.0	--	170	5,000
Nitrobenzene EXIT Disclaimer	--	5.0	--	170	5,000

Isophorone <u>EXIT Disclaimer</u>	-	5.0	--	170	5,000
2-Nitrophenol <u>EXIT Disclaimer</u>	-	5.0	--	170	5,000
2,4-Dimethylphenol	-	5.0	--	170	5,000
Bis(2-chloroethoxy) methane	-	5.0	--	170	5,000
2,4-Dichlorophenol <u>EXIT Disclaimer</u>	-	5.0	--	170	5,000
Naphthalene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
4-Chloroaniline	-	5.0	--	170	5,000
Hexachlorobutadiene <u>EXIT Disclaimer</u>	-	5.0	--	170	5,000
Caprolactam	-	5.0	--	170	5,000
4-Chloro-3-methylphenol	-	5.0	--	170	5,000
2-Methylnaphthalene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Hexachlorocyclopentadiene	-	5.0	--	170	5,000
2,4,6-Trichlorophenol <u>EXIT Disclaimer</u>	-	5.0	--	170	5,000
2,4,5-Trichlorophenol <u>EXIT Disclaimer</u>	-	5.0	--	170	5,000
1,1'-Biphenyl	-	5.0	--	170	5,000
2-Chloronaphthalene	-	5.0	--	170	5,000
2-Nitroaniline	-	10	--	330	10,000
Dimethylphthalate	-	5.0	--	170	5,000
2,6-Dinitrotoluene <u>EXIT Disclaimer</u>	-	5.0	--	170	5,000
Acenaphthylene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
3-Nitroaniline	-	10	--	330	10,000
Acenaphthene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
2,4-Dinitrophenol <u>EXIT Disclaimer</u>	-	10	--	330	10,000
4-Nitrophenol <u>EXIT Disclaimer</u>	-	10	--	330	10,000
Dibenzofuran	-	5.0	--	170	5,000
2,4-Dinitrotoluene <u>EXIT Disclaimer</u>	-	5.0	--	170	5,000

Diethylphthalate	-	5.0	-	170	5,000
Fluorene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
4-Chlorophenyl-phenyl ether	-	5.0	-	170	5,000
4-Nitroaniline	-	10	-	330	10,000
4,6-Dinitro-2-methylphenol	-	10	-	330	10,000
N-Nitrosodiphenylamine EXIT Disclaimer	-	5.0	-	170	5,000
1,2,4,5-Tetrachlorobenzene	-	5.0	-	170	5,000
4-Bromophenyl-phenylether	-	5.0	-	170	5,000
Hexachlorobenzene EXIT Disclaimer	-	5.0	-	170	5,000
Atrazine EXIT Disclaimer	-	5.0	-	170	5,000
Pentachlorophenol EXIT Disclaimer	0.20	10	6.7	330	10,000
Phenanthrene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
Anthracene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
Carbazole	-	5.0	-	170	5,000
Di-n-butylphthalate EXIT Disclaimer	-	5.0	-	170	5,000
Fluoranthene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
Pyrene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
Butylbenzylphthalate	-	5.0	-	170	5,000
3,3'-dichlorobenzidine EXIT Disclaimer	-	5.0	-	170	5,000
Benzo(a)anthracene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
Chrysene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
Bis(2-ethylhexyl) phthalate EXIT Disclaimer	-	5.0	-	170	5,000
Di-n-octylphthalate EXIT Disclaimer	-	5.0	-	170	5,000
Benzo(b) fluoranthene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
Benzo(k) fluoranthene EXIT Disclaimer	0.10	5.0	3.3	170	5,000
Benzo(a) pyrene EXIT Disclaimer	0.10	5.0	3.3	170	5,000

<u>Indeno(1,2,3-cd) pyrene</u> EXIT Disclaimer	0.10	5.0	3.3	170	5,000
<u>Dibenzo(a,h) anthracene</u> EXIT Disclaimer	0.10	5.0	3.3	170	5,000
<u>Benzo(g,h,i) perylene</u> EXIT Disclaimer	0.10	5.0	3.3	170	5,000
<u>2,3,4,6-Tetrachlorophenol</u> EXIT Disclaimer	--	5.0	--	170	5,000

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Metals/Cyanide Target Analyte List and Corresponding CRQLs

The following Analytes and CRQLs are taken from the ILM05.3 Statement of Work.

ANALYTES	CONTRACT REQUIRED QUANTITATION LIMITS		
	ICP-AES Water (ug/L)	ICP-AES Soil (mg/kg)	ICP-MS Water (ug/L)
Aluminum EXIT Disclaimer	200	20	—
Antimony EXIT Disclaimer	60	6	2
Arsenic	10	1	1
Barium	200	20	10
Beryllium EXIT Disclaimer	5	0.5	1
Cadmium EXIT Disclaimer	5	0.5	1
Calcium	5000	500	—
Chromium EXIT Disclaimer	10	1	2
Cobalt EXIT Disclaimer	50	5	1
Copper EXIT Disclaimer	25	2.5	2
Iron	100	10	—
Lead	10	1	1
Magnesium	5000	500	—
Manganese EXIT Disclaimer	15	1.5	1
Mercury EXIT Disclaimer	0.2	0.1	—

Nickel EXIT Disclaimer	40	4	1
Potassium	5000	500	—
Selenium EXIT Disclaimer	35	3.5	5
Silver EXIT Disclaimer	10	1	1
Sodium	5000	500	—
Thallium EXIT Disclaimer	25	2.5	1
Vanadium EXIT Disclaimer	50	5	1
Zinc EXIT Disclaimer	60	6	2
Cyanide EXIT Disclaimer	10	2.5	—

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APPENDIX B

EPD LABORATORY – QUALITY ASSURANCE/QUALITY CONTROL

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Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

A.7.3 EPA Method 624/8260B Volatile Organics Analyses**A.7.3.1 Scope and Application**

Method 624/8260B is used to determine volatile organic compounds in liquids, soils, and variety of multiphase samples. As part of the SW-846 requirement, the EPD laboratory analyzes the following:

Dichlorodifluoromethane
Chloromethane
Bromomethane
Vinyl chloride
Chloroethane
Methylene chloride
Trichlorofluoromethane
Acetone
Dibromomethane
trans-1,2-Dichloroethene
Iodomethane
Carbon disulfide
1,1-Dichloroethene
1,1-Dichloroethane
cis-1,2-Dichloroethene
2,2-Dichloropropane
Bromochloromethane
Chloroform
1,1-Dichloropropene
1,2-Dichloroethane
2-Butanone
1,1,1-Trichloroethane
Carbon tetrachloride
Vinyl acetate
Bromodichloromethane
1,2-Dichloropropane
Trichloroethene
Benzene
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
Dibromochloromethane
1,1,2-Trichloroethane
Bromoform
Acrylonitrile
trans-1,4-Dichloro-2-butene
1,2,3-Trichloropropane

Methyl tert-butyl ether

4-Methyl-2-pentanone
2-Hexanone
Tetrachloroethene
1,3-Dichloropropane
1,1,2,2-Tetrachloroethane
Toluene
1,2-Dibromoethane
Chlorobenzene
Ethylbenzene
1,1,1,2-Tetrachloroethane
Styrene
p,m-Xylene
o-Xylene
Isopropylbenzene
Bromobenzene
n-Propylbenzene
2-Chlorotoluene
1,3,5-Trimethylbenzene
4-Chlorotoluene
tert-Butylbenzene
1,2,4-Trimethylbenzene
sec-Butylbenzene
1,3-Dichlorobenzene
p-Isopropyltoluene
1,4-Dichlorobenzene
n-Butylbenzene
1,2-Dichlorobenzene
1,2-Dibromo-3-chloropropane
1,2,4-Trichlorobenzene
Hexachlorobutadiene
Naphthalene
1,2,3-Trichlorobenzene
1,1,2-Trichlorotrifluoroethane

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Methyl acetate
Cyclohexane
Methylcyclohexane

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Water samples for volatile organic compounds are collected in a 40 ml glass vial with 1:1 HCL as a preservative, samples must be cooled to 4°C. Four sample bottles are required for each sample. Holding time for persevered samples is 14 days.

Soil and sediment samples for volatile organic compounds are collected in EnCore™ samplers. The EnCore™ samplers must be cooled to 4°C after sample collection. Four EnCores™ are required for each sample; additionally, a single 4 oz wide mouth glass bottle is required for each sample. Samples must be preserved in the Laboratory within 48 hours and must then be analyzed within 14 days.

A.7.3.1.2 Samples are introduced into a gas chromatograph by the purge-and-trap method. Purged sample analytes are trapped using a Purge Trap K (VOCARB 3000). Upon completion of purging (11 minutes at 30°C at approximately 38-40ml/min for aqueous or 11 minutes at 40°C at approximately 38-40ml/min for soil), the trap is heated and back flushed with helium to desorb (desorb preheat at 240°C and then desorb at 250°C for 4 minutes) the analytes onto the GC column. The GC column is temperature programmed to separate the analytes and introduces them to the mass spectrometer detector (35°C for 4 minutes, then ramp up to 200°C at 8°C/min and baked at 200°C for 1 minutes.). The identification of target analytes is accomplished by the comparison of mass spectrum of known standards with the aid of a reference library. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard followed by a comparison to a seven point calibration curve.

A.7.3.2 Calibrations and Calculations**A.7.3.2.1 BFB Tuning Criteria**

GC/MS system calibration and sample analysis can not begin until the required BFB key ions and ion abundance criteria is met.

<u>Mass</u>	<u>Ion Abundance Criteria</u>
50	15.0 to 40.0 percent of m/e 95
75	30.0 to 60.0 percent of m/e 95
95	base peak, 100 percent relative abundance
96	5.0 to 9.0 percent of m/e 95
173	less than 2.0 percent of m/e 174
174	>50.0 but < 100 percent of m/e 95
175	5.0 to 9.0 percent of m/e 174
176	>95.0 but < 101.0 percent of m/e 174
177	5.0 to 9.0 percent of m/e 176

A.7.3.2.2 Calibration Curve

A seven-point calibration is generated for each matrix prior to analysis. The calibration system utilizes traceable standards containing a mixture of the above compounds listed volatile organic.

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A seven-point calibration curve is generated to determine the response factor and the percent relative standard deviation of each analyte. A mid-level standard, prepared from a second source, is analyzed as a confirmation of standard mix concentrations.

A.7.3.2.3 Calibration Standards

The calibration curve consists of the calibration standards at concentrations of 2, 5, 50, 100, 150, 200, and 400 (ug/L or ug/Kg). There are four internal standards, Pentafluorobenzene, 1,4-Difluorobenzene, Chlorobenzene-d5, 1,4-Dichlorobenzene-d4, and four surrogate standards, Dibromofluoromethane, Toluene-d8, 1,2-Dichloroethene-d4, Bromofluorobenzene. They are used in calibration, quality control, and sample analysis. The calibration is an average response factor curve fit and should result in a percent relative standard deviation for all compounds.

The System Performance Check Compounds (SPCCs) should be checked for a minimum average relative response factor before the calibration curve is used. The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The percent relative standard deviation (%RSD) for Calibration Check Compound (CCCs) must be less than 30%. The CCCs are:

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene, and
Vinyl chloride

The percent relative standard deviation (%RSD) should be less than 15% for each target analyte. If the %RSD of any compound is greater than 15%, then the analyst should select linear or quadratic regression fit of the seven calibration with the correlations must be greater or equal to 0.990.

A second source initial calibration standard should be analyzed with all performance analytes (SPCCs & CCCs). The %D should be between 70% to 130% limit, or a new initial calibration standard should be prepared.

A.7.3.2.4 Calibration Verification

A daily continuing calibration verification (CCV) is performed every 12-hour analysis period to monitor and validate the instrumentation, column, and mass spectrometer performance. The CCV consist of a 50ppb calibration standard.

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A.7.3.2.5 Record Keeping

Documentation of instrument calibration are reviewed for adherence to quality criteria and then stored in the calibration curve records.

A.7.3.2.6 Daily Calibration Verification and Continuing Calibration

A 50 ppb calibration standard ensures the instrument's SPCCs and CCCs meet method performance criteria. For any 12 hours analysis period, prior to samples analysis, a one point daily continuing calibration verification is performed. The System Performance Check Compounds (SPCCs) must meet the minimum average relative response factor (A.7.3.2.3). For the Calibration Check Compound (CCCs) the percent drift for each CCC is not to exceed 20% of the initial calibration. If the continuing calibration does not meet method performance criteria then the instrument must be recalibrated.

Calculate the percent drift using the following equation:

$$\% \text{Drift} = (C_i - C_c) / C_i \times 100$$

where:

C_i = Calibration Check Compound standard concentration.

C_c = Measured concentration using selected quantitation method.

A.7.3.2.7 Relative Response Factor (RRF)

Relative Response Factor: Calculate the relative response factors (RRF) for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

$$\text{Equation A.7.3.1} \quad RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where

RRF = Relative response factor

A_x = Area of the primary ion for the compound to be measured

A_{is} = Area of the primary ion for the internal standard

C_{is} = Concentration of internal standard spiking mixture, ppb

C_x = Concentration of the compound in the calibration standard, ppb

A.7.3.2.8 Mean Relative Response Factor (\overline{RRF})

$$\text{Equation A.7.3.2} \quad \overline{RRF} = \frac{\sum_{i=1}^n x_i}{n}$$

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Mean Relative Response Factor: Calculate the mean \overline{RRF} for each compound by averaging the values obtained at the seven concentrations using the following equation:

where:

$$\begin{aligned}\overline{RRF} &= \text{Mean relative response factor} \\ x_i &= \text{RRF of the compound} \\ n &= \text{Number of values}\end{aligned}$$

A.7.3.2.9 Percent Relative Standard Deviation (%RSD)

Using the RRFs from the initial calibration, calculate the %RSD for all target compounds using the following equations:

$$\text{Equation A.7.3.3} \quad \%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100$$

and

$$\text{Equation A.7.3.4} \quad SD_{RRF} = \sqrt{\frac{\sum_{i=1}^n (RRF_i - \overline{RRF})^2}{n - 1}}$$

where:

$$\begin{aligned}SD_{RRF} &= \text{Standard deviation of initial response factors (per compound)} \\ RRF_i &= \text{Relative response factor at a concentration level} \\ \overline{RRF} &= \text{Mean of initial relative response factors (per compound).} \\ n &= \text{Number of values}\end{aligned}$$

A.7.3.2.10 Relative Retention Times (RRT)

The retention time for each internal standard must be within ± 30 seconds of the retention time of the internal standard in the most recent valid calibration. Relative retention time of each analyte must be within ± 0.06 RRT units of the RRT. Calculate the RRTs for each target compound over the initial calibration range using the following equation.

$$\text{Equation A.7.3.5} \quad RRT = \frac{RT_c}{RT_{is}}$$

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where:

 Rt_c = Retention time of the target compound Rt_{IS} = Retention time of the internal standard.A.7.3.2.11 Mean of the Relative Retention Times (\overline{RRT}):

Calculate the mean of the relative retention times (\overline{RRT}) for each analyte target compound over the initial calibration range using the following equation:

Equation A.7.3.6
$$\overline{RRT} = \sum_{i=1}^n \frac{RRT}{n}$$

where:

 \overline{RRT} = Mean relative retention time for the target compound for each initial calibration standard RRT = Relative retention time for the target compound at each calibration level n = Number of values

Tabulate the area response (Y) of the primary ion and the corresponding concentration for each compound and internal standard.

A.7.3.2.12 Mean Area Response (\overline{Y}) for Internal Standard:

Calculate the mean area response (\overline{Y}) for each internal standard compound over the initial calibration range using the following equation:

Equation A.7.3.7
$$\overline{Y} = \sum_{i=1}^n \frac{Y_i}{n}$$

where:

 \overline{Y} Mean area response Y = Area response for the primary quantitation ion for the internal standard for each initial calibration standard.A.7.3.2.13 Mean of the Retention Times (\overline{RT}) For Internal Standard:

Calculate the mean of the retention times (\overline{RT}) for each internal standard over the initial calibration range using the following equation:

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Equation A.7.3.8

$$\overline{RT} = \sum_{i=1}^n \frac{RT_i}{n}$$

where:

 \overline{RT} = Mean retention time RT = Retention time for the internal standard for each initial calibration standard. n = Number of values

A.7.3.2.14 Percent Difference (%D):

Calculate the percent difference in the RRF of the daily RRF (24-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

Equation A.7.3.9

$$\%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

where:

 RRF_c = RRF of the compound in the continuing calibration standard $\overline{RRF_i}$ = Mean RRF of the compound in the most recent initial calibration.

A.7.3.2.15 Sample Concentration Calculation.

Equation A.7.3.10

$$C_x = \frac{A_x C_{is} DF}{A_{is} RRF}$$

where:

 C_x = Compound concentration, ppb A_x = Area of the characteristic ion for the compound to be measured A_{is} = Area of the characteristic ion for the specific internal standard C_{is} = Concentration of the internal standard spiking mixture, ppb RRF = Relative response factor from the analysis of the continuing calibration standard or the mid level standard of the initial calibration DF = Dilution factor. If no dilution is performed, $DF = 1$

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Table A.7.3.1 RLs for EPA 624/8260B

Parameter/ Method	Analyte	Matrix Water		Matrix Soil		Matrix Waste		TCLP	
		RL	Unit	RL	Unit	RL	Unit	RL	Unit
VOCs in 624/8260B	Dichlorodifluoromethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Chloromethane	10.0	ug/L	10.0	ug/Kg	0.5	mg/Kg		mg/L
	Bromomethane	10.0	ug/L	10.0	ug/Kg	0.5	mg/Kg		mg/L
	Vinyl chloride	2.0	ug/L	2.0	ug/Kg	0.1	mg/Kg	0.04	mg/L
	Chloroethane	10.0	ug/L	10.0	ug/Kg	0.5	mg/Kg		mg/L
	Methylene chloride	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Trichlorofluoromethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Acetone	100.0	ug/L	100.0	ug/Kg	5.0	mg/Kg		mg/L
	Dibromomethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	trans-1,2-Dichloroethene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Iodomethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Carbon disulfide	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	1,1-Dichloroethene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg	0.1	mg/L
	1,1-Dichloroethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	cis-1,2-Dichloroethene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	2,2-Dichloropropane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Bromochloromethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Chloroform	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg	0.1	mg/L
	1,1-Dichloropropene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	1,2-Dichloroethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg	0.1	mg/L
	2-Butanone	100.0	ug/L	100.0	ug/Kg	5.0	mg/Kg	2.0	mg/L
	1,1,1-Trichloroethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Carbon tetrachloride	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg	0.1	mg/L
	Vinyl acetate	50.0	ug/L	50.0	ug/Kg	2.5	mg/Kg		mg/L
	Bromodichloromethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	1,2-Dichloropropane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Trichloroethene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg	0.1	mg/L
	Benzene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg	0.1	mg/L
	cis-1,3-Dichloropropene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	trans-1,3-Dichloropropene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Dibromochloromethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	1,1,2-Trichloroethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Bromoform	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	1,2,3-Trichloropropane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	4-Methyl-2-pentanone	50.0	ug/L	50.0	ug/Kg	2.5	mg/Kg		mg/L
	2-Hexanone	50.0	ug/L	50.0	ug/Kg	2.5	mg/Kg		mg/L
	Tetrachloroethene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg	0.1	mg/L
	1,3-Dichloropropane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	1,1,2,2-Tetrachloroethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	Toluene	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L
	1,2-Dibromoethane	5.0	ug/L	5.0	ug/Kg	0.25	mg/Kg		mg/L

Modified 1/24/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

	Chlorobenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	0.1 mg/L
VOCs in 624/8260B	Ethylbenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,1,1,2-Tetrachloroethane	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	Styrene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	p,m-Xylene	10.0 ug/L	10.0 ug/Kg	0.5 mg/Kg	mg/L
	o-Xylene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	Isopropylbenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	Bromobenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	n-Propylbenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	2-Chlorotoluene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,3,5-Trimethylbenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	4-Chlorotoluene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	tert-Butylbenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,2,4-Trimethylbenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	sec-Butylbenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,3-Dichlorobenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	p-Isopropyltoluene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,4-Dichlorobenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	n-Butylbenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,2-Dichlorobenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,2-Dibromo-3-chloropropane	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,2,4-Trichlorobenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	Hexachlorobutadiene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	Naphthalene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,2,3-Trichlorobenzene	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	Acrylonitrile	200 ug/L	200 ug/Kg	10.0 mg/Kg	mg/L
	trans-1,4-Dichloro-2-butene	100 ug/L	100 ug/Kg	5.0 mg/Kg	mg/L
	Methyl tert-butyl ether	5.0 ug/L	5.0 ug/Kg	0.25 mg/Kg	mg/L
	1,1,2-Trichloro-1,2,2-trifluoroethane	10.0 ug/L	10.0 ug/Kg	0.5 mg/Kg	mg/L
	Methyl acetate	10.0 ug/L	10.0 ug/Kg	0.5 mg/Kg	mg/L
	Cyclohexane	10.0 ug/L	10.0 ug/Kg	0.5 mg/Kg	mg/L
	Methylcyclohexane	10.0 ug/L	10.0 ug/Kg	0.5 mg/Kg	mg/L
	Pentafluorobenzene (IS)	--	--	--	--
	1,4-Difluorobenzene (IS)	--	--	--	--
	Chlorobenzene (IS)	--	--	--	--
	1,4-Dichlorobenzene-d4 (IS)	--	--	--	--
	Dibromofluoromethane (SURR)	50.0 ug/L	50.0 ug/Kg	50 ug/Kg	50 ug/L
	Toluene-d8 (SURR)	50.0 ug/L	50.0 ug/Kg	50 ug/Kg	50 ug/L
	Bromofluorobenzene (SURR)	50.0 ug/L	50.0 ug/Kg	50 ug/Kg	50 ug/L
	1,2 Dichloroethane d4 (SURR)	50.0 ug/L	50.0 ug/Kg	50 ug/Kg	50 ug/L

Modified 1/24/2006

Georgia Department of Natural Resources
Environmental Protection Division
 EPD Laboratory, 455 14th Street, Atlanta GA 30318
 David Jones, Laboratory Director

Table A.7.2.2 Acceptance Criteria for EPA 624/8260B

[1 year upper and lower control limits (01/01/2005 12/31/2005)]

Method 624/8260B	Analyte	Accuracy Aqueous (%R)	Precision Aqueous (RPD)	Accuracy Soil (%R)	Precision Soil (RPD)	Accuracy Waste (%R)	Precision Waste (RPD)
LCS	1,1-Dichloroethene	88-115	≤ 30%	79-120	≤ 40%	86-109	≤ 50%
	Benzene	91-112	≤ 30%	87-108	≤ 40%	89-109	≤ 50%
	Trichloroethene	90-114	≤ 30%	84-113	≤ 40%	89-109	≤ 50%
	Toluene	91-111	≤ 30%	79-114	≤ 40%	89-109	≤ 50%
	Chlorobenzene	89-109	≤ 30%	82-111	≤ 40%	88-108	≤ 50%
MS	1,1-Dichloroethene	41-144	≤ 30%	19-149	≤ 40%	34-135	≤ 50%
	Benzene	61-160	≤ 30%	54-137	≤ 40%	69-126	≤ 50%
	Trichloroethene	62-149	≤ 30%	72-137	≤ 40%	80-116	≤ 50%
	Toluene	64-160	≤ 30%	57-150	≤ 40%	59-148	≤ 50%
	Chlorobenzene	62-159	≤ 30%	84-146	≤ 40%	74-141	≤ 50%
SS	Dibromofluoromethane	96-115	NA	93-121	NA	86-112	NA
	1,2-Dichloroethane-d4	95-115	NA	90-124	NA	88-112	NA
	Toluene-d8	91-111	NA	87-103	NA	89-109	NA
	Bromofluorobenzene	88-108	NA	76-105	NA	87-107	NA

Table A.7.3.3 Summary of Calibration and QC Procedures for Method EPA 624/8260B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
624/8260B	Volatile Organics	Seven -point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.10 ; and %RSD for CCCs ≤ 30% and RSD for all compounds ± 15% <i>option #1</i> linear regression for any analytes r ≥ 0.990 <i>option #2</i> non-linear regression CORR ≥ 0.990	Correct problem then repeat initial calibration.	
		Second-source calibration verification	Once per seven- point initial calibration (usually 50ug/L concentration level)	All performance analytes within ±30% of expected value (SPCCs & CCCs)	Correct problem . Repeat another second source run. If problem persist repeat initial calibration.	

Modified 1/24/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

624/8260B	Volatile Organics	Calibration verification	Daily, before sample analysis, every 12 hours of analysis time	SPCCs average RF0.10; and CCCs \pm 20% drift; and all calibration analytes within \pm 20% drift criterion if the CCCs are not required analyses by the permit	Correct problem then repeat initial calibration	
		Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table A.7.3.2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify	
		ISs	Immediately after or during data acquisition of calibration check standard	Retention time \pm 30 seconds: EICP area within -50% and \pm 100% of Initial Calibration from mid-point standard (50ug/L)	Inspect mass spectrometry or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	
		Method Blank	One per analytical batch	No analytes detected >RL	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	If unable to re-analyze, flag with a "B"
		LCS/LCSD for all analytes	One LCS/LCSD per analytical batch	QC acceptance criteria Table A.7.3.2	Correct problem then reanalyze the LCS and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		Matrix spike & Matrix spike dup	One MS & MSD per analytical batch	QC acceptance criteria Table A.7.3.2	Correct problem then reanalyze samples in the batch if and only if QC acceptance criteria of LCS is failed.	
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria Table A.7.3.2	Correct problem then reanalyze sample	
		MDL study	Once per year	Detection limits established shall be \leq the RLs in Table A.7.3.1	None	

Modified 1/24/2006

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David Jones, Laboratory Director

624/8260B	Volatile Organics	Estimated amount for analytes above the 7- pt calibration curve	none	All analytes >400ug/L. Except for m,p-Xylene# > 800ug/L	Sample must be diluted and reanalyzed.	Apply E to all analytes above initial calibration range..
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A.7.4 EPA Methods 625/8270C - Semi-Volatile Organics by Capillary GC/MS

Measurement of Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique:

Water samples for semivolatile organic compounds are collected in a 1-liter narrow mouth glass bottle. Sample bottles must be cooled to 4°C after sample collection. Two to four bottles are required for each sample. Samples must be extracted within 7 days and the extracts then analyzed within 40 days.

Soil and sediment samples for semivolatile organic compounds are collected in 8 oz wide mouth glass sample bottles. Sample bottles must be cooled to 4°C after sample collection. Two to four bottles are required for each sample. Samples must be extracted within 14 days and the extracts then analyzed within 40 days.

A.7.4.1 Scope and Application:

Methods 625/ 8270C is used to determine the concentration of semi-volatile organic compounds in extracts prepared from liquids, sediments, and a variety of multi-phase samples. The laboratory currently analyzes the following compounds:

1,4-Dioxane	2-Nitrophenol	Acenaphthylene
Pyridine	2,4-Dimethylphenol	2,6-Dinitrotoluene
n-Nitrosodimethylamine	Bis(2-chloroethoxy)methane	3-Nitroaniline
2-Picoline	Benzoic acid	Acenaphthene
Methylmethanesulfonate	2,4-Dichlorophenol	2,4-Dinitrophenol
Ethylmethanesulfonate	1,2,4-Trichlorobenzene	4-Nitrophenol
Aniline	aa-Dimethylphenethylamine	Dibenzofuran
Benzaldehyde	Naphthalene	Pentachlorobenzene
Phenol	4-Chloroaniline	2,4-Dinitrotoluene
Bis(2-chloroethyl)ether	2,6-Dichlorophenol	1-Naphthylamine
2-Chlorophenol	Hexachlorobutadiene	2-Naphthylamine
1,3-Dichlorobenzene	Caprolactam	2,3,4,6-Tetrachlorophenol
1,4-Dichlorobenzene	N-Nitroso-di-n-butylamine	Diethylphthalate
Benzyl alcohol	4-Chloro-3-methylphenol	Fluorene
1,2-Dichlorobenzene	2-Methylnaphthalene	4-Chlorophenyl phenyl ether
2-Methylphenol	1,2,4,5-Tetrachlorobenzene	4-Nitroaniline
Bis (2-chloroisopropyl)ether	Hexachlorocyclopentadiene	Diphenylamine
Acetophenone	2,4,6-Trichlorophenol	4,6-Dinitro-2-methylphenol
4-Methylphenol	2,4,5-Trichlorophenol	N-Nitrosodiphenylamine
N-Nitroso-di-n-propylamine	1,1'-Biphenyl	1,2-Diphenylhydrazine
Hexachloroethane	2-Chloronaphthalene	4-Bromophenyl phenyl ether
Nitrobenzene	1-Chloronaphthalene	Phenacetin
N-Nitrosopiperidine	2-Nitroaniline	Hexachlorobenzene
Isophorone	Dimethylphthalate	

Modified 1/24/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

Atrazine
 4-Aminobiphenyl
 Pentachlorophenol
 Pronamide
 Pentachlorodinitrobenzene
 Phenanthrene
 Anthracene
 Carbazole
 Di-n-butyl phthalate
 Fluoranthene
 Benzdine
 Pyrene
 p-Dimethylaminoazobenzene
 Butylbenzylphthalate
 Benzo[a]anthracene
 3,3'-Dichlorobenzidine
 Chrysene
 Bis(2-ethylhexyl)phthalate
 Di-n-octylphthalate

Benzo[b]fluoranthene
 Benzo[k]fluoranthene
 7,12- Dimethylbenz(a)anthracene
 Benzo[a]pyrene
 3-Methylchloranthrene
 Dibenz(a,j)acridine
 Indeno[1,2,3-cd]pyrene
 Dibenz[a,h]anthracene
 Benzo[g,h,i]perylene
 Alpha-BHC
 Gamma-BHC
 Beta-BHC
 Delta-BHC
 Heptachlor
 Aldrin
 Heptachlor epoxide
 Endosulfan I
 Dieldrin
 p,p'-DDE

Endrin
 Endosulfan 2
 p,p'-DDD
 Endrin aldehyde
 Endosulfan sulfate
 p,p'-DDT

Surrogate Standards:

2-Fluorophenol
 Phenol-d5
 Nitrobenzene-d5
 2-Fluorobiphenyl
 2,4,6-Tribromophenol
 Terphenyl-d14

A.7.4.1.1 The following EPA SW-846 extraction methods are used to prepare samples for 625/8270C analysis:

3510, 3520, 3541, 3550B & 3580A

A.7.4.1.2 Each of these extraction methods uses similar principles. A sample of water or solid is mixed with methylene chloride, which is collected and concentrated to a much smaller volume under controlled conditions. The resulting extracts are then analyzed for the compounds listed in section A.7.4.1, Scope.

A.7.4.1.2 Application

Methods 625/8270C can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

Samples are introduced into the GC/MS system by direct injection from an autosampler. The coating inside this column will separate the compounds of interest by a combination of molecular size and polarity. As each of these separated compounds exits the column it is introduced into the Mass Spectrometer which reduces the compound into several ions which form a unique pattern of ion sizes and intensity which will aid in identifying the compound and determining the concentration.

A.7.4.2 Calibration

Modified 1/24/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

A.7.4.2.1 Initial Calibration - Before any analysis of samples the GC/MS must be "tuned" and calibrated with a minimum of 5 different concentrations of standards that contain all compounds of interest.

The criteria for a passing tune are:

<u>Mass Ion</u>	<u>Abundance Criteria</u>
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Initial Calibration Curve: A minimum of five concentrations of all relative compounds should be analyzed and entered into the initial calibration section of the software. The range of concentrations should be between the reporting limit and a concentration that maintains linearity and does not saturate the column. An average response of less than 15% is considered acceptable for calculating results with the average response factor. If any compound has a higher %RSD it can be checked for Correlation Coefficient (CORR also call R^2) using linear or quadratic regression and it's curve can be used for calculation.

The recommended concentrations for a five-point curve are:

10, 20, 50, 120, and 160 mg/L for all compounds.

All analyses are corrected for drifts in the MS sensitivity by use of internal standards. This method utilizes 6 internal standards at 40 mg/L: 1,4-Dichlorobenzene- d_4 , Naphthalene- d_8 , Acenaphthene- d_{10} , Phenanthrene- d_{10} , Chrysene- d_{12} , and Perylene- d_{12} . All standards and samples are spiked with these compounds.

Independent Calibration Verification: A standard from a different supplier containing the analytes of interest is analyzed. The % Difference should be $\pm 15\%$ from the theoretical amount to verify the concentration of the standards used to make the curve. This is also called a "control" standard.

A.7.4.2.2 Calibration Verification: A mid-level concentration standard of all compounds of interest must be analyzed before each batch of analyses (every 12 hours). The % difference of all compounds should be no greater than "20% difference of the true value. Certain compounds have additional requirements for acceptance, see method 625/8270, section 8 for further requirements.

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

A.7.4.2.3 Calibration Verification

A daily continuing calibration is performed every 12-hour analysis period to monitor and validate the instrumentation, column, and Mass Spectrometer performance.

A.7.4.2.4 Record Keeping

Documentation of instrument calibration is reviewed for adherence to quality criteria and then stored in the calibration curve records.

A.7.4.2.5 Daily Calibration Verification and Continuing Calibration

A 50 ppm calibration standard ensures the instrument's SPCCs and CCCs meet method performance criteria. For any 12-hour analysis period, prior to samples analysis, a one point daily continuing calibration verification is performed. The System Performance Check Compounds (SPCCs) must meet the minimum average relative response factor of 0.050. For the Calibration Check Compound (CCCs) the percent drift for each CCC is not to exceed 20% of the initial calibration. If the continuing calibration does not meet method performance criteria then the instrument must be recalibrated.

Calculate the percent drift using the following equation:

$$\% \text{Drift} = (C_i - C_c) / C_i \times 100$$

where:

C_i = Calibration Check Compound standard concentration.

C_c = Measured concentration using selected quantitation method.

A.7.4.2.6 Relative Response Factor (RRF)

Relative Response Factor: Calculate the relative response factors (RRF) for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

$$\text{Equation A.7.4.1} \quad RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where

RRF = Relative response factor

A_x = Area of the primary ion for the compound to be measured

A_{is} = Area of the primary ion for the internal standard

C_{is} = Concentration of internal standard spiking mixture, ppb

C_x = Concentration of the compound in the calibration standard, ppb

A.7.4.2.7 Mean Relative Response Factor (\overline{RRF})

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

Equation A.7.4.2

$$\overline{RRF} = \sum_{i=1}^n \frac{x_i}{n}$$

Mean Relative Response Factor: Calculate the mean RRF (\overline{RRF}) for each compound by averaging the values obtained at the five concentrations using the following equation:

where:

$$\begin{aligned} \overline{RRF} &= \text{Mean relative response factor} \\ x_i &= \text{RRF of the compound} \\ n &= \text{Number of values} \end{aligned}$$

A.7.4.2.8 Percent Relative Standard Deviation (%RSD)

Using the RRFs from the initial calibration, calculate the %RSD for all target compounds using the following equations:

Equation A.7.4.3

$$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100$$

and

Equation A.7.4.4

$$SD_{RRF} = \sqrt{\sum_{i=1}^n \frac{(RRF_i - \overline{RRF})^2}{n-1}}$$

where:

$$\begin{aligned} SD_{RRF} &= \text{Standard deviation of initial response factors (per compound)} \\ RRF_i &= \text{Relative response factor at a concentration level} \\ \overline{RRF} &= \text{Mean of initial relative response factors (per compound).} \\ n &= \text{Number of values} \end{aligned}$$

A.7.4.2.9 Relative Retention Times (RRT)

The retention time for each internal standard must be within ± 30 seconds of the retention time of the internal standard in the most recent valid calibration. Relative retention time of each analyte within ± 0.06 RRT units of the RRT. Calculate the RRTs for each target compound over the initial calibration range using the following equation.

Equation A.7.4.5

$$RRT = \frac{RT_c}{RT_{IS}}$$

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

where:

Rt_c = Retention time of the target compound

RT_{is} = Retention time of the internal standard.

A.7.4.2.10 Mean of the Relative Retention Times (\overline{RRT}):

Calculate the mean of the relative retention times (\overline{RRT}) for each analyte target compound over the initial calibration range using the following equation:

$$\overline{RRT} = \sum_{i=1}^n \frac{RRT}{n}$$

Equation A.7.4.6

where:

\overline{RRT} = Mean relative retention time for the target compound for each initial calibration standard

RRT = Relative retention time for the target compound at each calibration level

N = Number of values

Tabulate the area response (Y) of the primary ion and the corresponding concentration for each compound and internal standard.

A.7.4.2.11 Mean Area Response (\bar{Y}) for Internal Standard:

Calculate the mean area response (\bar{Y}) for each internal standard compound over the initial calibration range using the following equation:

$$\bar{Y} = \sum_{i=1}^n \frac{Y_i}{n}$$

Equation A.7.4.7

where:

\bar{Y} = Mean area response

Y = Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

A.7.4.2.12 Mean of the Retention Times (\overline{RT}) For Internal Standard:

Calculate the mean of the retention times (\overline{RT}) for each internal standard over the initial calibration range using the following equation:

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

Equation A.7.4.8
$$\overline{RT} = \frac{\sum_{i=1}^n RT_i}{n}$$

where:

 \overline{RT} = Mean retention time RT = Retention time for the internal standard for each initial calibration standard. n = Number of values

A.7.4.2.13 Percent Difference (%D):

Calculate the percent difference in the RRF of the daily RRF (24-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

Equation A.7.4.9
$$\%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

where:

 RRF_c = RRF of the compound in the continuing calibration standard $\overline{RRF_i}$ = Mean RRF of the compound in the most recent initial calibration.

A.7.4.2.14 Sample Concentration Calculation.

Equation A.7.4.5
$$C_x = \frac{A_x C_{is} DF}{A_{is} RRF}$$

where:

 C_x = Compound concentration, ppm A_x = Area of the characteristic ion for the compound to be measured A_{is} = Area of the characteristic ion for the specific internal standard C_{is} = Concentration of the internal standard spiking mixture, ppm RRF = Relative response factor from the analysis of the continuing calibration standard or the mid level standard of the initial calibration DF = Dilution factor. If no dilution is performed, $DF = 1$

Table A.7.4.1 RLs for EPA 625 and SW-846/8270C

Parameter/ Method	Analyte	Matrix (Aqueous)	Matrix (Soil)
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		RL	Unit	RL	Unit
SVOCs EPA 625/SW-846 Method 8270C	Pyridine	10	ug/L	660	ug/kg
	n-Nitrosodimethylamine	10	ug/L	660	ug/kg
	2-Picoline	10	ug/L	660	ug/kg
	Methylmethanesulfonate	10	ug/L	660	ug/kg
	Ethylmethanesulfonate	20	ug/L	660	ug/kg
	Aniline	10	ug/L	1300	ug/kg
	Benzaldehyde	10	ug/L	660	ug/kg
	Phenol	10	ug/L	660	ug/kg
SVOCs EPA 625/SW-846 Method 8270C	bis(2-Chloroethyl)ether	10	ug/L	660	ug/kg
	2-Chlorophenol	10	ug/L	660	ug/kg
	1,3-Dichlorobenzene	10	ug/L	660	ug/kg
	1,4-Dichlorobenzene	10	ug/L	660	ug/kg
	Benzyl Alcohol	20	ug/L	1300	ug/kg
	1,2-Dichlorobenzene	10	ug/L	660	ug/kg
	2-Methylphenol	10	ug/L	660	ug/kg
	Bis (2-Chloroisopropyl) ether	10	ug/L	660	ug/kg
	Acetophenone	10	ug/L	660	ug/kg
	4-Methylphenol	10	ug/L	660	ug/kg
	N-Nitroso-di-n-propylamine	10	ug/L	660	ug/kg
	Hexachloroethane	10	ug/L	660	ug/kg
	Nitrobenzene	20	ug/L	1300	ug/kg
	N-Nitrosopiperidine	10	ug/L	660	ug/kg
	Isophorone	10	ug/L	660	ug/kg
	2-Nitrophenol	10	ug/L	660	ug/kg
	2,4-Dimethylphenol	10	ug/L	660	ug/kg
	Bis(2-Chloroethoxy)methane	10	ug/L	660	ug/kg
	Benzoic Acid	50	ug/L	3300	ug/kg
	2,4-Dichlorophenol	10	ug/L	660	ug/kg
	1,2,4-Trichlorobenzene	10	ug/L	660	ug/kg
	aa-Dimethyl-phentylamine	10	ug/L	660	ug/kg
	Naphthalene	20	ug/L	1300	ug/kg
	4-Chloroaniline	10	ug/L	660	ug/kg
	2,6-Dichlorophenol	10	ug/L	660	ug/kg
	Hexachlorobutadiene	10	ug/L	660	ug/kg
	Caprolactam	10	ug/L	660	ug/kg
	N-Nitroso-di-n-butylamine	10	ug/L	660	ug/kg
	4-Chloro-3-Methylphenol	20	ug/L	1300	ug/kg
	2-Methylnaphthalene	10	ug/L	660	ug/kg
	1,2,4,5-Tetrachlorobenzene	10	ug/L	660	ug/kg
	Hexachlorocyclopentadiene	10	ug/L	660	ug/kg
	2,4,6-Trichlorophenol	10	ug/L	660	ug/kg
	2,4,5-Trichlorophenol	10	ug/L	660	ug/kg
	1,1'Biphenyl	10	ug/L	660	ug/kg
	2-Chloronaphthalene	10	ug/L	660	ug/kg

Modified 1/24/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

	1-Chloronaphthalene	10	ug/L	660	ug/kg
	2-Nitroaniline	50	ug/L	3300	ug/kg
	Dimethylphthalate	10	ug/L	660	ug/kg
	Acenaphthylene	10	ug/L	660	ug/kg
	2,6-Dinitrotoluene	10	ug/L	660	ug/kg
	3-Nitroaniline	50	ug/L	3300	ug/kg
	Acenaphthene	10	ug/L	660	ug/kg
	2,4-Dinitrophenol	50	ug/L	3300	ug/kg
	4-Nitrophenol	50	ug/L	3300	ug/kg
	Dibenzofuran	10	ug/kg	660	ug/kg
SVOCs EPA 625/SW-846 Method 8270C	Pentachlorobenzene	10	ug/kg	660	ug/kg
	2,4-Dinitrotoluene	10	ug/L	660	ug/kg
	1-Naphthylamine	10	ug/L	660	ug/kg
	2-Naphthylamine	10	ug/L	660	ug/kg
	2,3,4,6-Tetrachlorophenol	10	ug/L	660	ug/kg
	Diethylphthalate	10	ug/L	660	ug/kg
	Fluorene	10	ug/L	660	ug/kg
	4-Chlorophenyl-phenylether	10	ug/L	660	ug/kg
	4-Nitroaniline	20	ug/L	1300	ug/kg
	Diphenylamine	10	ug/L	660	ug/kg
	4,6-Dinitro-2-methylphenol	50	ug/L	3300	ug/kg
	N-Nitrosodiphenylamine	10	ug/L	660	ug/kg
	1,2-Diphenylhydrazine	10	ug/L	660	ug/kg
	4-Bromophenyl-phenylether	10	ug/L	660	ug/kg
	Phenacetin	20	ug/L	1300	ug/kg
	Hexachlorobenzene	10	ug/L	660	ug/kg
	Atrazine	10	ug/L	660	ug/kg
	4-Amino-biphenyl	20	ug/L	1300	ug/kg
	Pentachlorophenol	50	ug/L	3300	ug/kg
	Pronamide	10	ug/L	660	ug/kg
	Pentachloronitrobenzene	20	ug/L	1300	ug/kg
	Phenanthrene	10	ug/L	660	ug/kg
	Anthracene	10	ug/L	660	ug/kg
	Carbazole	10	ug/L	660	ug/kg
	Di-n-butylphthalate	10	ug/L	660	ug/kg
	Fluoranthene	10	ug/L	660	ug/kg
	Benzidine	10	ug/L	660	ug/kg
	Pyrene	10	ug/L	660	ug/kg
	p-Dimethylaminoazobenzene	10	ug/L	660	ug/kg
	Butylbenzylphthalate	10	ug/L	660	ug/kg
	Benzo[a]anthracene	10	ug/L	660	ug/kg
	3,3'-Dichlorobenzidine	20	ug/L	1300	ug/kg
	Chrysene	10	ug/L	660	ug/kg
	Bis(2-ethylhexyl)phthalate	10	ug/L	660	ug/kg
	Di-n-octylphthalate	10	ug/L	660	ug/kg
	Benzo[b]fluoranthene	10	ug/L	660	ug/kg

Modified 1/24/2006

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Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

	Benzo[k]fluoranthene	10	ug/L	660	ug/kg
	7,12- Dimethylbenz(a)anthracene	10	ug/L	660	ug/kg
	Benzo[a]pyrene	10	ug/L	660	ug/kg
	3-Methylchloranthrene	10	ug/L	660	ug/kg
	Dibenz(a,j)acridine	10	ug/L	660	ug/kg
	Indeno[1,2,3-cd]pyrene	10	ug/L	660	ug/kg
	Dibenz[a,h]anthracene	10	ug/L	660	ug/kg
	Benzo[g,h,i]perylene	10	ug/L	660	ug/kg
	Alpha-BHC	10	ug/L	660	ug/kg
SVOCs EPA 625/SW-846 Method 8270C	Gamma-BHC	10	ug/L	660	ug/kg
	Beta-BHC	10	ug/L	660	ug/kg
	Delta-BHC	10	ug/L	660	ug/kg
	Heptachlor	10	ug/L	660	ug/kg
	Aldrin	10	ug/L	660	ug/kg
	Heptachlor epoxide	25	ug/L	1800	ug/kg
	Endosulfan 1	50	ug/L	3300	ug/kg
	Dieldrin	10	ug/L	660	ug/kg
	p,p'-DDE	10	ug/L	660	ug/kg
	Endrin	20	ug/L	1300	ug/kg
	Endosulfan 2	50	ug/L	3300	ug/kg
	P,p'-DDD	10	ug/L	660	ug/kg
	Endrin aldehyde	10	ug/L	660	ug/kg
	Endosulfan sulfate	25	ug/L	1800	ug/kg
	p,p'-DDT	10	ug/L	660	ug/kg

Table A.7.4.2 Acceptance Criteria for Method EPA 625 and SW-846/8270C

12-month upper and lower control limits (1/01/04 to 12/3/04)

Method 8270C	Analyte	Accuracy Aqueous (%R)	Precision Aqueous (RPD)	Accuracy Soil (%R)	Precision Soil (RPD)	Accuracy Waste (%R)	Precision Waste (RPD)
SS	2-Fluorophenol	15-90	≤ 30%	22-102	≤ 40%	24-95	≤ 50%
	Phenol-d5	16-101	≤ 30%	24-108	≤ 40%	46-94	≤ 50%
	Nitrobenzene-d5	31-95	≤ 30%	23-106	≤ 40%	50-91	≤ 50%
	2-Fluorobiphenyl	29-97	≤ 30%	33-114	≤ 40%	53-99	≤ 50%
	2,4,6-Tribromophenol	25-110	≤ 30%	31-112	≤ 40%	18-104	≤ 50%
	Terphenyl-d14	10-116	≤ 30%	56-114	≤ 40%	64-104	≤ 50%
MS	Phenol	10-111	≤ 30%	14-112	≤ 40%	32-120	≤ 50%
	2-Chlorophenol	11-100	≤ 30%	10-118	≤ 40%	44-96	≤ 50%
	1,4-Dichlorobenzene	16-80	≤ 30%	10-104	≤ 40%	40-87	≤ 50%
	n-Nitroso-di-n-propylamine	25-110	≤ 30%	19-131	≤ 40%	43-127	≤ 50%
	1,2,4-Trichlorobenzene	26-83	≤ 30%	10-118	≤ 40%	51-91	≤ 50%
	4-Chloro-3-methylphenol	20-111	≤ 30%	21-120	≤ 40%	49-104	≤ 50%
	Acenaphthene	24-100	≤ 30%	24-115	≤ 40%	49-101	≤ 50%

Modified 1/24/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

	4-Nitrophenol	10-122	≤ 30%	10-133	≤ 40%	13-132	≤ 50%
	2,4-Dinitrotoluene	30-117	≤ 30%	32-120	≤ 40%	53-102	≤ 50%
	Pentachlorophenol	10-122	≤ 30%	10-130	≤ 40%	21-100	≤ 50%
	Pyrene	21-126	≤ 30%	34-141	≤ 40%	44-115	≤ 50%
LCS	Phenol	44-83	≤ 30%	39-86	≤ 40%	42-90	≤ 50%
	2-Chlorophenol	49-82	≤ 30%	40-85	≤ 40%	43-89	≤ 50%
	1,4-Dichlorobenzene	42-68	≤ 30%	34-75	≤ 40%	35-85	≤ 50%
	n-Nitroso-di-n-propylamine	57-95	≤ 30%	47-97	≤ 40%	57-97	≤ 50%
	1,2,4-Trichlorobenzene	46-76	≤ 30%	40-87	≤ 40%	40-93	≤ 50%
	4-Chloro-3-methylphenol	50-98	≤ 30%	42-95	≤ 40%	52-90	≤ 50%
	Acenaphthene	53-95	≤ 30%	49-90	≤ 40%	42-96	≤ 50%
	4-Nitrophenol	38-107	≤ 30%	49-89	≤ 40%	43-102	≤ 50%
	2,4-Dinitrotoluene	58-105	≤ 30%	58-91	≤ 40%	45-103	≤ 50%
	Pentachlorophenol	31-82	≤ 30%	31-93	≤ 40%	22-107	≤ 50%
	Pyrene	61-122	≤ 30%	50-106	≤ 40%	33-114	≤ 50%

Table A.7.4.3 Summary of Calibration and QC Procedures for Method EPA 625 and 8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA 625/ SW846 - 8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ₂ ≥ 0.050 ; %RSD for CCCs ≤ 30% drift, should be less than 15% for all other analytes. If CORR used, > 0.990.	Correct problem then repeat initial calibration	
		Second-source calibration verification	Once per five-point initial calibration	Analytes within ±30% of expected value	Correct problem then repeat initial calibration	
		Calibration verification	Daily, before sample analysis, every 12 hours of analysis time	SPCCs average RF ₂ ≥ 0.050; and CCCs ≤ 20% drift; and all calibration analytes within ±20% of expected value	Correct problem (usually clipping column and changing insert) then repeat calibration ver. If fails, recalibrate.	
		Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four replicate analysis of a QC check sample	Once per analyst	QC limits set for LCS must be used and all tested analytes must fall within these limits for acceptable results.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify with DFTPP tune check again.	

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		Internal Standards	Immediately after or during data acquisition of calibration check standard	Retention time \pm 30 seconds: EICP area within -50% and + 100% of initial calibration midpoint	Inspect mass spectrometry or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	
EPA 625/ SW846 - 8270C	Semi-Volatile Organics	Method Blank	One per analytical batch	No analytes detected >RL	Ensure no contamination then reanalyze method blank and all samples processed with the contaminated blank	If unable to re-analyze, flag with a "B"
		LCS/LCSD for selected analytes	One LCS/LCSD per analytical batch	QC acceptance criteria established by control charts semi-annually. Precision depends on matrix, 30, 40, 50% for waters, soils, wastes	Correct problem then reanalyze the LCS and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria set by control charts semi-annually	Reanalyze sample. If still low, re-extract and reanalyze. If still low flag data.	If determined that sample matrix is interfering, flag as estimated values.
		Matrix Spike and Matrix Spike Duplicate	One MS&MSD per analytical batch	QC acceptance criteria set by control charts semi-annually	Reanalyze sample failed, flag data.	Matrix inference, flag as estimate.
		MDL Study	Once per year	Detection limits established shall be <the RLs in SOP	If MDL recoveries are less than 70%, repeat at a higher concentration.	
		Estimated amount for analytes above the 5-pt calibration curve	none	All analytes < 160ug/L waters < 5300ug/Kg soils < 160mg/Kg wastes.	Sample must be diluted and reanalyzed.	Apply E to all analytes out of range.

Modified 1/24/2006

Georgia Department of Natural Resources

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A.7.2 EPA Method 525.2 - Organic Compounds in D.W. by Liquid-Solid Extraction and GC/MS**A.7.2.1 Scope and Application**

A.7.2.1.1 This is a general purpose method for the identification and simultaneous measurement of organic compounds in finished drinking water, source water, or drinking water in any stage of treatment. The method is applicable to a wide range of organic compounds that are partitioned from the water sample onto C18 organic phase chemically bonded to disks and sufficiently volatile and thermally stable for gas chromatography. The target list includes the following:

Water samples for 525 semivolatile organic compounds are collected in a 1 liter amber glass bottle containing 50 mg of sodium sulfite to dechlorinate the sample. 5 ml of 1:1 HCl is then poured into the bottle as a preservative. All sample bottles must be cooled to 4°C. Two 1 liter sample bottles are required for each sample. Holding time for preserved samples is 14 days until extraction and then analysis within 30 days from collection.

<u>Compound</u>	<u>CAS Number</u>
Alachlor	151972-60-8
Aldrin	309-00-2
Atrazine	1912-24-9
Benzo(a)pyrene	50-32-8
Butachlor	23184-66-9
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
trans-Nonachlor	39765-80-5
Dieldrin	60-57-1
Di(2-ethylhexyl)adipate	103-23-1
Di(2-ethylhexyl)phthalate	117-81-7
Endrin	72-20-8
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorbenzene	118-74-1
Hexachlorocyclopentadiene	77-47-4
gamma-BHC(Lindane)	58-89-9
Methoxychlor	72-43-5
Metolachlor	51218-45-2
Metribuzin	21087-64-9
Pentachlorophenol	87-86-5
ProPachlor	1918-16-7
Simazine	122-34-9

A.7.2.2 Calibration and Calculation

Modified 1/26/2006

Georgia Department of Natural Resources

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A.7.2.2.1 Initial DFTPP Tune Verification of mass spectrometer**A.7.2.2.2 Calibration Curve**

A 6 point calibration is performed for initial calibration, the calibration is performed to determine the response factor using NIST traceable standards and set retention times. An alternate source standard is used to verify the initial calibration standard lot. If that lot is validated with the alternate source, the rest of that lot is deemed valid for future use for calibration. Software must recognize a peak in the retention time window of 99% of the target compounds and list it as detected in the data report. Problem compounds may use a 5 point curve at higher concentrations.

A.7.2.2.3 Calibration Standards

The 525 regulated calibration curve consists of 6 points and the calibration range is from 0.1 to 12 ug/L depending upon the reporting limit of the target compound, reference table A.7.2.1 for reporting limits. The calibration curve is an average response factor curve fit and should result in a RSD of less than 30% between the calibration levels. A linear least squares regression calibration fit have a correlation coefficient ≥ 0.995 .

A.7.2.2.3.1 Record Keeping

Documentation of instrument calibrations are reviewed for adherence to quality control criteria and then stored in calibration curve records file.

A.7.2.3.2 Daily calibration Verification and Continuing Calibration

A 6 PPB regulated standard ensures the instrument's target compounds' retention times and quantitation parameters meet method performance criteria. For a 12 hour period after the DFTPP has passed and prior to sample analysis a one point CCV is performed. The daily CCV instrument calibration check cannot exceed 30% RSD versus the initial calibration. If the CCV for a compound is lower than method performance criteria the instrument must be recalibrated before any samples are analyzed. If the CCV for a compound is higher than method performance analysis may proceed, however any compound that failed the criteria and is detected must be reanalyzed after the instrument is recalibrated. Software must recognize a peak in the retention time window of 99% of the target compounds and list it as detected in the data report.

Determine that the mass spectrometer is tuned for DFTPP by injecting 1 uL of the performance check solution at a concentration of 5 ug/L and verify that it meets the criteria described below as defined by 525.2. The entire peak may be averaged and used for DFTPP tune verification. Should the tune parameters not be met, maintenance must be performed and the DFTPP re-injected before any more injections are made.

Mass (M/z)	Relative Abundance Criteria
51	10-80% of mass 198
68	<2% of mass 69

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

70	<2% of mass 69
127	10-80% of mass 198
197	<2% of mass 198
198	>50% of mass 442
199	5-9% of mass 198
275	10-60% of mass 198
365	>1% of mass 198
441	Present and < mass 443
442	>50% of mass 198
443	15-24% of mass 442

Upon completion of the DFTPP tune, the parameters are saved to the DFTPPMMDD.U tune file and printout saved in a tune folder.

At the time of injection the performance check solution, the percent breakdown of both p,p' DDT and Endrin must be calculated and verified to be less than 20%. The formulas for calculating percent breakdown are as follows, using TIC area:

% p,p'-DDT Breakdown=

$$\frac{\text{Area 4,4'-DDE} + \text{Area 4,4'-DDD}}{[\text{Area DDE} + \text{DDD}] + \text{Area 4,4' DDT}} * 100$$

% Endrin Breakdown=

$$\frac{\text{Area Endrin aldehyde} + \text{Area Endrin ketone}}{[\text{Area EA} + \text{EK}] + \text{Area Endrin}} * 100$$

If breakdown of either endrin or p,p' DDT exceed 20%, maintenance on the GC inlet must be performed and the performance check re-injected before any other samples may be analyzed.

After the initial verification of the DFTPP tune, the performance check solution must be re-injected every 12 hours to verify consistency in conditions.

A.7.2.3 Relative Response Factor (RRF)

Relative Response Factor: Calculate the relative response factors (RRF) for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

$$\text{Equation A.7.2.1} \quad RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where

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David Jones, Laboratory Director

 RRF = Relative response factor A_x = Area of the primary ion for the compound to be measured A_{is} = Area of the primary ion for the internal standard C_{is} = Concentration of internal standard spiking mixture, ppb C_x = Concentration of the compound in the calibration standard, ppb

NOTE: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume of field and QC sample introduced into the GC is the same for each analysis. C_{is} and C_x must be in the same units.

A.7.2.4 Mean Relative Response Factor (\overline{RRF})

Mean Relative Response Factor: Calculate the mean RRF (\overline{RRF}) for each compound by averaging the values obtained at the five concentrations using the following equation:

Equation A.7.2.2
$$\overline{RRF} = \sum_{i=1}^n \frac{x_i}{n}$$

where:

 \overline{RRF} = Mean relative response factor x_i = RRF of the compound n = Number of valuesA.7.2.5 Percent Relative Standard Deviation (% RSD)

Using the $RRFs$ from the initial calibration, calculate the % RSD for all target compounds using the following equations:

a
$$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100$$
 nd

Equation A.7.2.3

Equation A.7.2.3
$$SD_{RRF} = \sqrt{\sum_{i=1}^n \frac{(RRF_i - \overline{RRF})^2}{n-1}}$$

where:

 SD_{RRF} = Standard deviation of initial response factors (per compound)

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

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$\frac{RRF_i}{RRF}$ = Relative response factor at a concentration level
 $\frac{RRF_i}{RRF}$ = Mean of initial relative response factors (per compound).
 n = Number of values

A.7.2.6 Relative Retention Times (*RRT*)

A compound's target ion is extracted from a window that is within ± 5 seconds of the expected retention time. The retention time for each internal standard must be within "0.33 minutes of the retention time of the internal standard in the most recent valid calibration. Calculate the *RRT*s for each target compound over the initial calibration range using the following equation

$$\text{Equation A.7.2.5} \quad RRT = \frac{RT_c}{RT_{is}}$$

where:

RT_c = Retention time of the target compound
 RT_{is} = Retention time of the internal standard.

A.7.2.7 Percent Difference (%D):

Calculate the percent difference in the RRF of the daily RRF (24-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

$$\text{Equation A.7.2.6} \quad \%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

where:

RRF_c = RRF of the compound in the continuing calibration standard
 $\overline{RRF_i}$ = Mean RRF of the compound in the most recent initial calibration.

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David Jones, Laboratory Director

Equation A.7.2.7
$$C_x = \frac{A_x C_{is} DF}{A_{is} RRF}$$

A.7.2.8 Sample Concentration Calculation.

where:

- C_x = Compound concentration, ppb
- A_x = Area of the characteristic ion for the compound to be measured
- A_{is} = Area of the characteristic ion for the specific internal standard
- C_{is} = Concentration of the internal standard spiking mixture, ppb
- RRF = Relative response factor from the analysis of the continuing calibration standard or the mid level standard of the initial calibration
- DF = Dilution factor calculated as described in section 2. If no dilution is performed, $DF = 1$

NOTE: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume of field and QC sample introduced into the trap is the same for each analysis.

A.7.2.9 Linear Calibration using Least Squares Regression

Linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for the 6 points are done using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.00 is a perfect fit, 525.2 requires a fit of 0.995 or better.

A.7.2.2.10 Sample Concentration Calculation

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.7.2.8 $y = a x + b$

- y = Instrument response
- a = Slope of the line
- x = Concentration of Sample
- b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

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Environmental Protection Division

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$$x = \frac{y - b}{a}$$

NOTE: The equation above is valid under the condition that the volume of 1000ml is the same from run to run, and that the volume of field and QC sample extracted are the same from run to run.

Table A.7.2.1 R.L. for EPA Method 525.2

Parameter/Method	Analyte	Matrix (Water)	
		RL	Unit
Semivolatiles /525.2	Alachlor	2.0	ug/L
	Aldrin	2.0	ug/L
	Atrazine	2.0	ug/L
	Benzo(a)pyrene	0.1	ug/L
	Butachlor	2.0	ug/L
	Alpha-Chlordane	1.0	ug/L
	gamma-Chlordane	1.0	ug/L
	Dieldrin	2.0	ug/L
	Di(2-ethylhexyl)adipate	2.0	ug/L
	Di(2-ethylhexyl)phthalate	4.0	ug/L
	Endrin	1.0	ug/L
	Heptachlor	0.1	ug/L
	Heptachlor epoxide	0.1	ug/L
	Hexachlorobenzene	1.0	ug/L
	Hexachlorocyclopentadiene	2.0	ug/L
	gamma BHC(Lindane)	0.1	ug/L
	Methoxychlor	2.0	ug/L
	Metolachlor	2.0	ug/L
	Metribuzin	2.0	ug/L
	Pentachlorophenol	1.0	ug/L
	Propachlor	2.0	Ug/L
	Simazine	1.0	Ug/L
	trans-Nonachlor	1.0	Ug/L

Table A.7.2.2 Acceptance Criteria for Method EPA 525.2

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)
Semivolatiles /525.2	Alachlor	70-130	30
	Aldrin	70-130	30
	Atrazine	70-130	30

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

	Benzo(a)pyrene	70-130	30
	Butachlor	70-130	30
	alpha-Chlordane	70-130	30
	gamma-Chlordane	70-130	30
	Dieldrin	70-130	30
	Di(2-ethylhexyl)adipate	70-130	30
	Di(2-ethylhexyl)phthalate	70-130	30
Semivolatiles /525.2	Endrin	70-130	30
	Heptachlor	70-130	30
	Heptachlor Epoxide	70-130	30
	Hexachlorobenzene	70-130	30
	Hexachlorocyclopentadiene	70-130	30
	gamma-BHC(Lindane)	70-130	30
	Methoxychlor	70-130	30
	Metolachlor	70-130	30
	Metribuzin	70-130	30
	Pentachlorophenol	70-130	30
	Propachlor	70-130	30
	Simazine	70-130	30
	trans-Nonachlor	70-130	30

Table A.7.2.3 Summary of Calibration and QC Procedures for Method 525.2

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
525.2	SVOCs	Six -point initial calibration for all analytes	Initial calibration prior to sample analysis	%RSD for all calibration analytes #<30%. A 5 point curve may be used for problem compounds.	Correct problem then repeat initial calibration	
		Second-source calibration verification	Once per six-point initial calibration using standard lot number. Lot is deemed valid	95 % of all analytes within $\pm 30\%$ of expected value.	Correct problem then repeat initial calibration	
		Calibration verification	Daily, before sample analysis, valid for 12 hours of analysis time for one QC batch of samples.	All calibration analytes within "30% RSD. Linear fit calib. conc. at " $\pm 30\%$ of true value. Internal Std within 50% area of curve.	If <30% negative RSD correct the problem. If > 30% positive RSD and analyte not detected in batch, note in corrective action.	
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Twice a year per analyst	All targets must meet at or below 30% RPD and >70% Recovery.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Check of mass spectral ion intensities using DFTPP	Daily, before sample analysis, every 12 hours of analysis shift time.	Refer to criteria listed in the 8.1.1.	Retune instrument and verify DFTPP again	
		MDL study	Once per year	MDL must be less than reporting limits	Check calculations, redo MDL analysis and redo results.	

Modified 1/26/2006

Georgia Department of Natural Resources

Environmental Protection Division

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David Jones, Laboratory Director

525.2	SVOCs	Internal Standard	Immediately after or during data acquisition of calibration check standard, and on every sample run.	Internal standard retention times should not drift by more than 30 seconds from one calibration check to the next (every 12 hours). Recovery for the internal standards must be $\geq 70\%$ based on the fortification standards.	Inspect mass spectrometry or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning, and need to build new calibration curve if needed.	
		Method Blank	One per analytical batch before any samples are run for the batch period.	No analytes detected >RL	Correct problem then reprep and analyze method blank	If unable to re-analyze, flag with a "B"
		LCS and LCSD	One LCS and LCS duplicate per analytical batch.	Refer to table 2	If not detected in batch any analytes that do not meet criteria noted in corrective action logbook and in sample comment field.	If unable to re-analyze, flag with "J".
		Surrogate spike	Every sample, spiked sample, standard, and method blank.	Range from 3.5 ug/L to 6.5 ug/L.	Correct problem then reanalyze sample.	
		Matrix spike/ Spike Duplicate	Once per batch	Refer to table 2.	If LCS is in control comment possible matrix problem; Corrective action	
		Retention time for compounds	Per analysis	Within 5 sec of daily CCV	Rerun samples	

Georgia Department of Natural Resources

Environmental Protection Division

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David Jones, Laboratory Director

A.7.1 EPA Method 524.2 - Purgeable Organics in D.W. by Capillary GC/MS

Measurement of purgeable organic compounds in water by capillary column gas chromatography/mass spectrometry

A.7.1.1 Scope and Application

This is a general-purpose method for the identification and simultaneous measurement of purgeable volatile organic compounds in surface water, ground water, and drinking water in any stage of treatment. The method is applicable to a wide range of organic compounds that have sufficiently high volatility and low water solubility to be removed from water samples with purge and trap procedures.

Water samples for volatile organic compounds are collected in a 125 ml glass bottle containing 75 mg of ascorbic acid to dechlorinate the sample then poured into two 40 ml glass sample vials containing 0.5ml 1:1 HCl as a preservative. All samples must be cooled to 4°C. Two 40 ml sample vials are required for each sample. Holding time for preserved samples is 14 days.

As THMS are not regulated by Method 524.2, the four trihalomethane disinfection by-products analyzed are reported for informational purposes only, due to the addition of HCl as a preservative in the sampling process. High levels of THMs encountered that fall above the calibration curve will be flagged as "E" (estimated amount) Also, results for any THM with failing QC, either in the calibration curve or the CCV, LCS/LCSD will be flagged as "E" (estimated amount).

Dichlorodifluoromethane	1,2-Dichloroethane	Bromoform
Chloroethene	Trichloroethene	Isopropylbenzene
Chloromethane	1,2-Dichloropropane	1,1,2,2-Tetrachloroethane
Bromomethane	Dibromomethane	Bromobenzene
Chloroethane	Bromodichloromethane	1,2,3-Trichloropropane
Trichlorofluoromethane	cis-1,3-Dichloropropene	2-Chlorotoluene
1,1-Dichloroethene	Toluene	4-Chlorotoluene
Methylene chloride	trans-1,3-Dichloropropene	tert-Butylbenzene
trans-1,2-Dichloroethene	1,1,2-Trichloroethane	1,3,5-Trimethylbenzene
1,1-Dichloroethane	Tetrachloroethene	1,2,4-Trimethylbenzene
2,2-Dichloropropane	1,3-Dichloropropane	4-Isopropyltoluene
cis-1,2-Dichloroethene	Dibromochloromethane	1,2-Dichlorobenzene
Bromochloromethane	1,2-Dibromoethane	1,3-Dichlorobenzene
Chloroform	Chlorobenzene	1,4-Dichlorobenzene
1,1,1-Trichloroethane	1,1,1,2-Tetrachloroethane	1,2-Dibromo-3-chloropropane
1,1-Dichloropropene	Ethylbenzene	1,2,4-Trichlorobenzene
Carbon tetrachloride	o,m& p-xylene	Hexachlorobutadiene
Benzene	Styrene	Naphthalene

Modified 1/26/2006

Georgia Department of Natural Resources

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1,2,3-Trichlorobenzene
2-Methoxy-2-methyl-propane

n-Propylbenzene
sec-Butylbenzene

n-Butylbenzene

A.7.1.1.1 Application

Volatile organic compounds and surrogates with low water solubility are extracted (purged) from the sample matrix by bubbling an inert gas through the aqueous sample. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete the sorbent tube is heated and back flushed with helium to desorb the trapped sample components into a capillary gas chromatography column interfaced to a mass spectrometer and identified by comparing their measured mass spectra and retention times to reference spectra and retention times in a data base. The concentration of each identified component is measured by relating the MS response of the quantitation ion produced by that compound to the MS response of the quantitation ion produced by a compound that is used as an internal standard.

A.7.1.2**Calibrations and Calculations****A.7.1.2.1 BFB Tuning Criteria**

GC/MS system calibration and sample analysis cannot begin until the required BFB key ions and ion abundance criteria are met, valid for 12 hours.

<u>Mass</u>	<u>Ion Abundance Criteria</u>
50	15.0 to 40.0 percent of m/e 95
75	30.0 to 80.0 percent of m/e 95
95	base peak, 100 percent relative abundance
96	5.0 to 9.0 percent of m/e 95
173	less than 2.0 percent of m/e 174
174	>50.0 percent of m/e 95
175	5.0 to 9.0 percent of m/e 174
176	>95.0 but < 101.0 percent of m/e 174
177	5.0 to 9.0 percent of m/e 176

A.7.1.2.2 Calibration Curve

A 4 point calibration is performed in initial calibration with the lowest being below the method detection limit to determine response factors using NIST traceable standards and to set retention times. Per method, a 3 point curve may also be with a concentration range factor of 20. An alternate second source is used to validate the initial calibration standard lot and if valid, all the standards of that lot number are considered valid. Column performance should be demonstrated by symmetrical peak shape and minimal tailing. If the chromatography is poor (i.e. unusually broad peaks, excessive tailing), corrective action must be taken before samples can be analyzed. The system software must be able to autofind 99% of the target compounds using mass spectra and retention time comparisons without manual integration. If fewer than 99% are properly identified corrective action must be taken before samples can be analyzed

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David Jones, Laboratory Director

A.7.1.2.3 Calibration Standards

The 4 point calibration curve consists of the calibration standards at concentrations of 0.4, 2, 5, and 10 ug/L. Internal standard, fluorobenzene, is used in calibration, quality control, and sample analysis. The calibration curve is an "average of response factor" curve fit and should result in a percent relative standard deviation less than 20% between the calibration levels. A linear least squares regression calibration fit must fit at ≥ 0.995 . The method detection limit for 524.2 is 0.5 ug/L.

A.7.1.2.3.1 Record Keeping

Documentation of instrument calibration are reviewed for adherence to quality criteria and then stored in the calibration curve records.

A.7.1.2.3.2 Daily Calibration Verification and Continuing Calibration

A 5 ppb calibration standard ensures the instrument's target compounds retention times and quantitation parameters meet method performance criteria. For any 12-hour period, prior to sample analysis, a one-point daily continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed 30% of the initial calibration. If the continuing calibration is lower than method performance criteria, the instrument must be recalibrated before any samples are analyzed. If the continuing calibration accuracy exceeds method performance criteria, analysis may proceed, however any compound that failed the criteria and is detected must be reanalyzed after the instrument is recalibrated. Column performance should be demonstrated by symmetrical peak shape and minimal tailing. If the chromatography is poor (i.e. unusually broad peaks, excessive tailing), corrective action must be taken before samples can be analyzed. The system software must be able to autofind 99% of the target compounds using mass spectra and retention time comparisons without manual integration. If fewer than 99% are properly identified corrective action must be taken before samples can be analyzed

A.7.1.2.4 Relative Response Factor (RRF)

Relative Response Factor: Calculate the relative response factors (*RRF*) for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

Equation A.7.1.1
$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where

RRF = Relative response factor

A_x = Area of the primary ion for the compound to be measured

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

- A_{is} = Area of the primary ion for the internal standard
 C_{is} = Concentration of internal standard spiking mixture, ppb
 C_x = Concentration of the compound in the calibration standard, ppbv

NOTE: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume of field and QC sample introduced into the GC is the same for each analysis. C_{is} and C_x must be in the same units.

A.7.1.2.5 Mean Relative Response Factor (\overline{RRF})

Mean Relative Response Factor: Calculate the mean RRF (\overline{RRF}) for each compound by averaging the values obtained at the five concentrations using the following equation:

Equation A.7.1.2

$$\overline{RRF} = \sum_{i=1}^n \frac{x_i}{n}$$

where:

- \overline{RRF} = Mean relative response factor
 x_i = RRF of the compound
 n = Number of values

A.7.1.2.6 Percent Relative Standard Deviation (%RSD)

Using the RRFs from the initial calibration, calculate the %RSD for all target compounds using the following equations:

a

$$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100 \quad \text{nd}$$

Equation A.7.1.3

$$SD_{RRF} = \sqrt{\sum_{i=1}^n \frac{(RRF_i - \overline{RRF})^2}{n-1}}$$

Equation A.7.1.3

where:

- SD_{RRF} = Standard deviation of initial response factors (per compound)
 RRF_i = Relative response factor at a concentration level
 \overline{RRF} = Mean of initial relative response factors (per compound).

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

n = Number of values

A.7.1.2.7 Relative Retention Times (*RRT*)

The retention time for each internal standard must be within "0.50 minutes of the retention time of the internal standard in the most recent valid calibration. Calculate the *RRT*s for each target compound over the initial calibration range using the following equation

$$\text{Equation A.7.1.5} \quad RRT = \frac{RT_c}{RT_{IS}}$$

where:

Rt_c = Retention time of the target compound

RT_{IS} = Retention time of the internal standard.

A compound's target ion is extracted from a window that is within ± 0.1 minutes of the expected retention time.

A.7.1.2.8 Percent Difference (%D):

Calculate the percent difference in the RRF of the daily RRF (12-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

$$\text{Equation A.7.1.6} \quad \%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

where:

RRF_c = RRF of the compound in the continuing calibration standard

$\overline{RRF_i}$ = Mean RRF of the compound in the most recent initial calibration.

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

Equation A.7.1.7
$$C_x = \frac{A_x C_{is} DF}{A_{is} RRF}$$

A.7.1.2.9 Sample Concentration Calculation.

where:

- C_x = Compound concentration, ppb
- A_x = Area of the characteristic ion for the compound to be measured
- A_{is} = Area of the characteristic ion for the specific internal standard
- C_{is} = Concentration of the internal standard spiking mixture, ppb
- RRF = Relative response factor from the analysis of the continuing calibration standard or the mid level standard of the initial calibration
- DF = Dilution factor calculated as described in section 2. If no dilution is performed, $DF = 1$

NOTE: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume of field and QC sample introduced into the trap is the same for each analysis.

A.7.1.2.10 Linear Calibration using Least Squares Regression

Linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for the 4 points is done using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.00 is a perfect fit, 524.2 requires a fit of 0.995 or better.

A.7.1.2.11 Sample Concentration Calculation

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.7.1.8 $y = a x + b$

- y = Instrument response
- a = Slope of the line
- x = Concentration of Sample
- b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

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Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

$$x = \frac{y - b}{a}$$

NOTE: The equation above is valid under the condition that the volume of 5 ml is the same from run to run, and that the volume of field and QC sample introduced into the trap is the same, 5 ml, for each analysis.

Table A.7.1.1 R.L. for EPA Method 524.2

Parameter/Method	Analyte	Matrix (Water)	
		MDL	Unit
Volatile Organics 524.2	Dichlorodifluoromethane	0.50	ug/L
	Chloroethene	0.50	ug/L
	Chloromethane	0.50	ug/L
	Bromomethane	0.50	ug/L
	Chloroethane	0.50	ug/L
	Trichlorofluoromethane	0.50	ug/L
	1,1-Dichloroethene	0.50	ug/L
	Methylene chloride	0.50	ug/L
	trans-1,2-Dichloroethene	0.50	ug/L
	1,1-Dichloroethane	0.50	ug/L
	2,2-Dichloropropane	0.50	ug/L
	cis-1,2-Dichloroethene	0.50	ug/L
	Bromochloromethane	0.50	ug/L
	Chloroform	0.50	ug/L
	1,1,1-Trichloroethane	0.50	ug/L
	1,1-Dichloropropene	0.50	ug/L
	Carbon tetrachloride	0.50	ug/L
	Benzene	0.50	ug/L
	1,2-Dichloroethane	0.50	ug/L
	Trichloroethene	0.50	ug/L
	1,2-Dichloropropane	0.50	ug/L
	Dibromomethane	0.50	ug/L
	Bromodichloromethane	0.50	ug/L
	cis-1,3-Dichloropropene	0.50	ug/L
	Toluene	0.50	ug/L
	trans-1,3-Dichloropropene	0.50	ug/L
	1,1,2-Trichloroethane	0.50	ug/L
	Tetrachloroethene	0.50	ug/L
	1,3-Dichloropropane	0.50	ug/L
	Dibromochloromethane	0.50	ug/L
	1,2-Dibromoethane	0.50	ug/L
	Chlorobenzene	0.50	ug/L

Modified 1/26/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

	1,1,1,2-Tetrachloroethane	0.50	ug/L
	Ethylbenzene	0.50	ug/L
	o,m& p-xylene	0.50	ug/L
	Styrene	0.50	ug/L
	Bromoform	0.50	ug/L
	Isopropylbenzene	0.50	ug/L
	1,1,2,2-Tetrachloroethane	0.50	ug/L
	Bromobenzene	0.50	ug/L
	1,2,3-Trichloropropane	0.50	ug/L
	2-Chlorotoluene	0.50	ug/L
Volatile Organics 524.2	4-Chlorotoluene	0.50	ug/L
	tert-Butylbenzene	0.50	ug/L
	1,3,5-Trimethylbenzene	0.50	ug/L
	1,2,4-Trimethylbenzene	0.50	ug/L
	4-Isopropyltoluene	0.50	ug/L
	1,2-Dichlorobenzene	0.50	ug/L
	1,3-Dichlorobenzene	0.50	ug/L
	1,4-Dichlorobenzene	0.50	ug/L
	1,2-Dibromo-3-chloropropane	0.50	ug/L
	1,2,4-Trichlorobenzene	0.50	ug/L
	Hexachlorobutadiene	0.50	ug/L
	Naphthalene	0.50	ug/L
	1,2,3-Trichlorobenzene	0.50	ug/L
	2-Methoxy-2-methyl-propane	0.50	ug/L
	n-Propylbenzene	0.50	ug/L
	sec-Butylbenzene	0.50	ug/L
	n-Butylbenzene	0.50	ug/L

Table A.7.1.2 Acceptance Criteria for Method EPA 524.2

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)
Volatile Organics 524.2	Dichlorodifluoromethane	70-130	20
	Chloroethene	70-130	20
	Chloromethane	70-130	20
	Bromomethane	70-130	20
	Chloroethane	70-130	20
	Trichlorofluoromethane	70-130	20
	1,1-Dichloroethene	70-130	20
	Methylene chloride	70-130	20
	trans-1,2-Dichloroethene	70-130	20
	1,1-Dichloroethane	70-130	20
	2,2-Dichloropropane	70-130	20
	cis-1,2-Dichloroethene	70-130	20
	Bromochloromethane	70-130	20

Modified 1/26/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

	Chloroform	70-130	20
	1,1,1-Trichloroethane	70-130	20
	1,1-Dichloropropene	70-130	20
	Carbon tetrachloride	70-130	20
	Benzene	70-130	20
	1,2-Dichloroethane	70-130	20
	Trichloroethene	70-130	20
Volatile Organics 524.2	1,2-Dichloropropane	70-130	20
	Dibromomethane	70-130	20
	Bromodichloromethane	70-130	20
	cis-1,3-Dichloropropene	70-130	20
	Toluene	70-130	20
	trans-1,3-Dichloropropene	70-130	20
	1,1,2-Trichloroethane	70-130	20
	Tetrachloroethene	70-130	20
	1,3-Dichloropropane	70-130	20
	Dibromochloromethane	70-130	20
	1,2-Dibromoethane	70-130	20
	Chlorobenzene	70-130	20
	1,1,1,2-Tetrachloroethane	70-130	20
	Ethylbenzene	70-130	20
	o,m& p-xylene	70-130	20
	Styrene	70-130	20
	Bromoform	70-130	20
	Isopropylbenzene	70-130	20
	1,1,2,2-Tetrachloroethane	70-130	20
	Bromobenzene	70-130	20
	1,2,3-Trichloropropane	70-130	20
	2-Chlorotoluene	70-130	20
	4-Chlorotoluene	70-130	20
	tert-Butylbenzene	70-130	20
	1,3,5-Trimethylbenzene	70-130	20
	1,2,4-Trimethylbenzene	70-130	20
	4-Isopropyltoluene	70-130	20
	1,2-Dichlorobenzene	70-130	20
	1,3-Dichlorobenzene	70-130	20
	1,4-Dichlorobenzene	70-130	20
	1,2-Dibromo-3-chloropropane	70-130	20
	1,2,4-Trichlorobenzene	70-130	20
	Hexachlorobutadiene	70-130	20
	Naphthalene	70-130	20
	1,2,3-Trichlorobenzene	70-130	20
	2-Methoxy-2-methyl-propane	70-130	20
	n-Propylbenzene	70-130	20
	sec-Butylbenzene	70-130	20
	n-Butylbenzene	70-130	20

Modified 1/26/2006

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Modified 1/26/2006

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Table A.7.1.3 Summary of Calibration and QC Procedures for Method 524.2

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
524.2	Volatile Organics	4 -point initial calibration for all analytes. Method minimum is three point to calibrate a range of 20.	Initial calibration prior to sample analysis	% RSD for all calibration analytes <20%. Linear ≥ 0.995 THM's may be excluded	Correct problem then repeat initial calibration	If THM is excluded flag as estimated
		Second-source calibration verification	Once per 4-point initial calibration of that standard lot number. If valid, lot is valid	95 % of all analytes within "30% RSD of expected value.	Correct problem then repeat initial calibration	
		Calibration verification	Daily, before sample analysis	All calibration analytes within "30% RSD. Linear fit calib. conc. within " 30%. THMs may be excluded	If <30% negative RSD, correct problem. If >30% positive RSD and analyte not detected in batch, note in corrective action.	If THM is excluded flag as estimated
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analytes of a QC check sample	Twice a year per analyst	Precision on all targets must meet at or below 20% RSD and 20% RSD recovery.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Check of mass spectral ion intensities using BFB	Daily, before sample analysis, starts 12 hours of analysis time batch.	Refer to criteria listed in the method description	Retune instrument and verify BFB again	
		MDL study	Once per year	Theoretical detection limit calculation must be at least 5 times lower than reporting MDL.	Check calculations, redo MDL analysis and redo results.	
		Internal Standard	Immediately after or during data acquisition of calibration check standard, and on every sample run.	Internal standard retention times should not drift by more than 30 seconds from most recent calibration. Also, the ion area for the internal standards cannot change by more than 50% from the last PM.	Inspect the instrument and correct the problem. Any failed sample must be reanalyzed, however if the QC fails the entire batch must be reanalyzed.	
		Method Blank	One per analytical batch before any samples are run for the batch period.	No analytes detected >MDL	Correct problem then reprep and analyze method blank	If unable to re-analyze, flag with a "B"
		LCS and LCSD precision	One LCS and LCS duplicate per analytical batch	Precision 20 RPD or less Table A.7.1.2	If not detected in batch, any analytes that do not meet criteria are noted in corrective action logbook and in sample comment field.	If unable to re-analyze, flag with a "J"
		Surrogate spike	Every sample, spiked sample, standard, and method blank	Range from 3.5 ug/L to 6.5 ug/L.	Correct problem then reanalyze sample	

Modified 1/26/2006

Georgia Department of Natural Resources

Environmental Protection Division

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David Jones, Laboratory Director

524.2	Volatile Organics	Estimated amount for analytes other than THM above calibration curve	None	All analytes < 10 ppb	Sample must be diluted	Apply E to all analytes out of range that cannot be diluted.
		Target retention time	Per analysis	Target ion within 0.1 min of expected R.T.	Reanalyze sample	

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EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.3.11 EPA Method 335.4 - Total Cyanide by Semi-Automated Colorimetry****A.3.11.1 Scope and Application**

This method is applicable to the determination of cyanide in drinking and surface waters, domestic and industrial wastes. The cyanide as hydrocyanic acid (HCN), is released from cyanide complexes by means of distillation. Cyanides are converted to cyanogen chloride by reactions with chloramine-T, which subsequently reacts with pyridine and barbituric acid to give a red-colored complex. The color is read at 570 nm. The method is modified to use the MIDI-VAP Model MCV-103 midi-cyanide distillation system and the Lachet 8000 auto analyzer.

Water samples for cyanide analysis are collected in a half gallon plastic narrow mouth bottles. Samples are preserved with sufficient NaOH to raise the pH above 12. Sample bottles must be cooled to 4°C after sample collection. Samples must be distilled and analyzed within 14 days.

A.3.11.2 Calibration Verification

The Lachet 8000 is calibrated daily. Seven standards are used to construct the calibration curve; 0 mg/L CN, 0.020 mg/L /CN, 0.050 mg/L CN, 0.10 mg/L CN, 0.20 mg/L CN, 0.30 mg/L CN, 0.40 mg/L and 0.50 mg/L CN. An ICV and ICB are run daily to check the calibration curve. An alternate source standard, where available, is used to verify initial calibration of the measurement system. The ICV value must be within $\pm 10\%$ of true value. The ICB value must be < 0.025 mg/L. Minimum correlation coefficient is 0.995 using linear regression. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

A.3.11.3 Calculation

A standard curve is prepared by plotting the absorbance value of standards versus the corresponding cyanide concentration. The concentration value of the sample is obtained directly from the standard curve.

Table A.3.11.1 RLs for Method EPA 335.4

Parameter/Method	Analyte	Matrix (aqueous)	
		RL	Unit
EPA 335.4	Total Cyanide	0.020	mg/L

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Table A.3.11.2 Acceptance Criteria for Method EPA 335.4

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)
EPA 335.4	Total Cyanide	85-115	30

Table A.3.11.3 Summary of Calibration and QC Procedures for Method
EPA 335.4

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
EPA 335.4	Total Cyanide	Seven point calibration curve	Initial calibration verification once per batch	Correlation coefficient \geq 0.995 linear regression	Correct problem then repeat initial calibration	
		Second source calibration verification	Once per batch	Cyanide concentration within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	
		Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four analysis of a QC check sample	Once per analyst	QC Acceptance Criteria Table and Initial Demonstration SOP	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per batch	Total Cyanide value must be < 0.025 mg/L	Correct problem then analyze method blank and all samples processed with the contaminated blank	If unable to re-analyze, flag with a "B"
		Laboratory Control Sample (LCS/LCSD)	One LCS/LCSD per analytical batch	QC Acceptance Criteria Table	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		MDL Study	Once per year	Detection limits established shall be $<$ the RL's in table	none	
		Matrix Spike (MS/MSD)	One MS/MSD per analytical batch	QC Acceptance Criteria Table	Evaluate out of control event, reanalyze or flag data	
		Continuing Calibration Check (CCC)	After every 10 samples	Concentration within $\pm 10\%$ of expected value	Correct problem then reanalyze all samples associated with out of control CCC.	
		Continuing Calibration Blank (CCB)	After every 10 samples	CN concentration must be < 0.025 mg/l	Correct problem then reanalyze all samples associated with out of control CCB.	

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A.3.19 EPA Method 9010B/9012A - Total Cyanide in Waste and Sediments- Manual Distillation with Automated Color Development

A.3.19.1 Scope and Application

This method is a reflux-distillation procedure used to extract soluble cyanide salts and many insoluble cyanide complexes from wastes and leachates. It is based on the decomposition of nearly all cyanides by a reflux distillation procedure using a strong acid and a magnesium catalyst. Cyanide, in the form of hydrocyanic acid (HCN) is purged from the sample and captured into an alkaline scrubber solution. Method 9010 maybe used as a reflux-distillation procedure for both total cyanide and cyanide amenable to chlorination.

A.3.19.2 Calibration Verification

The Lachat 8000 is calibrated daily. Seven standards are used to construct the calibration curve; 0.00 mg/L CN, 0.025 mg/L /CN, 0.050 mg/L CN, 0.10 mg/L CN, 0.20 mg/L CN, 0.30 mg/L CN, and 0.50 mg/L CN. An ICV and ICB are run daily to check the calibration curve. The ICV value must be within "10% of true value. The ICB value must be < 0.025 mg/L. An alternate source standard, where available, is used to verify initial calibration of the measurement system. Minimum correlation coefficient is 0.995 using linear regression.

A.3.19.3 Calculation

A standard curve is prepared by plotting the absorbance value of standards versus the corresponding cyanide concentration. The concentration of cyanide in the sample digestates is determined by plotting sample absorbance's against the standard curve. Calculation of final result is accomplished using the following equation:

$$\text{CN mg/kg} = \frac{(X)(Y)}{(\text{kg})(\%S)}$$

X
=
C
N

concentration in NaOH trapping solution

Y = Volume (in liters) of the trapping solution

kg = weight (in kg) of the sample (wet weight)

%S = percent solids in sediment, as a decimal fraction

Table A.3.19.1 RLs for Method SW846 9010B/9012A

Parameter/Method	Analyte	Matrix (Waste/Sediment)	
		RL	Unit
SW846 9010B	Total Cyanide in Waste and Sediments	9.0	mg/kg

Modified 2/1/2006

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Environmental Protection Division

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Table A.3.19.2 Acceptance Criteria for Method SW846 9010B/9012A

Method	Analyte	Accuracy Waste (%R)	Precision Waste (RPD)
SW846 9010B/9012A	Total Cyanide in Waste and Sediments	85-115	30

Table A.3.19.3 Summary of Calibration and QC Procedures for Method
SW846 9010B/9012A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
SW846 9010B/ 9012A	Total Cyanide in Waste and Sediments	Seven point calibration curve	Initial calibration verification once per batch	Correlation coefficient \geq 0.995 linear regression	Correct problem then repeat initial calibration	
		Second source calibration verification	Once per batch	Cyanide concentration within "10% of expected value	Correct problem then repeat initial calibration	
		Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four analysis of a QC check sample	Once per analyst	QC Acceptance Criteria Table and Initial Demonstration SOP	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per batch	Total Cyanide value must be < RL in table	Correct problem then analyze method blank and all samples processed with the contaminated blank	If unable to re- analyze, flag with a "B"
		Laboratory Control Sample (LCS/LCSD) spiked with Ottawa sand or glass beads	One LCS/LCSD per analytical batch	QC Acceptance Criteria Table	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re- analyze, flag with a "J"
		Matrix Spike (MS/MSD)	One MS/MSD per analytical batch	QC Acceptance Criteria Table	Evaluate out of control event, reanalyze or flag data	
		Continuing Calibration Check (CCC)	After every 10 samples	Concentration within "10 % of expected value	Correct problem and reanalyze all samples associated with out of control CCC.	
		Continuing Calibration Blank (CCB)	After every 10 samples	CN concentration must be < RL in table	Correct problem and reanalyze all samples associated with out of control CCB.	

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A.2.1 EPA Method 200.7 - Metals by Plasma Emission Spectroscopy**A.2.1.1 Scope and Application****A.2.1 Scope and Application**

Method 200.7 is used to determine metals in ground water, surface water, drinking water waste water, sludges and soils by ICP-OES. Samples analyzed by this method must be acceptable for analysis by direct aspiration or prepared by EPA Method 200.2.

Water samples and liquid waste samples for metal analysis are collected in 500 ml narrow mouth plastic (HDPE) bottles. Samples are preserved with sufficient HNO₃ to lower the pH below 2. One to two bottles are required for each sample. Analysis must be performed within 6 months after preservation.

Soil and sediment samples for metals analysis are collected in 500 ml wide mouth plastic (HDPE) bottles. Samples must be cooled to 4°C ± 2°C after sample collection. Analysis must be performed within 180 days.

A.2.1.2 ICP Calibration and Calculations**A.2.1.2.1 Calibration Curve**

The ICP-OES is calibrated daily using a minimum of a blank and a high standard. The minimum acceptable correlation coefficient is 0.995 using linear regression.

Calibration Verification:

An ICV, CCC, and CCB are analyzed immediately after calibration. A CCC and CCB are analyzed after calibration, after every ten samples, and as the last samples in an analytical sequence. An Interference check standard is analyzed prior to analyzing samples. If the low concentration calibration standard is not at the or below the lowest RL, an MDL check standard is analyzed.

Calculation

A calibration curve is obtained by plotting the absorbance of standards against analyte concentration. The sample concentration for liquid is computed directly from the standard curve and is expressed as ug/L for liquids or mg/Kg for solids and wastes. Solid and waste concentrations are calculated from the following formula:

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Liquid Sample Concentration:

Concentration = CD_f

Where C = concentration from instrument

 D_f = dilution factor

$$D_f = \frac{D}{S}$$

Where D = dilution volume in liters.

S = Sample aliquot volume in liters.

$$\frac{CVD_f}{DW}$$

Concentration = $\frac{CVD_f}{DW}$

Where C = concentration from instrument in mg/L.

V = final digestion volume in L.

 D_f = dilution factor.

DW = sample weight in kg after drying to constant weight at 60°C ± 5°C.

Use the following formula if there is insufficient sample to dry for digestion:

$$\frac{CVD_f P_s}{WW}$$

Concentration = $\frac{CVD_f P_s}{WW}$

Where C = concentration from instrument in mg/L

V = final digestion volume in L.

 D_f = dilution factor.

WW = wet weight of sample (not dried).

 P_s = percent solids

$$\frac{DW}{WW}$$

Percent Solids = $\frac{DW}{WW}$

Where DW = sample weight in kg after drying to constant weight at 60°C ± 5°C

WW = wet weight of sample (not dried).

Waste Concentration Calculation:

$$\frac{CVD_f}{WW}$$

Concentration = $\frac{CVD_f}{WW}$

Where C = concentration from instrument in mg/L

V = final digestion volume in L.

 D_f = dilution factor

WW = wet weight of sample.

Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

Table A.2.1.1 Reporting Limits for EPA 200.7

Method	Analyte	Matrix (Drinking Water)	
		RL	Unit
200.7	Aluminum	50	ug/L
	Barium	50	ug/L
	Chromium	25	ug/L
	Iron	50	ug/L
	Manganese	25	ug/L
	Sodium	1000	ug/L
	Nickel	40	ug/L
	Zinc	50	ug/L

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Table A.2.1.1 Reporting Limits for EPA 200.7

Method	Analyte	Matrix (Water, Water Quality Metals)	
		RL	Unit
200.7	Silver	10	ug/L
	Aluminum	60	ug/L
	Arsenic	80	ug/L
	Barium	10	ug/L
	Beryllium	10	ug/L
	Calcium	1000	ug/L
	Cadmium	10	ug/L
	Cobalt	10	ug/L
	Chromium	20	ug/L
	Copper	20	ug/L
	Iron	20	ug/L
	Potassium	5000	ug/L
	Magnesium	1000	ug/L
	Manganese	10	ug/L
	Sodium	1000	ug/L
	Nickel	20	ug/L
	Lead	90	ug/L
	Antimony	120	ug/L
	Selenium	190	ug/L
	Titanium	10	ug/L
	Thallium	200	ug/L
	Vanadium	10	ug/L
	Zinc	20	ug/L

Modified 5/25/2006

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Table A.2.1.1 Reporting Limits for EPA 200.7

Method	Analyte	Matrix (Water, Ground Water Management Metals)	
		RL	Unit
200.7	Silver	30	ug/L
	Aluminum	50	ug/L
	Barium	10	ug/L
	Bismuth	30	ug/L
	Calcium	1000	ug/L
	Cobalt	10	ug/L
	Chromium	20	ug/L
	Copper	20	ug/L
	Iron	20	ug/L
	Potassium	5000	ug/L
	Magnesium	1000	ug/L
	Manganese	10	ug/L
	Molybdenum	10	ug/L
	Sodium	1000	ug/L
	Lead	50	ug/L
	Nickel	20	ug/L
	Titanium	10	ug/L
	Vanadium	10	ug/L
	Zinc	20	ug/L

Table A.2.1.1 Reporting Limits for EPA 200.7

Method	Analyte	Matrix (Water, Munici Perm Prog)	
		RL	Unit
200.7	Aluminum	50	ug/L
	Beryllium	10	ug/L
	Calcium	1000	ug/L
	Cobalt	10	ug/L
	Iron	20	ug/L
	Potassium	5000	ug/L
	Magnesium	1000	ug/L
	Manganese	10	ug/L
	Sodium	1000	ug/L
	Titanium	10	ug/L
	Vanadium	10	ug/L

Modified 5/25/2006

Georgia Department of Natural Resources

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Table A.2.1.1 Reporting Limits for EPA 200.7

Method	Analyte	Matrix (Soil/Sediment)	
		RL	Unit
200.7	Silver	1.0	mg/kg
	Aluminum	6.0	mg/kg
	Arsenic	8.0	mg/kg
	Barium	1.0	mg/kg
	Beryllium	1.0	mg/kg
	Calcium	100	mg/kg
	Cadmium	1.0	mg/kg
	Cobalt	1.0	mg/kg
	Chromium	2.0	mg/kg
	Copper	2.0	mg/kg
	Iron	2.0	mg/kg
	Potassium	500	mg/kg
	Magnesium	100	mg/kg
	Manganese	1.0	mg/kg
	Sodium	100	mg/kg
	Nickel	2.0	mg/kg
	Lead	9.0	mg/kg
	Antimony	12	mg/kg
	Selenium	19	mg/kg
	Titanium	1.0	mg/kg
	Thallium	20	mg/kg
	Vanadium	1.0	mg/kg
	Zinc	2.0	mg/kg

Table A.2.1.2 Acceptance Criteria for EPA Method 200.7 (All acceptance criteria are the same for each type matrix)

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)	Accuracy Solids (%R)	Precision Solids (RPD)
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Modified 5/25/2006

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200.7	Aluminum	85-115	≤ 15	85-115	≤ 15
	Antimony	85-115	≤ 15	85-115	≤ 15
	Arsenic	85-115	≤ 15	85-115	≤ 15
	Barium	85-115	≤ 15	85-115	≤ 15
	Beryllium	85-115	≤ 15	85-115	≤ 15
	Bismuth	85-115	≤ 15	85-115	≤ 15
	Cadmium	85-115	≤ 15	85-115	≤ 15
	Calcium	85-115	≤ 15	85-115	≤ 15
	Chromium	85-115	≤ 15	85-115	≤ 15
	Cobalt	85-115	≤ 15	85-115	≤ 15
	Copper	85-115	≤ 15	85-115	≤ 15
	Iron	85-115	≤ 15	85-115	≤ 15
	Lead	85-115	≤ 15	85-115	≤ 15
	Magnesium	85-115	≤ 15	85-115	≤ 15
200.7	Manganese	85-115	≤ 15	85-115	≤ 15
	Molybdenum	85-115	≤ 15	85-115	≤ 15
	Nickel	85-115	≤ 15	85-115	≤ 15
	Potassium	85-115	≤ 15	85-115	≤ 15
	Selenium	85-115	≤ 15	85-115	≤ 15
	Silver	85-115	≤ 15	85-115	≤ 15
	Sodium	85-115	≤ 15	85-115	≤ 15
	Strontium	85-115	≤ 15	85-115	≤ 15
	Thallium	85-115	≤ 15	85-115	≤ 15
	Tin	85-115	≤ 15	85-115	≤ 15
	Titanium	85-115	≤ 15	85-115	≤ 15
	Vanadium	85-115	≤ 15	85-115	≤ 15
	Yttrium	85-115	≤ 15	85-115	≤ 15
	Zinc	85-115	≤ 15	85-115	≤ 15

Table A.2.1.3 Summary of Calibration and QC Procedures for EPA Methods 200.7

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
200.7	Aluminum, Antimony, Arsenic, Barium, Beryllium, Bismuth, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Vanadium, Yttrium, Zinc	Analyst Initial Demonstration.	Once per analyst	Average of 4 LCS recoveries between 85-115%. Recovery of unknown sample within QC limits.	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	
		Continuing Demonstration	Every 6 Months	Average of 4 LCS recoveries between 85%-115%, mb<RL, Unknown or PE.	Correct the problem.	

Modified 5/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

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David Jones, Laboratory Director

Ray Terhune, Quality Assurance Manager

		MDL study.	Once every 12 months.	All analyte MDLs must be <RL.	Correct the problem.	
		Analysis of PE sample.	Once every 12 months	All analyte results acceptable per the auditing agency.	Correct the problem	
		Initial Calibration. Minimum of 2 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient \geq 0.995.	Correct the problem and recalibrate	
200.7	Aluminum, Antimony, Arsenic, Barium, Beryllium, Bismuth, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Vanadium, Yttrium, Zinc	Interference Check Sample (SIC).	Daily after calibration.	Spiked element recoveries between 80% and 120% the absolute value of the true value, other element concentrations must be below the reporting limit.	Correct the problem, calculate new interelement correction factors and recalibrate.	
		MDL Check	Daily after calibration.	All analyte recoveries between 50-150%.	Correct the problem and recalibrate.	
		IDL Calculation	Daily after calibration.	All analyte IDLs < RL.	Correct the problem, recalibrate.	
		Initial Calibration Verification (ICV)	Daily after calibration.	All analyte recoveries between 95% and 105% of true value.	Correct the problem and recalibrate.	
		Continuing Calibration Blank (CCB).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte concentrations <RL.	Rerun once, if still out of control, correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	If unable to re-analyze, flag with a "B".
		Continuing Calibration Check (CCC).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	Initial analyte recoveries between 95% and 105%, subsequent analyte recoveries between 90% and 110%	Rerun once, if still out of control, correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCC.	
		Instrument detection limit standard.	Daily after calibration but before analyzing samples.	All analyte recoveries between 50% and 150% of the true value.	Rerun once, if still out of control, correct problem, recalibrate, and reanalyze the samples.	
		Laboratory Control Sample (LCS).	Once per batch.	All analyte recoveries between 85% and 115%.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J".

Modified 5/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

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		Laboratory Control Sample Duplicate (LCSD).	Once per batch.	≤ 15 RPD.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to reanalyze, flag with a "J".
		Matrix Blank.	Once per batch.	All analyte concentrations must <RL.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to reanalyze, flag with a "B".
200.7	Aluminum, Antimony, Arsenic, Barium, Beryllium, Bismuth, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Vanadium, Yttrium, Zinc	Matrix Spike.	Every 10 samples.	All analyte recoveries between 70% and 130%.	If recovery outside QC limits but CCC, CCB, ICV, and LCSD are acceptable, matrix effect is suspected.	Inform data user that data is suspect due to matrix effect.
		Matrix Spike Duplicate.	Every 10 samples.	≤15 RPD.		Inform data user that data is suspect due to matrix effect.

Modified 5/25/2006

Georgia Department of Natural Resources
Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.2.2 EPA Method 200.8 - Metals in Water by Plasma Mass Spectroscopy

A.2.2.1 Scope and Application

Method 200.8 is used to determine trace metals in ground waters, surface waters, drinking waters, wastewaters, sludges, and soils by ICP-MS. Samples analyzed by this method must be acceptable for analysis by direct aspiration or prepared by EPA methods 200.2 or 200.3.

Water samples and liquid waste samples are collected in 500 ml narrow mouth plastic (HDPE) bottles. Copper and Lead samples are collected in 1L HDPE bottles, IOC samples are collected in 250 ml HDPE bottles. Samples are preserved with sufficient HNO₃ to lower the pH below 2. One to two bottles are required for each sample. Analysis must be performed within 6 months after preservation. Soil and sediment samples for metals analysis are collected in 500 ml wide mouth plastic (HDPE) bottles. Samples must be cooled to 4°C ± 2°C after sample collection. Analysis must be performed within 180 days.

A.2.2.2 ICP Calibration and Calculations

A.2.2.2.1 Calibration Curve

The ICP-MS is calibrated daily using a multipoint calibration curve. Refer to the calibration standard concentration table for standard concentrations. Minimum acceptable correlation coefficient is 0.995 using linear regression.

Calibration Standard Concentrations
Concentration (ug/L)

Element	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5	Standard 6	Standard 7
Metals	0.00	0.500	1.00	5.00	10.0	25.0	50.0
Ag	0.00	0.100	0.200	1.000	2.00	5.00	10.0
Se	0.00	2.5	5.00	25.0	50.0	125	250
AL, Fe, Na, Ca, K, Mg	0.00	10.0	20.0	100	200	500	1000

Calibration Verification

An ICV, CCC, and CCB are analyzed immediately after calibration. A CCC and CCB are also analyzed after every ten samples and as the last samples in the analytical batch.

Calculations

Modified 2/7/2006

Georgia Department of Natural Resources
Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

$$\text{Concentration} = \frac{CVD_f}{DW}$$

Where C = concentration from instrument in mg/L.
 V = final digestion volume in L.
 D_f = dilution factor.
 DW = sample weight in kg after drying to constant weight at $60^\circ\text{C} \pm 5^\circ\text{C}$.

Use the following formula if there is insufficient sample to dry for digestion:

$$\text{Concentration} = \frac{CVD_f}{WW} \times \frac{100}{P_s}$$

Where C = concentration from instrument in mg/L.
 V = final digestion volume in L.
 D_f = dilution factor.
 WW = wet weight of sample (not dried).
 P_s = percent solids

$$\text{Percent Solids} = \frac{DW}{WW} \times 100$$

Where DW = sample weight in kg after drying to constant weight at $60^\circ\text{C} \pm 5^\circ\text{C}$
 WW = wet weight of sample (not dried).

Waste Concentration Calculation:

$$\text{Concentration} = \frac{CVD_f}{WW}$$

Where C = concentration from instrument in mg/L
 V = final digestion volume in L.
 D_f = dilution factor
 WW = wet weight of sample.

Aqueous sample results are reported in ug/L, biological tissue is reported in mg/kg wet weight, solids are reported in mg/kg, and wastes in mg/kg wet weight.

Table A.2.2.1 Reporting Limits for Method 200.8

Modified 2/7/2006

Georgia Department of Natural Resources
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Parameter/Method	Analyte	Matrix IOC Water	
		RL	Units
200.8	Beryllium	2	ug/L
	Sodium	1000	ug/L
	Aluminum	50	ug/L
200.8	Chromium	25	ug/L
	Iron	50	ug/L
	Manganese	25	ug/L
	Nickel	40	ug/L
	Zinc	50	ug/L
	Arsenic	5	ug/L
	Selenium	25	ug/L
	Cadmium	2.5	ug/L
	Antimony	3	ug/L
	Barium	50	ug/L
	Thallium	1	ug/L

Table A.2.2.2 Reporting Limits for Method 200.8

Parameter/Method	Analyte	Matrix Fish	
		RL	Units
200.8	Beryllium	1	mg/kg
	Chromium	2	mg/kg
	Nickel	2	mg/kg
	Copper	2	mg/kg
	Zinc	5	mg/kg
	Arsenic	2	mg/kg
	Selenium	2	mg/kg
	Silver	1	mg/kg
	Cadmium	1	mg/kg
	Antimony	2	mg/kg
	Thallium	2	mg/kg
	Lead	1	mg/kg

Modified 2/7/2006

Georgia Department of Natural Resources
Environmental Protection Division
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Table A.2.2.3 Reporting Limits for Method 200.8

Parameter/Method	Analyte	Matrix WQ Water	
		RL	Units
200.8	Chromium	5	ug/L
	Nickel	10	ug/L
	Copper	5	ug/L
	Zinc	10	ug/L
	Arsenic	5	ug/L
	Selenium	5	ug/L
	Molybdenum	5	ug/L
	Silver	5	ug/L
	Cadmium	0.7	ug/L
	Tin	30	ug/L
	Antimony	5	ug/L
	Barium	2	ug/L
	Thallium	1	ug/L
	Lead	1	ug/L
	Uranium	1	ug/L

Table A.2.2.4 Reporting Limits for Method 200.8

Parameter/Method	Analyte	Matrix Appendix I Water	
		RL	Units
200.8	Beryllium	1	ug/L
	Vanadium	1	ug/L
	Chromium	5	ug/L
	Cobalt	5	ug/L
	Nickel	5	ug/L
	Copper	5	ug/L
	Zinc	10	ug/L
	Arsenic	5	ug/L
	Selenium	5	ug/L
	Silver	5	ug/L
	Cadmium	1	ug/L
	Tin	10	ug/L
	Antimony	5	ug/L

Modified 2/7/2006

Georgia Department of Natural Resources
Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

200.8	Barium	1	ug/L
	Thallium	1	ug/L
	Lead	1	ug/L

Table A.2.2.5 Acceptance Criteria for Method 200.8

Parameter/Method	Analyte	Matrix IOC Water	
		Accuracy %R	Precision RPD
200.8	Beryllium	85-115	≤15
	Sodium	85-115	≤15
	Aluminum	85-115	≤15
	Chromium	85-115	≤15
	Iron	85-115	≤15
	Manganese	85-115	≤15
	Nickel	85-115	≤15
	Zinc	85-115	≤15
	Arsenic	85-115	≤15
	Selenium	85-115	≤15
	Cadmium	85-115	≤15
	Antimony	85-115	≤15
	Barium	85-115	≤15
	Thallium	85-115	≤15

Table A.2.2.6 Acceptance Criteria for Method 200.8

Parameter/Method	Analyte	Matrix Fish	
		Accuracy %R	Precision RPD
200.8	Beryllium	85-115	≤15
	Chromium	85-115	≤15
200.8	Nickel	85-115	≤15
	Copper	85-115	≤15
	Zinc	85-115	≤15

Modified 2/7/2006

Georgia Department of Natural Resources
Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

	Arsenic	85-115	≤15
	Selenium	85-115	≤15
	Silver	85-115	≤15
	Cadmium	85-115	≤15
	Antimony	85-115	≤15
	Thallium	85-115	≤15
	Lead	85-115	≤15

Table A.2.2.7 Acceptance Criteria for Method 200.8

Parameter/Method	Analyte	Matrix WQ Water	
		Accuracy %R	Precision RPD
200.8	Chromium	85-115	≤15
	Nickel	85-115	≤15
	Copper	85-115	≤15
	Zinc	85-115	≤15
	Arsenic	85-115	≤15
	Selenium	85-115	≤15
	Molybdenum	85-115	≤15
	Silver	85-115	≤15
	Cadmium	85-115	≤15
	Tin	85-115	≤15
	Antimony	85-115	≤15
	Barium	85-115	≤15
	Thallium	85-115	≤15
	Lead	85-115	≤15
	Uranium	85-115	≤15

Table A.2.2.8 Acceptance Criteria for Method 200.8

Parameter/Method	Analyte	Matrix Appendix I Water	
		Accuracy %R	Precision RPD
200.8	Beryllium	85-115	≤15
	Vanadium	85-115	≤15
	Chromium	85-115	≤15

Modified 2/7/2006

Georgia Department of Natural Resources
Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

Cobalt	85-115	≤15
Nickel	85-115	≤15
Copper	85-115	≤15
Zinc	85-115	≤15
Arsenic	85-115	≤15
Selenium	85-115	≤15
Silver	85-115	≤15
Cadmium	85-115	≤15
Tin	85-115	≤15
Antimony	85-115	≤15
Barium	85-115	≤15
Thallium	85-115	≤15
Lead	85-115	≤15

**Table A.2.2.9 Summary of Calibration and QC Procedures for Method
EPA 200.8**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
200.8	Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Manganese, Molybdenum, Nickel, Selenium, Silver, Sodium, Thallium, Tin, Uranium, Vanadium, Zinc	Analyst Initial Demonstration.	Once per analyst	2 matrix blanks < RL. Average of 4 LCS recoveries between 85-115%. Recovery of unknown sample within established QC limits.	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	
		Continuing Demonstration	Every 6 Months	Average of 4 LCS recoveries between 85%-115% mb<RL, Unknown or PE.	Correct the problem. Then rerun the continuing demonstration for those analytes that did not meet criteria.	

Modified 2/7/2006

Georgia Department of Natural Resources
Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

200.8	Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Manganese, Molybdenum, Nickel, Selenium, Silver, Sodium, Thallium, Tin, Uranium, Vanadium, Zinc	MDL study.	Once every 12 months.	All analyte MDLs must be < RL.	Correct the problem.	
		Interference Check Sample	Once every 12 months	Spiked element recoveries between 80-120% recovery, the absolute value of unspiked element concentrations < RL.	Correct the problem; and recalibrate.	
		Analysis of PE sample.	Once every 12 months	All analyte results acceptable per the auditing agency.	Correct the problem	
		Instrument Tune	Daily before calibration	See table.	Correct problem and recalibrate.	
		Initial Calibration. Minimum of 2 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient \geq 0.995	Correct the problem and recalibrate	
		Initial Calibration Verification (ICV)	Daily after calibration.	All analyte recoveries between 90% and 110% of the true value..	Correct the problem and recalibrate.	
		Continuing Calibration Blank (CCB).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte concentrations must be <RL.	Rerun once, if still out of control, correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	
		Instrument Detection Limit (IDL)	After first calibration of the day.	3x Standard deviation of 10 reps < analyte RL	Recalibrate	

Modified 2/7/2006

Georgia Department of Natural Resources
Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

200.8	Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Manganese, Molybdenum, Nickel, Selenium, Silver, Sodium, Thallium, Tin, Uranium, Vanadium, Zinc	Continuing Calibration Check (CCC).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte recoveries between 90-110%	Rerun once, if still out of control, correct the problem, recalibrate, reanalyze all samples since the last acceptable CCC.	
		Laboratory Control Sample (LCS).	Once per batch.	All analyte recoveries between 85-115%.	Rerun one, if still out of control correct the problem, redigest batch if sample amounts permit, and reanalyze all samples in the batch.	If insufficient sample for redigestion, flag with "J".
		Laboratory Control Sample Duplicate (LCSD).	Once per batch.	Relative Percent Difference ≤ 15 .	Rerun once, if still out of control correct the problem, redigest batch if sample amounts permit, and reanalyze all samples in batch	If insufficient sample for redigestion, flag with "J".
		Matrix Blank	Once per batch.	All analyte concentrations must be less than the reporting limit.	Comment report if reanalysis has contamination.	Flag data with "B"
		Matrix Spike	Every 10 samples.	All analyte recoveries between 70-130%.	If recovery exceeds QC limits but CCC, CCB, ICV, LCS, and LCSD are acceptable, matrix effect is suspected.	Comment report.
		Matrix Spike Duplicate.	Every 10 samples.	Relative Percent Difference ≤ 15 .		If RPD > 15 inform data user data suspect due to matrix effect.
200.8	Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Manganese, Molybdenum, Nickel, Selenium, Silver, Sodium, Thallium, Tin, Uranium, Vanadium, Zinc	Internal Standards	Every sample and standard except tuning solution	All internal standard recoveries must be between 60% - 125% of the original response of the calibration blank.	Dilute and reanalyze.	

Modified 2/7/2006

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Modified 2/7/2006

Georgia Department of Natural Resources
Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

Table A.2.2.10 Tuning Criteria Method 200.8

Mass 220 counts	<100
Cerium Oxide ratio	<3%
Ba ⁺⁺ ratio	<5%
Mass calibration of ^{24, 25, 26} Mg and ^{206, 207, 208} Pb	± 0.1 AMU of unit mass.
RSD of 5 replicates of a 10 ug/L solution of ⁹ Be, ²⁴ Mg, ⁵⁹ Co, ¹¹⁵ In, and ²⁰⁸ Pb	< 5
²⁴ Mg counts of a 10 ug/L solution	>5,000 CPS
¹¹⁵ In counts of a 10 ug/L solution	>10,000 CPS
²⁰⁸ Pb counts of a 10 ug/L solution	>7,500 CPS
Peak width of ^{24, 25, 26} Mg and ^{206, 207, 208} Pb	Between 0.6 and 0.8 AMU at 10% peak height.

Modified 2/7/2006

Georgia Department of Natural Resources

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A.2.3 EPA Method 245.1 - Mercury in Water by cold vapor Atomic Absorption

Method 245.1 is used to determine Mercury in drinking, surface, and saline waters and industrial wastes by cold vapor atomic absorption spectroscopy. An aliquot of the sample is accurately measured and transferred to a clean 50 ml centrifuge tube. The sample is then digested in dilute Potassium Permanganate-Potassium Persulfate solution and oxidized for 2 hours at 95°C. Mercury in the sample is then reduced by Stannous Chloride to elemental Mercury and analyzed by flow injection cold vapor atomic absorption.

Water and liquid waste samples for Mercury analysis are collected in 500 ml narrow mouth plastic (HDPE) bottles. Samples are preserved with sufficient HNO₃ to lower the pH below 2. One to two bottles are required for each sample. Mercury analysis must be performed within 28 days.

A.2.3.1 Calibration and Calculations**A.2.3.1.1 Calibration Curve**

The Mercury analyzer is calibrated daily. A multipoint calibration curve is used. The concentrations of the calibration standards are (in ug/L) 0.0, 0.2, 0.4, 1.0, 2.0, 3.0, and 6.0. Minimum acceptable correlation coefficient is 0.995 using linear regression. An ICV, CCB and CCC are analyzed immediately after calibration. A CCC and CCB are analyzed after calibration, after every ten samples and as the last samples in an analytical sequence.

A.2.3.1.2 Calculation

A standard curve is obtained by plotting the absorbance of standards against analyte concentration. The sample concentrations are computed directly from the standard curve and are reported as ug/L.

$$\text{Concentration} = CD_f$$

Where C = concentration from instrument

D_f = dilution factor

$$D_f = \frac{D}{S}$$

D = dilution volume in liters.

S = Sample aliquot volume in liters.

Georgia Department of Natural Resources

Environmental Protection Division

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Table A.2.3.1 RLs for Method 245.1

Parameter/Method	Analyte	Matrix (Water)	
		RL	Unit
Mercury by Cold Vapor Atomic Absorption Spectrometry	Mercury	0.2	ug/L

Table A.2.3.2 Acceptance Criteria for Method 245.1

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)
Mercury by Cold Vapor Atomic Absorption Spectrometry	Mercury	85-115	≤ 15

Table A.2.3.3 Summary of Calibration and QC Procedures for Method
EPA 245.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
245.1	Mercury	Analyst Initial Demonstration.	Once per analyst.	Average of 4 LCS recoveries between 85-115%. Recovery of unknown sample between 70%-130%.	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	
		Continuing Demonstration	Every 6 Months	Average of 4 LCS recoveries between 85%-115%, mb<RL, Unknown or PE.	Correct the problem.	
		MDL study.	Once every 12 months.	Analyte MDL must be SRL	Correct the problem.	
		Analysis of PE sample.	Once every 12 months	All analyte results acceptable per the auditing agency.	Correct the problem.	
		Initial Calibration. Minimum of 4 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct the problem and recalibrate.	
		IDL Calculation	Daily after calibration.	Analyte IDL <RL.	Correct the problem, clean the torch, recalibrate.	

Modified 2/9/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

Ray Terhune, Quality Assurance Manager

		Initial Calibration Verification (ICV)	Daily after calibration.	Analyte recovery between 90% and 110% .	Correct the problem and recalibrate.	
245.1	Mercury	Continuing Calibration Blank (CCB).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	Analyte concentration \leq RL.	Correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	If unable to re-analyze, flag with a "B"
		Continuing Calibration Check (CCC).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	Initial recovery between 95-105%, subsequent recoveries between 90%-110%.	Correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCC.	
		Laboratory Control Sample (LCS).	Once per batch.	All analyte recoveries between 85%-115%.	Correct the problem, redigest and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J".
		Laboratory Control Sample Duplicate (LCSD).	Once per batch.	≤ 15 RPD	Correct the problem, redigest and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J".
		Matrix Blank	Once per batch.	Analyte concentration $<RL$	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "B".
		Matrix Spike	Every 10 samples.	Analyte recovery between 70%-130%.	Comment sample report.	
		Matrix Spike Duplicate.	Every 10 samples.	≤ 15 RPD.	Comment sample report.	

Modified 2/9/2006

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A.2.9 EPA Method 7471 - Mercury by Cold Vapor AA Spectroscopy in Solids**A.2.9.1 Scope and Application**

Method 7471 is used to determine Mercury in soils, sediments, bottom deposits, and sludge-like materials by cold vapor atomic absorption spectroscopy. An aliquot of the sample is accurately measured and transferred to a 50 ml centrifuge tube. The sample is then digested in dilute Potassium Permanganate-aqua regia solution and oxidized at 95°C. Mercury in the sample is then reduced by Stannous Chloride to elemental Mercury and analyzed by flow injection cold vapor atomic absorption.

Soil and sediment samples for metal analysis are collected in 500 ml wide mouth plastic (HDPE) bottles. Sample bottles must be cooled to 4°C ± 2°C after sample collection. Samples must be analyzed within 180 days.

A.2.9.2 Calibration and Calculations**A.2.9.2.1 Calibration Curve**

The Mercury analyzer is calibrated daily. A multipoint calibration curve is used. The concentrations of the calibration standards are (in mg/Kg): 0.0, 0.0002, 0.0005, 0.001, 0.002, 0.003, and 0.006. An ICV is analyzed immediately after calibration. A CCC and CCB are analyzed after calibration, after every ten samples, and as the last samples in an analytical sequence.

A.2.9.2.2 Calculation

A standard curve is obtained by plotting the absorbance of standards against analyte concentration. The sample concentrations are computed by the following formula and are reported in mg/kg, ug/kg for TAL solids. The reporting limit for the diluted analyte is also multiplied by the dilution factor.

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

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Solids Concentration Calculation

Use the following formula if there is insufficient sample to dry for digestion:

$$\frac{CVD_F P_s}{WW}$$

$$\text{Concentration} = \frac{CVD_F P_s}{WW}$$

Where C = concentration from instrument in mg/L V = final digestion volume in L. D_f = dilution factor. WW = wet weight of sample (not dried). P_s = percent solids

$$\frac{DW}{WW}$$

$$\text{Percent Solids} = \frac{DW}{WW}$$

Where DW = sample weight in kg after drying to constant weight at $60^\circ\text{C} \pm 5^\circ\text{C}$ WW = wet weight of sample (not dried)

Waste Concentration Calculation:

$$\frac{CVD_F}{WW}$$

$$\text{Concentration} = \frac{CVD_F}{WW}$$

Where C = concentration from instrument in mg/L V = final digestion volume in L. D_f = dilution factor WW = wet weight of sample.

Table A.2.9.1 RLs for Method 7471A

Parameter/Method	Analyte	Matrix (Solids)		Matrix (TAL)	
		RL	Unit	RL	Unit
Mercury by Cold Vapor Atomic Absorption Spectrometry	Mercury	0.1	mg/Kg	100	ug/Kg

Table A.2.9.2 Acceptance Criteria for Method 7471A

Method	Analyte	Accuracy Solids (%R)	Precision Solids (RPD)
Mercury by Cold Vapor Atomic Absorption Spectrometry	Mercury	85-115%	15

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

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Table A.2.9.3 Summary of Calibration QC Procedures for Method SW846-7471A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
7471A	Mercury	Analyst Initial Demonstration.	Once per analyst	Average of 4 LCS recoveries between 85-115%. Recovery of unknown sample between 75%-130%.	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	
		Continuing Demonstration	Every 6 Months	Average of 4 LCS recoveries between 85%-115%, mb<RL, Unknown or PE.	Correct the problem.	
		MDL study.	Once every 12 months.	Analyte MDLs must be < RL.	Correct the problem.	
		Analysis of PE sample.	Once every 12 months	Analyte results acceptable per the auditing agency.	Correct the problem	
		Initial Calibration. Minimum of 4 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient \geq 0.995	Correct the problem and recalibrate	
		Initial Calibration Verification (ICV)	Daily after calibration.	Analyte recoveries between 90% and 110% of true value.	Correct the problem and recalibrate.	
		Continuing Calibration Blank (CCB).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	Analyte concentrations must be < RL.	Correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	
		Continuing Calibration Check (CCC).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	Initial analyte recovery between 90% and 110%, subsequent recoveries between 80% and 120%.	Correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCC.	
		Laboratory Control Sample (LCS).	Once per batch.	Analyte recoveries between 85% and 115%.	Correct the problem, recalibrate, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J".
		Laboratory Control Sample Duplicate (LCSD).	Once per batch.	\leq 15 RPD	Correct the problem, recalibrate, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J".

Modified 2/22/2005

Georgia Department of Natural Resources**Environmental Protection Division**EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

Ray Terhune, Quality Assurance Manager

		Matrix Blank	Once per batch.	Analyte concentrations must be < RL.	Correct the problem, recalibrate, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "B".
		Matrix Spike	Every 10 samples.	Analyte recoveries between 70% and 130%.	Comment Sample Report.	
7471A	Mercury	Matrix Spike Duplicate.	Every 10 samples.	≤ 15 RPD	Comment Sample Report.	

Modified 2/22/2005

Georgia Department of Natural Resources

Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.2.6 EPA Method 6010B - Metals by Plasma Emission Spectroscopy

A.2.6.1 Scope and Application

Method SW846-6010B is used to determine metals in ground water, aqueous matrices, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments and other solid wastes by ICP optical spectroscopy. An aliquot of the sample is accurately measured and refluxed with Hydrochloric and Nitric acids to solubilize analytes. Samples analyzed by this method must first be prepared by one of the following methods: SW846-3010A, SW846-3015, SW846-3050B, SW846-3051. The sample is centrifuged or allowed to settle overnight prior to analysis.

Water samples and liquid waste samples for analysis by method 6010B are collected in 500 ml narrow mouth plastic (HDPE) bottles. Samples are preserved with sufficient HNO_3 to lower the pH below 2. Solid samples are collected in 500 ml wide mouth HDPE bottles and preserved by cooling to $4^\circ\text{C} \pm 2^\circ\text{C}$. Samples must be analyzed within 180 days of collection.

A.2.6.2 ICP Calibration and Calculations

A.2.6.2.1 Calibration Curve

Calibration Verification:

An ICV, CCC, and CCB are analyzed immediately after calibration. A CCC and CCB are analyzed after calibration, after every ten samples, and as the last samples in an analytical sequence. An Interference check standard is analyzed prior to analyzing samples. If the low concentration calibration standard is not at the or below the lowest RL, an MDL check standard is analyzed.

Calculation

A calibration curve is obtained by plotting the absorbance of standards against analyte concentration. The sample concentration for liquid is computed directly from the standard curve and is expressed as ug/L for liquids or mg/Kg for solids and wastes. Solid and waste concentrations are calculated from the following formula:

Modified 5/25/2006

Georgia Department of Natural Resources

Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

Liquid Sample Concentration:

$$\text{Concentration} = CD_f$$

Where C = concentration from instrument

 D_f = dilution factor

$$D_f = \frac{D}{S}$$

Where D = dilution volume in liters.

S = Sample aliquot volume in liters.

$$\frac{CVD_f}{DW}$$

$$\text{Concentration} = \frac{CVD_f}{DW}$$

Where C = concentration from instrument in mg/L.

V = final digestion volume in L.

 D_f = dilution factor.

DW = sample weight in kg after drying to constant weight at 60°C ± 5°C.

Use the following formula if there is insufficient sample to dry for digestion:

$$\frac{CVD_f P_s}{WW}$$

$$\text{Concentration} = \frac{CVD_f P_s}{WW}$$

Where C = concentration from instrument in mg/L

V = final digestion volume in L.

 D_f = dilution factor.

WW = wet weight of sample (not dried).

 P_s = percent solids

$$\frac{DW}{WW}$$

$$\text{Percent Solids} = \frac{DW}{WW}$$

Where DW = sample weight in kg after drying to constant weight at 60°C ± 5°C

WW = wet weight of sample (not dried).

Waste Concentration Calculation:

$$\frac{CVD_f}{WW}$$

$$\text{Concentration} = \frac{CVD_f}{WW}$$

Where C = concentration from instrument in mg/L

V = final digestion volume in L.

 D_f = dilution factor

WW = wet weight of sample.

Table A.2.6.1 RLs for EPA Method SW846-6010B

Method	Analyte	Matrix (Aqueous and Extracts)		Matrix (Soils, Solids and Wastes)	
		RL	Unit	RL	Unit

Modified 5/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

6010B	Aluminum	0.06	mg/L	6.0	mg/kg
	Antimony	0.12	mg/L	12	mg/kg
6010B	Arsenic	0.08	mg/L	8.0	mg/kg
	Barium	0.01	mg/L	1.0	mg/kg
	Beryllium	0.01	mg/L	1.0	mg/kg
	Bismuth	0.09	mg/L	9.0	mg/kg
	Cadmium	0.01	mg/L	1.0	mg/kg
	Calcium	1.0	mg/L	100	mg/kg
	Chromium	0.02	mg/L	2.0	mg/kg
	Cobalt	0.01	mg/L	1.0	mg/kg
	Copper	0.02	mg/L	2.0	mg/kg
	Iron	0.02	mg/L	2.0	mg/kg
	Lead	0.09	mg/L	9.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.04	mg/L	4.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	5.0	mg/L	5.0	mg/kg
	Selenium	0.19	mg/L	19	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Strontium	0.01	mg/L	1.0	mg/kg
	Thallium	0.20	mg/L	2.0	mg/kg
	Titanium	0.01	mg/L	1.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

*RL for solids must be multiplied by the dilution factor, usually 100.

Table A.2.6.2 RLs for EPA Method SW846-6010B for Target Analyte List (TAL)

Method	Analyte	Matrix (Water)		Matrix (Solids and Wastes)	
		RL	Unit	RL	Unit
6010B	Silver			10000	ug/kg
	Aluminum	200	ug/L	200000	ug/kg
	Arsenic			80000	ug/kg
	Barium			200000	ug/kg
	Beryllium			5000	ug/kg
	Calcium	5000	ug/L	5000000	ug/kg
	Cadmium			5000	ug/kg
	Cobalt			50000	ug/kg
	Chromium			100000	ug/kg
	Copper			25000	ug/kg
	Iron	100	ug/L	100000	ug/kg
	Potassium	5000	ug/L	5000000	ug/kg
	Magnesium	5000	ug/L	5000000	ug/kg

Modified 5/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

	Manganese	15	ug/L	15000	ug/kg
	Sodium	5000	ug/L	5000000	ug/kg
	Nickel			40000	ug/kg
6010B	Lead			90000	ug/kg
	Antimony			60000	ug/kg
	Selenium			190000	ug/kg
	Thallium			200000	ug/kg
	Vanadium			50000	ug/kg
	Zinc			20000	ug/kg

*RL for solids must be multiplied by the dilution factor, usually 100.

Table A.2.6.3 Acceptance Criteria for EPA Method SW846-6010B

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)	Accuracy Solids (%R)	Precision Solids (RPD)
6010B	Aluminum	85-115	≤ 15	85-115	≤ 15
	Antimony	85-115	≤ 15	85-115	≤ 15
	Arsenic	85-115	≤ 15	85-115	≤ 15
	Barium	85-115	≤ 15	85-115	≤ 15
	Beryllium	85-115	≤ 15	85-115	≤ 15
	Bismuth	85-115	≤ 15	85-115	≤ 15
	Cadmium	85-115	≤ 15	85-115	≤ 15
	Calcium	85-115	≤ 15	85-115	≤ 15
	Chromium	85-115	≤ 15	85-115	≤ 15
	Cobalt	85-115	≤ 15	85-115	≤ 15
	Copper	85-115	≤ 15	85-115	≤ 15
	Iron	85-115	≤ 15	85-115	≤ 15
	Lead	85-115	≤ 15	85-115	≤ 15
	Magnesium	85-115	≤ 15	85-115	≤ 15
	Manganese	85-115	≤ 15	85-115	≤ 15
	Molybdenum	85-115	≤ 15	85-115	≤ 15
	Nickel	85-115	≤ 15	85-115	≤ 15
	Potassium	85-115	≤ 15	85-115	≤ 15
	Selenium	85-115	≤ 15	85-115	≤ 15
	Silver	85-115	≤ 15	85-115	≤ 15
	Sodium	85-115	≤ 15	85-115	≤ 15
	Strontium	85-115	≤ 15	85-115	≤ 15
	Thallium	85-115	≤ 15	85-115	≤ 15
	Titanium	85-115	≤ 15	85-115	≤ 15
	Vanadium	85-115	≤ 15	85-115	≤ 15
	Zinc	85-115	≤ 15	85-115	≤ 15

Table A.2.6.4 Summary of Calibration and QC Procedures for EPA Method 6010B

Modified 5/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

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Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
6010B	Aluminum, Antimony, Arsenic, Barium, Beryllium, Bismuth, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Vanadium, Zinc	Analyst Initial Demonstration	Once per analyst	Average of 4 LCS recoveries between 85-115%. Recovery of unknown sample between 75%-125%, TCLP limits are between 50%-150%	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	
		MDL study.	Once every 12 months.	All analyte MDLs <RL	Correct the problem.	
		Analysis of PE sample.	Once every 12 months	All analyte results acceptable per the auditing agency.	Correct the problem	
		Initial Calibration. Minimum of 2 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient ≥ 0.995	Correct the problem and recalibrate	
		Interference Check Sample (ICS).	Daily after calibration.	Spiked element recoveries between 80-120% recovery, the absolute value of the other element concentrations must be <RL.	Correct the problem, calculate new interelement correction factors and recalibrate.	
		MDL Check	Daily after calibration.	All analyte recoveries between 50-150%.	Correct the problem and recalibrate.	
		IDL Calculation	Daily after calibration.	All analyte IDLs < RL.	Correct the problem, clean the torch, recalibrate.	
		Initial Calibration Verification (ICV)	Daily after calibration.	All analyte recoveries between 90% and 110% of true value.	Correct the problem and recalibrate.	
		Continuing Calibration Blank (CCB).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte concentrations < RL.	Correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	If unable to re-analyze, flag with a "B"
		Continuing Calibration Check (CCC).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte recoveries between 90%-110%.	Correct the problem; recalibrate, and reanalyze all samples since the last acceptable CCC.	
		Laboratory Control Sample (LCS).	Once per batch.	All analyte recoveries between 85%-115%.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J"

Modified 5/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

6010B	Aluminum, Antimony, Arsenic, Barium, Beryllium, Bismuth, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Vanadium, Zinc	Laboratory Control Sample Duplicate (LCSD).	Once per batch.	≤ 15 RPD	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J"
		Matrix Blank	Once per batch.	All analyte concentrations < RL.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "B"
		Matrix Spike	Every 10 samples.	All analyte recoveries between 75%-125%, TCLP recoveries between 50%-150%	Comment Sample report.	
		Matrix Spike Duplicate.	Every 10 samples.	≤ 20 RPD.	Comment Sample report.	
		TCLP duplicate	One every batch	≤ 20 RPD	Sample Commented.	

Modified 5/25/2006

Georgia Department of Natural ResourcesEnvironmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.2.7 EPA Method 6020 - Metals in Water by Plasma Mass Spectroscopy****A.2.7.1 Scope and Application**

Method 6020 is used to determine trace metals in ground waters, surface waters, wastewaters, solids and wastes by ICP-MS. An aliquot of the sample is accurately measured and refluxed with Hydrochloric and Nitric acids to solubilize analytes. Samples analyzed by this method must first be prepared by one of the following EPA methods: SW846-3010A, SW846-3015, SW846-3050B, SW846-3051. The digested sample is centrifuged or allowed to settle overnight prior to analysis.

Water samples and liquid waste samples for metal analysis are collected in 500 ml narrow mouth plastic (HDPE) bottles. Samples are preserved with sufficient HNO₃ to lower the pH below 2. One to two bottles are required for each sample. Samples must be analyzed within 6 months. Soil and sediment samples are collected in 500 ml wide mouth HDPE bottles and are preserved by cooling to 4°C ± 2°C. Samples must be analyzed within 6 months.

A.2.7.2 ICP Calibration and Calculations**A.2.7.2.1 Calibration Curve**

The ICP-MS is calibrated daily using a multipoint calibration curve. Refer to the calibration standard concentration table for standard concentrations. Minimum acceptable correlation coefficient is 0.995 using linear regression.

Calibration Standard Concentrations
Concentration (ug/L)

Element	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5	Standard 6	Standard 7
Metals	0.00	0.500	1.00	5.00	10.0	25.0	50.0
Ag	0.00	0.100	0.200	1.000	2.00	5.00	10.0
Se	0.00	2.5	5.00	25.0	50.0	125	250
AL, Fe, Na, Ca, K, Mg	0.00	10.0	20.0	100	200	500	1000

Calibration Verification

An ICV, CCC, and CCB are analyzed immediately after calibration. A CCC and CCB are also analyzed after every ten samples and as the last samples in an analytical batch.

Calculation

A standard curve is obtain by plotting the standard count against analyte concentration.

Modified 2/7/2006

Georgia Department of Natural Resources

Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

The sample concentration is computed directly from the standard curve and is reported in ug/L.

$$\text{Concentration} = \frac{CVD_f}{DW}$$

Where C = concentration from instrument in mg/L.
 V = final digestion volume in L.
 D_f = dilution factor.
 DW = sample weight in kg after drying to constant weight at $60^\circ\text{C} \pm 5^\circ\text{C}$.

Use the following formula if there is insufficient sample to dry for digestion:

$$\text{Concentration} = \frac{CVD_f}{WW} \times \frac{100}{P_s}$$

Where C = concentration from instrument in mg/L
 V = final digestion volume in L.
 D_f = dilution factor.
 WW = wet weight of sample (not dried).
 P_s = percent solids

$$\text{Percent Solids} = \frac{DW}{WW} \times 100$$

Where DW = sample weight in kg after drying to constant weight at $60^\circ\text{C} \pm 5^\circ\text{C}$
 WW = wet weight of sample (not dried).

Waste Concentration Calculation:

$$\text{Concentration} = \frac{CVD_f}{WW}$$

Where C = concentration from instrument in mg/L
 V = final digestion volume in L.
 D_f = dilution factor
 WW = wet weight of sample.

Aqueous sample results are reported in ug/L, solids are reported in mg/kg dry weight, wastes are reported in mg/kg wet weight..

Modified 2/7/2006

Georgia Department of Natural Resources

Environmental Protection Division
EPD Laboratory, 455 14th Street, Atlanta GA 30318

Modified 2/7/2006

Georgia Department of Natural Resources

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Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

Table 2.7.1 Reporting Limits for Method 6020 TAL

Parameter/Method	Analyte	Matrix Aqueous	
		RL	Units
6020	Antimony	60	ug/L
	Arsenic	10	ug/L
	Barium	200	ug/L
	Beryllium	5	ug/L
	Cadmium	5	ug/L
	Chromium	10	ug/L
	Cobalt	50	ug/L
	Copper	25	ug/L
	Lead	3	ug/L
	Nickel	40	ug/L
	Selenium	5	ug/L
	Silver	10	ug/L
	Thallium	10	ug/L
	Vanadium	50	ug/L
	Zinc	20	ug/L

Table 2.7.2 Acceptance Criteria for Method 6020 TAL

Method	Analyte	Accuracy Aqueous (%R)	Precision Aqueous (RPD)
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Modified 2/7/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

6020	Antimony	85-115	≤15
	Arsenic	85-115	≤15
	Barium	85-115	≤15
	Beryllium	85-115	≤15
	Cadmium	85-115	≤15
	Chromium	85-115	≤15
	Cobalt	85-115	≤15
	Copper	85-115	≤15
	Lead	85-115	≤15
	Molybdenum	85-115	≤15
	Nickel	85-115	≤15
	Selenium	85-115	≤15
	Silver	85-115	≤15
	Thallium	85-115	≤15
	Tin	85-115	≤15
6020	Vanadium	85-115	≤15
	Zinc	85-115	≤15

**Table 2.7.3 Summary of Calibration and QC Procedures for Method
EPA 6020**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
6020	Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Lead, Molybdenum, Nickel, Selenium, Silver, Thallium, Tin, Vanadium, Zinc	Analyst Initial Demonstration.	Once per analyst	2 matrix blanks <RL. Average of 4 LCS recoveries between 85-115%. Recovery of unknown sample between 75%-125%.	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	
		Continuing Demonstration	Every 6 Months	Average of 4 LCS recoveries between 85%-115% mb<RL, Unknown or PE.	Correct the problem.	
		MDL study.	Once every 12 months.	All analyte MDLs must be < RL.	Correct the problem.	
		Analysis of PE sample.	Once every 12 months	All analyte results acceptable per the auditing agency.	Correct the problem	

Modified 2/7/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

6020	Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Lead, Molybdenum, Nickel, Selenium, Silver, Thallium, Tin, Vanadium, Zinc	Initial Calibration. Minimum of 2 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient \geq 0.995	Correct the problem and recalibrate	
		Interference Check Sample (ICS).	Daily after calibration.	Spiked element recoveries between 80-120% recovery, the absolute value of the other element concentrations must be below the reporting level.	Correct the problem, calculate new interelement correction factors and recalibrate.	
		IDL Calculation	Daily after calibration.	All analyte IDLs <RL.	Correct the problem, recalibrate.	
		Initial Calibration Verification (ICV)	Daily after calibration.	All analyte recoveries between 90% and 110% of the true value.	Rerun once, if still out of control correct the problem and recalibrate.	
		Continuing Calibration Blank (CCB).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte concentrations must be below the analyte reporting limit.	Rerun once, if still out of control, correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	
		Continuing Calibration Check (CCC).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	All analyte recoveries between 90-110%.	Rerun once, if still out of control, correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCC.	
		Laboratory Control Sample (LCS).	Once per batch.	All analyte recoveries between 85-115%.	Rerun once, if still out correct the problem, redigest batch if sample amounts permit, and reanalyze all samples in the batch.	If insufficient sample for redigestion, flag with "J".

Modified 2/7/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

6020	Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Lead, Molybdenum, Nickel, Selenium, Silver, Thallium, Tin, Vanadium, Zinc	Laboratory Control Sample Duplicate (LCSD).	Once per batch.	≤ 15 RPD	Rerun once, if still out correct the problem, redigest batch if sample amounts permit, and reanalyze all samples in the batch.	If insufficient sample for redigestion, flag with "J".
		Matrix Blank	Once per batch.	All analyte concentrations must be <RL.	Comment report if reanalysis has contamination.	Flag data with "B"
		Matrix Spike	Every 10 samples.	All analyte recoveries between 75-125%.	If recovery exceeds QC limits but CCC, CCB, ICV, LCS and LCSD are acceptable, matrix effect is suspected.	Comment report.
		Matrix Spike Duplicate.	Every 10 samples.	≤ 15 RPD	Comment sample.	If RPD > 15 inform data user data suspect due to matrix effect.
		Dilution Test: dilute one matrix spike per batch fivefold with matrix blank.	Once per batch	Original and diluted results must be within 10 RPD of each other.	If RPD > 10 matrix interference must be suspected.	Comment data about suspected matrix interference.

Table 13.10 Tuning Criteria Method 6020

Mass 220 counts	<100
Cerium Oxide ratio	<3%
Ba ⁺⁺ ratio	<5%
Mass calibration of ²⁴ , ²⁵ , ²⁶ Mg and ²⁰⁶ , ²⁰⁷ , ²⁰⁸ Pb	± 0.1 AMU of unit mass.
RSD of 5 replicates of a 10 ug/L solution of ⁹ Be, ²⁴ Mg, ⁵⁹ Co, ¹¹⁵ In, and ²⁰⁸ Pb	< 5
²⁴ Mg counts of a 10 ug/L solution	>5,000 CPS
¹¹⁵ In counts of a 10 ug/L solution	>10,000 CPS
²⁰⁸ Pb counts of a 10 ug/L solution	>7,500 CPS
Peak width of ²⁴ , ²⁵ , ²⁶ Mg and ²⁰⁶ , ²⁰⁷ , ²⁰⁸ Pb	Between 0.6 and 0.8 AMU at 10% peak height.

Modified 2/7/2006

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Ray Terhune, Quality Assurance Manager

A.2.8 EPA Method 7470 - Mercury by cold vapor AA**A.2.8.1 Scope and Application**

Method 7470 is used to determine Mercury in mobility extraction procedures, TCLP extracts, aqueous wastes and ground water by cold vapor atomic absorption spectroscopy. An aliquot of the sample is accurately measured and transferred to a clean 50 ml centrifuge tube. The sample is then digested in dilute Potassium Permanganate-Potassium Persulfate solutions and oxidized at 95°C. Mercury in the sample is then reduced by Stannous Chloride to elemental Mercury and analyzed by flow injection cold vapor atomic absorption.

Water samples and liquid waste samples for Mercury analysis are collected in 500 ml narrow mouth plastic (HDPE) bottles. Samples are preserved with sufficient HNO₃ to lower the pH below 2. One to two bottles are required for each sample. Mercury analysis must be performed within 28 days.

A.2.8.2 Calibration and Calculations**A.2.8.2.1 Calibration Curve**

The Mercury instrument is calibrated daily. A multipoint calibration curve is used. The concentrations of the calibration standards are (in ug/L): 0.0, 0.2, 0.5, 1.0, 2.0, 3.0, and 6.0. An ICV is analyzed immediately after calibration. A CCC and CCB are analyzed after calibration, after every ten samples, and as the last sample in an analytical sequence.

A.2.8.2.2 Calculation

A standard curve is obtained by plotting the absorbance of standards against analyte concentration. The sample concentrations are computed directly from the standard curve and are reported in ug/L, TCLP extracts are in mg/L.

Table A.2.8.1 RLs for Method 7470A

Parameter/Method	Analyte	Matrix Aqueous	
		RL	Unit
Mercury by Cold Vapor Atomic Absorption Spectrometry	Mercury	0.2	ug/l

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Ray Terhune, Quality Assurance Manager

Table A.2.8.2 Acceptance Criteria for Method 7470A

Method	Analyte	Accuracy Aqueous (%R)	Precision Aqueous (RPD)
Mercury by Cold Vapor Atomic Absorption Spectrometry	Mercury	85-115	≤ 15

Table A.2.8.3 Summary of Calibration and QC Procedures for Method 7470A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
7470A	Mercury	Analyst Initial Demonstration.	Once per analyst	Average of 4 LCS recoveries between 85-115%. Recovery of unknown sample between 70%-130%, 50%-150% for TCLP.	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	
		Continuing Demonstration	Every 6 Months	Average of 4 LCS recoveries between 85%-115%, mb<RL, Unknown or PE.	Correct the problem.	
		MDL study.	Once every 12 months.	Analyte MDL <RL.	Correct the problem.	
		Analysis of PE sample.	Once every 12 months	Analyte results acceptable per the auditing agency.	Correct the problem.	
		Initial Calibration. Minimum of 4 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct the problem and recalibrate.	
		Initial Calibration Verification (ICV)	Daily after calibration.	Analyte recovery between 90% and 110% of the true value.	Correct the problem and recalibrate.	
		Continuing Calibration Blank (CCB).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	Analyte concentration <RL.	Correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	
		Continuing Calibration Check (CCC).	Daily after calibration, after every 10 samples, and at end of analysis sequence.	Initial analyte recovery between 90%-110%, subsequent recoveries between 80%-120%.	Correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCC.	

Modified 2/16/2005

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7470A	Mercury	Laboratory Control Sample (LCS).	Once per batch.	Analyte recovery between 85-115%.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J".
		Laboratory Control Sample Duplicate (LCSD).	Once per batch.	≤ 15 RPD.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "J".
		Matrix Blank	Once per batch.	Analyte concentration <RL.	Correct the problem, redigest, and reanalyze all samples in the batch.	If unable to re-analyze, flag with a "B".
		Matrix Spike	Every 10 samples.	Analyte recovery between 70%-130%, 50% - 150% for TCLP.	Comment sample report.	
		Matrix Spike Duplicate.	Every 10 samples.	≤15 RPD.	Comment sample report.	

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.1.1 EPA Method 504.1 - EDB and DBCP in Drinking Water****A.1.1.1 Scope and Application**

Method 504.1 is used to determine the concentrations of EDB and DBCP in extracts from drinking water. Water samples are extracted with hexane. The extract is injected into a temperature programmable gas chromatograph with an electron capture detector. Identifications are obtained by analyzing a standard curve under identical conditions used for samples and comparing results and retention times. Analytes are quantitated using procedural standard calibration.

Drinking water samples for EPA Method 504.1 are collected in clear pre-certified 40 ml vials with a Teflon lined screw cap, and preserved with 75 ul of 40mg/ml sodium thiosulfate solution. Sample vials are cooled to 4°C after sample collection. Three vials are required for each sample. Samples must be extracted within 14 days and the extracts then analyzed within 24 hours.

A.1.1.2 Calibrations and Calculations**A.1.1.2.1 Calibration Curve**

A minimum 5-point calibration is performed for all single peak components. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than 20% between calibration levels for each analyte or linear calibration using least squares regression with a correlation coefficient of 0.990 or better. An alternate source standard is used to verify initial calibration of the measurement system.

A.1.1.2.2 Calibration Standards

The calibration curve consists of the calibration standards at the following concentrations in ug/l:

NAME	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
EDB	0.01	0.02	0.06	0.10	0.20	0.40
DBCP	0.01	0.02	0.06	0.10	0.20	0.40

A.1.1.2.3 Calibration Verification

A daily continuing calibration is performed every twelve-hour analysis period to monitor and validate the instrumentation, column and detector performance.

A.1.1.2.4 Record Keeping

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

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Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.1.1.2.5 Daily Calibration Verification and Continuing Calibration

A continuing calibration standard ensures the target compound retention times and quantitation parameters meet method performance criteria. For any 12 hour analysis period, prior to sample analysis, a one point daily continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed 20% of the initial calibration. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

A.1.1.2.6 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

Equation A.1.1.1
$$RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

Equation A.1.1.2
$$\overline{RF} = \sum \frac{RF_i}{n}$$

Equation A.1.1.3
$$RSD = \frac{SD}{\overline{RF}}$$

Equation A.1.1.4
$$SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n - 1}}$$

Where:

RF = Response Factor
 RF_i = Response Factor for compound at each calibration level
 n = Number of calibration standards
 \overline{RF} = Mean Response Factor
 SD = Standard Deviation
 RSD = Relative Standard Deviation

A.1.1.2.7 Retention Time Windows

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

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The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72-hour period.

Equation A.1.1.5

$$\overline{RT} = \sum \frac{RT}{n}$$

where:

\overline{RT} = Mean retention time for target compound
 RT = Retention time for the target compound
 n = number of values

A.1.1.2.8 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % drift may be no more than " 20%.

Equation A.1.1.6

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

Equation A.1.1.7

$$\text{Concentration(water) } \mu\text{g/L} = \frac{(A_s)(D)(V_T)}{(\overline{RF})(V_s)(V_i)}$$

A.1.1.2.9 Sample Concentration

where:

A_s = Area of the peak for the analyte in the sample
 D = Dilution factor
 \overline{RF} = Mean response factor (Area per μg)
 V_i = Volume of sample injected in microliters
 V_s = Volume of original sample in liters
 V_T = Total Volume of concentrated extract in microliters

A.1.1.2.10 Linear Calibration using Least Squares Regression

Linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for the 5 points is done using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 is a perfect fit.

A.1.1.2.11 Alternate Sample Concentration Calculation using linear regression

The regression's slope and intercept terms for the linear equation is in the form:

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318Equation A.1.1.8 $y = a x + b$

y = Instrument response

a = Slope of the line

x = Concentration of Sample

b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

$$x = \frac{y - b}{a}$$

Table A.1.1.1 RLs for Method 504.1

Parameter/Method	Analyte	Matrix (WATER)	
		RL	Unit
504.1	EDB	0.022	ug/l
	DBCP	0.044	ug/l

Table A.1.1.2 Acceptance Criteria for Method EPA 504.1

Method	Analyte	Accuracy (%R)	Precision (RPD)
504.1	EDB	70-130	20
	DBCP	70-130	20
	SS: 1,2,3-Trichloropropane	70-130	

Table A.1.1.3 Summary of Calibration and QC Procedures for Method EPA 504.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
504.1	EDB& DBCP	Minimum of five-point initial calibration for all analytes	Initial calibration prior to sample analysis	Linear mean RSD for all analytes \leq 20% linear-least squares regression $r^2 > 0.980$	Correct problem then repeat initial calibration	
		Second-source Calibration verification (ICV)	Once per five-point initial calibration	All analytes within \pm 20% of expected value	Correct problem then repeat initial calibration	

Modified 1/25/2006

Georgia Department of Natural Resources
Environmental Protection Division
 EPD Laboratory, 455 14th Street, Atlanta GA 30318

		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzes since the last retention time check	
		Calibration verification (CCC)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 20\%$ of the expected value	Correct problem then repeat initial calibration	
504.1	EDB& DBCP	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria Table A.1.1.2.2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per analytical batch	No analyte detected > RL	Reextract and reanalyze batch	If unable to re-analyze, flag with a "B"
		LCS/LCSD for all analytes	One LCS/LCSD per analytical batch	QC acceptance criteria Table A.1.1.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		MS/MSD for all analytes	One MS/MSD per analytical batch	QC acceptance criteria EDB 65-135% and DBCP 65-135%	Flag report	
		Surrogate spike	Every sample, spiked sample, standard and method blank	QC acceptance criteria Table A.1.1.2	Flag Report	
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis, if used for quantitation.	
		MDL study	Once per year	Detection limits established shall be <the RLs in Table A.1.1.1	None	
		Results reported between MDL and RL	None	none	none	
		MDL Check 0.02 ug/L std	Each Sequence	60%-140% for EDB and DBCP	Correct problem and rerun	

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.1.2 EPA Method 508.1 - Chlorinated Hydrocarbon Pesticides in Drinking Water****A.1.2.1 Scope and Application**

Method 508.1 is used to determine the concentrations of various chlorinated hydrocarbon pesticides in drinking water. Samples are extracted with SDB-XC Empore disks followed by elution with methylene chloride. The extract is analyzed by injection into a temperature programmable gas chromatograph with an electron capture detector. Identifications are obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. Concentrations of the identified components are measured by relating the response produced for that compound to the standard curve response.

Drinking water samples for EPA Method 508.1 are collected in a 500 ml amber glass bottle with 25 mg Sodium Sulfite as preservative. Samples must be cooled to 4°C after collection. Samples must be extracted within 14 days, and extracts analyzed within 14 days.

A.1.2.2 Calibrations and Calculations**A.1.2.2.1 Calibration Curve**

A minimum five-point calibration is performed for all single peak components. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than 20% between calibration levels for each analyte or linear calibration, using least squares regression with a correlation coefficient of 0.990 or greater. An alternate source standard, where available, is used to verify initial calibration of the measurement system.

A.1.2.2.2 Calibration Standards

The calibration curve consists of the calibration standards at the following concentrations in ug/ml:

NAME	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Chlordane(tech)	0.08	0.2	0.4	0.5	0.6	0.8	1.0
Toxaphene	0.1	0.3	0.5	0.7	1.0	1.4	2.0
Aroclor 1016	0.1	0.2	0.4	0.5	0.8	1.0	1.2
Aroclor 1221	0.1	0.2	0.4	0.5	0.8	1.0	1.2
Aroclor 1232	0.1	0.2	0.4	0.5	0.8	1.0	1.2
Aroclor 1242	0.1	0.2	0.4	0.5	0.8	1.0	1.2
Aroclor 1248	0.1	0.2	0.4	0.5	0.8	1.0	1.2
Aroclor 1254	0.1	0.2	0.4	0.5	0.8	1.0	1.2
Aroclor 1260	0.1	0.2	0.4	0.5	0.8	1.0	1.2

A.1.2.2.3 Calibration Verification

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

A daily continuing calibration is performed every twelve-hour analysis period to monitor and validate the instrumentation, column and detector performance.

A.1.2.2.4 Record Keeping

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

A.1.2.2.5 Daily Calibration Verification and Continuing Calibration

A continuing calibration standard ensures the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12-hour analysis period, prior to sample analysis, a one point daily continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed 30% of the initial calibration. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Two different levels of calibration are alternated throughout the run. CCC is required after running the standard curve initial calibration verification.

A.1.2.2.6 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

$$\overline{RF} = \sum \frac{RF_i}{n}$$

Equation A.1.2.1

$$RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

Equation A.1.2.2

$$RSD = \frac{SD}{\overline{RF}}$$

Equation A.1.2.3

$$SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n - 1}}$$

Equation A.1.2.4

Where:

RF = Response Factor

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

RF_i = Response Factor for compound at each calibration level
 n = Number of calibration standards
 \overline{RF} = Mean Response Factor
 SD = Standard Deviation
 RSD = Relative Standard Deviation

A.1.2.2.7 Retention Time Windows

The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72 hour period.

Equation A.1.2.5

$$\overline{RT} = \sum \frac{RT}{n}$$

where:

\overline{RT} = Mean retention time for target compound
 RT = Retention time for the target compound
 n = number of values

A.1.2.2.8 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % drift may be no more than " 30%.

Equation A.1.2.6

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

A.1.2.2.9 Sample Concentration

Equation A.1.2.7

$$\text{Concentration(water)} \text{ } \mu\text{g/L} = \frac{(A_s)(D)(V_T)}{(RF)V_s(V_i)}$$

where:

A_s = Area of the peak for the analyte in the sample
 D = Dilution factor
 RF = Mean Response factor (Area per μg)
 V_i = Volume of sample injected in microliters
 V_s = Volume of original sample in liters
 V_T = Total Volume of concentrated extract in microliters

A.1.2.2.10 Linear Calibration using Least Squares Regression

Linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for the 5 points is done using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 is a perfect fit. An acceptable correlation coefficient should be ≥ 0.990

A.1.2.2.11 Alternate Sample Concentration Calculation using linear regression

The regression's slope and intercept terms for the linear equation is in the form:

$$\text{Equation A.1.2.8 } y = a x + b$$

y = Instrument response

a = Slope of the line

x = Concentration of Sample

b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

$$x = \frac{y - b}{a}$$

Table A.1.2.1 RLs for Method 508.1

Parameter/Method	Analyte	Matrix (WATER)	
		RL	Unit
508.1	Chlordane(tech)	0.80	ug/l
	Toxaphene	1.20	ug/l
	Aroclor 1016	1.0	ug/l
	Aroclor 1221	1.0	ug/l
	Aroclor 1232	1.0	ug/l
	Aroclor 1242	1.0	ug/l
	Aroclor 1248	1.0	ug/l
	Aroclor 1254	1.0	ug/l
	Aroclor 1260	1.0	ug/l

Table A.1.2.2 Acceptance Criteria for Method EPA 508.1

Method	Analyte	Accuracy (%R)	Precision (RPD)
508.1	Chlordane(tech)	70-130	30
	Toxaphene	70-130	30
	Aroclor 1016	70-130	30
	Aroclor 1221	70-130	30
	Aroclor 1232	70-130	30
	Aroclor 1242	70-130	30
	Aroclor 1248	70-130	30
	Aroclor 1254	70-130	30
	Aroclor 1260	70-130	30

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

	SS: 2,4,5,6-Tetrachloro-m-xylene	70-130	
	SS: Decachlorobiphenyl	70-130	

Modified 1/25/2006

Georgia Department of Natural Resources
 Environmental Protection Division
 EPD Laboratory, 455 14th Street, Atlanta GA 30318

Table A.1.2.3 Summary of Calibration and QC Procedures for Method EPA 508.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
508.1	Chlorinated Hydrocarbon Pesticides	Minimum five point initial calibration for all analytes	Initial calibration prior to sample analysis	Linear Mean RSD for all analytes# $\leq 20\%$ linear -least squares regression $r \geq 0.990$	Correct problem then repeat initial calibration	
		Second-source calibration verification (ICV)	Once per five-point initial calibration	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration	
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzes since the last retention time check	
		Calibration verification (CCC)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 30\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration	
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria Table A.1.2.2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per analytical batch	No analytes detected > RL	Re-extract and reanalyze batch of samples.	If unable to re-analyze, flag with a "B"
		LCS/LCSD for all analytes	One LCS/LCSD per analytical batch	QC acceptance criteria Table A.1.2.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		MS/MSD	One MS/MSD per analytical batch	RPD ≤ 30 . 65-135% recovery for all compounds.	Flag Report	
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis.	Same as for initial or primary column analysis, if used for quantitation.	
		MDL study	Once per year	Detection limits established shall be <the RLs in Table A.1.2.1	None	
		Results reported between MDL and RL	none	none	none	

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

508.1	Chlorinated hydrocarbon pesticides	DDT/Endrin Breakdown	Prior to analysis then every 12 hours before pattern profile	Degradation $\leq 20\%$	Repeat breakdown check	
		Lab Performance Check (LPC)	Once at the beginning of a run.	Lowest standard run with visual peaks to distinguish from noise.	Fix problem and repeat.	

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.1.3 EPA Method 515.1 - Chlorinated Acids in Drinking Water

A.1.3.1 Scope and Application

A.1.3.1.1 Method 515.1 is a capillary gas chromatographic method for determining chlorinated acid herbicides in drinking water. Herbicide esters and salts are hydrolyzed to their free acid. Samples are extracted with ethyl ether under acidic conditions and esterified with diazomethane. The extract is injected in a temperature programmable gas chromatograph with an electron capture detector. Identifications are obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. Concentrations of the identified components are measured by relating the response produced for that compound to the standard curve response.

Drinking water samples for EPA Method 515.1 are collected in a 500 ml amber glass bottle, with a preservative of 25 mg Sodium Sulfite. Samples must be cooled to 4⁰ C after sample collection. Samples must be extracted within 14 days and the extracts must be analyzed within 14 days

A.1.3.2 Calibrations and Calculations

A.1.3.2.1 Calibration Curve

A minimum five-point calibration is performed for all single peak components. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than 20% between calibration levels for each analyte or linear calibration using least squares regression with a coefficient of 0.990 or better. An alternate source standard, where available, is used to verify initial calibration of the measurement system.

A.1.3.2.2 Calibration Standards

The calibration curve consists of the calibration standards at the following concentrations in ug/ml:

NAME	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Dalapon	0.20	0.40	0.60	0.80	1.00	1.20	1.50
Dicamba	0.02	0.04	0.06	0.08	0.10	0.12	0.15
2,4-D	0.06	0.12	0.18	0.24	0.30	0.36	0.45
PCP	0.01	0.02	0.03	0.04	0.05	0.06	0.075
Silvex	0.06	0.12	0.18	0.24	0.30	0.36	0.45
Dinoseb	0.06	0.12	0.18	0.24	0.30	0.36	0.45
Picloram	0.02	0.04	0.06	0.08	0.10	0.12	0.15
DCPA, di-acid	0.02	0.04	0.06	0.08	0.10	0.12	0.15

A.1.3.2.3 Calibration Verification

A daily continuing calibration is performed every twelve-hour analysis period to monitor and

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

validate the instrumentation, column and detector performance.

A.1.3.2.4 Record Keeping

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

A.1.3.2.5 Daily Calibration Verification and Continuing Calibration

A continuing calibration standard ensures the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12-hour analysis period, prior to sample analysis, a one point daily continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed 20% of the initial calibration. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Two different levels of calibration are alternated throughout the run. CCC is required after running the standard curve and initial calibration.

A.1.3.2.6 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

$$\text{Equation A.1.3.1} \quad RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

$$\text{Equation A.1.3.2} \quad RSD = \frac{SD}{\overline{RF}}$$

$$\text{Equation A.1.3.3} \quad SD = \sqrt{\sum_{i=1}^n \frac{(RF_i - \overline{RF})^2}{n-1}}$$

$$\text{Equation A.1.3.4} \quad \overline{RF} = \frac{\sum RF_i}{n}$$

Where:

RF = Response Factor

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318 RF_i = Response Factor for compound at each calibration level n = Number of calibration standards \overline{RF} = Mean Response Factor SD = Standard Deviation RSD = Relative Standard Deviation

A.1.3.2.7 Retention Time Windows

The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72 hour period.

$$\overline{RT} = \sum \frac{RT}{n}$$

Equation A.1.3.5

where:

 \overline{RT} = Mean retention time for target compound RT = Retention time for the target compound n = number of values

A.1.3.2.8 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % drift may be no more than " 20%.

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

Equation A.1.3.6

A.1.3.2.9 Sample Concentration

$$\text{Concentration(water)} \text{ ug/L} = \frac{(A_s)(D)(V_T)}{(RF)V_s(V_i)}$$

Equation A.1.3.7

where:

 A_s = Area of the peak for the analyte in the sample D = Dilution factor RF = Mean Response factor (Area per μg) V_i = Volume of sample injected in microliters V_s = Volume of original sample in liters V_T = Total Volume of concentrated extract in microliters

A.1.3.2.10 Linear Calibration using Least Squares Regression

Linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for the 5 points is done using the instrument

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 is a perfect fit. An acceptable correlation coefficient should be ≥ 0.990

A.1.3.2.11 Alternate Sample Concentration Calculation using linear regression

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.1.3.8 $y = a x + b$

y = Instrument response

a = Slope of the line

x = Concentration of Sample

b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

$$x = \frac{y - b}{a}$$

Table A.1.3.1 RLs for Method 515.1

Parameter/Method	Analyte	Matrix (WATER)	
		RL	Unit
515.1	2,4-D	0.88	ug/L
	Dalapon	4.40	ug/L
	Dicamba	0.44	ug/L
	Dinoseb	0.88	ug/L
	Pentachlorophenol	0.18	ug/L
	Picloram	0.44	ug/L
	Silvex	0.88	ug/L
	DCPA, di-acid	0.13	ug/L

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.1.4 EPA Method 531.1 - N-Methyl Carbamate/Oxime Compounds in Drinking Water

A.1.4.1 Scope and Application

Method 531.1 is used to determine the concentrations of various n-methyl carbamate and oxime compounds in drinking water. Samples are hydrolyzed with sodium hydroxide to methylamine, which reacts with Thioflur to form isoindole. The analysis occurs on a high-pressure liquid chromatograph equipped with post-column derivatization and fluorescence detector. Identification is obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. Concentrations of the identified components are measured by relating the response produced for that compound to the standard curve response.

Drinking water samples for EPA method 531.1 are collected in clear 40-ml vials with 1.2ml monochloroacetic acid buffer as a preservative, added prior to shipping; then 80 ul of 40ml/ml sodium thiosulfate solution is added upon return. Samples vials must be cooled to 4°C. Samples must be analyzed within 28 days.

A.1.4.2 Calibrations and Calculations

A.1.4.2.1 Calibration Curve

A minimum five-point calibration is performed for all single peak components. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than 20% between calibration levels for each analyte or linear calibration using least squares regression with a correlation coefficient of 0.980 or better. An alternate source standard, where available, is used to verify initial calibration of the measurement system.

A.1.4.2.2 Calibration Standards

The calibration curve consists of the calibration standards at the following concentrations in ug/L:

NAME	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aldicarb	2	5	8	10	15	20	30
Aldicarb sulfone	2	5	8	10	15	20	30
Aldicarb sulfoxide	2	5	8	10	15	20	30
Baygon	2	5	8	10	15	20	30
Carbaryl	2	5	8	10	15	20	30
Carbofuran	2	5	8	10	15	20	30
3-Hydroxycarbofuran	2	5	8	10	15	20	30
Methiocarb	2	5	8	10	15	20	30
Methomyl	2	5	8	10	15	20	30
Oxamyl	2	5	8	10	15	20	30

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.1.4.2.3 Calibration Verification

A daily continuing calibration is performed every twelve-hour analysis period to monitor and validate the instrumentation, column and detector performance.

A.1.4.2.4 Record Keeping

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

A.1.4.2.5 Daily Calibration Verification and Continuing Calibration

A continuing calibration standard ensures the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12-hour analysis period, prior to sample analysis, a one-point daily continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed 20% of the initial calibration. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Two levels of calibration are alternated throughout the run. CCC is required after running the standard curve and initial calibration verification.

A.1.4.2.6 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

Equation A.1.4.1

$$RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

Equation A.1.4.2

$$RSD = \frac{SD}{\overline{RF}}$$

Equation A.1.4.3

$$SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}}$$

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

Equation A.1.4.4

$$\overline{RF} = \sum \frac{RF_i}{n}$$

Where:

RF = Response Factor
 RF_i = Response Factor for compound at each calibration level
 n = Number of calibration standards
 \overline{RF} = Mean Response Factor
 SD = Standard Deviation
 RSD = Relative Standard Deviation

A.1.4.2.7 Retention Time Windows

The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72 hour period.

Equation A.1.4.5

$$\overline{RT} = \sum \frac{RT}{n}$$

where:

\overline{RT} = Mean retention time for target compound
 RT = Retention time for the target compound
 n = number of values

A.1.4.2.8 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % drift may be no more than " 20%.

Equation A.1.4.6

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

A.1.4.2.9 Sample Concentration

$$\text{Concentration(water)} \text{ ug/L} = \frac{(A_s)(D)(V_T)}{(RF)(V_s)(V_i)}$$

Equation A.1.4.7

where:

A_s = Area of the peak for the analyte in the sample
 D = Dilution factor
 RF = Mean response factor (Area per ug)
 V_i = Volume of sample injected in microliters

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

V_s = Volume of original sample in liters

V_T = Total Volume of concentrated extract in microliters

A.1.4.2.10 Linear Calibration using Least Squares Regression

Linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for the 5 points is done using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 is a perfect fit. An acceptable correlation coefficient should be ≥ 0.980 .

A.1.4.2.11 Alternate Sample Concentration Calculation using linear regression

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.1.4.8 $y = a x + b$

y = Instrument response

a = Slope of the line

x = Concentration of Sample

b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

$$x = \frac{y - b}{a}$$

Table A.1.4.1 RLs for Method 531.1

Parameter/Method	Analyte	Matrix (WATER)	
		RL	Unit
531.1	Aldicarb	2.9	ug/L
	Aldicarb sulfone	3.2	ug/L
	Aldicarb sulfoxide	4.4	ug/L
	Baygon	2.1	ug/L
	Carbaryl	2.2	ug/L
	Carbofuran	4.2	ug/L
	3-Hydroxycarbofuran	3.7	ug/L
	Methiocarb	3.0	ug/L
	Methomyl	2.9	ug/L
	Oxamyl	2.4	ug/L

Table A.1.4.2 Acceptance Criteria for Method EPA 531.1

Modified 1/25/2006

Georgia Department of Natural Resources
Environmental Protection Division
 EPD Laboratory, 455 14th Street, Atlanta GA 30318

Method	Analyte	Accuracy (%R)	Precision (RPD)
531.1	Aldicarb	80-120	30
	Aldicarb sulfone	80-120	30
	Aldicarb sulfoxide	80-120	30
	Baygon	80-120	30
	Carbaryl	80-120	30
	Carbofuran	80-120	30
	3-Hydroxycarbofuran	80-120	30
	Methiocarb	80-120	30
	Methomyl	80-120	30
	Oxamyl	80-120	30
531.1	SS: 4-Bromo-3,5-Dimethylphenyl n-methylcarbamate	80-130	

Table A.1.4.3 Summary of Calibration and QC Procedures for Method EPA 531.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
531.1	N-Methyl Carbamate and Oxine compounds	Minimum five - point initial calibration for all analytes	Initial calibration prior to sample analysis	Linear mean RSD for all analytes 20% linear-least squares regression $r \geq 0.980$	Correct problem then repeat initial calibration	
		Second-source calibration verification (ICV)	Once per five point initial calibration	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration	
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzes since the last retention time check	
		Calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 20\%$ of expected value.	Correct problem, then repeat initial calibration	
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria Table A.1.4.2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per analytical batch	No analytes detected > RL	Reextract batch samples.	If unable to re-analyze, flag with a "B"

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

531.1	N-Methyl Carbamate and Oxine compounds	LCS/LCSD for all analytes	One LCS/LCSD per analytical batch	QC acceptance criteria Table A.1.4.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		Surrogate spike	Every sample, spiked sample, standard and method blank	QC acceptance criteria Table A.1.4.2	Flag report	
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria in Table A.1.4.2	Flag Report	
		MDL study	Once per year	Detection limits established shall be < the RLs in Table A.1.4.1	None	
		Results reported between MDL and RL	none	None	none	
		Lab performance check solution	Once at beginning of a run	Lowest standard run with visual peaks to distinguish from base line	Fix problem and repeat	

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.1.5 EPA Method 547 - Glyphosate in Drinking Water****A.1.5.1 Scope and Application**

Method 547 is used to determine the concentrations of Glyphosate in drinking water. The water sample is injected into a high-pressure liquid chromatograph with separation achieved using an isocratic elution. The analyte is oxidized to glycine with calcium hypochlorite. Glycine is then coupled with Thioflur to give a Fluorophor detected by a fluorescence detector. Identification is obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. Concentrations of the identified components are measured by relating the response produced for that compound to the standard curve response.

Drinking water samples for EPA Method 547 are collected in 40 ml vials, with 100 ul of 40 mg/ml sodium thiosulfate solution as a preservative. Sample vials must be cooled to 4°C after sample collection. Samples must be analyzed within 14 days.

A.1.5.2 Calibrations and Calculations**A.1.5.2.1 Calibration Curve**

A minimum five-point calibration is performed for all single peak components. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than or equal to 20% between calibration levels for each analyte or linear calibration using least squares regression with a correlation coefficient of 0.980 or better. An alternate source standard, where available, is used to verify initial calibration of the measurement system.

A.1.5.2.2 Calibration Standards

The calibration curve consists of the calibration standards at the following concentrations in ug/L:

NAME	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Glyphosate	10	13	20	40	60	80	100

A.1.5.2.3 Calibration Verification

A daily continuing calibration is performed every twelve-hour analysis period to monitor and validate the instrumentation, column and detector performance.

A.1.5.2.4 Record Keeping

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

A.1.5.2.5 Daily Calibration Verification and Continuing Calibration

A continuing calibration standard ensures the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12 hour analysis period, prior to sample analysis, a one point daily continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed 20% of the initial calibration. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Two levels of calibration standards are alternated throughout the run. CCC is required after running the standard curve and initial calibration verification.

A.1.5.2.6 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

$$\text{Equation A.1.5.1} \quad RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

$$\text{Equation A.1.5.2} \quad RSD = \frac{SD}{RF}$$

$$\text{Equation A.1.5.3} \quad SD = \sqrt{\sum_{i=1}^n \frac{(RF_i - \overline{RF})^2}{n-1}}$$

$$\text{Equation A.1.5.4} \quad \overline{RF} = \frac{\sum RF_i}{n}$$

Where:

RF = Response Factor
 RF_i = Response Factor for compound at each calibration level
 n = Number of calibration standards
 \overline{RF} = Mean Response Factor
 SD = Standard Deviation

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

RSD = Relative Standard Deviation

A.1.5.2.7 Retention Time Windows

The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72-hour period.

Equation A.1.5.5

$$\overline{RT} = \sum \frac{RT}{n}$$

where:

\overline{RT} = Mean retention time for target compound
 RT = Retention time for the target compound
 n = number of values

A.1.5.2.8 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The percentage drift may be no more than " 20%.

Equation A.1.5.6

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

A.1.5.2.9 Sample Concentration

Equation A.1.5.7

$$\text{Concentration(water)} \text{ } \mu\text{g/L} = \frac{(A_s)}{(RF)}$$

where:

A_s = Area of the peak for the analyte in the sample
 RF = Mean Response factor (Area per $\mu\text{g/L}$)

A.1.5.2.10 Linear Calibration using Least Squares Regression

Linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for the minimum 5 points is done using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 is a perfect fit. An acceptable correlation coefficient should be ≥ 0.980

A.1.5.2.11 Alternate Sample Concentration Calculation using linear regression

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.1.5.8 $y = a x + b$

y = Instrument response

a = Slope of the line

x = Concentration of Sample

b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

Table A.1.5.1 RLs for Method 547

Parameter/Method	Analyte	Matrix (Water)	
		RL	Unit
547	Glyphosate	13	ug/L

Table A.1.5.2 Acceptance Criteria for Method EPA 547

Method	Analyte	Accuracy (%R)	Precision (RPD)
547	Glyphosate	80-120	35

Table A.1.5.3 Summary of Calibration and QC Procedures for Method 547

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
547	Glyphosate	5 point minimum initial calibration for all analytes	Initial calibration prior to sample analysis	Linear mean RSD for all analytes $\leq 20\%$ with linear least squares regression $r \geq 0.980$	Correct problem then repeat initial calibration	
		Second source calibration verification (ICV)	Once per 5 point initial calibration	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration	
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72 hour study	Correct problem then reanalyze all samples since the last retention time check	
		Calibration verification	After every 10 samples and @ the end of the analysis sequence	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat ICV and reanalyze samples since last calibration verification	
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration	

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

		Demonstrate ability to generate acceptable accuracy and precision using 4 replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria Table A.1.5.2	Recalculate results; locate and fix problem w/system and then rerun demonstration for those analytes that did not meet criteria	
547	Glyphosate	Method blank	One per analytical batch	No analytes detected >RL	Correct problem then reprep and analyze method blank and all samples processed w/the contaminated blank	If unable to re-extract, flag samples with a "B"
		MS/MSD for all analytes	One MS/MSD per batch	QC acceptance criteria in Table A.1.5.2	Flag report if recoveries are out of acceptable range	
		LCS/LCSD for all analytes	One LCS/LCSD per batch	QC acceptance criteria in Table A.1.5.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-extract, flag samples with a "J"
		MDL study	Once per year	Detection limits established shall be < RL's in Table A.1.5.1	None	
		Results reported between MDL and RL	None	None	None	

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.1.6 EPA Method 548.1 - Endothall in Drinking Water****A.1.6.1 Scope and Application**

Method 548.1 is used to determine the concentrations of Endothall in drinking water. Samples are extracted with an anion exchange disk eluted with acidic methanol and methylene chloride. The dimethyl ester of endothall is formed by heating the extract at 50°C. After addition of salted reagent water, the ester is partitioned into methylene chloride. The extract is analyzed by injection into a temperature programmable gas chromatograph with a flame ionization detector. Identification is obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. Concentrations of identified components are measured by relating the response produced for that compound to the standard curve response.

Drinking water samples for EPA Method 548.1 are collected in a 500-ml amber glass bottle with 40 mg sodium thiosulfate as a preservative. Samples must be cooled to 4°C after sample collection. Samples must be extracted within 7 days and the extracts analyzed within 14 days.

A.1.6.2 Calibrations and Calculations**A.1.6.2.1 Calibration Curve**

A minimum five-point calibration is performed. The calibration system uses traceable certified standards. The calibration is a procedural external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than 20% between calibration levels for each analyte or linear calibration using least squares regression with a correlation coefficient of 0.980 or better. An alternate source standard is used to verify initial calibration of the measurement system.

A.1.6.2.2 Calibration Standards

The calibration curve consists of the calibration standards at the following concentrations in ug/ml:

NAME	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Endothall	1.5	3	4	5	8	10	15

A.1.6.2.3 Calibration Verification

A daily continuing calibration is performed every twelve-hour analysis period to monitor and validate the instrumentation, column and detector performance.

A.1.6.2.4 Record Keeping

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

A.1.6.2.5 Daily Calibration Verification and Continuing Calibration

A continuing calibration standard ensures the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12-hour analysis period, prior to sample analysis, a one-point daily continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed 30% of the initial calibration. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

A.1.6.2.6 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 30% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

$$\text{Equation A.1.6.1} \quad RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

$$\text{Equation A.1.6.2} \quad RSD = \frac{SD}{\overline{RF}}$$

$$\text{Equation A.1.6.3} \quad SD = \sqrt{\sum_{i=1}^n \frac{(RF_i - \overline{RF})^2}{n-1}}$$

$$\text{Equation A.1.6.4} \quad \overline{RF} = \sum \frac{RF_i}{n}$$

Where:

RF = Response Factor
 RF_i = Response Factor for compound at each calibration level
 n = Number of calibration standards
 \overline{RF} = Mean Response Factor
 SD = Standard Deviation
 RSD = Relative Standard Deviation

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.1.6.2.7 Retention Time Windows

The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72-hour period.

Equation A.1.6.5

$$\overline{RT} = \sum \frac{RT}{n}$$

where:

\overline{RT} = Mean retention time for target compound
 RT = Retention time for the target compound
 n = number of values

A.1.6.2.8 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % drift may be no more than " 30%.

Equation A.1.6.6

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

A.1.6.2.9 Sample Concentration

Equation A.1.6.7

$$\text{Concentration(water)} \text{ ug/L} = \frac{(A_s)(D)(V_T)}{(RF)V_s)(V_i)}$$

where:

A_s = Area of the peak for the analyte in the sample
 D = Dilution factor
 RF = Mean response factor (Area per μg)
 V_i = Volume of sample injected in microliters
 V_s = Volume of original sample in liters
 V_T = Total Volume of concentrated extract in microliters

A.1.6.2.10 Linear Calibration using Least Squares Regression

Linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for the minimum 5 points is done using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 is a perfect fit. An acceptable correlation coefficient should be ≥ 0.980

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.1.6.2.11 Alternate Sample Concentration Calculation using linear regression

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.1.6.8 $y = a x + b$

y = Instrument response

a = Slope of the line

x = Concentration of Sample

b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

$$x = \frac{y - b}{a}$$

Table A.1.6.1 RLs for Method 548.1

Parameter/Method	Analyte	Matrix (WATER)	
		RL	Unit
548.1	Endothall	20	ug/L

Table A.1.6.2 Acceptance Criteria for Method EPA 548.1

Method	Analyte	Accuracy (%R)	Precision (RPD)
548.1	Endothall	10-135	54
	SS: Phthalic acid	10-161	

Table A.1.6.3 Summary of Calibration and QC Procedures for Method 548.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
548.1	Endothall	5 point minimum initial calibration for all analytes	Initial calibration prior to sample analysis	Linear mean RSD for all analytes $\leq 20\%$ with linear least squares regression $r \geq 0.980$	Correct problem then repeat initial calibration	
		Second source calibration verification (ICV)	Once per 5 point initial calibration	All analytes within $\pm 30\%$ of expected value	Correct problem then repeat initial calibration	

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

548.1	Endothall	Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72 hour study	Correct problem then reanalyze all samples since the last retention time check	
		Initial calibration verification	Daily, before sample analysis	All analytes within $\pm 30\%$ of expected value	Correct problem then repeat initial calibration	
		Calibration verification	After every 10 samples and @ the end of the analysis sequence	All analytes within $\pm 30\%$ of expected value	Correct problem then repeat ICV and reanalyze samples since last calibration verification	
		Demonstrate ability to generate acceptable accuracy and precision using 4 replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria Table A.1.6.2	Recalculate results; locate and fix problem w/system and then rerun demonstration for those analytes that did not meet criteria	
		Method blank	One per analytical batch	No analytes detected >RL	Correct problem then reprep and analyze method blank and all samples processed w/the contaminated blank	If unable to re-extract, flag samples with a "B"
		MS/MSD for all analytes	One MS/MSD per batch	QC acceptance criteria in Table A.1.6.2	Flag report if recoveries are out of acceptable range	
		LCS/LCSD for all analytes	One LCS/LCSD per batch	QC acceptance criteria in Table A.1.6.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-extract, flag samples with a "J"
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria in Table A.1.6.2	Flag report	
		Second column confirmation	100% for all positive results	Same as for primary column analysis	Same as for primary column analysis if used for quantitation	
		MDL study	Once per year	Detection limits established shall be < RLs in Table A.1.6.1	None	
		Results reported between MDL and RL	None	None	None	

Modified 1/25/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.1.7 EPA Method 549.2 - Diquat in Drinking Water****A.1.7.1 Scope and Application**

Method 549.2 is used to determine the concentrations of Diquat in drinking water. The sample is extracted with a C₈ Empore disk, which has been prepared for the reversed-phase, ion-pair mode. The disk is eluted with acidic aqueous solvent. The extract is injected into a high-pressure liquid chromatograph with an ultraviolet absorbance detector. Identification is obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. The concentration of Diquat is measured by relating the response produced for Diquat to the standard curve response.

Drinking water samples for EPA method 549.2 are collected in 500-ml plastic amber bottles with 50mg sodium thiosulfate as preservative. Sample bottles must be cooled to 4°C after sample collection. Samples must be extracted within 7 days and the extracts analyzed within 21 days.

A.1.7.2 Calibrations and Calculations**A.1.7.2.1 Calibration Curve**

A minimum five-point calibration is performed. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than 20% between calibration levels for each analyte. An alternate source standard, where available, is used to verify initial calibration of the measurement system. Or linear calibration using least squares regression with a correction coefficient of 0.980 or better.

A.1.7.2.2 Calibration Standards

The calibration curve consists of the calibration standards at the following concentrations in ug/ml:

NAME	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Diquat	0.01	0.02	0.06	0.10	0.20	0.40

A.1.7.2.3 Calibration Verification

A daily continuing calibration is performed every twelve-hour analysis period to monitor and validate the instrumentation, column and detector performance.

A.1.7.2.4 Record Keeping

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

A.1.7.2.5 Daily Calibration Verification and Continuing Calibration

A continuing calibration check standard (CCC) ensures the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12-hour analysis period, prior to sample analysis, a one-point daily continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed 20% of the initial calibration. If these criteria cannot be met, the continuing calibration checks standard is reanalyzed in order to determine if the response deviations observed from the initial analysis are repeated. If the CCC meets method criteria, this time then the samples that were bracketed by the failed CCC must also be reanalyzed. On the other hand, if these criteria still cannot be met, then the instrument is considered out of calibration for those specific analytes beyond the acceptance range and the instrument needs to be recalibrated. Then the samples bracketed by the failed CCC must be reanalyzed. Two different levels of calibration standards are alternated throughout the run.

A.1.7.2.6 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

$$\text{Equation A.1.7.1} \quad RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

$$\text{Equation A.1.7.2} \quad R = \sum \frac{RF_i}{n}$$

$$\text{Equation A.1.7.3} \quad RSD = \frac{SD}{\overline{RF}}$$

$$\text{Equation A.1.7.4} \quad SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}}$$

Where:

RF = Response Factor
 RF_i = Response Factor for compound at each calibration level
 n = Number of calibration standards
 \overline{RF} = Mean Response Factor

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318*SD* = Standard Deviation*RSD* = Relative Standard Deviation

A.1.7.2.7 Retention Time Windows

The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72 hour period.

Equation A.1.7.5

$$\overline{RT} - 3 SD$$

where:

 \overline{RT} Mean
retention
time for

target compound
 RT = Retention time for the target compound
 n = number of values

A.1.7.2.8 Linear Calibration using Least Squares Regression

The instrument response (peak areas) versus the concentration of the standards for the minimum 5-point is done using the instrument data management software and the regression will produce the slope intercept terms for a linear equation. The regression calculation generates a correlation coefficient (r), which is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 being a perfect fit. An acceptable correlation coefficient should be ≥ 0.980 .

Note: the use of linear regression may not be used as rational for reporting results below the calibration range demonstrated by the analysis of the standards

A.1.7.2.9 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % drift may be no more than " 20%.

Equation A.1.7.6

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

A.1.7.2.10 Sample Concentration

Equation A.1.7.7

$$\text{Concentration(water)} \text{ ug/L} = \frac{(A_s)(D)(V_T)}{(RF)V_s(V_i)}$$

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

where:

- A_s = Area of the peak for the analyte in the sample
- D = Dilution factor
- RF = Mean Response factor (Area per μg)
- V_i = Volume of sample injected in microliters
- V_s = Volume of original sample in liters
- V_T = Total Volume of concentrated extract in microliters

A.1.7.2.11 Sample Concentration Calculation using linear regression.

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.1.7.8 $y = a x + b$

- y = Instrument response (area of peak)
- a = Slope of the line
- x = Concentration of Sample or Calibration Standard
- b = the Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

$$x = \frac{y - b}{a}$$

NOTE: The equation above is valid under the condition that the initial volume of sample extracted, the final volume of sample extract, the volume and concentration of the internal standard, plus the volume of sample extract introduced into the GC all remain constant for all samples, QC samples and standards.

Table A.1.7.1 RLs for Method 549.2

Parameter/Method	Analyte	Matrix (WATER)	
		RL	Unit
549.2	Diquat	0.88	$\mu\text{g/l}$

Table A.1.7.2 Acceptance Criteria for Method EPA 549.2

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Method	Analyte	Accuracy (%R)	Precision (RPD)
549.2	Diquat	20-126	49

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Table A.1.7.3 Summary of Calibration and QC Procedures for Method EPA 549.2

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
549.2	Diquat	Minimum Five - point initial calibration for all analytes	Initial calibration prior to sample analysis	Linear mean RSD for analyte $\pm 20\%$ linear-least squares regression $r \geq 0.980$	Correct problem then repeat initial calibration	
		Second-source calibration verification (ICV)	Once per five-point initial calibration	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration	
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzes since the last retention time check	
		Calibration verification (CCC)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	
		Initial calibration verification		All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration verification	
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria Table A.1.7.2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per analytical batch	No analyte detected > RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	If unable to re-analyze, flag with a "B"
		LCS/LCSD for all analytes	One LCS/LCSD per analytical batch	QC acceptance criteria Table A.1.7.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		MDL study	Once per year	Detection limits established shall be \leq the RLs in Table A.1.7.1	None	
		Results reported between MDL and RL	none	none	none	

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		MS/MSD for all analytes	One MS/MSD per analytical batch	QC acceptance criteria Table A.1.7.2	Flag Report	
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EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.1.14 EPA Method 8081A – Org-Cl Pesticides in Water/Solids by GC/ECD and 608-Org-Cl Pesticides in water.****A.1.14.1 Scope and Application**

Method 8081A is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices. This method is applicable to groundwater, surface water, soils, sediments and industrial waste. Water samples are extracted at neutral pH with Methylene Chloride by method 3510C. Solid samples are extracted with acetone-hexane (1:1) using Method 3541. Samples are cleaned up using Method 3620B and Method 3640A. The extract is analyzed by injection into a temperature programmable gas chromatograph with a silica capillary column and electron capture detector. Identifications are obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant GC retention times. Concentrations of the identified components are measured by relating the response produced for that compound to the standard curve response.

Water samples for semivolatile organic compounds are collected in a 1-liter narrow mouth glass bottle. Sample bottles must be cooled to 4°C after sample collection. Two to four bottles are required for each sample. Samples must be extracted within 7 days and the extracts then analyzed within 40 days.

Soil and sediment samples for semivolatile organic compounds are collected in 8 oz wide mouth glass sample bottles. Sample bottles must be cooled to 4°C after sample collection. Two to four bottles are required for each sample. Samples must be extracted within 14 days and the extracts then analyzed within 40 days.

A.1.14.2 Calibrations and Calculations**A.1.14.2.1 Calibration Curve**

A minimum five-point calibration is performed for all single peak components. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than 20% between calibration levels for each analyte. If linear regression is used to evaluate the calibration curve, a correlation coefficient r not lower than 0.990 (or coefficient of determination r^2 not lower than 0.980) is required per compound. An alternate source standard, where available, is used to verify initial calibration of the measurement system.

A.1.14.2.2 Calibration Standards

The calibration curve consists of the calibration standards at the following concentrations:
ug/ml

Name	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
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Environmental Protection Division

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α -BHC	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
β -BHC	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
δ -BHC	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
Lindane	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
Chlordane	0.10	0.50	0.80	1.0	1.2	1.5	2.0	
Gamma-Chlordane	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
alpha-Chlordane	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
p,p'-DDD	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
p,p'-DDE	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
p,p'-DDT	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
Dieldrin	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
Endosulfan I	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
Endosulfan II	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
Endosulfan Sulfate	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
Endrin	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
Endrin Aldehyde	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
Heptachlor	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
Heptachlor Epoxide	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
Toxaphene	0.10	0.50	0.80	1.0	1.2	1.5	2.0	
Chlorpyrifos	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
Hexachlorobenzene	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
Methoxychlor	0.020	0.040	0.20	0.32	0.40	0.48	0.60	0.80
Mirex	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160
Aldrin	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
SS: TCMX	0.002	0.004	0.020	0.032	0.040	0.048	0.060	0.080
SS:DCB	0.004	0.008	0.040	0.064	0.080	0.096	0.120	0.160

A.1.14.2.3 Record Keeping

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

A.1.14.2.4 Daily Calibration Verification and Continuing Calibration

A continuing calibration standard ensures the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12-hour period, prior to sample analysis, a one-point continuing calibration verification is performed. Following analysis of samples, a one-point continuing calibration verification is performed. Continuing calibration standards are analyzed during the analysis period to verify that the instrument calibration accuracy does not exceed 15%D (%D = %Drift, see sec. A.1.14.2.7) of the initial calibration. If the %D for one or more of the analytes exceeds 15%, the continuing calibration is acceptable if the mean of the %D values for all analytes in the continuing calibration standard is $\leq 15\%$ and there are no reportable concentrations of high failing compounds (+15%D, high bias) in any associated samples and no reportable analyte fails low (-15%D, low bias). If the continuing calibration does not meet method performance criteria, then the source of the failure should be determined and corrected. Recalibration may be required.

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A.1.14.2.5 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

Equation A.1.14.1
$$RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

Equation A.1.14.2
$$\overline{RF} = \sum \frac{RF_i}{n}$$

Equation A.1.14.3
$$RSD = \frac{SD}{\overline{RF}}$$

Equation A.1.14.4
$$SD = \sqrt{\sum_{i=1}^n \frac{(RF_i - \overline{RF})^2}{n - 1}}$$

Where:

- RF = Response Factor
- RF_i = Response Factor for compound at each calibration level
- n = Number of calibration standards
- \overline{RF} = Mean Response Factor
- SD = Standard Deviation
- RSD = Relative Standard Deviation

A.1.14.2.6 Retention Time Windows

The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72-hour period.

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Equation A.1.14.5

$$\overline{RT} = \sum \frac{RT}{n}$$

where:

\overline{RT} = Mean retention time for target compound
 RT = Retention time for the target compound
 n = number of values

A.1.14.2.7 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent Drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % Drift may be no more than " 15%.

Equation A.1.14.6

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

A.1.14.2.8 Sample Concentration

Equation A1.14.7

$$\text{Concentration(water)} \text{ ug/L} = \frac{(A_s)(D)(V_T)}{(RF)(V_s)(V_i)}$$

where:

A_s = Area of the peak for the analyte in the sample
 D = Dilution factor
 RF = Mean response factor (Area per ug)
 V_i = Volume of sample injected in microliters
 V_s = Volume of original sample in liters
 V_T = Total Volume of concentrated extract in microliters

A.1.14.2.9 Sample Concentration

Equation A.1.14.8

$$\text{Concentration(soil)} \text{ ug / kg} = \frac{(A_s)(D)(V_T)}{(RF)(W_s)(V_i)}$$

where:

A_s = Area of the peak for the analyte in the sample
 D = Dilution factor
 RF = Mean response factor (Area per ug)
 V_i = Volume of sample injected in microliters
 W_s = Weight of original sample in kilograms
 V_T = Total Volume of concentrated extract in microliters

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A.1.14.2.10 Alternate Sample Concentration Calculation using linear regression

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.1.14.8 $y = a x + b$

y = Instrument response

a = Slope of the line

x = Concentration of Sample

b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

$$x = \frac{y - b}{a}$$

Note: The equation above is valid under the condition that the volume of sample extract introduced into the GC all remains constant for all samples, QC samples and standards

Table A.1.14.1 RLs for Method 8081A

Parameter/Method	Analyte	Matrix (Water)		Matrix(Soil)	
		RL	Unit	RL	Unit
8081A Org-Cl Pesticides	a-BHC	0.05	ug/L	2.0	ug/kg
	b-BHC	0.06	ug/L	3.0	ug/kg
	d-BHC	0.15	ug/L	4.5	ug/kg
	Lindane	0.05	ug/L	1.0	ug/kg
	Chlordane	2.0	ug/L	50	ug/kg
	p,p'-DDD	0.10	ug/L	7.5	ug/kg
	p,p'-DDE	0.05	ug/L	3.0	ug/kg
	p,p'-DDT	0.06	ug/L	6.5	ug/kg
	Dieldrin	0.05	ug/L	2.0	ug/kg
	Endosulfan I	0.10	ug/L	5.0	ug/kg
	Endosulfan II	0.10	ug/L	7.5	ug/kg
	Endosulfan Sulfate	0.10	ug/L	8.0	ug/kg
	Endrin	0.10	ug/L	7.5	ug/kg
	Endrin Aldehyde	0.10	ug/L	3.5	ug/kg
	Heptachlor	0.05	ug/L	5.0	ug/kg
	Heptachlor Epoxide	0.05	ug/L	4.0	ug/kg
	Toxaphene	3.0	ug/L	130	ug/kg
	Chlorpyrifos	0.10	ug/L	5.0	ug/kg
	Hexachlorobenzene	0.05	ug/L	1.0	ug/kg

Modified 1/26/2006

Georgia Department of Natural Resources

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Methoxychlor	0.20	ug/L	20	ug/kg
Mirex	0.30	ug/L	3.5	ug/kg
Aldrin	0.05	ug/L	3.5	ug/kg
alpha-Chlordane	0.10	ug/L	5.0	ug/kg
gamma-Chlordane	0.10	ug/L	5.0	ug/kg

Table A.1.14.2 Acceptance Criteria for Method EPA 8081A

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)	Accuracy Soil (%R)	Precision Soil (RPD)
8081A	a-BHC	65-130	30	50-150	35
	b-BHC	65-140	30	50-150	35
	d-BHC	40-140	30	50-150	35
	Lindane	70-125	35	50-150	30
	Chlordane	60-125	30	50-120	30
	gamma-Chlordane	60-140	30	50-150	30
	alpha-Chlordane	60-140	30	50-150	30
	p,p'-DDD	70-130	30	50-150	35
	p,p'-DDE	65-135	30	50-150	30
	p,p'-DDT	50-140	30	50-150	30
	Dieldrin	75-125	30	50-150	30
	Endosulfan I	60-130	30	50-150	35
	Endosulfan II	70-135	30	50-150	35
	Endosulfan Sulfate	60-145	30	50-150	35
	Endrin	75-120	30	50-150	40
	Endrin Aldehyde	55-130	40	50-150	40
	Heptachlor	55-125	40	50-150	30
	Heptachlor Epoxide	60-135	30	50-150	30
	Toxaphene	60-140	30	50-150	35
	Chlorpyrifos	65-140	30	50-150	30
	Hexachlorobenzene	55-125	30	50-150	35
	Methoxychlor	65-135	30	50-150	40
	Mirex	60-130	30	50-150	35
	Aldrin	50-120	30	50-150	30
	2,4,5,6-TCMX	40-120	NA	45-120	NA
	Decachlorobiphenyl	40-145	NA	55-120	NA

Table A.1.14.3 Summary of Calibration and QC Procedures for Method EPA 8081A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
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8081A	Organochlorine pesticides	Minimum five-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD for all analytes $\leq 20\%$ linear-least squares regression $r \geq 0.990$	Correct problem then repeat initial calibration	
		Second-source calibration verification (ICV)	Once per five-point initial calibration	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration	
8081A	Organochlorine pesticides	Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem and verify retention times then reanalyze all samples analyzed since the last retention time check.	
		Calibration verification (CCC)	Prior to the analysis of samples, at the beginning of every 12 hour shift and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	
		Breakdown check (Endrin and DDT)	Daily prior to analysis of samples	Degradation $\leq 15\%$	Repeat breakdown check. If it still fails correct problem.	
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria Table A.1.14.2.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per analytical batch of 20 samples or less	No analytes detected $> RL$	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	If re-extract not possible flag with "B"
		LCS/LCSD for all analytes	One LCS/LCSD per analytical batch of 20 or less samples	QC acceptance criteria Table A.1.14.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria Table A.1.14.2	Flag Report	
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria Table A.1.14.2	Flag Report	
		Second-column confirmation	100% for all positive results	If used for quantitation same as for initial or primary column analysis	Same as for initial or primary column analysis	
		MDL study	Once per year	Detection limits established shall be $< RL$ s in Table A.1.14.2.1	None	
		Results reported between MDL and RL	none	None	none	

Modified 1/26/2006

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Modified 1/26/2006

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EPD Laboratory, 455 14th Street, Atlanta GA 30318**A.1.17 EPA Method 8082 - PCBs in Water and Solids by GC/ECD****A.1.17.1 Scope and Application**

Method 8082 is used to determine the concentrations of Polychlorinated Biphenyls (PCBs) as Aroclors in extracts from solid and aqueous matrices. The names PCBs and Aroclor are used interchangeable by this laboratory. This method is applicable to groundwater, surface water, soils, sediments and industrial waste. Water samples are extracted at neutral pH with Methylene Chloride by Method 3510C. Solid samples are extracted with Acetone-Methylene Chloride (1:1) using Method 3541. Samples may be cleaned up using Method 3665A when pesticides are not requested for the sample. The extract is analyzed by injection into a temperature programmable gas chromatograph with a fused silica capillary column and electron capture detector. Identifications are obtained by analyzing a standard curve of PCB 1016/1260 mixture under identical conditions used for samples and comparing resultant GC retention times. Concentrations of the identified Aroclors are measured by relating the Aroclors' response to the standard curve response.

Water samples for semivolatile organic compounds are collected in a 1-liter narrow mouth glass bottle. Sample bottles must be cooled to 4°C after sample collection. Two to four bottles are required for each sample. Samples must be extracted within 7 days and the extracts then analyzed within 40 days.

Soil, sediment and waste samples for semivolatile organic compounds are collected in 8 oz wide mouth glass sample bottles. Sample bottles must be cooled to 4°C after sample collection. Two to four bottles are required for each sample. Samples must be extracted within 14 days and the extracts then analyzed within 40 days.

A.1.17.2 Calibrations and Calculations**A.1.17.2.1 Calibration Curve**

A minimum five-point calibration is performed for a PCB 1016/1260 mixture. For all other reported PCBs, one standard of equivalent concentration to the mid-point of the PCB 1016/1260 curve is analyzed at least once under the same conditions as samples and the curve and kept on file for pattern ID purposes. If a PCB other than PCB 1016 or PCB 1260 is found, then a minimum 3-point curve will be run for that PCB. Alternately, three-point calibration of PCBs other than 1016/1260 may be performed prior to sample analysis and verified per A.1.17.2.4. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an "average of response factor linear curve fit" and should result in a percent relative standard deviation of less than 20% between calibration levels for each analyte. An alternate source standard, where available, is used to verify initial calibration of the measurement system.

A.1.17.2.2 Calibration Standards

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The calibration curve consists of the calibration standards at the following concentrations in ug/ml:

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Name	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Aroclor 1016	0.05	0.1	0.5	0.8	1.0	1.2	1.5	2.0
Aroclor 1260	0.05	0.1	0.5	0.8	1.0	1.2	1.5	2.0
SS: TCMX	0.002	0.004	0.02	0.032	0.04	0.048	0.06	0.08
SS: DCB	0.004	0.008	0.004	0.064	0.08	0.096	0.12	0.16

A.1.17.2.3 Record Keeping

Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

A.1.17.2.4 Daily Calibration Verification and Continuing Calibration

A continuing calibration standard ensured the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12-hour period, prior to sample analysis, a one-point continuing calibration verification of the PCB 1016/1260 mixture is performed. Additionally, verification of current calibration of other PCBs may be performed. For any instrument sequence, one-point daily calibration verification is performed prior to sample analysis and at the end of the sequence rotating through the additional calibrated PCBs. If a PCB other than 1016 or 1260 is found, a continuing calibration verification standard of that specific PCB must be analyzed within 48 hours of that sample. Continuing calibration standards are analyzed during the analysis period to verify that the instrument calibration accuracy does not exceed 15%D (%D= % Drift, see section A.1.17.2.7) the %D for one or more analytes exceeds 15%, the continuing calibration is acceptable if the mean of the %D values for all analytes in the continuing calibration standard is ≤15% and there are no reportable concentrations of failing compounds in any associated samples. If the continuing calibration does not meet method performance criteria, then the instrument must be recalibrated.

A.1.17.2.5 Evaluation of the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean response factor (\overline{RF}), the standard deviation (SD) and the relative standard deviation expressed as a percentage. If the RSD of the calibration or response factors is less than 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

$$\text{Equation A.1.17.1} \quad RF = \frac{\text{Peak area of the compound standard}}{\text{micrograms of the compound injected}}$$

$$\text{Equation A.1.17.2} \quad \overline{RF} = \sum \frac{RF_i}{n}$$

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Equation A.1.17.3

$$RSD = \frac{SD}{\overline{CF}}$$

Equation A.1.17.4

$$SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}}$$

Where:

RF = Response Factor
 RF_i = Response Factor for compound at each calibration level
 n = Number of calibration standards
 \overline{RF} = Mean Response Factor
 SD = Standard Deviation
 RSD = Relative Standard Deviation

The instrument responses versus the concentrations of the standards for the 5 points is done using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 is a perfect fit. 8082 requires a fit of 0.990 or better for r (0.980 or better for r²)

A.1.17.2.6 Retention Time Windows

The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as " 3 times the standard deviation of the mean absolute retention time established over a 72 hour period.

Equation A.1.17.5

$$\overline{RT} \pm \sigma RT$$

where:

\overline{RT} Mean
 retention
 time for

target compound
 RT = Retention time for the target compound
 n = number of values

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A.1.17.2.7 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of percent Drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % Drift may be no more than " 15%.

$$\text{Equation A.1.17.6} \quad \% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100$$

A.1.17.2.8 Sample Concentration

$$\text{Equation A.1.17.7} \quad \text{Concentration(water)} \text{ ug/L} = \frac{(A_s)(D)(V_T)}{(RF)(V_s)(V_i)}$$

where:

- A_s = Area of the peak for the analyte in the sample
- D = Dilution factor
- RF = Mean response factor (Area per ug)
- V_i = Volume of sample injected in microliters
- V_s = Volume of original sample in liters
- V_T = Total Volume of concentrated extract in microliters

A.1.17.2.9 Sample Concentration

$$\text{Equation A.1.17.8} \quad \text{Concentration(soil)} \text{ mg / kg} = \frac{(A_s)(D)(V_T)}{(RF)(W_s)(V_i)}$$

where:

- A_s = Area of the peak for the analyte in the sample
- D = Dilution factor
- RF = Mean response factor (Area per ug)
- V_i = Volume of sample injected in microliters
- W_s = Weight of original sample in kilograms
- V_T = Total Volume of concentrated extract in microliters

$$\text{Equation A.1.17.9} \quad \text{Concentration(waste)} \text{ mg / kg} = \frac{(A_s)(D)(V_T)}{(RF)(W_s)(V_i)}$$

Modified 1/26/2006

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where:

- A_s = Area of the peak for the analyte in the sample
 D = Dilution factor
 RF = Mean response factor (Area per ug)
 V_i = Volume of sample injected in microliters
 W_s = Weight of original sample in kilograms
 V_T = Total Volume of concentrated extract in microliters

A.1.17.2.10 Alternate Sample Concentration Calculation using linear regression

The regression's slope and intercept terms for the linear equation is in the form:

Equation A.1.17.10 $y = a x + b$ y = Instrument response a = Slope of the line x = Concentration of Sample b = Intercept

To use the equation to calculate sample concentrations, the equation is rearranged:

$$x = \frac{y - b}{a}$$

Note: The equation above is valid under the condition that the volume of sample extract introduced into the GC all remain constant for all samples, QC samples and standards.

Table A.1.17.1 RLs for Method 8082

Parameter/Method	Analyte	Matrix (Water)		Matrix (Soil)	
		RL	Unit	RL	Unit
8082	Aroclor 1016	1.0	ug/L	33	ug/kg
	Aroclor 1221	1.0	ug/L	33	ug/kg
	Aroclor 1232	1.0	ug/L	33	ug/kg
	Aroclor 1242	1.0	ug/L	33	ug/kg
	Aroclor 1248	1.0	ug/L	33	ug/kg
	Aroclor 1254	1.0	ug/L	33	ug/kg
	Aroclor 1260	1.0	ug/L	33	ug/kg
	Aroclor 1262	1.0	ug/L	33	ug/kg

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Table A.1.17.2 Acceptance Criteria for Method EPA 8082

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)	Accuracy Soil (%R)	Precision Soil (RPD)
8082	Aroclor 1016	65-130	30	55-120	30
	Aroclor 1260	70-125	30	65-120	30
	SS: 2,4,5,6-Tetrachloro-m-xylene	50-120	N/A	60-120	N/A
	SS: Decachlorobiphenyl	65-125	N/A	65-120	N/A

Table A.1.17.3 Summary of Calibration and QC Procedures for Method EPA 8082 Water & Soil

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
8082	PCBs	Minimum five-point initial calibration for PCBs 1016/1260 TCMX and DCB, Minimum three point for other PCBs.	Initial calibration prior to sample analysis	RSD for all analytes $\leq 20\%$ linear -least squares regression $r \geq 0.990$	Correct problem then repeat initial calibration	
		Second-source calibration verification PCBs (ICV)	Once per five or three-point initial calibration	All analytes within $\pm 20\%$ of expected value	Correct problem then repeat initial calibration	
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check. redo retention time study	
		Calibration verification for PCB 1016/1260 mix (CCC) and for other PCBs with calibration and reanalysis after OCB is identified	Prior to the analysis of samples, at the beginning of every 12 hour shift and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	
		Calibration verification for other PCBs	Optional: at the beginning and end of instrument sequence (rotating through PCBs) and within 48 hours of found PCB	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	

Modified 1/26/2006

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8082	PCBs	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria Table A.1.17.2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per analytical batch of 20 samples or less	No analytes detected >RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	If re-extract not possible flag with "B"
		LCS/LCSD for all analytes	One LCS/LCSD per analytical batch of 20 or less samples	QC acceptance criteria Table A.1.17.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria Table A.1.17.2	Flag Report	
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria Table A.1.17.2	Flag Report	
		Second-column confirmation	100% for all positive results (confirmation must be within 40% of primary quantitation)	If used for quantitation same as for initial or primary column analysis	Same as for initial or primary column analysis	
		MDL study	Once per year	Detection limits established shall be <the RLs in Table A.1.17..1	None	
		Results reported between MDL and RL	none	none	none	