

**SITE INSPECTION
SAMPLING PLAN**

SITE: Seven Out LLC
BREAK: 1.9
OTHER: no

**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**

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CONTENTS

1.0	INTRODUCTION	4
2.0	BACKGROUND	4
3.0	SITE DESCRIPTION AND HISTORY	5
3.1	Site History	5
3.2	Waste Characterization	7
4.0	PATHWAYS	10
4.1	Air Migration	10
4.2	Ground Water Pathway	10
4.3	Soil Exposure Pathway	12
4.4	Surface Water Pathway	14
5.0	SAMPLING ACTIVITIES	14
5.1	Surface Water/Sediment Sampling	16
5.2	Groundwater Sampling	16
5.3	Soil Sampling	17
5.4	Data Quality Assurance/Objectives	17
5.5	Field Activities	18
5.6	Quality Control Procedures	18
6.0	INVESTIGATORY DERIVED WASTE PLAN	18
7.0	PROJECT MANAGEMENT	18
7.1	Health and Safety	19
7.2	Project Schedule	19
7.3	Site Access	19
8.0	REFERENCES	20

TABLES

Table 1	Parcels Associated with Seven Out at 901 Francis Street	4
Table 2	Constituents Detected in Samples Taken from From On-Site Containers at Seven Out	6
Table 3	Qualifying Releases to Soil at Seven Out	8
Table 4	Identified Source Areas at Seven Out	9
Table 5	Population Within 4-Mile Radius of Seven Out	10
Table 6	Groundwater Users Within 4-Mile Radius of Seven Out	12
Table 7	Nearby Resident Population to Seven Out	14
Table 8	Proposed Samples	15
Table 9	EPD DQO for Seven Out	17
Table 10	Personnel Associated with Seven Out Site Inspection	19

FIGURES (in back)

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

APPENDICES (in back)

Appendix A	List of Analytes
Appendix B	EPD Laboratory - Quality Assurance/Quality Control
Appendix C	Seven Out Safety and Health Plan
Appendix D	Site Access Authorizations
Appendix E	References

PHOTOGRAPHS (see Reference 4)

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) will complete a Site Inspection (SI) Report for the site known as Seven Out LLC Tank located in Waycross, Ware County, Georgia (Reference 1).

2.0 BACKGROUND

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).

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 6).

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 6)

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, 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 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 6). 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).

3.0 SITE HISTORY AND WASTE CHARACTERIZATION

3.1 Site 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 7), a hay storage warehouse in 1908 (Reference 8), the W.N. Gramling Wholesale Grocer Warehouse in 1913 (Reference 9) and the John D. Hopkins Hay and Grain facility in 1922 (Reference 10). Some of these buildings were enlarged prior to 1922 to form the Hopkins Hay and Grain facility (References 9 and 10). In 1930, a grocery was added to the John D. Hopkins Hay and Grain (Reference 11). The 1930/1947 maps indicate occupation of the site by a wholesale hay, grain, and grocery (Reference 12). 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 13). 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 9 and 10). 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 14). 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 15). 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 16). 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 17). 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 18). These violations of the facility's discharge permit resulted in a Consent Order issued by the City on January 27, 2004 (Reference 19). 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 20). 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 21). 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 22). 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 23). EPD inspected the site and took soil samples from the spill area on June 23, 2004 (Reference 24). 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 25). EPD received responses from representatives of the facility, however the Consent Order was never signed by the facility (Reference 26). 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. A summary of the sampling that identifies the constituents that were detected in those containers is provided in Table 2.

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

Constituent	Highest Concentration	Container ID
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 8)

Complete sampling results are included in the Removal Assessment Report prepared by Tetra Tech (References 14 and 27).

EPD requested assistance from EPA with the remediation of the facility by letter dated January 21, 2005 (Reference 28). The EPA initiated removal operations at the facility on January 28, 2005 (Reference 29) and removal activities continued until EPA left the site on June 10, 2005 (References 30 and 31). 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 32).

3.2 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 handled 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 9). 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 33). 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 9 and 10). Foundries operated during the time period of the Gillon Machine Shop and Foundry generally were cupola foundries (Reference 34). 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 8, 9, and 10). 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, undocumented removal action was completed at the frac tank area by CSX Railroad. The frac tanks have been removed and some soils have been removed, however, no documentation could be obtained to document the removals. With the exception of the frac tank area, those contaminants already identified as having qualified releases to soil are listed in the following table. No soil removals have occurred in the drainage ditch or along the south wall of the secondary containment areas (References 3 and 4).

Table 3 – Qualifying Releases to Soil at Seven Out LLC Tank

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
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)				
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
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 8)

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).

Table 4 – Identified Source Areas at the Seven Out

Area	Description	Secondary Containment	Area/Volume	Included as a Source Area	Source Area ID#
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	Source 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	Source No. 2
Soil at Tank Farm south wall.	Contaminated Soil outside of south wall of Tank Farm.	Not Present	22' X 50' Soil area is approximately 1,100 square feet.	Yes	Source No. 3

Area	Description	Secondary Containment	Area/Volume	Included as a Source Area	Source Area ID #
Drainage Ditch.	Contaminated Soil in Drainage Ditch south of Tank Farm.	Not Present	50' X 1' Soil/ sediment area is approximately 50 square feet	Yes	Source No. 4

(References 3 and 4)

Although over 300,000 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.

4.0 PATHWAYS

4.1 Air Migration

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 (Reference 4). 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 5 (Reference 35). No primary targets were identified. The surrounding residents constitute a secondary target. No release into the air is suspected at the Seven Out LLC Tank site.

Table 5 – 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 35)

4.2 Groundwater Pathway

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 36). 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 37).

The site is underlain by Pleistocene and Pliocene sands and gravels, primarily associated with the Brandywine, Coharie, and Suderland 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 38).

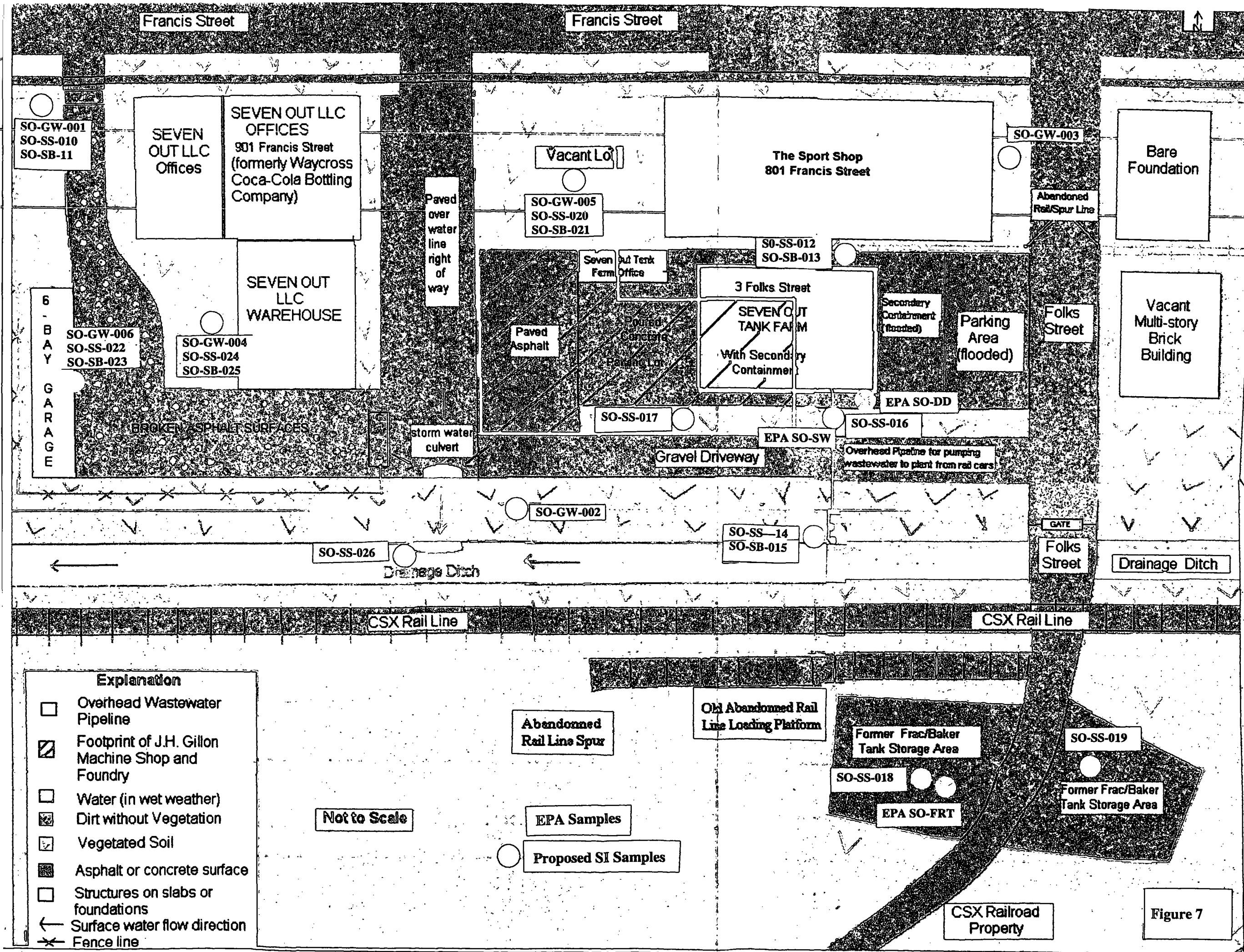
The sources at the site were either relatively small (contaminated soils in the frac tank area and in the area behind the secondary containment berm) or were primarily contained in a concrete secondary containment system (References 4, 14, 27). As evidenced during rainfall during the VSI for the previous PA reconnaissance, infiltration rate is not high and the subsurface to a great depth does not appear to be highly permeable (Reference 39). 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 40).

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 41 and 42). 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 43). 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 44 and 45). 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 46).

The Seven Out LLC Tank site lies within an area determined to have a higher (Drastic Rating > 141) groundwater pollution susceptibility rating (Reference 47). The site is not located within a significant groundwater recharge area (Reference 48).

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 1/4-1/2 mile radius, Satilla Regional Water and Sewer Authority has three (3) wells, and the Waycross/Ware County Industrial Park has one (1) well. 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 44, 45, and 49). Additional wells include numerous residential wells located within the four (4) mile radius (References 35 and 50). Table 6 provides a complete listing of groundwater wells and user population identified within the 4-mile radius. Figure 3 shows the 4-



mile radius from the site with the major groundwater wells identified (References 35, 44, 45, 49, and 51).

Table 6 – 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 35, 44, 45, 49, 50)

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 wells that serve 19,114 people are within 0.25-0.50 mile from the site. 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. The Coca Cola Bottling Company well identified on the Well Survey Maps is no longer in service (References 35 and 52).

4.3 Surface Water Pathway

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 53). 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 4 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 5). 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 54). Figure 5 traces the 15-mile surface water migration pathway TDL from the PPE (Reference 54). 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 6)(Reference 55). 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 56). Rainfall in the region averages 51 inches annually with average annual runoff at 11 inches (Reference 57).

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 58 and 59). 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 60).

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, 49 and 54). 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 61, 62, and 63). 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 4 and 61). 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 38). From this point downstream the remaining 12 miles are bordered almost continuously by wetlands (Figure 6)(Reference 38).

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 is proposed.

4.4 Soil Exposure Pathway

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 64). 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 39).

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 14). 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 (References 4 and 6).

Nearby resident populations, as provided by the Census of Population and Housing, 2000 (Reference 35) are summarized in Table 7. Approximately 5,498 people live within one mile of the site (Reference 35).

Table 7. Nearby Resident Population to Seven Out

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

(Reference 35)

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 65 and 66). These species have not been observed in habitat at the site (Reference 4).

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 is warranted to better define the areas of contamination on all parcels of property relevant to this SI.

5.0 SAMPLING ACTIVITIES

All sampling activities will adhere to the November 2001 edition of the EPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual

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

August 1, 2006

VIA MAIL COURIER

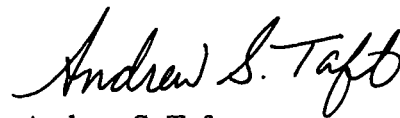
Ms. Carolyn Callihan
Superfund Site Assessment Manager
U.S. Environmental Protection Agency
Waste Management Division
Superfund Site Evaluation Section
61 Forsyth Street, SW
Atlanta, Georgia 30303-3104

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

Dear Ms. Callihan:

Enclosed you will find a Site Inspection (SI) Sampling Plan that has been completed for the above referenced site. Should you have any questions or comments regarding the SI Sampling Plan, 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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found @ <http://www.epa.gov/region4/sesd/eisopqam/eisopqam.html>. All background samples will be collected first in each medium to prevent cross contamination.

The EPD Laboratory located in Atlanta, Georgia will conduct all analyses. At a minimum, the sampled media will be analyzed for those compounds and analytes included in the Contract Laboratory Program (CLP) Target Compound List (TCL) and Target Analyte List (TAL) found at <http://www.epa.gov/superfund/programs/clp/target.htm>. For this investigation, analyses for pesticides/pesticides and dioxins/furans will not be included. See Appendix A for TCL/TAL lists applicable to this SI. Table 8 depicts the number of proposed samples for each media.

Table 8. Proposed Samples

MEDIA	SAMPLE #	SAMPLING JUSTIFICATION
Shallow Groundwater	SO-GW-001	Background sample taken from off-site, preferably north or south of site.
Shallow Groundwater	SO-GW-002	On-site sample taken on tank farm site to determine if a release to the shallow groundwater has occurred.
Shallow Groundwater	SO-GW-003	On-site sample taken on tank farm site to establish if a release to the shallow groundwater has occurred.
Shallow Groundwater	SO-GW-004	On-site sample taken on facility storage site to establish if a release to the shallow groundwater has occurred.
Shallow Groundwater	SO-GW-005	On-site sample taken on facility former foundry site to establish if a release to shallow groundwater has occurred
Shallow Groundwater	SO-GW-006	On-site sample taken on facility former laundry site to establish if a release to shallow groundwater has occurred.
Shallow Groundwater	SO-GW-007	Residential well sample taken at nearby residence to establish if a target well has been impacted.
Shallow Groundwater	SO-GW-008	Residential well sample taken at other nearby residence to establish if a target well has been impacted.
Deep Groundwater	SO-GW-009	Municipal well sample taken at one of wells within ¼-1/2 mile radius to establish if a potential target well has been impacted.
Surface Soil (0-2')	SO-SS-10	Background surface sample from area north of the facility
Subsurface Soil (3-5')	SO-SB-11	Background subsurface sample from area north of the facility
Surface Soil (0-2')	SO-SS-12	Surface sample taken from north of secondary containment where containment was breached to identify a source area.
Subsurface Soil (3-5')	SO-SB-13	Subsurface sample taken from north of secondary containment where containment was breached to identify a source area.
Surface Soil (0-2')	SO-SS-14	Surface sample taken in area of waste/wastewaterwater pump station near railroad tracks to identify a source area.

MEDIA	SAMPLE #	SAMPLING JUSTIFICATION
Subsurface Soil (3-5')	SO-SB-15	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	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	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	Surface sample taken in area of frac tank spill to identify a source area.
Surface Soil (0-2')	SO-SS-19	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	Surface sample taken in area of former foundry to identify a source area.
Subsurface Soil (3-5')	SO-SB-21	Subsurface sample taken in area of former foundry to identify a source area.
Surface Soil (0-2')	SO-SS-22	Surface sample taken in area of former laundry to identify a source area.
Subsurface Soil (3-5')	SO-SB-23	Subsurface sample taken in area of former laundry to identify a source area.
Surface Soil (0-2')	SO-SS-24	Surface sample taken in area of waste storage facility to identify a source area.
Subsurface Soil (3-5')	SO-SB-25	Subsurface sample taken in area of waste storage facility to identify a source area.
Surface Soil (0-2')	SO-SS-26	Surface sample taken in area of culvert drainage pipe discharge to ditch to identify a source area.
Surface Soil (0-2') Duplicate	SO-SS-Duplicate	Duplicate of Surface Soil Sample
Subsurface Soil (0-2') Duplicate	SO-SB-Duplicate	Duplicate of Subsurface Soil Sample
Groundwater Duplicate	SO-GW-Duplicate	Duplicate of Groundwater Sample

Figure 7 shows the proposed soil and groundwater sampling locations.

5.1 Surface Water Sampling

No sampling of the surface water pathway is proposed for this site.

5.2 Groundwater Sampling

Groundwater sampling of the shallow aquifer is planned for this site. Five shallow (20-25') Geoprobe groundwater samples will be attempted on the parcels at this site. Shallow groundwater samples will be attempted at five locations. Three (3) of those locations will be where subsurface soil samples are proposed. These will attempt to determine if a release to shallow groundwater has occurred on the site. One background sample will be taken. At least one residential well, located within two blocks of the site will be sampled. Records on this well

indicate that the well is completed in the shallow aquifer. Should an additional shallow well be located during the sampling event, that well will also be sampled. Additionally, at least one City of Waycross municipal well will be sampled.

5.3 Soil Sampling

Soil sampling is planned for this site. At some of the locations, a surface (0-2') sample and a subsurface (3-5') sample will be taken. Ten (10) surface soil samples and five (5) subsurface soil samples will be taken. The sample locations will be selected based on soil discoloration, waste overflow areas, drainage discharge areas, and suspected waste generation areas. Sample locations will include the drainage ditch, the pump station, the secondary containment overflow area, the waste facility storage area, the former foundry area, the former laundry area, and the frac tank area.. An additional background surface soil sample and a background subsurface soil sample will be taken.

5.4 Data Quality Assurance/Objectives

Quality assurance and quality control (QA/QC) for this sampling event will be provided by collecting duplicate samples. One duplicate sample for each matrix sample will test the reliability of sampling procedures and results. Appropriate trip blanks and rinsate blanks, if decontamination is required, will be taken. All sample collection, preservation, QA/QC preparation of duplicates and chain-of-custody procedure used during the sampling activities will be in accordance with the standard operating procedures specified in the latest version of EPA Region IV SOP/QAM. The EPD Laboratory QA/QC documentation for VOCs, SVOCs, and metals analyses is attached in Appendix B. The data quality objectives are listed below in Table 9.

Table 9. EPD DQO for Seven Out

DQO Seven Steps		Relevant Section(s)
1. State the Problem The contamination problem is summarized in Sections 3.1 and 3.2. Personnel and additional resources for this investigation are outlined in Section 7.0		3.1, 3.2, 7.0
2. Identify the Decision The principal objective of this investigation is to provide analytical data to make site management decisions and possibly complete a Hazard Ranking System (HRS) score. Samples from relevant pathways will be taken to document contaminant migration and define waste sources.		1.0, 4.0
3. Identify Inputs to the Decision Environmental samples will be collected to and will provide analytical data for making site management decisions. Samples will be collected as noted in Section 5.0. Analytical methods appropriate to provide necessary data for this investigation are discussed in 5.0		5.0
4. Define the Study Boundaries Sampling activities have been scheduled as discussed in Section 7.2. Site characteristics and targeted populations are discussed in subsections of Section 4.0.		4.0, 7.2
5. Develop a Decision Rule Analytical detection limits have been chosen to provide data sufficient for site management decisions. This data will be applied to individual pathways of scoring scenarios and other pre-remedial concerns. Statistical based sampling does not apply to this investigation.		5.0
6. Specify Limits on Decision Does not apply because statistical sampling is not part of this investigation		-
7. Optimize the Design Approval by USEPA Region IV Remedial Project Managers is required prior to resource allocation		-

5.5 Field Activities

Field personnel are scheduled to collect samples at the site the week of August 28 – September 1, 2006. Site access will be obtained by EPD prior to the sampling date. All environmental samples and non-sampling information will be collected during that week. Field personnel are scheduled to complete the sampling activities and leave the site by September 1, 2006.

Exact sample locations will be chosen on or prior to the initial sampling. The proposed sample locations are based on previous visits to the site in 2006. Upon verifying the sample locations with all the involved personnel and after any modifications are determined, the sampling will begin. Photographs will document all sample locations, and GPS coordinates will be taken at each sample location for further documentation. All EPD personnel participating in the sampling activity are properly OSHA certified. If required, proper site restrictions will be implemented. Sampling will be done in level D, with exceptions when needed.

5.6 Quality Control Procedures

All sampling will be performed with dedicated sampling equipment. All samples will be stored in coolers on ice and, if required, preserved until they are delivered to the Georgia EPD Laboratory. Chain-of-Custody will be maintained according to Section 4.2 of the Georgia EPD Standard Operating Procedures by field personnel until samples are handed over to the Georgia EPD Laboratory. Copies of the EPD Laboratory Quality Control Procedures are located in Appendix B.

6.0 INVESTIGATORY DERIVED WASTE PLAN

Disposable sampling equipment and personal protective equipment (PPE) will be bagged or containerized on-site and returned to the EPD Laboratory for disposal. Reusable sampling equipment will be cleaned in the field with detergent and water to remove residuals and then transported to the EPD Laboratory for further decontamination. Decontamination wastewater and purge water will be containerized and returned to the site. A sample from each container may be obtained if necessary to determine the proper disposition of the wastewaters. The container(s) will be properly labeled and left on site until analytical results are received. Once wastewaters have been characterized, proper disposal will occur in accordance with applicable federal, state and local regulations.

7.0 PROJECT MANAGEMENT

The Project Leader for the Seven Out LLC Tank Site Inspection will be Eddie Williams, Advanced Geologist. The Project Leader will schedule field activities, personnel requirements, obtain site access authorization, direct and oversee all onsite and offsite activities associated with the investigation. The team will document and manage all collected samples. Table 10 identifies all the field personnel that will be performing any field activities pertaining to the Seven Out LLC Tank Site Inspection.

Table 10. Personnel Associated with the Seven Out Site Inspection Sampling Event

PERSONNEL	JOB TITLE	SITE RESPONSIBILITIES
Eddie Williams	Geologist	Coordinate all sampling activities.
Billy Hendricks	Environmental Specialist	Assist in team sampling activities.
Fred Rowe	Engineer	Assist in team sampling activities.
Brett Blackwelder	Engineer	Assist in team sampling activities.
Daniel Braverman	Engineer	Assist in team sampling activities.
Bruce Khaleghi	Unit Coordinator	Transport samples

Eddie Williams is the qualified Geoprobe operator. He will be in charge of equipment operation during the sampling event. He has approximately ten (10) years experience taking soil and groundwater samples with the Geoprobe.

7.1 Health and Safety

Field protection will be in Level D during the site visit. Level D includes work boots, disposable gloves, hardhats, and earplugs (if required). Disposable gloves will be worn during all sampling events and during decontamination activities. All personnel involved in the Seven Out LLC Tank site sampling event will be OSHA certified.

Local emergency numbers are as follows:

- Waycross Police Department (912) 287-2921
- Waycross Fire Department (912) 287-2937
- Satilla Regional Medical Center (912) 283-3030

The Seven Out Health and Safety Plan is included in Appendix C.

7.2 Project Schedule

Fieldwork will take place during the week of August 28, 2006. When the field tasks are completed, preparation of the Site Investigation Report will begin. Upon receipt of the analytical results, the scoring package for the site will be completed. The Final Site Inspection Report will be completed and submitted to EPA as required.

7.3 Site Access

Authorization for site access will be the responsibility of the Project Leader, Eddie Williams. Verbal or written authorization will be obtained prior to sampling on the associated Seven Out parcels. Access authorizations that had been obtained at the time of the submittal of this sampling plan are included in Appendix D.

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FIGURES

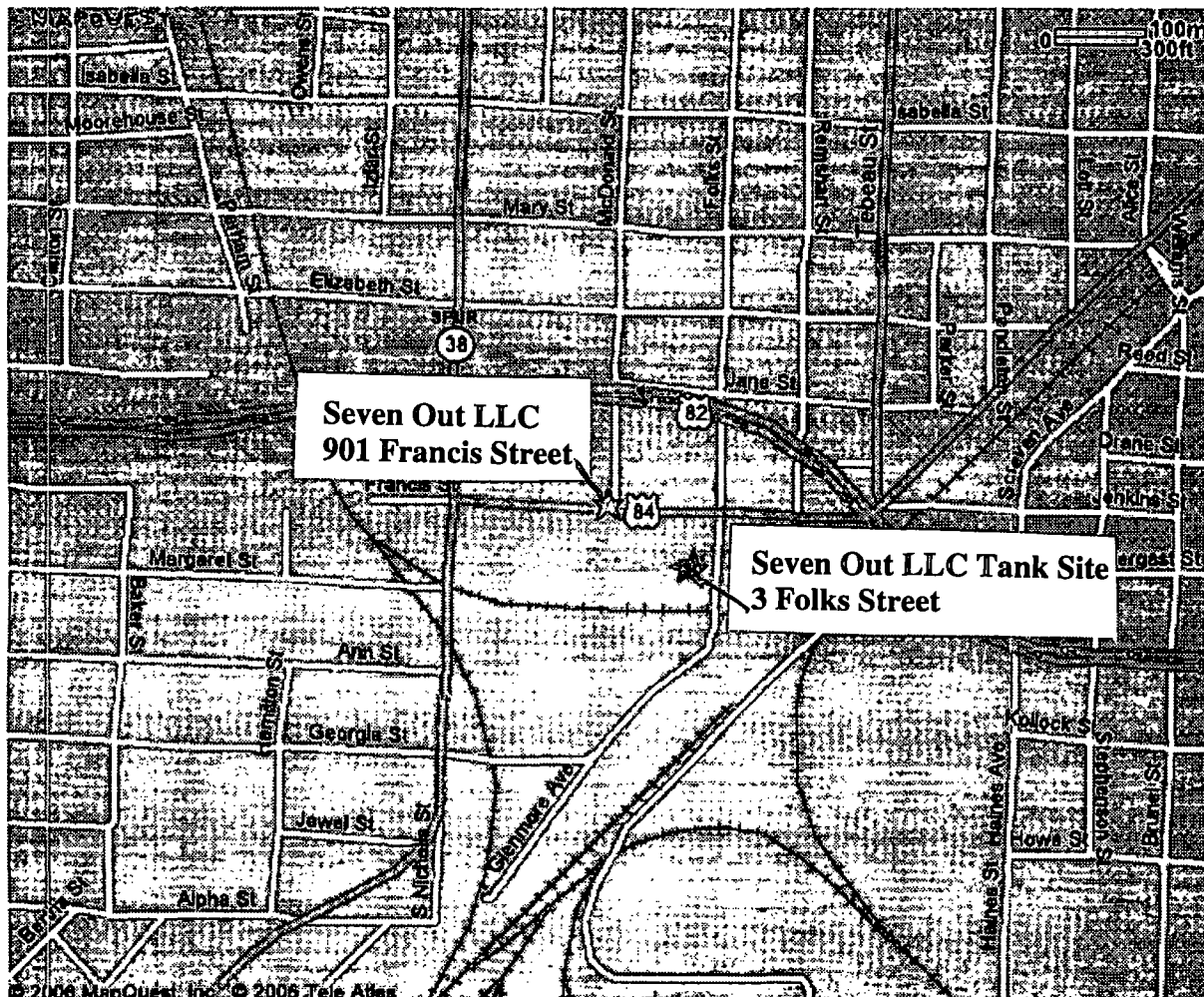
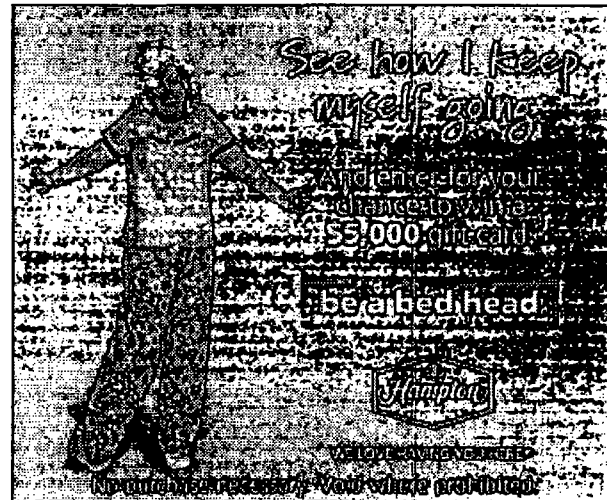
MAPQUEST.

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

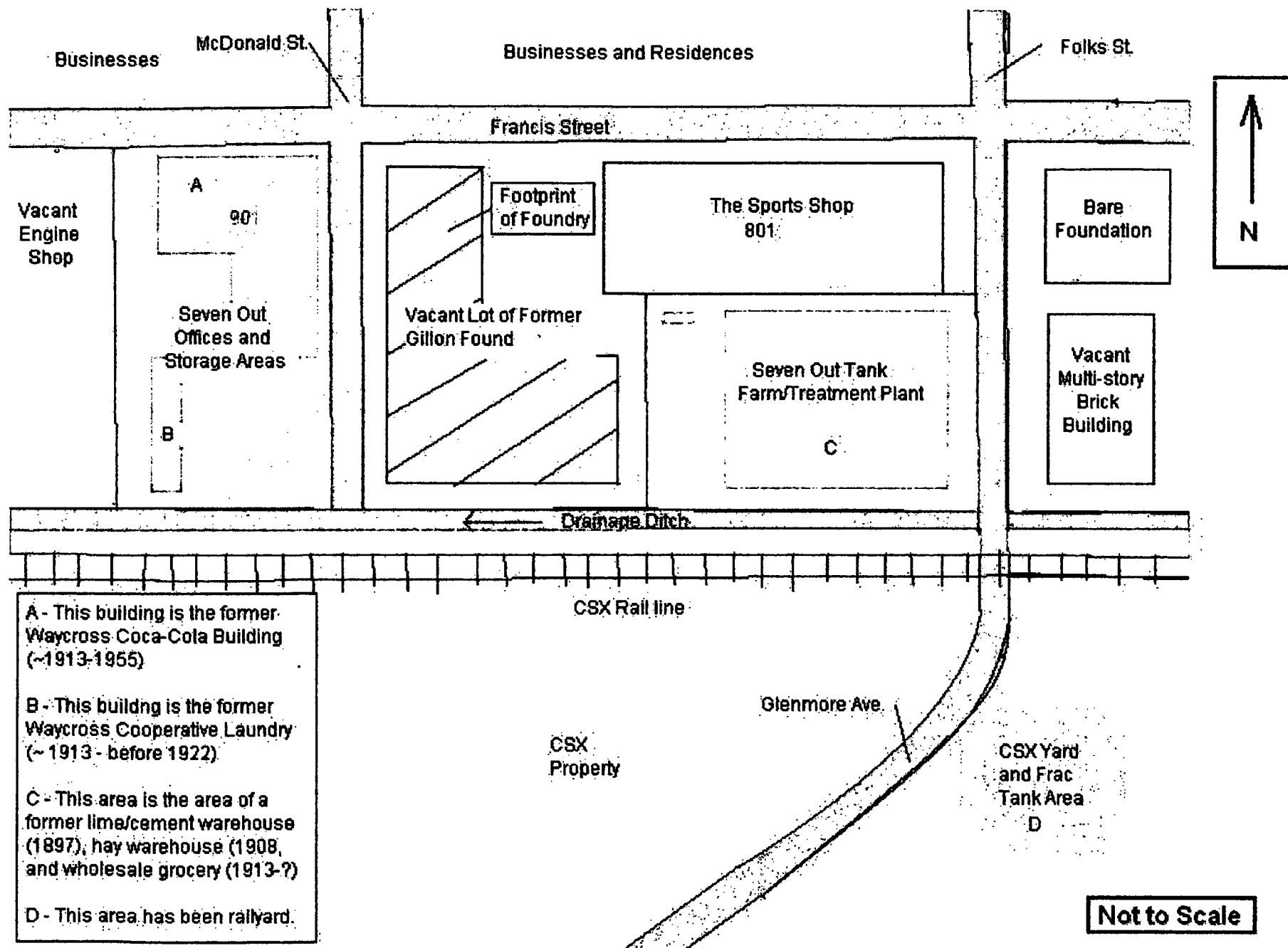


Figure 2

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

SDMS

Unscannable Material Target Sheet

DocID: 10508637 Site ID: GAL000407811

Site Name: James Out LLC

Nature of Material:

Map: ✓

Computer Disks:

Photos:

CD-ROM:

Blueprints:

Oversized Report:

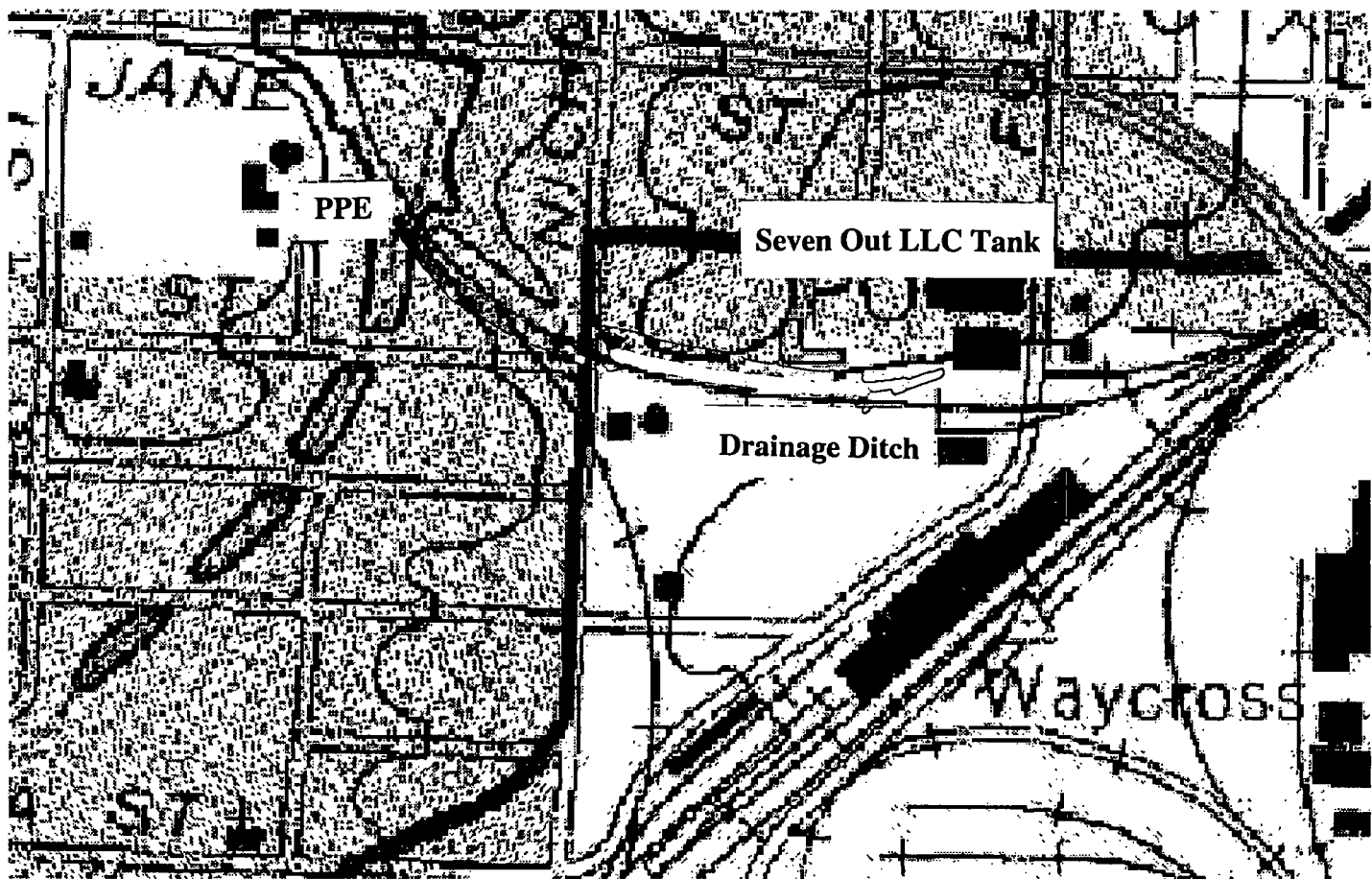
Slides:

Log Book:

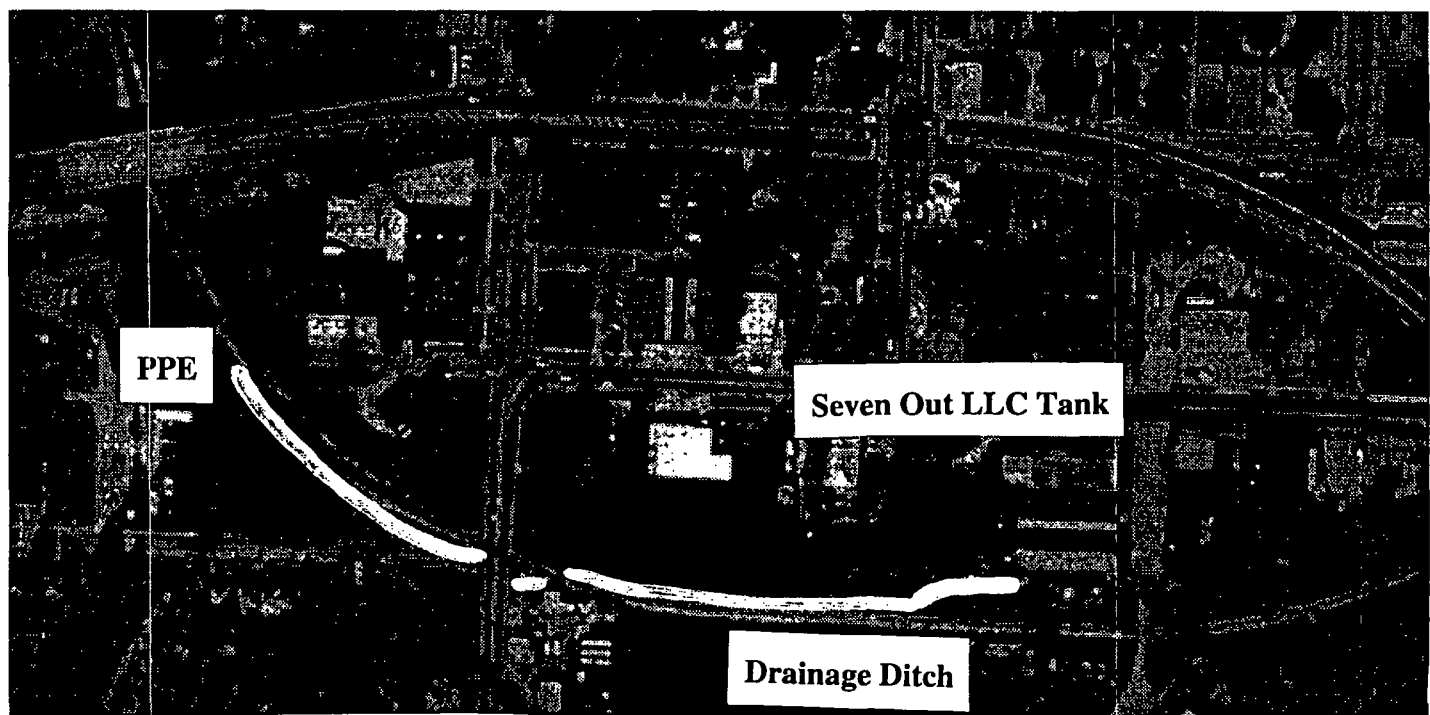
Other (describe): 4 mile radius map

Amount of material:

Please contact the appropriate Records Center to view the material.



Seven Out LLC Tank Site Overland Runoff Route



(Modified from References 13 and 51)

Figure 4

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

SDMS

Unscannable Material Target Sheet

DocID: 10508637 Site ID: CAAL000407811

Site Name: Seven Oaks 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.

SDMS

Site Name: James Oat LLC

*** Please contact the appropriate Records Center to view the material.***

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LIST OF ANALYTES



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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 <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
cis-1,2-Dichloroethene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
2-Butanone <u>EXIT Disclaimer</u>	--	5.0	10	10	500
Bromochloromethane	--	0.50	5.0	5.0	250
Chloroform <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
1,1,1-Trichloroethane <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
Cyclohexane	--	0.50	5.0	5.0	250
Carbon tetrachloride <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
Benzene	--	0.50	5.0	5.0	250
1,2-Dichloroethane <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
1,4-Dioxane	2.0	20	100	100	5000
Trichloroethene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
Methylcyclohexane	--	0.50	5.0	5.0	250
1,2-Dichloropropane <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
Bromodichloromethane <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
cis-1,3-Dichloropropene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
4-Methyl-2-pentanone	--	5.0	10	10	500
Toluene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
trans-1,3-Dichloropropene	--	0.50	5.0	5.0	250
1,1,2-Trichloroethane <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
Tetrachloroethene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
2-Hexanone <u>EXIT Disclaimer</u>	--	5.0	10	10	500
Dibromochloromethane <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
1,2-Dibromoethane <u>EXIT Disclaimer</u>	0.050	0.50	5.0	5.0	250
Chlorobenzene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
Ethylbenzene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
o-Xylene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
m,p-Xylene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
Styrene <u>EXIT Disclaimer</u>	--	0.50	5.0	5.0	250
Bromoform <u>EXIT Disclaimer</u>	--	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 <u>EXIT Disclaimer</u>	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 <u>EXIT Disclaimer</u>	--	5.0	--	170	5,000
1,2,4,5-Tetrachlorobenzene	--	5.0	--	170	5,000
4-Bromophenyl-phenylether	--	5.0	--	170	5,000
Hexachlorobenzene <u>EXIT Disclaimer</u>	--	5.0	--	170	5,000
Atrazine <u>EXIT Disclaimer</u>	--	5.0	--	170	5,000
Pentachlorophenol <u>EXIT Disclaimer</u>	0.20	10	6.7	330	10,000
Phenanthrene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Anthracene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Carbazole	--	5.0	--	170	5,000
Di-n-butylphthalate <u>EXIT Disclaimer</u>	--	5.0	--	170	5,000
Fluoranthene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Pyrene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Butylbenzylphthalate	--	5.0	--	170	5,000
3,3'-dichlorobenzidine <u>EXIT Disclaimer</u>	--	5.0	--	170	5,000
Benzo(a)anthracene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Chrysene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Bis(2-ethylhexyl) phthalate <u>EXIT Disclaimer</u>	--	5.0	--	170	5,000
Di-n-octylphthalate <u>EXIT Disclaimer</u>	--	5.0	--	170	5,000
Benzo(b) fluoranthene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Benzo(k) fluoranthene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000
Benzo(a) pyrene <u>EXIT Disclaimer</u>	0.10	5.0	3.3	170	5,000

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

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

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	

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

Atrazine	Benzo[b]fluoranthene	Endrin
4-Aminobiphenyl	Benzo[k]fluoranthene	Endosulfan 2
Pentachlorophenol	7,12- Dimethylbenz(a)anthracene	p,p'-DDD
Pronamide	Benzo[a]pyrene	Endrin aldehyde
Pentachlorodinitrobenzene	3-Methylchloranthrene	Endosulfan sulfate
Phenanthrene	Dibenz(a,j)acridine	p,p'-DDT
Anthracene	Indeno[1,2,3-cd]pyrene	
Carbazole	Dibenz[a,h]anthracene	
Di-n-butyl phthalate	Benzo[g,h,i]perylene	<u>Surrogate Standards:</u>
Fluoranthene	Alpha-BHC	2-Fluorophenol
Benidine	Gamma-BHC	Phenol-d5
Pyrene	Beta-BHC	Nitrobenzene-d5
p-Dimethylaminoazobenzene	Delta-BHC	2-Fluorobiphenyl
Butylbenzylphthalate	Heptachlor	2,4,6-Tribromophenol
Benzo[a]anthracene	Aldrin	Terphenyl-d14
3,3'-Dichlorobenzidine	Heptachlor epoxide	
Chrysene	Endosulfan 1	
Bis(2-ethylhexyl)phthalate	Dieldrin	
Di-n-octylphthalate	p,p'-DDE	

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

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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.

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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:

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

where:

C_i = Calibration Check Compound standard concentration.

C_e = 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})

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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:

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

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

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

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).
 n = Number of values

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

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

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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:

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David Jones, Laboratory Director

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

where:

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

A.7.4.2.13 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
	bis(2-Chloroethyl)ether	10	ug/L	660	ug/kg
SVOCs EPA 625/SW-846 Method 8270C	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-phenthylamine	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

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Environmental Protection Division

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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

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	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%

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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.

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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**

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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

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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 values

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

where:

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

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David Jones, Laboratory Director

$\frac{RRF_i}{RRF}$ = 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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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

Modified 1/26/2006

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 analytes 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.	

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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; <i>Corrective action</i>	
		Retention time for compounds	Per analysis	Within 5 sec of daily CCV	Rerun samples	

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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
Chloroethene
Chloromethane
Bromomethane
Chloroethane
Trichlorofluoromethane
1,1-Dichloroethene
Methylene chloride
trans-1,2-Dichloroethene
1,1-Dichloroethane
2,2-Dichloropropane
cis-1,2-Dichloroethene
Bromochloromethane
Chloroform
1,1,1-Trichloroethane
1,1-Dichloropropene
Carbon tetrachloride
Benzene

1,2-Dichloroethane
Trichloroethene
1,2-Dichloropropane
Dibromomethane
Bromodichloromethane
cis-1,3-Dichloropropene
Toluene
trans-1,3-Dichloropropene
1,1,2-Trichloroethane
Tetrachloroethene
1,3-Dichloropropane
Dibromochloromethane
1,2-Dibromoethane
Chlorobenzene
1,1,1,2-Tetrachloroethane
Ethylbenzene
o,m& p-xylene
Styrene

Bromoform
Isopropylbenzene
1,1,2,2-Tetrachloroethane
Bromobenzene
1,2,3-Trichloropropane
2-Chlorotoluene
4-Chlorotoluene
tert-Butylbenzene
1,3,5-Trimethylbenzene
1,2,4-Trimethylbenzene
4-Isopropyltoluene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dibromo-3-chloropropane
1,2,4-Trichlorobenzene
Hexachlorobutadiene
Naphthalene

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David Jones, Laboratory Director

1,2,3-Trichlorobenzene
2-Methoxy-2-methyl-propanen-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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Environmental Protection Division

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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:

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

Equation A.7.1.1

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

Equation A.7.1.3

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

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

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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.

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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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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

Georgia Department of Natural Resources

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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

EPD Laboratory, 455 14th Street, Atlanta GA 30318

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	

Georgia Department of Natural Resources

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

Georgia Department of Natural Resources

Environmental Protection Division

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David Jones, Laboratory Director

Methyl acetate
Cyclohexane
Methylcyclohexane

Georgia Department of Natural Resources

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David Jones, Laboratory Director

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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David Jones, Laboratory Director

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.

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

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})

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

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

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

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

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.7.3.2.10 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.

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

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.7.3.2.11 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:

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

Equation A.7.3.6

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.7.3.2.12 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.7.3.7

where:

 \bar{Y} Mean area response Y = Area response for the primary quantitation ion for the internal standard for each initial calibration standard.A.7.3.2.13 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:

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

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, $DF = 1$

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

David Jones, Laboratory Director

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	0.04	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		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	0.1	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		mg/L
	2-Butanone	100.0	ug/L	100.0	ug/Kg	5.0	mg/Kg		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		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	0.1	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		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

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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% option #1 linear regression for any analytes r ≥ 0.990 option #2 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.	

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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 RFO.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 <the RLs in Table A.7.3.1	None	

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

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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Georgia Department of Natural Resources

Environmental Protection Division

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

Modified 2/1/2006

Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

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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Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

Modified 2/1/2006

Georgia Department of Natural Resources

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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

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Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

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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Georgia Department of Natural Resources

Environmental Protection Division

EPD Laboratory, 455 14th Street, Atlanta GA 30318

APPENDIX C
SAFETY AND HEALTH PLAN

**SITE HEALTH AND SAFETY PLAN SITE INSPECTION
SEVEN OUT LLC TANK SITE a.k.a. BCX
901 FRANCIS STREET
WAYCROSS, WARE COUNTY, GEORGIA
EPA ID NO. GAN000407811**

DATE: July 26, 2006

HAZARDS: Chemical and physical hazards include potential exposure to regulated substances associated with historical activities conducted at the former Seven Out LLC Tank Site, which processed wastewaters for the past 5 years. The property has also been used historically as a machine shop and foundry and as a laundry. Hazards may include the risk of exposure to metals, hydrocarbons, semi-volatile organic compounds, volatile organic compounds, slip-trip-fall hazards head-bumping hazards, snakes, wasps, fire ants, and physical hazards associated with sampling activities, including noise and vibration hazards. Because of the time of the year that the sampling will be done, dehydration and heat exposure risks are considered real hazards.

AREA AFFECTED: Impacted soil has been identified on the property during previous investigations.

ADDITIONAL INFORMATION:

The proposed assessment activities will include the collection of surface soil, subsurface soil, and groundwater samples. Various metals, including lead and arsenic, and polycyclic aromatic hydrocarbons (PAHs) have been detected in site soils. Tanks on the site have been documented to contain various metals, volatile organics, and semi-volatile organics. The potential for exposure to organic compounds, especially solvents, exists also because a machine shop and a laundry existed on the site during the early 20th Century.

ENTRY OBJECTIVES:

(Actions, tasks to be accomplished, etc.)

- On-site and off-site collection of non-sampling data (photographs, inventory, measurements,
- On-site collection of soil and groundwater samples, and
- Off-site collection of soil and groundwater samples.

ONSITE ORGANIZATION/COORDINATION:

Team Leader: Eddie Williams

Team Members: Billy Hendricks, Fred Rowe, Brett Blackwelder, Daniel Braverman, and Bruce Khaleghi

ONSITE CONTROL:

The site is currently abandoned. On-site and off-site access permission will be obtained prior to field sampling. Eddie Williams will be the site contact during site activities.

HAZARD EVALUATION:

Chemical hazards based on substances in site soil are:

Substances	Highest Soil Concentration	Primary hazard
Arsenic	151 mg/kg	Inhalation, skin absorption, ingestion, skin/eye contact
Barium	75.2 mg/kg	Inhalation, skin absorption, ingestion, skin/eye contact
Coal Tar Pitch Volatiles	Fluoranthene (4.6 mg/kg), Pyrene (4.0 mg/kg)	Inhalation, ingestion, absorption, skin/eye contact
Chromium	8.69 mg/kg	Inhalation, skin absorption, ingestion, skin/eye contact
Copper	59.2 mg/kg	Inhalation, ingestion, skin/eye contact
Lead	264 mg/kg (TCLP of 8.13 mg/l)	Inhalation, skin absorption, ingestion, skin/eye contact
Manganese	169 mg/kg	Inhalation, ingestion
Mercury	0.35 mg/kg	Inhalation, ingestion, absorption, skin/eye contact

Physical hazards on site include: slips, trips, falls, bumped heads (on overhead tanks and pipes), and noise and vibration (from operation of sampling rig).

Biological hazards on site include: insect bites and stings, snake bites, sunburn, heat stress, heat exhaustion, and heat stroke.

PERSONAL PROTECTIVE EQUIPMENT:

Based on an evaluation of the potential hazards, the following levels of personal protection have been designated for the applicable work areas or tasks:

Level D: Steel-toe boots, long-sleeve shirts, long pants, eye protection, nitrile disposable sampling gloves during sampling, hardhat and earplugs (for those working near the sampling rig).

First-Aid Equipment: A first-aid kit will be kept in a vehicle located in a central site location (all field vehicles should be equipped with a first aid kit).

Additionally, an overhead canopy will be on site to provide shade and exposure protection from the sun and heat that is expected in south Georgia during the month of August. Water and sunscreen will be available on site and all personnel will be monitored for heat stress.

SITE SAFETY and HEALTH PLAN:

Site Safety Officer: Eddie Williams is the designated Site Safety Officer and is responsible for safety recommendations on site.

Hospital: Satilla Regional Medical Center is the nearest facility offering emergency services. The facility is 1.9 miles from the site and is accessible by traveling east on Francis Street to its intersection with U.S. 84. Travel north on U.S. 84 to Darling Avenue, where the hospital is located. Emergency assistance should be obtained by dialing 911.

A map showing the directions to the facility is attached on the last page of this plan.

PERSONAL MONITORING:

All personnel participate in a medical monitoring program and are subject to an annual physical.

All personnel will be current with their 8-hour OSHA Refresher Training prior to the sampling event.

The undersigned site personnel of the Georgia Environmental Protection Division have read the above plan and are familiar with its contents:

Name	Responsibility	Med. Scrn.	OSHA 8-hr	Signature
Eddie Williams	Project Leader/Site Safety Officer	6/21/2006	8/1/2005*	
Billy Hendricks	Sampling Team	9/12/2005	10/26/2005	
Brett Blackwelder	Sampling Team	7/14/2005	1/4/2006	
Fred Rowe	Sampling Team	6/12/2006	5/1/2005*	
Daniel Braverman	Sampling Team	1/25/2006	1/4/2006	
Bruce Khaleghi	Sample Transporter	12/6/2005	Scheduled for 8/24/2006	

Refresher scheduled for 8/24/2006



Start: 901 Francis St
Waycross, GA 31503-2335, US

End: Satilla Regional Medical Ctr:
912-283-3030
410 Darling Ave, Waycross, GA
31501, US

Notes:

Directions from Seven Out LLC to Satilla
Regional Medical Center



With Accident Forgiveness,
life has a rewind button.



Directions

Distance

Total Est. Time: 5 minutes

Total Est. Distance: 1.90 miles

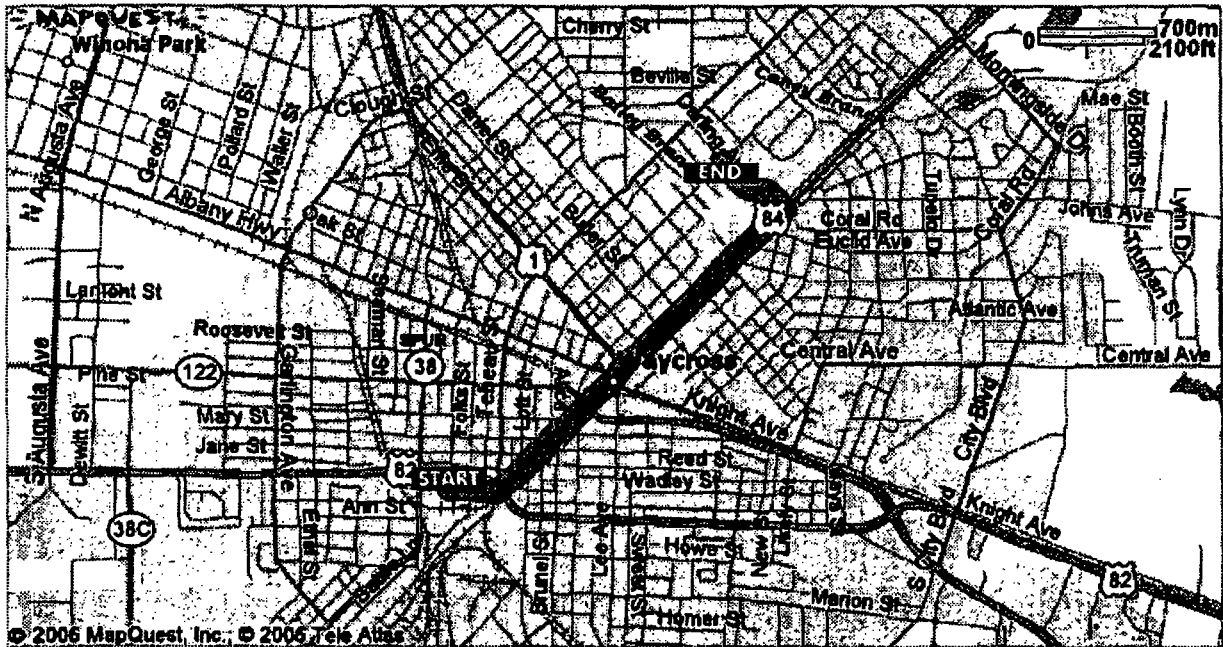
- | | | |
|--------------|---|------------|
| START | 1: Start out going EAST on FRANCIS ST / GA-38 toward MCDONALD ST / US-84. Continue to follow FRANCIS ST. | 0.1 miles |
| | 2: FRANCIS ST becomes US-84 / GA-38 / PLANT AVE. | 1.4 miles |
| | 3: Turn LEFT onto DARLING AVE. | 0.2 miles |
| | 4: Turn RIGHT onto ALICE ST. | <0.1 miles |
| | 5: Turn LEFT onto DARLING AVE. | <0.1 miles |
| END | 6: End at Satilla Regional Medical Ctr
410 Darling Ave, Waycross, GA 31501, US | |

Total Est. Time: 5 minutes

Total Est. Distance: 1.90 miles

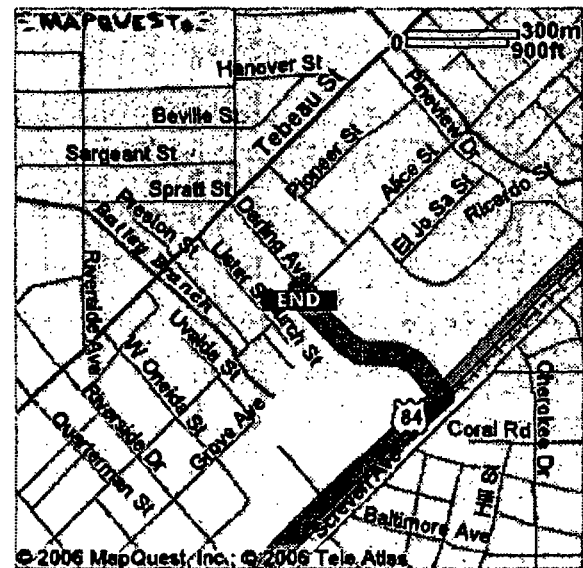


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Start:
901 Francis St
 Waycross, GA 31503-2335, US

End:
Satilla Regional Medical Ctr:
 912-283-3030
 410 Darling Ave, Waycross, GA 31501,
 US



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These directions are informational only. No representation is made or warranty given as to their content, road conditions or route usability or expeditiousness. User assumes all risk of use. MapQuest and its suppliers assume no responsibility for any loss or delay resulting from such use.

APPENDIX D
SITE ACCESS AUTHORIZATIONS

**RECORD OF TELEPHONIC CONVERSATION
HAZARDOUS WASTE MANAGEMENT PROGRAM**

DATE: July 26, 2006

TIME: 10:50 am

FILE: Seven Out LLC

SPOKE WITH: Vicki Carden

TITLE: Wife of Ferrell Carden, Principal Officer of Seven Out LLC.

CITY: Jacksonville

STATE/ZIP: FL

TELEPHONE NUMBER:

904/229-6059

SUBJECT: Authorization to access Seven Out Parcels at 901 Francis Street and 3 Folks Street in Waycross, GA

SUMMARY OF CALL:

The purpose of the call was to obtain verbal permission from Ms. Carden, who is currently acting on behalf of her husband, Ferrell Carden, to access the Seven Out LLC parcels for the purpose of taking soil and groundwater samples as required for the EPA Site Inspection. Ms. Carden said it was OK and that she would contact me or have her husband contact me if they were going to be present to collect splits. At this time, she would be happy with getting copies of our sample results.

She stated that she did not know where the keys to the gates at 901 Francis Street were. I suggested to her that I cut the locks on the property gates and replace them with new locks and keys. Upon my completion of the sampling, I would mail her the keys to the new locks. She was agreeable to that suggestion.

I thanked her for her time.

ACTION REQUIRED:

None

FOLLOW-UP RESPONSES/ADDITIONAL COMMENTS:

Mail keys and sampling results to: Condo Unit #17, 1307 East River Hills Circle, Jacksonville, FL 32211.

SIGNATURE:

Edward L. Willis

**RECORD OF TELEPHONIC CONVERSATION
HAZARDOUS WASTE MANAGEMENT PROGRAM**

DATE: June 20, 2006

TIME: 1:00 pm

FILE: Seven Out LLC

SPOKE WITH: Mr. Lawrence Woodard

TITLE: Administrator, Woodard Living Trust.

CITY: Waycross

STATE/ZIP: GA

TELEPHONE NUMBER:

912/283-8739

SUBJECT: Information on ownership of property identified on tax maps at belonging to the Woodard Living Trust (especially, the Seven Out LLC tank farm)

SUMMARY OF CALL:

Mr. Woodard stated that the property of the living trust was located between the CSX railway tracks and Remshart on the opposite side of the block from Seven Out LLC. This information is in disagreement with the tax plat maps and tax database that was accessed online and at the Ware County courthouse. Those records show that parcels 15 and 16 of block 6 on Map WA11 belong to the Woodard Living Trust. Mr. Woodard clarified that his trust does not include the properties on which the Seven Out LLC tank farm is located.

ACTION REQUIRED:

None

FOLLOW-UP RESPONSES/ADDITIONAL COMMENTS:

None at this time.

SIGNATURE:

Edward L. Williams

**RECORD OF PERSONAL CONVERSATION
HAZARDOUS WASTE MANAGEMENT PROGRAM**

DATE: June 16, 2006

TIME: 10:50 am

FILE: Seven Out LLC

SPOKE WITH: Bennie James

TITLE: Owner, The Sports Shop, 801 Francis Street

CITY: Waycross

STATE/ZIP: GA

TELEPHONE NUMBER:

SUBJECT: Authorization to access Sports Shop Property at 801 Francis Street in Waycross, GA

SUMMARY OF CALL:

The purpose of the in-person visit was to obtain verbal permission from Mr. James, owner of The Sports Shop, a facility next door to the Seven Out LLC site, to sample on his property between the Seven Out Tank Site and The Sports Shop. Without doing a physical survey, it was not clear where the property boundary was located.

Mr. James stated that it was fine with him wherever I wanted to sample and access was granted. He will be provided a copy of the sample analyses and did not express an interest in splits.

I thanked him for his time.

ACTION REQUIRED:

None

FOLLOW-UP RESPONSES/ADDITIONAL COMMENTS:

Mail sampling results to: Bennie James, c/o The Sports Shop, 801 Francis Street, Waycross, GA 81501

SIGNATURE:

Edw. L. Allen

**RECORD OF TELEPHONIC CONVERSATION
HAZARDOUS WASTE MANAGEMENT PROGRAM**

DATE: July 27, 2006

TIME: 1:05 pm

FILE: Seven Out LLC, BCX

SPOKE WITH: Ira Walker

TITLE: Regional Supervisor, CSX Transportation

CITY: Atlanta/Waycross

STATE/ZIP: GA

TELEPHONE NUMBER:

912/288-2418 (cell)

SUBJECT: Authorization to access CSX Rail Property Behind 3 Folks Street in Waycross, GA

SUMMARY OF CALL:

The purpose of the telephone call was to obtain verbal permission from a CSX authorized person to sample on the CSX property behind the 3 Folks Street property where Seven Out had leased space to store Frac/Baker Tanks that contained wastewater from the facility. Mr. Walker told me that he did not expect any problems with EPD taking samples on this property. He stated that the contact person in Waycross for CSX is Mr. Tommy Pierson (Haynes Street) and that Mr. Matt Atkinson with the CSX Remediation Dept. (770/235-9969) probably assisted Mr. Pierson in the CSX removal of the frac tanks and sampling/removal of impacted soils at the site. He stated that one of those two gentlemen would be calling me when they return to the office.

I thanked him for his time.

ACTION REQUIRED:

None

FOLLOW-UP RESPONSES/ADDITIONAL COMMENTS:

Mail sampling results to: CSX at the contact person's specified mailing address. Obtain documentation on the removals that were completed by CSX on this parcel of property.

SIGNATURE: