LAW OFFICES
GALLAGHER & PATTON
A PARTNERSHIP INCLUDING A PROFESSIONAL CORPORATION
ORDWAY BUILDING SUITE 2450
ONE KAISER PLAZA
OAKLAND, CALIFORNIA 94612-3685

(415) 836-1266
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May 17, 1990

<u>By Hand Delivery</u>

ALAMEDA COUNTY ENVIRONMENTAL
HEALTH DEPARTMENT
Hazardous Materials Program
80 Swan Way, Room 200
Oakland, CA 94621

Attention: Pam Evans

Re: 23555 Saklan Road, Hayward

Dear Pam:

Pursuant to our telephone conversation on May 17, I enclose herewith one copy of the March 26, 1990, Initial Site Survey and Preliminary Assessment Plan for the 23555 Saklan Road property in Hayward.

I believe the enclosure will satisfy your various requirements as discussed in our telephone conversation. I would very much appreciate your review of the materials prior to our planned Tuesday drilling schedule.

Incidentally, if you have not yet received the corrected check from Berkeley Land Company, please advise and I will arrange delivery of same immediately.

Best regards,

GALLAGHER & PATTON

BARRY M. GALLAGHER

BMG:lw
Enclosure

ENVIRONMENTAL WORK PLAN

INITIAL SITE SURVEY REPORT AND PRELIMINARY ASSESSMENT PLAN FOR HAYWARD SITE

Submitted to:

BERKELEY LAND COMPANY
1211 Newell Avenue, Suite 120
Walnut Creek, California

March 26, 1990

Submitted By:

CERTIFIED ENVIRONMENTAL CONSULTING, INC.
140 West Industrial Way
Benicia, California 94510
(707) 745-0171
(800) 447-0171

Registered Environmental Assessor
Michael T. Noble
No. 01138
Expires 6/90
State of California

M. Noble
Michael T. Noble, CIH
Vice President

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Appendix

EXECUTIVE SUMMARY

In June 1988 Berkeley Land Company removed an underground fuel tank located on their property at 23555 Saklan Road, Hayward, California. Berkeley Farms and the previous owner had stored gasoline in the tank. Berkeley Land Co. has records from the their fuel supplier which indicate that the only fuel purchased for the site was gasoline. Apparently the previous owner has indicated to Berkeley Land Co. and their attorney Mr. Ronald Hufft that the tank was not used for several years before the sale of the property and was then only used for gasoline storage.

The Alameda County Department of Environmental Health wrote Berkeley Land Co. on October 13, 1989 requesting that a preliminary assessment be conducted on the site. The purpose of the assessment is to determine the need for additional actions. The letter also indicated that soil samples collected from the bottom of the pit at the time the tank was removed had total hydrocarbon concentrations of 2,076 ppm and 24,144 ppm. Two composite soil samples collected by Kaprealian Engineering, Inc. on November 7, 1988 from approximately 130 cubic yards of stockpiled soil at the site had Total Petroleum Hydrocarbon concentrations as gasoline as 57 ppm and Total Petroleum Hydrocarbon concentrations as diesel as 140 ppm. There was no benzene, toluene, ethyl benzene or xylenes detected in the samples.

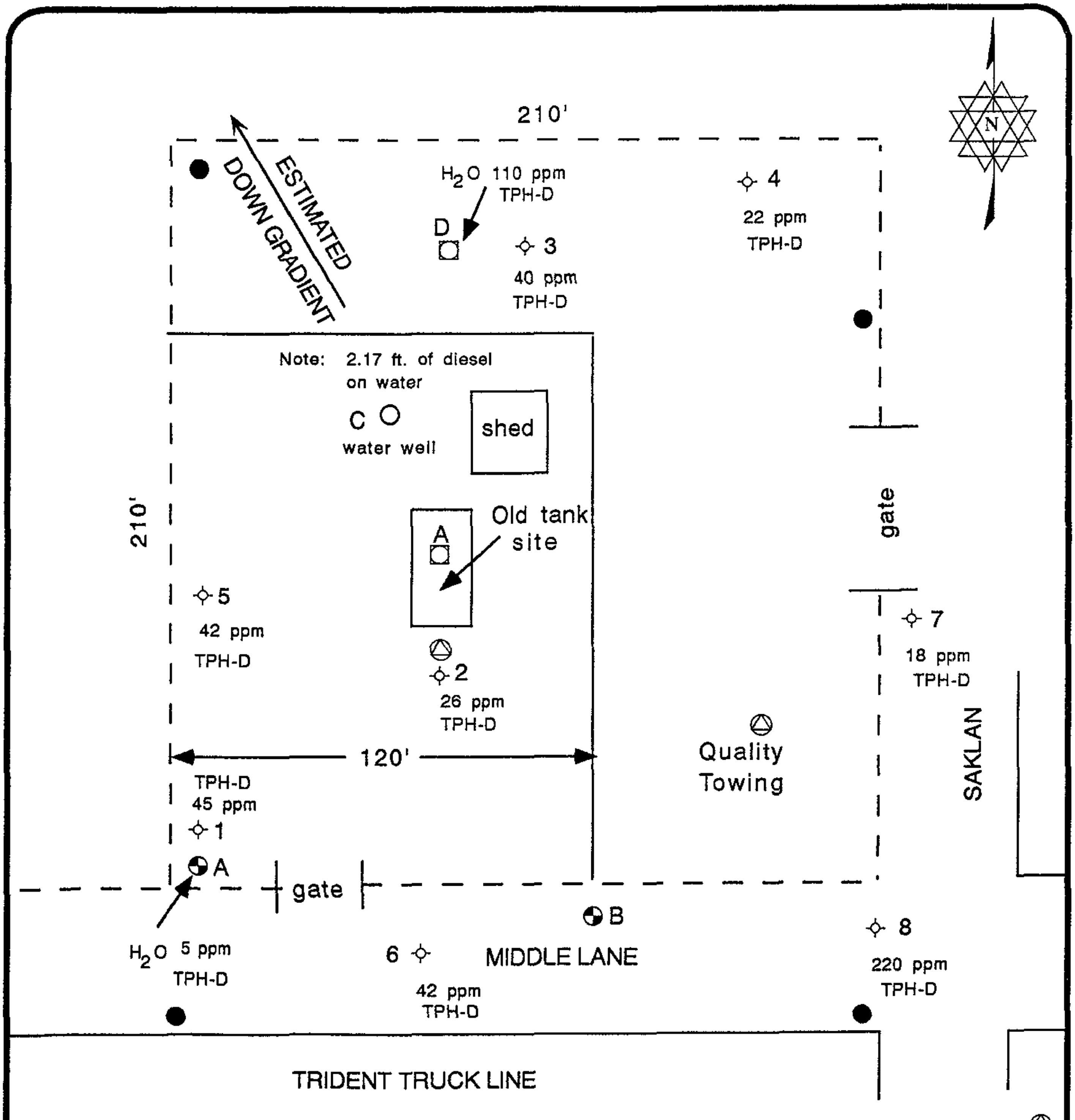
Berkeley Land Co. indicated that there was two feet of diesel floating in the well which was verified by CEC during the initial assessment on February 27, and March 1, 1990. Samples of product collected by Berkeley Land Co. in the well indicated that the oil is Diesel No. 2. Figure 1 and Table 1 summarize the results of the survey performed by CEC. Sampling consisted of collecting 3 ground water samples, 3 soil samples. Eight soil vapor probe samples were collected at depths of 5 to 15 feet at different locations on the site, including property leased by Quality Tow.

Although this initial survey is inconclusive, the vapor probe study tends to support Berkeley Land Co.'s position that the diesel contamination is from off site. Additional data is necessary to determine the sources of the diesel, including a need for samples on Saklan Road between Berkeley Land Co. and Trident Trucking. The evaluations performed by CEC to date appear to implicate Trident Trucking, Inc. as the diesel source.

Many of the homeowner's properties along Saklan Road appear to have their own drinking water pumps. Since the well water on the Berkeley Land Company site has at least 2.2 feet of diesel floating on it CEC has recommended that the Office of Emergency

Services (OES) be notified. Part of the Preliminary Assessment for the County will include coordination between CEC and the regulators to collect water samples on the neighboring properties.

The recommendations in this report are consistent with those required by the Regional Water Quality Control Board, and Alameda County in their November letter to Berkeley Land Co.



⌖ Vapor probe location

◐ Existing Piezometer

□ Borehole location

● Proposed boring and monitor well

△ Proposed sample bore

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Figure 1

Berkeley Land Co.
23555 Saklan Road
Hayward, CA.

March 23,1990 89-57-237

SURVEY SUMMARY
AND
TEST RESULTS

TABLE 1

Groundwater Gradient: Slightly North West, based on existing wells

Depth to Groundwater:	Monitoring Well (Pt.A)-13.99 Monitoring Well (Pt.B)-13.68' Water Well (Pt.C)-17.2' Hand Augured Well (Pt.D)-15.51'
Survey Corrected Relative Depth to Groundwater:	Monitoring Well (Pt.A)-18.66' Monitoring Well (Pt.B)-20.78' Water Well (Pt.C)-18.84' Hand Augured Well (Pt.D)-19.17'

WATER SAMPLE ANALYSIS FOR TOTAL PETROLEUM HYDROCARBONS

Sample #	Location	Gasoline	Diesel	Benzene	Toluene	Ethyl Benzene	Xylene
BC-0301-W1	Monitoring Well (Pt.A)	ND	110 ppm	ND	ND	4.4	15
BC-0301-W2	Hand Auguered Well	ND	5 ppm	0.4	14	ND	0.6
BC-0301-W3	Product Sample from water well	ND	100 %	Diesel	———	———	———

SOIL SAMPLES ANALYSIS FOR TOTAL PETROLEUM HYDROCARBONS, BENXENE, TOLUENE, ETHYL BENZENE AND XYLENES

Sample #	Location	Gasoline	Diesel	Benzene	Toluene	Ethyl Benxene	Xylene
BC-0227-120	Tank	ND	40 ppm	ND	15 ppm	6 ppm	13 ppm
BC-0227-B163	Quality Tow	ND	40 ppm	ND	ND	ND	ND
BC-0227-B186	Quality Tow	ND	550 ppm	ND	ND	4 ppm	10 ppm

NOTE: ND indicates below the analytical level of detection

INTRODUCTION/BACKGROUND

In June 1988 Berkeley Land Company removed an underground fuel tank located on their property at 23555 Saklan Road, Hayward, California. Berkeley Farms and the previous owner had stored gasoline in the tank. Berkeley Land Co. has records from the their fuel supplier which indicate that the only product purchased for storage in the tank was gasoline. The previous owner has indicated to Berkeley Land Co. and their attorney Mr. Ronald Hufft that the tank was not used for several years before the sale of the property and then was only used for gasoline storage.

The Alameda County Department of Environmental Health wrote Berkeley Land Co. on October 13, 1989 requesting that a preliminary assessment be conducted on the site. The purpose of the assessment was to determine the need for additional actions. The letter also indicated that soil samples collected from the bottom of the pit at the time the tank was removed in June 1988, had total hydrocarbon concentrations of 2,076 ppm and 24,144 ppm. (See Appendix A.)

Two composite soil samples collected by Kaprealian Engineering, Inc. on November 7, 1988 from approximately 130 cubic yards of stockpiled soil at the site had Total Petroleum Hydrocarbon concentrations as gasoline at 57 ppm and Total Petroleum Hydrocarbon concentrations as diesel at 140 ppm. There was no benzene, toluene, ethyl benzene or xylenes detected in the samples. (See Appendix D.)

Berkeley Land Co. indicated that there was two feet of diesel floating in the well which was verified by CEC during the initial assessment on February 27, and March 1. 1990. Samples of product collected by Berkeley Land Co. in the well indicated that it is Diesel No. 2. Figure l and Table 1 summarize the results of the assessment performed by CEC which consisted of collecting 3 ground water samples, and 3 soil samples. Ten soil vapor probes were performed at depths of 5 to 15 feet, at various locations on the site including property leased by Quality Tow.

The results of this preliminary survey are inconclusive. The vapor probe survey tends to support Berkeley Land Co.'s position that the diesel contamination is from off site. Vapor probe readings are a relative sampling of the actual concentrations of hydrocarbons in the soil. They can be effected by a number of variables which include the physical state of the contaminated soil, and depth of the probe especially through clay. Clay tends to clog the probe and is less porous, reducing the volume of vapors that can be drawn through to the organic vapor meter. The highest concentrations of organic vapors found in the soil were along the southern boarder of the property parallel to Middle Lane and down Saklan Road which is up gradient and in line with Trident Trucking Inc. An attempt was made to perform a vapor probe survey on Saklan Road, south towards Trident Trucking. Two bore samples on Saklan Road are recommended to evaluate the potential contaminants along this street.

Regional Water Quality Control Board and Hayward Fire Department records generally show that the ground water gradient flows in a west to slightly north westerly direction. However, there were at least two files of over 20 reviewed which estimated that the ground water flowed southwest, at approximately a 90 degree angle from the north west direction determined by CEC.

This report includes the proposal for the "Preliminary Assessment" requested by the county which will require coordination with the Regional Water Quality Control Board. Appendix B is a copy of the letter from Alameda County requesting the development and execution of a "Preliminary Assessment" at Berkeley Land Co.s Hayward site.

Many of the homeowner's properties along Saklan Road appear to have their own drinking water wells. Since the well water on the Berkeley Land Company site has at least 2.2 feet of diesel floating on it is recommended that the Office of Emergency Services (OES) be notified. Part of the Preliminary Assessment for the County will include the coordination between CEC and the regulators to collect water samples on the neighboring properties.

SAMPLING AND SURVEY METHODOLOGY:

To estimate the ground water gradient CEC surveyed the property, determined the depth to ground water at the water well and two monitoring wells on the site, and the distances between them. A fourth hand augured well was bored north of the water well because there was significant fluctuation in the ground water depth in one monitoring well and information on how the monitoring wells were put in was not readily available. After the water level in the hand augured well appeared to stabilize, the depth to ground water was measured. It appears from the data collected that ground water is at and average depth of 13 to 14 feet and flows west to slightly north west with a gradient of 0.011 feet/foot.

Before conducting a Vapor Probe study on the site USA was called to determine where there might be underground utilities buried. USA is unit of PG&E who check with utilities companies, the city, etc. to determine where the water, sewer, electrical, communications, and gas lines lie on or adjacent to the site. Part of their assessment includes marking the ground with up to five different colored lines to identify approximately where utilities are located (See Appendix A).

Eight to ten locations were initially identified which could be expected to give a reasonable characterization of any subsurface contamination. The goals of the survey include estimating the size, concentration and boundaries of the plume. Vapor probe samples were measured using an Organic Vapor Meter at depths ranging from 5 feet to 15 feet below grade. Because of the difficult sample collection caused by clay clogging the probe during installation, all sample locations were resampled after the probe was driven to maximum test depth, removed, cleaned and reinserted. Figure 1 and Table 1 provide a summary of the results of this sampling.

In addition to the vapor sampling three soil samples were collected at the locations identified in Figure 1. TPH(D) concentrations near the former tank ranged from 40 ppm at a 12 foot depth, to 550 ppm at a 15 foot depth fifty feet north of the tank location. This data appears to contradict the results of the vapor probe survey. However, the tank location had been previously backfilled with clean soil. CEC was also unable to sample below 12 feet because of a concrete slab at that depth which precluded going deeper with a hand auger.

Soil samples were collected by driving 4" long by 2" in diameter brass tubes into soil using a hand auger. After removing the sample from the auger it's ends were wrapped in aluminum foil, sample labels applied, plastic caps put on and taped in place. A sample data sheet was completed, as well as a Chain of Custody form which accompanied the samples to the laboratory. All samples were immediately chilled and kept chilled until they were analyzed.

Two water samples were collected with a Total Petroleum Hydrocarbon (Diesel) concentration ranging from 5 ppm to 110 ppm. The water samples were collected from the monitoring well located on the southwest corner of the site, and the hand augured well located on the Tow yard lease property. These two samples were collected in glass VOA bottles and sealed with a teflon lined cap. The samples were collected using the protocol outlined in this document. The filled VOA bottles were labeled, immediately chilled, and were accompanied by a Chain of Custody form to the laboratory.

Appendix B is a list of the regulatory agency files CEC requested to review. There were over twenty sites with a regulatory record within a one mile radius of Berkeley Land Co.'s. The results of the file review are summarized in Table 2 and Figure 2 which provides maximum hydrocarbon concentrations in the soil and/or water prior to any remediation actions initiated by the property owner.

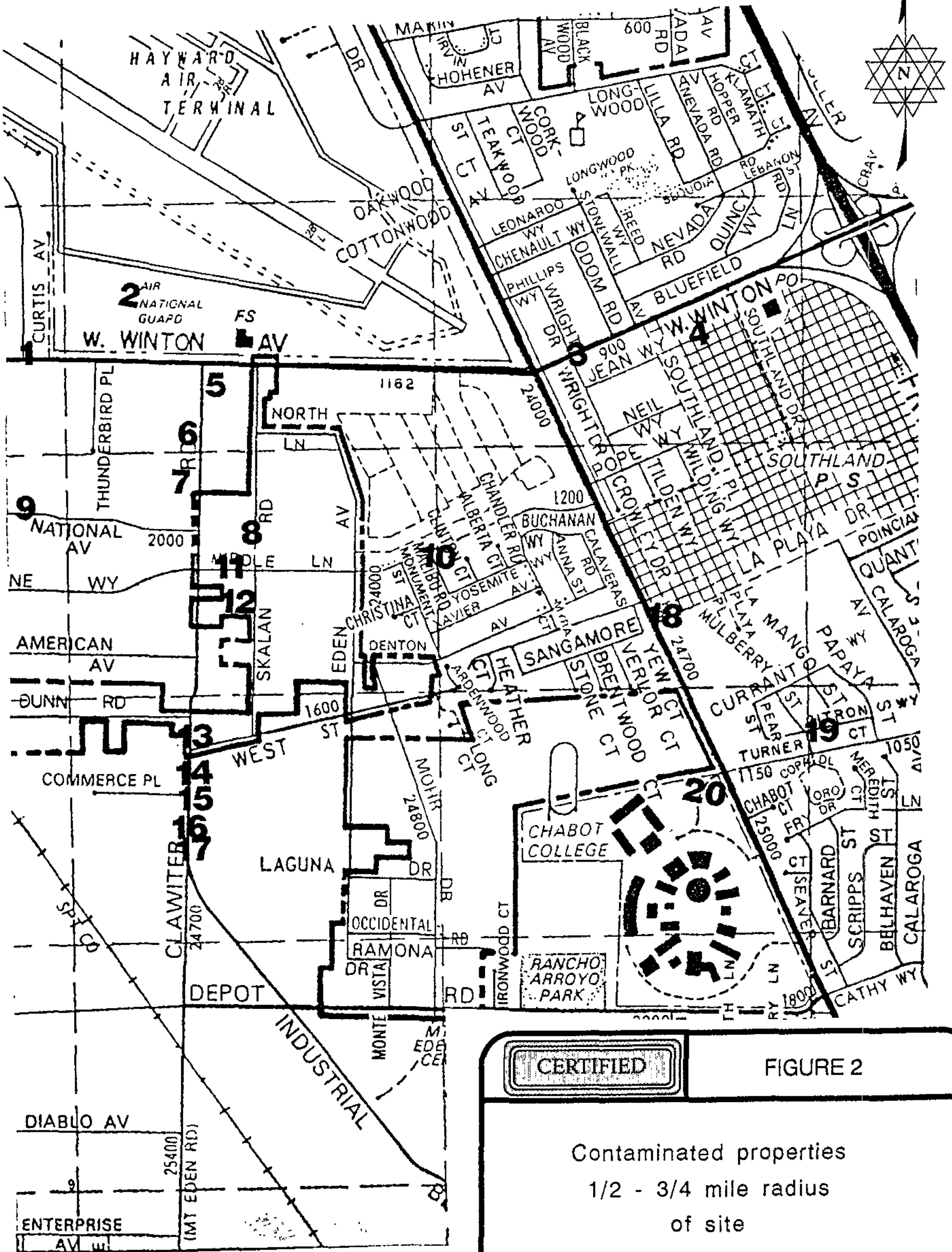

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FIGURE 2

Contaminated properties
1/2 - 3/4 mile radius
of site

INDEX

CONTAMINATED PROPERTIES

1/4 - 3/4 MILE RADIUS OF SITE (FIGURE 2)

1. Oakland Fence Co.
2. Air National Guard
3. Ferret Properties
4. Sears
5. Chevron
6. Continental
7. Oliver DeSilva
8. Alhambra
9. Tosconna
10. Homes with privite water wells
11. Berkeley Land Company
12. Trident Truckline
13. Clarks Building Supply
14. PCI
15. P G & E
16. Herrick
17. Yellow Freight
18. Equity Property
19. Alameda County Turner Court
20. Chabot College

FUEL RELEASE SUMMARY

1/2 TO 3/4 MILE RADIUS FROM BERKELEY LAND COMPANY SITE

TABLE 2

Company	Address	Maximum Soil Concentration	Maximum Conc.
Oakland Fence Co.	1580 Winton Ave	File not available (3/5/90)	
Air National Guard	1525 Winton Ave		8.3 ppb
Farret Properties	944 Winton Ave	130 ppm	
Sears	660 Winton Ave	20,000 ppm TPA	83 ppm
Chevron	1490 Winton Ave	21,000 ppm TPA	(1.3 of product or the Water)
Continental	22493 Clawiter		Produced in well
Oliver DeSilva	22991 Clawiter	24,000 ppm diesel 18,000 ppm 2,900 ppm gas	
Alhambra	22900 Clawiter	520 ppm	
Tosconna	2227 National	30 ppm	
Homes with privite water wells			
Berkeley Farms			
Trident Truckline			
Clarks Building Supply	23040 Clawiter	3,500 ppm (gas)	
PCI	23520 Clawiter	17,900 ppm (gas)	
PG & E	24300 Clawiter	18,000 ppm	250 ppm
Herrick	25450 Clawiter	32,000 ppm	110 ppm
Yellow Freight	25555 Clawiter	21,000 ppm	45 ppm
Equity Property	24668 Hesperian	1,800 ppm	180 ppb
Alameda County Turner Court	951 Turner Court		
Chabot College	25555 Hesperian Blvd		198 ppm
Walker Concrete	1844 Winton Avenue	File not available (3/5/90)	
Super Sturctures Wendland Trucking	24200 Clawiter 220 Laguna	File not available (3/5/90) Soil aeration on site	

REGULATORY FILE REVIEW:

Initially nine properties were identified within a few block radius of the site which had a potential for leaking underground tanks. The Regional Water Quality Control Board's (RWQCB) Fuel List was also consulted to identify fuel leaks within 1 mile of the property. EPA maintains a list of potential National Priority List candidates. This list is commonly referred to as the CERCLIS and currently has over 25,000 sites on it.

Five properties were identified on the CERCLIS that lie within a 1/2 mile radius of the site. Two of these properties were also on the RWQCB's list or the states Hazardous Waste Site Lists (6/89). There were 22 locations within a 1/2 to 1 mile radius of the Berkeley Land Co. site. CEC requested to review approximately 38 regulatory files at the Regional Water Quality Control Board, and nine each at the County Department of Environmental Health, and the Hayward Fire Department. Copies of the file lists are attached in Appendix C.

The following lists were used to identify sites with known environmental problems:

- California Office of Planning & Research,
  Hazardous Material Site list (6/89)
  (22 sites within a 1/2 to 3/4 mile radius)

- The Regional Water Quality Control Board,
  Oakland, Calif. (31 case files)

- CERCLIS (National Priority List, Approximately 5
  cases

- Department of Environmental Health, Oakland and the
  city of Hayward Fire Department. (9 files each).

Figure 2, identifies the majority of the sites around Berkeley Land Co. with known soil and ground water contamination. The following summarizes the findings of the file reviews conducted at the Regional Water Quality Control Board and the Hayward Fire Department.

Sears Automotive Center, is located at 660 W. Winton Avenue in Hayward. Twelve monitoring wells were installed in 1985 and 1986. Floating fuel was found in the wells with a maximum thickness of 1.34 feet. There was less than 2,000 gallons of fuel estimated to have been lost from an old pipe leak. Total petroleum hydrocarbons found in the soil ranged from 0.18 to 150 parts per million (ppm). Ground water samples had up to 83 ppm TPH as gasoline and 1 ppm TPH as diesel.

Chabot College, 25555 Hesperian Boulevard, was found to have total petroleum hydrocarbon (TPH) concentrations of 198 ppm in the ground near the two underground tanks.

California Air National Guard, 1525 W. Winton Ave. A 5,000 gallon gasoline tank was found to have a leak during a stick test. In addition there is a 6,000 gallon diesel underground tank and two above ground fuel tanks, trailers or tanks ranging from 300 to 1,200 gallons. Approximately 50 gallons of fuel was estimated to have been lost. The property is owned by the city and leased by the U. S. Airforce.

Alhambra Water, 2290 Clawiter Road has a 12,000 gallon gasoline tank. The tank is double walled and has a leak alert system. Soil samples during removal of the old tank had 520 ppm TPH on the east end of the tank.

Beacon Ultramart (former Exxon Service Station), 26115 Hesperian Boulevard has both ground water and soil contamination from a release of waste oil. The ground water samples contained TPH concentrations of 1 ppm grease and 130 ppb oil.

Ferrer Property, 944 W. Winton Avenue had some soil contamination beneath underground storage tanks. There is a 10,000 gallon gasoline tank on the property. Soil was found to have 130 ppm TPH with 210 ppb in the water.

PG &E, 24300 Clawiter had soil contamination to a depth of approximately 12 feet covering an 8' X 10' area in one location and excavation of 60' X 120' X 130' or about 3,500 cubic yards excavated and 1,500 cubic yards treated on site with aeration. There was approximately 2,000 gallon of mineral oil lost. Soil concentrations of TPH ranged from 3,500 to 18,000 ppm TPH and 190 ppm benzene underneath the tank. Ground water contamination of 250 ppm TPH dissolved gasoline extended approximately 200 feet down gradient from the tank storage area. On December 5, 1986 when 5 underground storage tanks were removed, there was 22 ppm TPH and 2 ppm benzene with 0.23' of floating fuel in the ground water. Soil with concentrations of fuel greater 1,000 ppm was hauled to a Class I site.

Oliver de Silva, 22991 Clawiter had soil excavated to a depth of 17 feet along the fuel trench line. Soil was not removed from the west and south walls because of existing heavy use driveways. The property has a 12,000 gallon diesel, 12,000 gallon gasoline, and 4,000 gallon oil tanks on it. The ground water gradient was determined to be slightly north of west. There was approximately 0.3 feet of fuel found in the monitoring wells. TPH levels as gasoline in the soil was found to be as high as 2,900 ppm, as diesel 5,000 ppm. Benzene concentrations were 120 ppb. The Hayward fire department's notes indicated that the extent of contamination in the immediate area only was defined. Monitoring wells were installed in early 1986. The tanks and piping were removed on May 25 and June 27, 1988.

Trident Truck Line, Inc., 23725 Saklan Road has a 10,000 & 5,000 gallon carbon steel tanks apparently with no leak detection system.

Casade Chevron, 1490 W. Winton has two 10,000 gallon gasoline tanks, a 5,000 gallon gasoline tank, and one 500 gallon waste oil tank which was removed last March. Ground water monitoring showed no detectable concentrations of benzene, 130 ppb tetrachloroethane, and soil samples contained 740 ppm TPH.

Yellow Freight, 25555 Clawiter Road had a 1,000 gallon fresh oil tank replaced and a 550 gallon waste oil tank removed on October 5, 1987. Two holes were discovered in the bottom, north end of the fresh oil tank. Ground water samples had 45 ppm TPH. Two monitoring wells were installed on June 6, 1986. The company has four 10,000 gallon diesel, and 10,000 gallon gasoline tanks on site.

Clarks Building Material, 23040 Clawiter Road had a 500 gallon waste oil tank, 3,000 gallon gasoline tank, and a 1,000 gallon diesel tank on it. There was 24,000 ppm TPH as diesel and 3,500 ppm TPH as gasoline in the soil.

Equity Properties, 24668 Hesperian lies approximately 3/4 of a mile southeast directly upgradient from the Berkeley Land Co. site on Hesperian Blvd. and La Playa Drive. There is a 5,000 gallon gasoline tank, 7,500 gallon gasoline tank, and 280 gallon waste oil tank on the property. Samples collected on March 2, 1989 had 1,800 ppm TPH in the soil and 180 ppb in the water.

Drewry Photo Color, 27105 Industrial Way had a 1,200 gallon gasoline tank on it.

Toscanna Bakery, 2227 National Ave has a 10,000 gallon gasoline tank on it. TPH contamination in the soil was approximately 30 ppm and with 3.4 ppb benzene.

Hayward Corporate Yard, 3050 W. Winton Ave. had 8,000 gallon tank on it. There was approximately 14 ppm TPH in the soil and 3.8 ppm TPH in the water.

Foodmaker Inc., 2395 American Ave. had 5.5 ppm TPH in the water and 9 ppm TPH in the soil.

EKC Technology, 1739 Sabre Ave had 18 ppm TPH in the soil and 1.8 ppb TPH in the ground water. Alameda County, Turner Court has had leaking fuel tanks and is undergoing remediation.

HYDROGEOLOGY:

The site is located in the San Francisco plain, a northwest-southeast trending structurally downward, bordered on the east by the Hayward fault and the Diablo Range, and bordered on the west by San Francisco Bay. Berkeley Land Co. is approximately 30 feet above sea level and 4.5 miles from the bay. The area is underlain by late Quaternary alluvial deposits that are generally comprised of clays, silts, sands and gravels. The alluvial deposits are laterally continuous, poorly permeable, and generally prevent significant quantities of surface water and precipitation from percolating into deeper water bearing strata. In this area, these alluvial deposits are generally underlain by fine sands, silts and gravels of the Santa Clara Formation. Regional ground-water flow is generally southwest toward the bay. The area is principally drained by two streams, San Lorenzo Creek which is approximately 4 miles north of the site and Alameda Creek, both of which discharge directly into San Francisco Bay.

The area is underlain by unconsolidated alluvial fan deposits of Pliocene-Pleistocene age. It is located on a discrete alluvial fan deposit known as the San Lorenzo Cone (AFCD, 1984). The deposits were laid down by the ancestral San Lorenzo Creek as it emerged from the foothills of the Diablo Range. Westward sloping sand and gravel beds were deposited by the braided creek, and during times of flood, finer grain layers of silt and clay were deposited. The total thickness of the unconsolidated deposits reportedly is in excess of 1,000 feet.

The Alameda County Bay Plain Ground-Water Study indicated that the depth of water supplies range from 32 to 581 feet and the depth to ground-water ranges from 16 to 90 feet. The city's water is currently derived from water imported from Hetch Hetchy Reservoir in the Sierra Nevada. Ground-water has not been actively used by the city for drinking water for approximately 30 years.

Aquifers in the San Lorenzo Cone are comprised of well sorted, permeable gravel and sand beds deposited by the San Lorenzo Creek. The aquifers are generally confined by the inter-bedded, finer grain silt and clay deposits. The deposits in the San Lorenzo Cone can be divided into five distinct zones: 1) shallow relatively permeable deposits within 500 feet of the land surface; 2) aquifers between depths of about 50 to 100 feet, known collectively as the Newark Aquifer; 3) aquifers between depths of about 130 and 220 feet, known collectively as the Centerville Aquifer; 4) aquifers between depths of 250 and 400 feet, known collectively as the Fremont Aquifer; and 5) aquifers deeper than 400 feet.

The shallow relatively permeable zone within the San Lorenzo Cone generally is comprised of silty sand, is of limited areal extent and often occurs under unconfined, perched conditions (ACFCD, 1984). Recharge to the shallow zone occurs by infiltration of direct precipitation, irrigational streamflow. Ground-water in the shallow relatively permeable zone generally flows towards San Francisco Bay.

The Newark aquifer is semi-confined by a 25 to 60 foot thick clay layer and generally consists of interfingering sand and gravel lenses separated by clay layers approximately 5 to 10 feet thick. Infiltrations of streamflow at the apex of the San Lorenzo Cone and leakage through the upper clay confining layer allow recharge of this aquifer. Ground water in the Newark Aquifer generally flows towards San Francisco Bay (ACFCD, 1984).

The Centerville and Fremont Aquifers are both confined by thick clay layers that extend westward beneath San Francisco Bay. These Aquifers consist of discrete sand and gravel lenses ranging from 5 to 60 feet in thickness. Recharge to these aquifers likely occurs by lateral ground water flow and leakage. The direction of ground water flow in both aquifers has not been documented (ACFCD, 1984).

The deeper aquifers are generally comprised of sand, gravel and cobbles deposited by Alameda Creek to the south. They are believed to be recharged by subsurface inflow from the south and leakage from the Fremont Aquifer. The direction of ground water flow in the deeper aquifers has not been documented (ACFCD, 1984).

The initial hand augured borings at the site showed evidence of gravelly sand and fill material to about a 3-4 foot depth followed by a few feet of black and brown clay, and then a layer of firm silts, sands and brown clays to a depth of about 12 to 13 feet, followed by another layer of brown clays.

AERIAL PHOTOGRAPH REVIEW:

Aerial photographs were reviewed at Pacific Aerial Survey in Oakland. Photographs were reviewed from 1947 to 1981. The site is located south of the Hayward airport in what appears to be the most industrialized area south of the airport in photos dating back to 1947. The 1953 photographs appeared to have a small orchard on it. The site is industrialized with residential developments boarding on the east. Through 1981 the Berkeley Land Company's property was a single lot and not subdivided as it currently is between Quality Tow and Berkeley Farms.

Trident trucking was not evident until 1975, older photos prior to that time show that the lot was vacant or had trees on. The aerial photographs revealed a large lot in back of several houses along Saklan Road across from Trident Trucking. The access to this lot was a narrow gravel driveway between two houses, which from the street tended to disguise the fact that there was a truck lot behind the houses. On March 14, 1990 the site was revisited and was found to be an extension of Tridents facilities with a fuel island located in the middle of the lot. It should be noted that this fuel island is directly upgradient approximately 500 to 600 feet from Berkeley Land Company and "appears" to be the source of diesel contamination.

DISCUSSION OF RESULTS/CONCLUSIONS:

The soil/water and vapor probe samples appear to contradict each other with regards to the level of contamination and areas of highest concentration on the property. Berkeley Land Companies records were inadequate with regards to how the two monitoring wells were placed. Apparently the contractor was released before the job was completed. It appears that if additional soil samples were collected and at the same depth as the vapor probe samples they would support the vapor probe survey. Which tends to show that the contamination is coming from off site especially since Berkeley Land Company or Berkeley Farms does not have any records of diesel purchases for this site.

Of primary concern is the fact that there is 2.17 feet of diesel floating in site's water well which tends to indicate that there is contamination of a deeper aquifer. The contamination has been found to be 100% diesel No. 2. However, soil samples collected by previous consultants and CEC had small amounts of more volatile compounds in them, including some benzene which would be expected in gasoline. The soil and water samples collected in the next phase of this assessment will need to be tested for benzene, ethyl benzene, toluene, and xylenes.

The ground water gradient was estimated to be northwest. Although ground water flows only in one direction, the gradients may be influenced by non-horizontal direction, especially in ground water recharge or discharge areas. Ground water may also flow in different directions beneath a site due to seasonal recharge, discharge from wells etc.

Ground water gradient can be determined by three monitoring wells, however, four wells allow the separate determinations of the gradient. If each of the four determinations of ground water gradient is the same then the flow direction has been determined. The frequency of ground water monitoring will be determined by the Regional Board which is made after evaluating the site assessment report and remediation plan.

There are over twenty sites within a 3/4 mile radius of this site that have had leaking fuel tanks, some with significant contamination. However, because of the distance and direction they don't appear to be the most likely cause of the contamination on Berkeley Farms Lot. The source of the contamination appears to be southeast of Berkeley Land Co. and possibly Trident Trucking Inc. This conclusion was drawn because of the increasing soil contamination measured as the vapor probes were inserted closer to the fuel island off Saklan road, and Tridents's relative closeness to the site.

It appears that there is potential for contamination of the drinking water aquifer which supplies the homes along Saklan Road. Many of these homes have wells visible on their properties which may or may not be in use. The depth of the Berkeley Farms water well is approximately 42 feet according to Berkeley Land Co. A possible reason for the large amount of diesel in Berkeley Land Co. well is that the well is directly in line with the water gradient and has in effect acted as a recovery well drawing the contamination towards it.

If Trident is the source of the contamination recovery wells will be needed between Berkeley Land Co. and Trident's tanks to prevent additional contamination from being brought on site.

Additional study is required to meet the assessment requirements of Alameda County and to verify the potential source of and extent of the contamination plume, including an evaluation with the county or Hayward to determine if the Saklan Road drinking water aquifer has been contaminated. This initial assessment appears to indicate that the source of the diesel contamination is Trident Trucking Inc.

Remediation plans can not be effectively designed or implemented until a more in depth assessment has been completed which includes placement of four sample borings, four monitoring wells and one recovery well on site. The sample borings and placement of the wells would also include complete logging of the core material, soil samples at 5 foot intervals for Total Petroleum Hydrocarbons as diesel and gasoline, and ground water samples. Placement of these wells and the collection of additional samples will allow CEC to quantify the contamination on the site, and better characterize the plume which will be necessary to develop effective remediation plans.

State regulations require that investigative work be conducted to assess the magnitude and extent of petroleum hydrocarbon contamination and evaluate the beneficial ground water usage in the vicinity of the site. To meet these requirements CEC will work with the county to determine the location of both private and municipal wells within a half 1/2 mile radius and survey selected wells especially those upgradient for contamination (Note only a couple of close down gradient wells will be surveyed initially). A remediation plan including cost estimates will be developed for Berkeley Land Co., and the regulator once the magnitude of the contamination to ground and drinking water aquifers has been determined.

Both the Hayward Fire Department and Regional Water Quality Control Board require that soil containing gasoline or waste oil at concentrations over 1,000 ppm be removed. Because the extent of soil contamination is not know the quantity of soil to be removed or treated can not be determined until additional sampling and analysis is completed. Ground waters monitoring wells are recommended in the locations indicated in Figure 1.

Before starting any remediation or further investigation, this report and its recommendations should be submitted to the Hayward Fire Department Hazardous Materials Section and Regional Water Quality Control Board. A meeting should be scheduled with the Fire Department and county to discuss the report and proposal as soon as possible.

RECOMMENDATIONS:

The California Water Resources Control Board LUFT Field Manual of April 5, 1989 requires that if there is known or suspected ground water contamination from a leaking underground tank the following actions must be taken:

1. Collect water and soil samples;
2. Laboratory analysis of samples for BTX&E and TPH;
3. Determine ground water gradient and drill wells as necessary;
4. Consult with the Regional Board to access impact on ground water use, and determine required remedial action;
5. Monitor site to ensure effectiveness of remedial action.

To complete the preliminary assessment requested by Alameda county the following actions are recommended:

1. Install 4 monitoring wells as recommended in Figure 1 and Figure 2, collect soil and water samples;
2. Place four sample borings on Saklan Road to a depth of 20 feet and as indicated in Figure 1 on Berkeley Land Company's lot;
3. Borings will be logged by a CEC geologist and soil samples collected at 5 foot intervals will be submitted to a State certified laboratory for TPH and BTX&E analysis using EPA methods 8015/8020/5030 procedures as outlined in this report;
4. The ground water level should be measured in accordance with the procedures outlined in this report;
5. Ground water samples will be analyzed for TPH and BTX&E using EPA method 8015/5030/602 in accordance with the procedures outlined in this report;
6. If free floating fuel is found in any of these wells the thickness of product and the corrected water level will be measured according to procedures outlined in this report;

7. All soil cuttings and fluids produced during drilling, well development and ground water quality sampling will be drummed and temporarily stored on the site until classified and appropriate disposal of the material is determined;

8. Resurvey the site, relative well elevations and confirm the site ground water gradient;

9. Conduct slug tests and physical soil analysis to determine ground water velocity and hydraulic conductivity.

10. Determine the location of public and private wells within a 1/2 mile radius of the site and collect 3 water samples upgradient and down gradient of the site;

11. Provide Berkeley Land Co., and their attorney with technical support to notify OES of drinking water contamination;

12. Install a recovery well as indicated in Figure 1 an initiate ground water clean up using an extraction type depression pump and skimmer. Note: The fire department will be notified to oversee or provide guidance for product removal. The removed product will be stored in an above ground tank.

WELL CONSTRUCTION AND SAMPLING SPECIFICATIONS

CEC's standard procedures for well installation and soil/water sampling meet or exceeds guidelines set forth by the EPA and the California State Regional Water Quality Control Board.

Wells are drilled with a hollow-stem, continuous-flight auger. All borings and logging will be overseen by a registered engineer or geologist with special attention given to the avoidance of cross contaminating underlying aquifers. Although each boring presents unique conditions, several methods used by CEC geologists and engineers prevent pollution of clean aquifer zones underlying contaminated zones. These methods include: (1) when boring in a zone of saturated permeable materials, drilling will cease if 5 feet of impermeable material (usually clays) are reached. The clay layer is most likely an aquiclude separating the shallow and deep aquifers and should not be penetrated. (2) In areas where the depth to the water table is not know, the hole will be bored down to groundwater or to a maximum depth of 45 feet if groundwater is not encountered unless local guidelines specify greater depths. This is above nearly all deep aquifers currently supplying groundwater in the Bay Area counties. If a saturated shallow aquifer is found which is greater that 20 feet thick drilling will be terminated 20 feet below the top of the aquifer.

If water is not found within 45 feet of the surface, a vapor monitoring system will be installed at a depth lower than the bottom of the tank. The access hole will be filled with a mixture of cement and bentonite to prevent downward movement of contaminants.

The PVC well casing used for both vapor and groundwater monitoring is carefully cleaned with trisodium phosphate and then steam cleaned. The casing is slotted throughout the zone of saturation and sections are connected with dry thread or slip joints. Using any type of glue to connect casing sections could cause false analytical interpretations of water quality. The casing extends one foot above ground surface and is capped to prevent surface contamination. When monitoring wells are placed in traffic areas where they cannot extend above the surface, cast iron boxes and covers are installed.

After the casing has been installed the space between the outside of the casing and sides of the boring is filled with washed gravel through the entire perforated zone. the casing is cemented from the top of the perforated zone to one foot below the surface.

An exploratory boring (pilot hole) will be drilled for monitoring wells to obtain samples of the subsurface strata. Monitoring wells are designed to measure the potentiometric surface and water quality of specific hydro-stratigraphic zone. The proposed locations of the monitoring wells are shown on Figure 1 of the report.

Exploratory borings for the monitoring wells will be drilled using hollow-stem augers. The augers have an inside diameter 3-5/8 inches and an outside diameter of 8 inches. The actual depth of each boring will be specified by the geologist assigned to the drill rig. If floating product is found its thickness will be measured using an interface probe.

All drilling equipment will be steam cleaned before and after drilling of each exploratory hole. Only clean water from a municipal supply will be used for decontamination of drilling equipment.

The final borehole diameter will be sufficiently large to allow placement of a specified type and size of well casing, screen ad filter.

The engineer shall specify to the driller the penetration rate, depth of soil damage. Final disposal of the soil. All drums will be the responsibility of Berkeley Land co, through CEC.

Excavated soil may be stockpiled in an area that is away from storm drains (at least 100) inside the fenced area and away from surface water flow channels. Once the excavation or borings are complete the stockpiled contaminated soil will be sampled. A composite sample will be collected to ensure that the sample is representative of the stockpiled soil.

Options for Handling Soil

Some options will include disposal as residue or hazardous solids. Treat the soil on-site or send to a recycler such as asphalt manufacturing. Disposal options will depend on the amount of soil, contamination type and concentration, costs and long term liability for disposal methods.

1. Logging of Boreholes

   The drill rig operator and the CEC geologist will discuss significant changes in material penetrated by the drill, changes in drilling conditions, hydraulic pressure and drilling action. The CEC geologist will be present during drilling of exploratory borings and will observe and record changes by time and depth and evaluate the relative moisture and content of the samples and note water produce zones. This record will be used later to prepare a detailed lithologic log. The lithologic description of the log will include soil or rock type, color, grain size, texture, hardness, degree of induration, calcareous content, presence of fossils and other materials (gypsum, hydrocarbons) and other pertinent information. A copy of the logs will be retained in the field file at the project site.

2. Exploratory borings will either be completed as monitoring wells (see PROTOCOL No. 2) or will be abandoned by filling a hole with cement grout. Cement shall be placed in one continuous pour through a cementing pipe. The cementing pipe initially will be placed near the bottom of the hole and will remain submerged int he

grout during the entire cementing operations. Cement will continue to be pumped until return of fresh grout (uncontaminated by cuttings) is witnessed at the ground surface.

Installation of Monitoring Wells

Each monitoring well will be designed to register the potentiometric surface, facilitate soil sample permit water sampling. Using the lithologic log for control the CEC geologist in consultation with the Project Manager will specify the exact depths of screened intervals. Drilling and logging of the exploratory borings for the monitoring wells will be in conformance with procedures in this manual. Construction and completion of all monitoring wells will be in conformance with the following provisions.

Screen and Riser Pipe

The monitoring well assembly will consist of four-inch diameter polyvinyl chloride (PVC) schedule 40 (minimum) casing.

The perforated casing (well screen) shall be factory slotted. The perforations will be 0.020 inches in size. These perforated casing sections are not intended to provide optimum flow but will provide hydraulic connection between the pervious material in the water-bearing zone and the monitoring well.

Setting Screens and Riser Casings

Upon completion of drilling, the monitoring well will be assembled and lowered to the bottom of the boring. The monitoring well will be designed so that the well screen is approximately opposite the water-bearing zone that will be monitored. The bottom of the screen shall be approximately flush with the bottom of the well and shall be capped with a threaded PVC cap or plug. The PVC joints will be flush coupled and threaded. No PVC cement or other solvents are permitted to be used to fasten the joints of casing or screen.

After the monitoring well assembly has been lowered to the specified depth, filter sand or gravel will be placed in the annular space to a level of about 1 foot above the top of the perforated casing. The depth to the top of the filter pack will be verified using the tremie pipe or a weighted steel tape.

Once the depth to the top of the filter pack has been verified, a layer of bentonite pellets will be placed with space to fill the annular space to a level of about 1 foot above the tope of the filter pack. The depth to the top of the bentonite pellets layer must be verified using the tremie pipe or a weighted steel tape.

A bentonite/cement grout seal will be placed above the bentonite pellets layer by pumping through a 1-inch diameter PVC pipe lowered to the bottom of the grouted zone. The bentonite/cement grout will be a mixture of Portland cement, approximately 5 percent by weight (of cement) powdered bentonite and approximately 9 gallons of water. Only clean water from a municipal supply shall be used to prepare the grout. The grout seal will extend from the top of the bentonite pellets layer to the ground surface. After grouting, no work will be done on the monitoring well until the grout has set a minimum of 48 hours.

Development of Monitoring Wells

When the monitoring well installation is complete, the well will be developed by surging, and bailing and/or pumping. Well development generally suppresses damage to the formation by drilling operations, restores natural hydraulic properties to the adjacent soils and improves hydraulic properties near the borehole so the water flows more freely in the well.

During development, pH, specific conductance, and temperature of the return water will be measured. Well development will proceed until these field water quality parameters have stabilized and the water is, in the judgment of the geologist, at its maximum possible clarity. Wells will be developed by removing the finer material from the formation and filter pack surrounding the wells. The procedure consists of two steps, bailing the well followed by pumping it until it produces clean water.

Water generated during development procedures will be placed in 55-gallon drums for temporary disposal. Final disposal will be the responsibility of Berkeley Land Co.

Capping Monitoring Well

Upon completion of the work, a suitable vented screw cap will be fitted on the end of the riser casing to prevent the entry of surface runoff of foreign matter. A steel well cover with a locking top will be set in mortar around the riser casing for protection. The steel protective well cover will be completed below the ground surface in a pre-cost concrete valve box with a traffic-rates cover.

Documentation

A well construction Diagram for each monitoring well will be completed by the geologist and submitted to the Project Manager when the work has ben completed. In addition, the details of well installation, construction, development and field measurements of water quality parameters will be summarized as daily entries in a field notebook or data sheets which will be submitted to the Project Manager when the work has been completed.

Cleaning of Drilling Equipment

Procedures for all drilling equipment will be therefore steam cleaned after drilling using clear water from a municipal service.

Well casing, whether constructed of PVC or steel, will be cleaned thoroughly before installed to prevent cross contamination.

The following cleaning procedure may be used or adapted as appropriate for conditions.

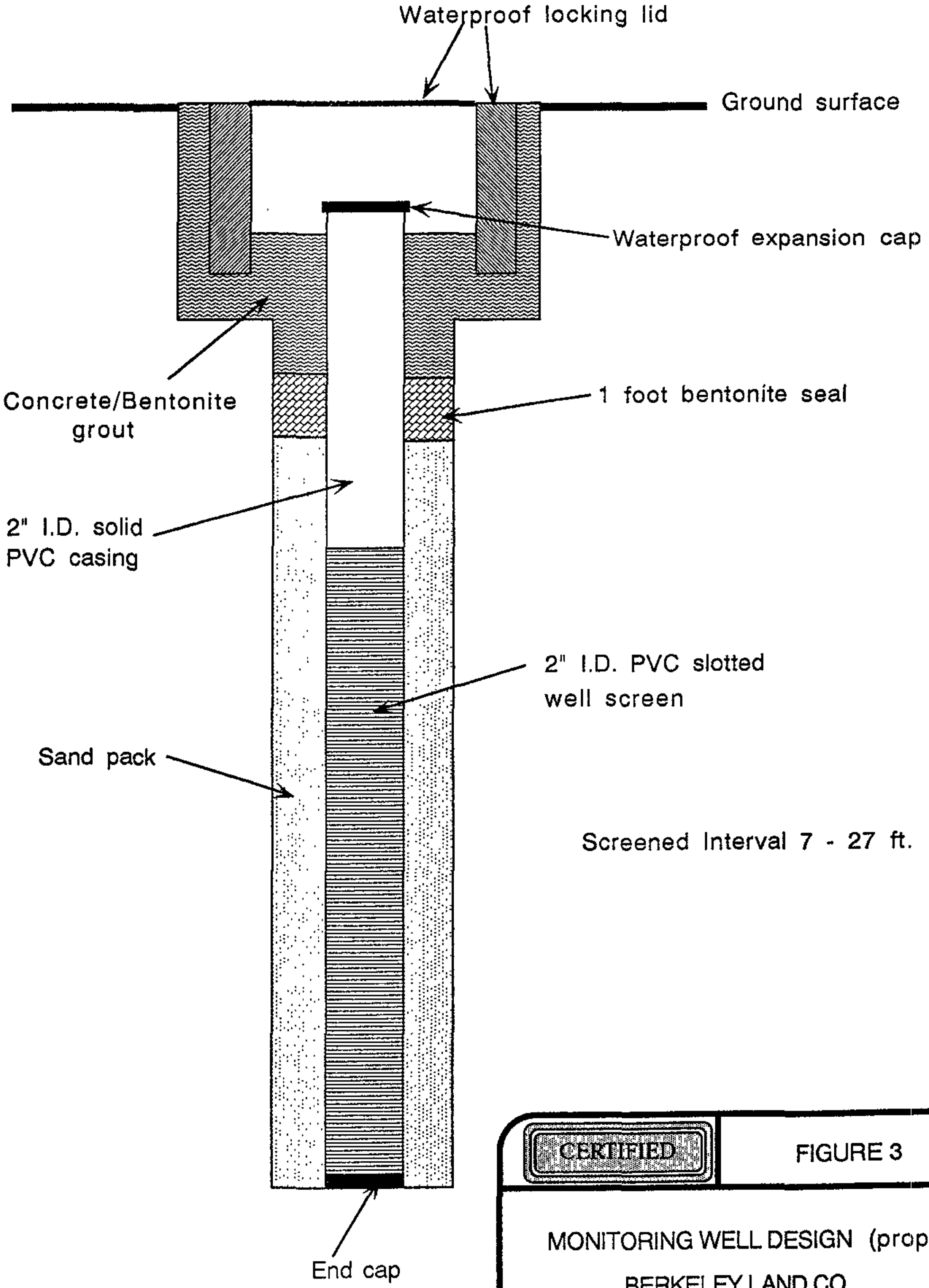
1. Swab surfaces, inside and out, with detergent-water solution.

2. Steam wash with a detergent-potable water solution.

3. Steam rinse with clean potable water.

4. Cover with plastic to protect equipment from contact with solvents, dust or other contaminants.

Equipment used for well development will be steamed clean.



CERTIFIED

FIGURE 3

MONITORING WELL DESIGN (proposed)

BERKELEY LAND CO.
23555 SAKLAN RD.
HAYWARD, CA.

SAMPLING METHODOLOGIES

GENERAL PRACTICES

U.S. Environmental Protection Agency standards serve as the foundation for all field sampling operations performed by CEC. The EPA SW 846 is the primary publication from which procedures are derived. Sampling related to underground storage tanks and tank related threats to groundwater are regulated by the California Water Resources Control Board and its Regional Water Quality Control Boards. While some aspects of field and laboratory work may be delegated to the California Department of Health Services, the CWRCB and the Regional Water Quality Control Boards establish the general and specific criteria for sampling performed in connection with underground storage tanks.

Other agencies, may require additional samples, but these are usually <u>in addition to</u> samples required by the RWQCB. Local regulatory agency inspectors are frequently present during the tank removal phase of a project and either direct or request that samples be taken according to RWQCB specifications.

Based on field conditions directly observable by the regulator CEC may be asked to collect samples that are tailored to the specific site and which the inspector judges will provide significant information. quite often these directions coincide with the sampling areas established by the RWQCB as directions coincide with the sampling areas established by the RWQCB as the proper collection points for samples which will be used as the Primary Criteria for a Regulatory Agency Determination on whether additional exploration or remediation will be required at a particular site.

<u>Hand Driven Core Sampling</u>: This is another term for undisturbed soil sampling. This is the preferred sampling method or both geotechnical and environmental investigations because the method captures a relatively undisturbed cylinder of soil which can be retained in its sealed brass liner during transport to a laboratory for examination Whether driven by a drill rig a hand auger or hand operated slide hammer, the methodology remains the same.

Because of the tons of force which can be exerted by a drill rig, the samplers, drill rod and hammers are, necessarily, quite massive. Equipment used in hand augured borings is usually much lighter and more subject to wear and breakage. Specialized hand tools that enable a person to drive samples consist of a sampling show (which contains the brass liners), light weight drill rod, and a small slide hammer. These hand operated drive samples collect samples in the same two inch diameter brass liners used in many drill rig samplers, but collect only a four or six inch long core rather than twelve to twenty-four inches of soil commonly obtained by a drilling rig.

Common uses for hand operated drive samplers include those applications where an undisturbed soil sample is desired. Typical applications include the collection of soil samples from the bottom of a hand augured boring, capillary zone sampling where a drill rod is used to extend the sampler across an open pit to a selected location on the wall of the excavation, and when sampling soil from the backhoe bucket that is too hard to allow a brass sample liner to be pushed into the soil by hand.

Sampling and Logging: Representative samples of drive-sample cores will be obtained, evaluated and retained. A detailed log of these samples will be made by a CEC geologist. Grain-size (sieve) analyses may be undertaken on selected samples in potential well completion zones. These analyses will be recorded on standard grain-size analysis sheets. Moisture content and dry density measurements may be performed on selected drive-sample cores to evaluate the degree of saturation.

Obtaining Samples: Undisturbed soil samples shall be obtained for chemical analysis and geotechnical classification at approximate 5-foot intervals or continuously beginning at 5 feet below grade and excluding its depth using a modified California split-spoon sampler. The sample will be lined with clean, thin-walled, segmented brass tubes. Selected samples for analysis will be sealed with aluminum foil, plastic end caps and vinyl electrical tape., The samples will be labelled and placed in an ice-cooled chest for delivery to a state-certified laboratory for chemical analysis. Chain-of-custody records will be completed for each sample delivery.

Soil Sampling: Continuous samples will be obtained from one shallow well boring near the center of the site. Sampling will be conducted using a 2- or 2.5 inch I. D. Modified California sampler containing four 4-inch long or three 6-inch long brass tubes. The sampler and tubes will be decontaminated before and after each use by steam cleaning, or an Alconox solution wash, and tap water followed by deionized water rinses. The sampler will be driven ahead of the augers using a 140 pound drop hammer. The average blow counts required to drive the sampler the last 12 inches will be recorded on the boring logs. The soils exposed at the ends of each brass tube will be examined by a geologist for soil type and the obvious presence of contamination and classified according to the Uniform Soil Classification System. These observations will be recorded in the boring logs.

The sample tubes, will be sealed at each end with aluminum foil or Teflon sheeting, PVC end caps, and placed in ziplock bags before placing in an ice chest for delivery to the laboratory. The middle tube will be extruded into a ziplock bag, sealed and left in the sun for 5 minutes to enhance the volatilization of hydrocarbons that may be present in the soils. A headspace reading will be taken from the interior of the bag using an organic vapor meter. These measurements will be recorded on the field boring logs. Soil cuttings generated during drilling will be placed in drums, labeled, sealed and left on-site for subsequent disposal pending the sample results.

Sample Selection: Selection of samples for laboratory analysis will be based primarily on headspace readings and position within the boring, although some discretion by the site geologist or engineer will be required. In general, samples with headspace readings over 50 ppm or that have visual or olfactory indications of contamination will be submitted for analysis. One sample will also be selected from one to two intervals below the apparent lower limit of contamination to obtain a "zero line" value. In addition, the sample closest to the depth of the storage tank invert (i.e. 12-13 feet) will be submitted for analysis. If the water table is above the tank invert, the sample closest to the water table will be selected.

In the event that no contamination is apparent, the sample closest to the depth of the tank invert and a composite of all other samples down to the water table will be submitted for analysis. The laboratory conducting the analysis will be responsible for compositing the samples

Samples selected for analysis will be labeled according to boring/well number and sample depth and transported under chain-of-custody to state-certified laboratory. An example of a chain-of-custody form is provided in Figure 1.f

Sample Analyses Chemical. Based on the site's history and nature of the released product the samples will be tested for the material(s) released or stored at the site, otherwise the samples will be analyzed for both heavy and light hydrocarbons.

Soil Classification
The United Soil Classification System will be used to categorize soils. See the summary of this system on the next page.

Abandoned Sample Bores Abandoned sample bores will be sealed with bentonite or a bentonite concrete grout mix.

Unified Soil Classification System

Major Divisions			Graph Symbol	Letter Symbol	Typical Descriptions
Coarse Grained Soils More than 50% of the material is Larger than number 200 sieve size.	Gravel and Gravelly Soils More than 50% of coarse fraction retained on number 4 sieve.	Clean Gravels (little or no fines)	▨	GW	Well - graded gravels, gravel-sand mixtures, little or no fines.
			▨	GP	Poorly - graded gravels, gravel-sand mixtures, little or no fines.
		Gravels with fines (appreciable amount of fines)	▨	GM	Silty gravels, gravel-sand-silt mixtures.
			▨	GC	Clayey gravels, gravel-sand-clay mixtures.
	Sand and Sandy Soils More than 50% of coarse fraction passing number 4 sieve.	Clean Sand (Little or no fines)	▨	SW	Well - graded sands, gravelly sands, little or no fines.
			▨	SP	Poorly - graded sands, gravelly sands, little or no fines.
		Sands with fines (Appreciable amount of fines)	▨	SM	Silty sands, sand-silt mixtures.
			▨	SC	Clayey sands, sand-clay mixtures.
Fine Grained Soils More than 60% of the material is Smaller than number 200 sieve size.	Silts and Clays	Liquid limit Less than 50	▨	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands or clayey silts with slight plasticity.
			▨	CL	Inorganic clays or low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
			▨	OL	Organic silts and organic silty clays of low plasticity.
	Silts and Clays	Liquid limit Greater than 50	▨	MH	Inorganic silts, micaceous or diatomaceous fine sand or silty soils.
			▨	CH	Inorganic clays of high plasticity, fat clays.
			▨	OH	Organic clays of medium to high plasticity, organic silts.
Highly organic soils			▨	PT	Peat, humus swamp soils with high organic content.

TABLE 4-1
SAMPLING PROCEDURES TO BE USED FOR BARREL AUGER

1. The sampler will be placed at the selected soil sampling location and depth.

2. With the sampler and extension rods in the vertical position, the sampler will be rotated into the sampling zone.

3. When the sampler penetrates the subsurface to a selected depth, or is filled with a selected volume of soil, the assembly will be pulled out of the cavity.

4. The auger will be carefully removed from the cavity and the barrel head gently placed on a clean board or other support. The soil contained in the barrel will be scraped into the sample container using a clean spatula or other tool. The remaining soil will be discarded.

5. The sample containers will be labeled pursuant to the project chain-of-custody procedures.

6. All components of the samplers that come into contact with the soils will be decontaminated prior to their use for the following sampling event.

In addition to the auger sampler used in conjunction with auger drilling, soil samples will be taken with a core sampler. This sampler consists of a 6-inch long hollow tube that contains a metal liner that is driven into the ground. Figure 4-2 illustrates components of the core sampler.

A weighted drop hammer fits onto the tube to facilitate driving the sampler into the soil. Pulling on the hammer forces the sampler out of the cavity. Components of this sampler are constructed from hardened metal.

The core sampler will be driven to a depth of 6-inches into the ground at each sampling depth and location. Samples obtained in this way are referred to as "undisturbed samples" in this document. Table 4-2 describes the sampling procedures to be used for the core sampler.

Upon extraction from the boring, inner metal liner will be removed from the sampler and sealed for shipment to the certified project laboratory.

TABLE 4-2
SAMPLING PROCEDURES TO BE USED FOR THE CORE SAMPLER

1. The sampler tip will be placed on the soil surface at the desired sampling depth and location after removing surface debris.

2. The tube will be positioned vertically above the ground surface. The hammer will be raised and dropped with both hands. The hammering will continue until the barrel of the core sample has been driven completely below the soil surface.

3. The hammer will be then pulled upward to force the tube out of the ground.

4. The unit will be carefully removed from the cavity. The sampler will be placed on cardboard or a clean board. The housing will be unscrewed and removed. The brass liner will be removed and the ends of the liner will be capped and sealed.

5. The sample container will be labeled pursuant to the project chain-of-custody procedures.

6. All components of the sampler that come into contact with the soils will be decontaminated prior to their use for the following sampling event.

GROUNDWATER SAMPLING

If groundwater is encountered during the excavation a sample will be collected. VOA bottles with teflon line caps will be used.

Sample Containers CEC uses new sample containers specified by either EPA or the RWQCB for the sample collection. Water samples are contained in 40 ml volatile organic analysis vials (VOAs) when analysis for gasoline and similar light volatile compounds is intended. These containers are prepared according to EPA SW 849 and will contain a small amount of preservative when the analysis is for TPH as gasoline or EPA 602. Vials intended for EPA 601 analysis and EPA 624 GCMS procedures are not preserved. Closure is accomplished with an open headed (syringe accessible) plastic screw cap brought down on top of a Teflon faced septum which is used to seal the sample without headspace.

Sample Containers and Volumes Water samples intended for semivolatile and nonvolatile analysis such as total oil and grease (TOG) and diesel (TPH HBF) are collected and transported in prepared dark new glass liter bottles to reduce any adverse effect on the sample by sunlight. Antimicrobial preservative may be added to the sample liquid if a prolonged holding time is expected prior to analysis.

The sample containers will be either glass or polyethylene and will be obtained from the laboratory or other approved source. Separate containers will be required for specific groups of analytes in accordance with EPA methods (40 CFR Part 136 and SW-846).

Sample containers, either glass or polyethylene, will be labelled with self-adhesive, preprinted tags having the following information written on them.

1. Project number (or name)
2. Sample number (or name)
3. Sample location (well number, etc.)
4. Date and time samples were obtained
5. Treatment (preservative added, filtered, etc.)
6. Signature (first initial, last name) of sample collector

Sample tags shall be filled out using water proof ink.

Sample Preservation and Storage Preservation and storage of samples is dependent on the chemical constituents to be analyzed and will be discussed with the laboratory prior to sample collection.

Water Sampling Water Samples are collected in any of several appropriate devices such as bailers, Coliwasas, Middleburg sampling pumps etc. Sample liquid is decanted into new sample containers in a manner which reduces the loss of volatile constituents and follows the applicable EPA procedures for handling volatile organic and semi-volatile compounds. Only two variations from the EPA methods are generally employed. First, preservative is added to the sample container prior to addition of the sample liquid.

Monitoring wells will be constructed by inserting a 4-inch flush threaded schedule 40 PVC well casing from the bottom of the boring to the ground surface. From approximately 5 feet above the ground water surface to the base of the well, the casing will consist of machine slotted well screen. The remainder of the well will be solid PVC casing. The filler pack will be placed in the annular space between the 4-inch well casing and borehole and will extend from the bottom of the well to approximately 2 feet above the top of the screened casing. A layer of bentonite approximately 1 foot thick will be placed above the filled packs and charged with water: The remaining space will be backfilled using a çement bentonite grout mixture. A locking aluminum well cap will be connected to the top of the well to protect it from ground surface traffic. The well will be completed at the ground surface and will be developed until the groundwater is visually clear and free of sediment.

The wells will be developed and sampled using a teflon bailer, washed with TSP, rinsed with tap water and then distilled water. A minimum of four well volumes will be removed from the well prior to sampling. To insure that water in the well was exchanged: (1) bailing will commence at the top and work downward; (2) bailing will be done briskly to draw the well down, thereby reducing the percentage of recharge water in relation to possible stagnant water within the well. The well will be allowed to return to 80 percent of the original water level before sampling. The sample vials and bottles will be filled to overflowing; (1) the precludes air bubbles passing through the sample during filling, and (2) sealed so that no air is trapped in the vial. Once filled, samples will be inverted and tapped to test for air bubbles. Samples will be placed on ice and delivered to the lab as soon as possible.

Water Level Measurement All water-level measurements will be recorded to the nearest one hundredth of a foot, and all depth measurements shall be noted to the nearest half foot, and all depth measurements shall be noted to the nearest half foot. For a period of 48 to 72 hours strip chart recorders will be used to document changes in groundwater levels.

1. Water-Level Measurements in Non-Flowing Wells

   Water-level measurements in non-flowing wells will be done by one of the following methods: Following the water level measurement a submersible pump well be lowered into the well and pumped unit three to four well volumes of water have been removed from the well. This is necessary to ensure representative sampling of the groundwater aquifer vs standing well water.

2. Wetted-Tape Method

A steel surveyors tape (e.g., Lufkin Highway Nubian Type B) shall be prepared by coating several feet of the lower end of the tape with chalk. A lead weight is attached to the lower end of the steel tape to keep it taut. The tape is lowered into the well until a foot or two of the chalked portion is submerged.

a. Lower and hold the tape at an even foot mark at the Measuring Point (MP) and note this tape reading.

b. Remove the steel tape from the well. Subtract the wetted length from the even foot mark noted in Step 1 and record this as Water Level Below MP.

3. Electric Sounder Method

An electric sounder consists of a contact electrode that is suspended by an insulated electric cable from the reel that has an ammeter, a buzzer, or other closed circuit indicator attached. The indicator shows a closed circuit and flow of current when the electrode touches the water surface. The electric sounders used in this project shall be calibrated by measuring each interval and remarking them where necessary.

The procedure for measuring water levels with an electric sounder is as follows:

a. Switch on.

b. Lower the electric cable into the well until the ammeter or buzzer indicates a closed circuit. Raise and lower the electric cable slightly until the shortest length of cable gives the maximum response on the indicator is found.

c. With the cable in this fixed position, note the length of cable at the Measuring Point (MP).

WELL TOTAL DEPTH MEASUREMENTS

Total depth of a well shall be measured by sounding with a weighted steel surveying tape or an electric sounding line, weighted when possible. Procedures to be followed are described below.

1. Measure the distance between the zero mark on the end of the measuring line and the bottom of the weight.

2. Lower the weighted steel tape into the well until the tape becomes slack or there is a noticeable decrease in weight which indicates the bottom of the well. Raise the tape slowly until it just becomes taut (this may have to be done several times to feel

that taut point), and with the tape in this fixed position note the reading at the Measuring Point. Adjust it for the difference described in Step 1, and record the resulting value as Well Depth.

3. Record the Well Depth value in a bound field notebook or on a data sheet.

FREE-PRODUCT MEASUREMENT

In wells where there is free product, the depth to the free-product surface will be measured using the wetted-tape method. The thickness of free product then will be measured using a graduated, clear acrylic bailer.

A corrected water level elevation will be calculated by subtracting the free-product thickness from the free-product level elevation and accounting for the buoyancy effect of the free-product.

SAMPLE COLLECTION

Individual ground or other samples will be collected for quality analysis.

1. Wells will be pumped or bailed at least until the volume of water pumped is equal to or four times the volume of the well's casing (purging of well) prior to sampling. To assure that the water samples are representative of the aquifer, periodic measurements of the temperature, Ph and specific conductance will be made. The sample will be collected only when the last temperature measure is within +/- 0.5° C, the pH is within 0.1 unit and the specific conductance is within +/- 5 percent of the previous reading.

2. A positive-displacement pump (blader or reciprocating) and/or bailer will be used for evacuating the casing of the monitoring wells in the ground-water sampling network. A Teflon or disposable bailer will be used to collect the water samples for laboratory chemical analysis.

3. A sample container and cap will first be rinsed thoroughly with the water to be sampled. The container will then be filled to overflowing to avoid trapping air at the top of the container. Splashing of the water in the sample container and exposure to the atmosphere shall be minimized during sampling. A container cap will be screwed on tightly immediately after filling the sample container. Samples for volatile organics analysis (VOA) will be collected in duplicate in VOA bottles and checked to be sure no headspace is present in the sample container.

4. The sampling sequence for the wells will begin with the well expected to have the least contamination. Successive samples will be obtained from wells of increasing suspected contamination. If the relative degree of suspected contamination cannot

be reasonably assumed, sampling will proceed from the perimeter of the site towards its center. The sampling sequence will be arranged such that wells are sampled in order of increasing proximity to the suspected source of contamination.

5. A map will be used in the field to record sample locations. A field log book or data sheets will be used to record the following information:

    a. Sample number (or name)

    b. Date and time sampled

    c. Sample location (well number)

    d. Depth at which sample was taken

    e. Conditions of source (flowing, pumping, etc.)

    f. Extraordinary circumstances (if any)

    g. Name collector

    h. Results of field measurements (temperature, pH, specific conductance)

QUALITY CONTROL SAMPLES

To document the precision and accuracy of analytical data generated by the subcontracted laboratory, one or more of the following procedures will be employed periodically:

1. <u>Duplicate Sample</u> - A double volume of water will be collected and preserved. The sample will then be divided into identical containers that are given different location identification names or sample numbers. These numbers are recorded in a field log book or on a data sheet for later reference. Both samples are submitted to the laboratory for identical analyses.

2. <u>Blank Sample</u> - Blank samples will consist of travel blanks and field blanks. A travel blank will consist of deionized water that is added to the various sample bottles. Preservatives supplied ;by the laboratory will also be added to sample bottles as appropriate. At least one set of travel blanks will be submitted for each set of samples collected and delivered to the laboratory. Travel blanks will be kept in separate storage containers from the groundwater samples. A field blank will consist of deionized water that is added to the sample bottles at the time of sample collection. The field blank sample bottles are preserved in the same manner as the groundwater samples and may be stored with the groundwater samples.

STORAGE AND TRANSPORTATION

Sample exposure to dust, direct sunlight, high temperature, and adverse weather conditions will be avoided whenever possible. Samples shall be transported to the laboratory as soon as possible and on no more that 48 hours from the time they were collected. All samples will be stored in and kept in plastic "ziplock" bags, a closed ice chest until delivery to the laboratory.

FIELD MEASUREMENTS

The following field measurements will be performed on groundwater samples. Data obtained from field water quality measurements will be recorded in the field log book or data sheets. Separate aliquot of groundwater from a monitoring well will be used to make field measurements (samples for laboratory analysis shall not be reopened).

TEMPERATURE MEASUREMENT

Temperature measurements will be made with a good grade mercury-filled Centigrade thermometer, bimetallic-element thermometer, or electronic thermistor.

PH MEASUREMENT

pH measurements will be made as soon as possible after collection of the sample preferably within a few minutes.

SPECIFIC CONDUCTANCE MEASUREMENT

Conductivity will be measured by dipping the conductivity probe in the water source or sample. The probe must be immersed above the vent. The temperature of the sample will be used to calculate specific conductance from the conductivity measurement. Measurements shall be reported in units of micromhos per centimeter at 25 degrees Centigrade.

EQUIPMENT CALIBRATION

Equipment used for field measurement of organic, vapor, pH and specific conductance shall be calibrated per manufacturers guidelines. Calibration shall be documented in the field log book or data sheets and will include a description of the calibration method, identification number of equipment and/or regents used in calibration.

DOCUMENTATION

Log Book or Data Sheets A log book containing information collected during sampling operations, as described above, will be maintained. It will be in a bound notebook with consecutively number pages. Pages may not be removed for any reason. Entries in the log book shall be made with indelible ink. Errata may be marked out with a single line, and initials of person making the change.

Alternatively, specially formatted field data sheets may be used to record the information collected during water quality sampling.

The log book and data sheets will be placed in the project file when sampling is completed.

Equipment Cleaning

Sample bottles and bottle caps will be cleaned by the laboratory using standard EPA-approved protocols or new unused containers. Sample bottles and bottle caps will be protected from solvent contact, dust or other contamination between time of receipt by CEC sampling.

Sampling equipment for this project may be dedicated to a particular well. Prior to installation of this equipment, all equipment surfaces that will be placed in the well or come in contact with groundwater will be cleaned to prevent cross contaminates.

Sampling equipment that will be reused will be cleaned after sampling at each well is completed.

FIELD EQUIPMENT DECONTAMINATION

A major concern with soil and groundwater sampling is the potential of cross-contamination of samples. Therefore, care will be taken to properly decontaminate all sampling equipment. Drilling and sampling tools, sample containers, and any other equipment which comes into contact with the samples will be cleaned or disposed of after each sample in order to preserve the integrity of the sample.

All tools, auger segments, and samplers used in the drilling and sampling program will be cleaned prior to being used. All such items will be cleaned prior to beginning work at each subsequent site, between each boring, and between each sample location.

Cleaning will include washing with appropriate detergents and solvents followed by a distilled water rinse and air drying. Wash water and solvent rinses will be collected at each site and containerized. Field equipment decontamination procedures to be used are summarized in Table 4-4.

SAMPLE HANDLING

Soil and groundwater samples collected using the techniques described previously in this section will be handled in the manner described in Table 4-3. Sample containers will be sealed with labels, and the samples will be delivered to the project laboratory for analysis. The samples will be accompanied by chain-of-custody records (discussed in Section 5.0) and sample analysis request sheets. The person who last signed the chain-of-custody record will accompany the samples to the laboratory and sign the samples over to the laboratory personnel authorized to receive the samples. In turn, the authorized laboratory staff will sign the chain-of-custody record and take possession of the samples. The laboratory will then assign their internal quality control and sample inventory codes to each sample, and record the codes on the chain-of-custody sheets. Copies of these records will be sent to CEC for further sample inventory, and placed in the permanent project file.

TABLE 4-3

SAMPLE HANDLING PROCEDURES TO BE USED IN THIS PROJECT

1. Soil samples will be placed into sample containers or liners sealed immediately upon their extraction from samplers. When brass sample tubes are used, the tube will be completely filled with soil so there is no head space. Next the ends of the tube will be sealed with aluminum foil or teflon sheeting prior to attaching the snugly-fitting plastic caps.

2. The caps will be replaced on the containers. The containers will be labelled and sealed to prevent loosening of the cap during transport to the laboratory and to protect the integrity of the sample.

3. The samples will be immediately chilled for collection. Ice will be sealed in plastic bags to avoid wetting sample bottles. (Note: Dry ice can be used for soil samples). Temperature in the cooler will be maintained at 4° C from the time of collection until they are received by the laboratory.

TABLE 4-4

FIELD EQUIPMENT DECONTAMINATION PROCEDURES TO BE USED IN THIS PROJECT

1. Equipment will be washed and scrubbed with an Alconox detergent solution using a bottle brush.

2. Equipment will then be double rinsed with distilled water.

3. The equipment will then be air dried.

4. Package and seal equipment in plastic bags or other sealed containers to prevent contact with solvents, dusts or other contamination.

QUALITY ASSURANCE AND QUALITY CONTROL (QA/QC)

In order to assure the quality of the analytical data, quality control blanks will be collected and analyzed for the same parameters as the investigation samples.

For each shipment (and type) of sample containers from the analytical laboratory, one container will be filled with Type II reagent grade water at the laboratory and sealed. The blanks will be transported to the site with the balance of the sample containers and be handled in the same manner as the samples. The trip blanks will be returned to the laboratory with the samples for analysis.

Additionally, the QA/QC of the laboratory will be checked by the collection and analysis of one blind duplicate sample each sampling day (water samples only). The duplicate will be labelled with a sample number which will not conflict with the other samples, but will not be discernible to the laboratory as a duplicate. One blind duplicate water sample will be collected and analyzed each sampling day.

Laboratory Custody Procedures

Laboratory custody procedures concerning the acquisition and handling of samples and the documentation required will be in accordance with the procedures outlined below.

1. Receipt of Samples at the Laboratory

   One person will be designated as the sample custodian and be responsible for the receipt of the samples at the laboratory. Upon delivery the custodian will:

   - Observe the physical condition of the sample container(s) noting any broken seals or other evidence of tampering.
   - Open the container and crosscheck to verify that the information on the Chain of Custody Record corresponds to that on the sample labels.
   - Records the information on the sample tags in a laboratory notebook.
   - Reports any damage to, or leakage of samples to the lab supervisor and the CEC Project Manager, and records this information in the laboratory notebook.
   - Signs the Chain of Custody Record, including the date and time received.
   - Places the samples in a secured storage area.

2. Damaged Samples In the event of sample leakage, headspace in the sample bottle or any other evidence of damage to a sample, the CEC Project Manager will determine sample disposition.

3. Sample Storage Samples, prepared samples, and standard solutions will remain in a secured area at all times unless being used for analysis. Access to this area will be limited to the laboratory supervisor, the sample custodian and analytical personnel.

TEST METHODS

1. Skougstand, M.W., and others, 1979, Methods for Determination of Inorganic Substances in Water and Fluvial Sediments: U. S. Geological Survey, Techniques of Water Resources Investigations, Book 5, Chapter AI, pages 7-15.

2. American Public Health Association, 1980, Standard Methods for the Examination of Water and Waste Water (15th ed.), Washington D. C. pages 3-41.

3. Federal Register, v. 49, n. 209, Friday, October 26, 1984, Part VIII (40 CFR 136).

Table 4-5 lists the chemical constituents to be analyzed for selected samples and specifies the method that will be used for each constituent.

In the event that one of these methods proves to be unsatisfactory for accurate and precise determination of a constituent, the laboratory will consult with CEC about an alternate method. The alternate method must be approved by the CEC Project Manager before it is performed; if approved, the reference document detailing the analytical method will be added to the list of references in Table 3.

TABLE 3

WATER QUALITY CONSTITUENTS AND ANALYTICAL METHODS

Constituents	Method	Reference
Total Petroleum Fuel Hydrocarbons (gasoline)	GC/FID[1]	EPA, 1982, Method 5020 for sample preparation
Benzene, Toluene, Xylenes	GC/FID[1]	EPA, 1982, Method 8020
Waste Oil	Gravimetric	EPA, 1984b, Method 413.1
Purgeable Organics	GC/MS[2]	EPA, 1984a, Method 624

References

American Public health Association ("Standard Methods"), 1980, Standard methods for the Examination of Water and Wastewater (15th ed.) Washington, D.C. 1134 p.

RWQCB, 1985, Guidelines for Addressing Fuel Leaks. September 1985.

U.S. Environmental Protection Agency (EPA), 1984, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act (40 CFR Part 136): Federal Register, v. 49, n. 209, Friday, October 26, 1984, Part VIII, Table 1B.

U.S. EPA, 1982, Test Methods for Evaluating Solid Waste -- Physical/Chemical methods -- SW-846, Second Edition, July.

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[1] GC/FID - Gas Chromatography with Flame Ionization Detector

[2] GC/MS - Gas Chromatography with Mass Spectrometer Detector

CHAIN OF CUSTODY PROCEDURES

After samples have been collected and labelled, they will be maintained under chain of custody procedures. These procedures document the transfer of custody of samples from the field to a designated laboratory.

A Chain of Custody Record will be filled out for each shipment of samples to be sent to the laboratory for analysis (Figure 4-1. Information shall include:

- Date and time the sample was taken
- Sample number and the number of sample bottles
- Analyses required
- Remarks including preservatives added and any special conditions
- Container number in which sample has been packaged.

Cross out any blank space on the Chain of Custody Record between last sample number listed and signatures at the bottom of the sheet.

After carefully packaging the sample into numbered containers (e.g., ice chests) for transfer to the laboratory, the field sampler will sign the Chain of Custody Record and record the time and the date. The original imprint of the Chain of Custody Record will accompany the sample containers. A duplicate copy will be retained by CEC.

Sample containers will be sealed with tape such that they may not be opened without breaking the seal. This seal is not to be removed until the container is opened at the laboratory.

When shipping the containers by a common carrier, a Bill of Lading will be used. Receipts of Bill of Lading should be retained as part of the permanent documentation in the project file.

Chain of Custody procedures for tracing the path of a given sample from the moment of collection through all the intervening processes required to deliver the specimen to an analytical laboratory. A sample is considered in custody if:

- It is in the sampler's or the transferee's actual possession.
- It is in the sampler's or the transferee's view, after being in his/her physical possession.

- It was in the sampler's or the transferee's physical possession and then he/she secured it to prevent tampering.
- It is placed in a designated secure area.

Chain-of-custody procedures for this project will establish the documentation and control necessary to identify and trace the soil and groundwater samples from their collection to their analysis. Such documentation includes labeling to prevent mix-up, container seals to prevent unauthorized tampering with contents of the sample containers, and secure custody. These procedures are described in the Environmental Protection Agency (EPA) document entitled "Test Methods for Evaluating Solid Wastes" (EPA, 1982).

Appropriate sections dealing with solids and liquids handling were used in developing the chain-of-custody procedures, which will be used throughout this project.

SAMPLE LABELS

Sample labels are necessary to prevent misidentification of samples. The sample label to be used in this project is a gummed tag, with peel-away backing. The label includes the following information:

- Name of collector
- Date and time of collection
- Place of collection
- Collector's sample number, which uniquely identifies the sample.

A copy of the label is shown in Figure 5-1.

CUSTODY SEALS

Custody seals are used to preserve the integrity of the sample from the time it is collected until it is opened in the laboratory. A copy of the custody seal to be used in this project is shown in Figure 5-2. The seal is gummed, with peel-away backing. It is attached in such a way that it would be necessary to break it in order to open the sample container. The seal will include the following information:

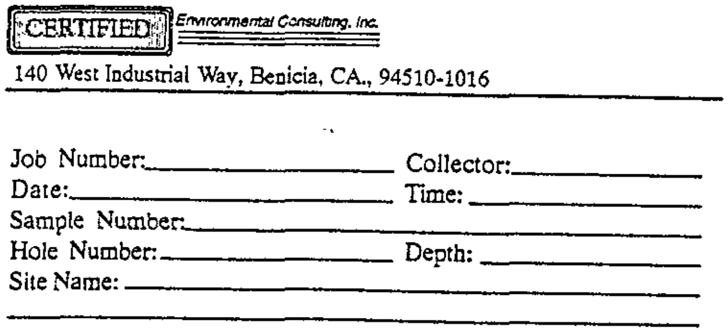
- Date of sampling

- Collector's sample number (This number must be identical with the number on the sample label)

- Collector's signature

FIELD LOG BOOK

Information pertinent to the investigation and sampling will be recorded in a log book. Entries in the log book typically include the following:

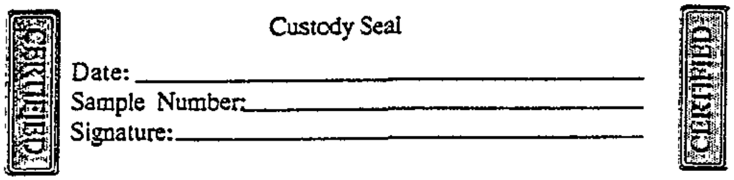


CERTIFIED Environmental Consulting, Inc.
140 West Industrial Way, Benicia, CA., 94510-1016

Job Number:________________ Collector:____________
Date:____________________ Time:____________
Sample Number:______________________________
Hole Number:______________ Depth:____________
Site Name:___________________________________

Figure 5

Sample Label



Custody Seal

Date: ____________________

Sample Number: ____________________

Signature: ____________________

Figure 6

Custody Seal

- Purpose of sampling
- Location of sampling point
- Suspected contaminant (chromium, lead, waste oil etc.) composition
- Number and volume of sample taken
- Description of sampling point and sampling methodology
- Date and time of collection
- Collector's sample identification number(s)
- Sample distribution and how transported (e.g., name of laboratory)
- References such as maps or photographs of the sampling site
- Field observations
- Any field measurements made (e.g., screening of volatile organic with a field gas chromatograph)

The log book is retained in CEC's permanent project file.

CHAIN-OF-CUSTODY RECORD AND SAMPLE ANALYSIS REQUEST SHEET

To establish the documentation necessary to trace sample possession from the time of collection, a chain-of-custody record will be filled out and will accompany every sample. The record will contain the following information:

- Collector's sample number
- Signature of collector
- Date and time of collection
- Place and address of collection
- Suspected contaminants
- Signatures of persons involved in the chain of possession

- Inclusive dates and times of possession.

  Sample analysis request is also included on the chain-of-custody record and accompanies the sample to the laboratory. The laboratory portion of the ill be completed by laboratory personnel and includes:

- Name of person receiving the sample

- Laboratory sample number

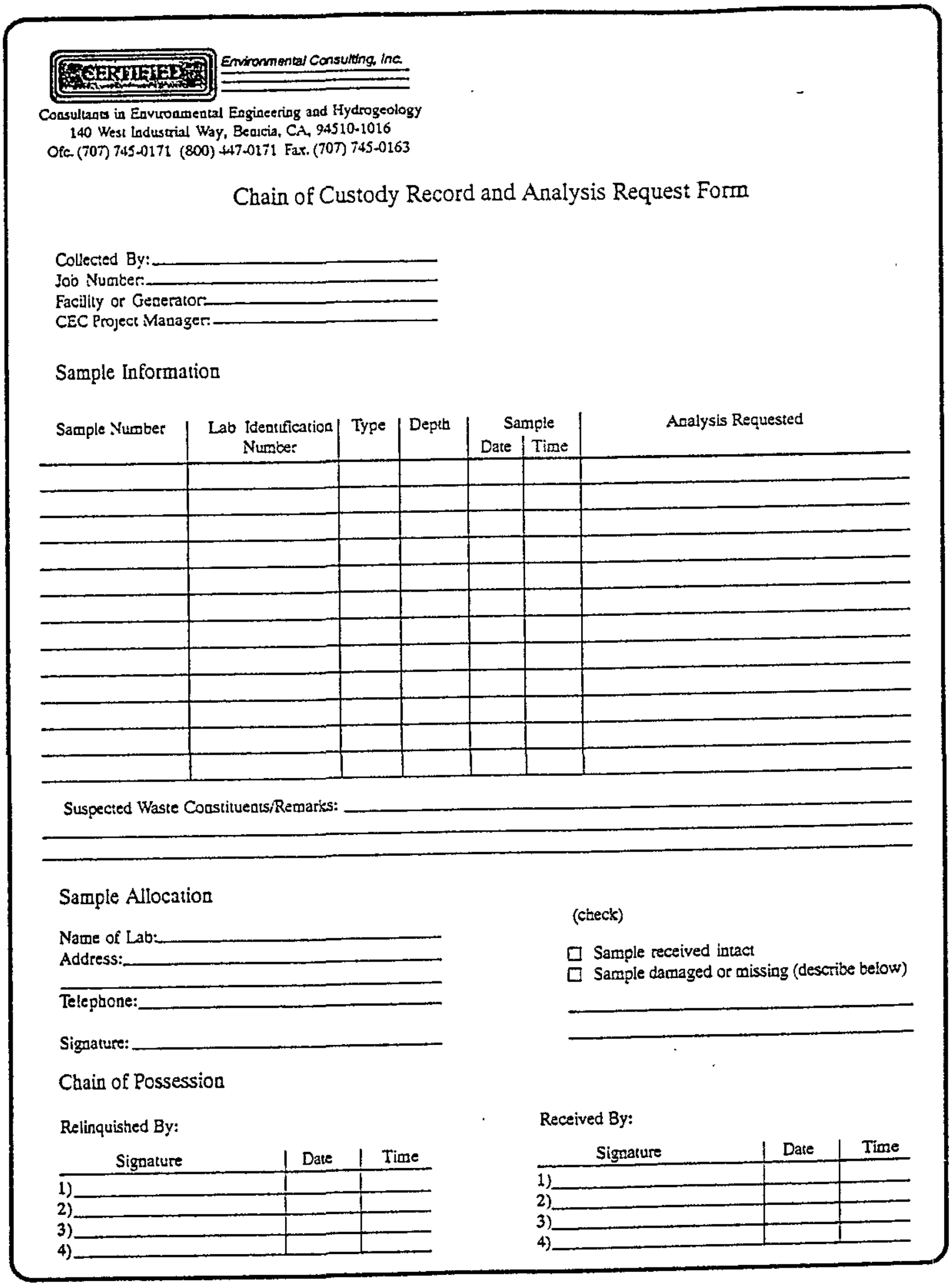
- Date of sample receipt

- Analyses to be performed

A copy of the Chain-of Custody and Sample Analysis Request Sheet to be used in this project is shown in Figure 5-3.

RECEIPT AND LOGGING OF SAMPLE

Upon delivery to the project laboratory, the samples will be turned over to a sample custodian assigned by the laboratory to receive the samples. Upon receipt of a sample, the custodian inspects the condition of the sample and the sample seal, reconciles the information on the sample label and seal against that on the chain-of-custody record, assigns a laboratory number, enters the sample in the laboratory log book, and stores the sample in a secured sample storage room or cabinet until assigned to an analyst for testing.



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Consultants in Environmental Engineering and Hydrogeology
140 West Industrial Way, Benicia, CA, 94510-1016
Ofc. (707) 745-0171 (800) 447-0171 Fax. (707) 745-0163

Chain of Custody Record and Analysis Request Form

Collected By:
Job Number:
Facility or Generator:
CEC Project Manager:

Sample Information

Sample Number	Lab Identification Number	Type	Depth	Sample		Analysis Requested
				Date	Time	

Suspected Waste Constituents/Remarks:

Sample Allocation

Name of Lab:
Address:
Telephone:
Signature:

(check)

☐ Sample received intact
☐ Sample damaged or missing (describe below)

Chain of Possession

Relinquished By:

Signature	Date	Time
1)		
2)		
3)		
4)		

Received By:

Signature	Date	Time
1)		
2)		
3)		
4)		

Figure 7

Chain-of-Custody Record

ANALYTICAL METHODS

The soil samples collected will be analyzed for total oil and grease. The protocol outlined in the Environmental Protection Agency's (EPA) method 9071 will be used for this analysis. A copy of the method from SW-846 is provided in the enclosures.

quantitative confirmation of results for most of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a halide-specific detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.3), 7.7 cm of 2,6-diphenylene oxide polymer (Section 6.3.2), 7.7 cm of silica gel (Section 6.3.4), 7.7 cm of coconut charcoal (Section 6.3.1). If it is not necessary to analyze for dichlorodifluoromethane, the charcoal can be eliminated, and the polymer section lengthened to 15 cm. The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—8 ft long x 0.1 in. ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—6 ft long x 0.1 in. ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120 mesh) or equivalent.

5.3.3 Detector—Electrolytic conductivity or microcoulometric detector. These types of detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). The electrolytic conductivity detector was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-μL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap Materials:

6.3.1 Coconut charcoal—6/10 mesh sieved to 26 mesh, Barnabey Cheney, CA-580-26 lot # M-2649 or equivalent.

6.3.2 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.3 Methyl silicone packing—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.4 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976; 49 FR 43251, 43258, 43259, Oct. 26, 1984; 50 FR 691, 692, 695, Jan. 4, 1985; 51 FR 23693, June 30, 1986; 52 FR 33543, Sept. 3, 1987]

EDITORIAL NOTE: Information collection requirements contained in § 136.3(e) have not been approved by the Office of Management and Budget and are not effective, pending OMB approval.

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an application for an alternate test procedure may be made by letter in triplicate. Any application for an alternate test procedure under this paragraph (c) shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976]

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will occur has final responsibility for approval of any alternate test procedure proposed by the responsible person or firm making the discharge.

(b) Within thirty days of receipt of an application, the Director will forward such application proposed by the responsible person or firm making the discharge, together with his recommendations, to the Regional Administrator. Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application, and shall forward a copy of the rejected application and his decision to the Director of the State Permit Program and to the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(c) Before approving any application for an alternate test procedure proposed by the responsible person or firm making the discharge, the Regional Administrator shall forward a copy of the application to the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(d) Within ninety days of receipt by the Regional Administrator of an application for an alternate test procedure, proposed by the responsible person or firm making the discharge, the Regional Administrator shall notify the applicant and the appropriate State agency of approval or rejection, or shall specify the additional information which is required to determine whether to approve the proposed test procedure. Prior to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati. A copy of all approval and rejection notifications will be forwarded to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, for the purposes of national coordination.

(e) Within ninety days of the receipt by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati of an application for an alternate test procedure for nationwide use, the Director of the Environmental Monitoring and Support Laboratory, Cincinnati shall notify the applicant of his recommendation to the Administrator to approve or reject the application, or shall specify additional information which is required to determine whether to approve the proposed test procedure. After such notification, an alternate method determined by the Administrator to satisfy the applicable requirements of this part shall be approved for nationwide use to satisfy the requirements of this subchapter; alternate test procedures determined by the Administrator not to meet the applicable requirements of this part shall be rejected. Notice of these determinations shall be submitted for publication in the FEDERAL REGISTER not later than 15 days after such notification and determination is made.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976]

APPENDIX A TO PART 136—METHODS FOR ORGANIC CHEMICAL ANALYSIS OF MUNICIPAL AND INDUSTRIAL WASTEWATER

METHOD 601—PURGEABLE HALOCARBONS

1. Scope and Application

1.1 This method covers the determination of 29 purgeable halocarbons.

The following parameters may be determined by this method:

Parameter	STORET No	CAS No
Bromodichloromethane	32101	75-27-4
Bromoform †D32104.	75-25-2	
Bromomethane.	34413	74-83-9
Carbon tetrachloride	32102	56-23-5
Chlorobenzene	34301	108-90-7
Chloroethane	34311	75-00-3
2-Chloroethylvinyl ether	34576	100-75-8
Chloroform	32106	67-66-3
Chloromethane	34418	74-87-3
Dibromochloromethane	32105	124-48-1
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
Dichlorodifluoromethane	34668	75-71-8
1,1-Dichloroethane	34496	75-34-3
1,2-Dichloroethane	34531	107-06-2
1,1-Dichloroethane	34501	75-35-4
trans-1,2-Dichloroethene	34546	156-60-5
1,2-Dichloropropane	34541	78-87-5
cis-1,3-Dichloropropene	34704	10061-01-5
trans-1,3-Dichloropropene	34699	10061-02-6
Methylene chloride	34423	75-09-2
1,1,2,2-Tetrachloroethane	34516	79-34-5
Tetrachloroethene	34475	127-18-4
1,1,1-Trichloroethane	34506	71-55-6
1,1,2-Trichloroethane	34511	79-00-5
Tetrachloroethene	39180	79-01-6
Trichlorofluoromethane	34488	75-69-4
Vinyl chloride	39715	75-01-4

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for most of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater *may differ from those listed, de*pending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a halide-specific detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is en*countered, it should be followed by an anal*ysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105 °C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4,6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. *Primary standards of these toxic com*pounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.3), 7.7 cm of 2,6-diphenylene oxide polymer (Section 6.3.2), 7.7 cm of silica gel (Section 6.3.4), 7.7 cm of coconut charcoal (Section 6.3.1). If it is not necessary to analyze for dichlorodifluoromethane, the charcoal can be eliminated, and the polymer section lengthened to 15 cm. The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—8 ft long x 0.1 in. ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—6 ft long x 0.1 in. ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120 mesh) or equivalent.

5.3.3 Detector—Electrolytic conductivity or microcoulometric detector. These types of detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). The electrolytic conductivity detector was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-μL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap Materials:

6.3.1 Coconut charcoal—6/10 mesh sieved to 26 mesh, Barnabey Cheney, CA-580-26 lot # M-2649 or equivalent.

6.3.2 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.3 Methyl silicone packing—3% OV 1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.4 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquid—Using a 100 μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the six halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve into the methanol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in μg/μL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the six gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.6 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards can be stored up to 24 h, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after 1 h.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes in Section 8.7 have been used successfully as internal standards, because of their generally unique retention times.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

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

Equation 1

where:

A_s = Response for the parameter to be measured.

A_{is} = Response for the internal standard.

C_{is} = Concentration of the internal standard.

C_s = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, proceed according to Section 7.5.4.

NOTE: The large number of parameters in Table 2 present a substantial probability that one or more will not meet the calibration acceptance criteria when all parameters are analyzed.

7.5.4 Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10 μg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and

Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 μg/L of each parameter by adding 200 μL of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, then the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 2 present a substantial probability that *one or more* will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of *the samples from* each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for *the background* concentrations in the sample. Spike a second 5-mL sample aliquot with 10 μL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A-B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 μg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as $(100\ X'/T) \pm 2.44(100\ S'/T)\%$.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 2 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spiked sample.

8.4.1 Prepare the QC check standard by adding 10 μL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent *recovery* (s_p). *Express the accuracy assessment* as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 6.5, add a volume to give 750 μg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume for a concentration of 15 ng/μL. Add 10 μL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis. If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2).

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.[3]

10. Procedure

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Column 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the surrogate spiking solution (Section 8.7) and 10.0 µL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 11.0±0.1 min at ambient temperature.

10.7 After the 11-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry or random retention time problems persist) instead of the initial program temperature of 45 °C

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2.

Equation 2

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

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard.

11.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentration listed in Table 1 were obtained using reagent water.[11]. Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for use in the concentration range from the MDL to 1000×MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1000×MDL.

12.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 8.0 to 500 µg/L.[9] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

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4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.

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9. "EPA Method Study 24, Method 601—Purgeable Halocarbons by the Purge and Trap Method," EPA 600/4-84-064, National Technical Information Service, PB84-212448, Springfield, Virginia 22161, July 1984.

10. "Method Validation Data for EPA Method 601," Memorandum from B. Potter, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, November 10, 1983.

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TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
Chloromethane	1.50	5.28	0.08
Bromomethane	2.17	7.05	1.18
Dichlorodifluoromethane	2.62	nd	1.81
Vinyl chloride	2.67	5.28	0.18
Chloroethane	3.33	8.68	0.52
Methylene chloride	5.25	10.1	0.25
Trichlorofluoromethane	7.18	nd	nd
1,1-Dichloroethene	7.93	7.72	0.13
1,1-Dichloroethane	9.30	12.6	0.07
trans-1,2-Dichloroethene	10.1	9.38	0.10
Chloroform	10.7	12.1	0.05
1,2-Dichloroethane	11.4	15.4	0.03
1,1,1-Trichloroethane	12.6	13.1	0.03
Carbon tetrachloride	13.0	14.4	0.12
Bromodichloromethane	13.7	14.6	0.10
1,2-Dichloropropane	14.9	16.6	0.04
cis-1,3-Dichloropropene	15.2	16.6	0.34
Trichloroethene	15.8	13.1	0.12
Dibromochloromethane	16.5	16.6	0.09
1,1,2-Trichloroethane	16.5	18.1	0.02
trans-1,3-Dichloropropene	16.5	18.0	0.20
2-Chloroethylvinyl ether	18.0	nd	0.13
Bromoform	19.2	19.2	0.20
1,1,2,2-Tetrachloroethane	21.6	nd	0.03
Tetrachloroethene	21.7	15.0	0.03
Chlorobenzene	24.2	18.8	0.25
1,3 Dichlorobenzene	34.0	22.4	0.32
1,2-Dichlorobenzene	34.9	23.5	0.15

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

Parameter	Retention time (min)		Method detection limit (μg/L)
	Column 1	Column 2	
1,4-Dichlorobenzene	35.4	22.3	0.24

Column 1 conditions. Carbopack B (60/80 mesh) coated with 1% SP-1000 packed in an 8 ft x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 45 °C for 3 min then programmed at 8 °C/min to 220 °C and held for 15 min.

Column 2 conditions: Porisil-C (100/120 mesh) coated with n-octane packed in a 6 ft x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 50 °C for 3 min then programmed at 6 °C/min to 170 °C and held for 4 min.

nd=not determined.

TABLE 2.—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 601 [a]

Parameter	Range for Q (μg/L)	Limit for s (μg/L)	Range for $\bar{X}$ (μg/L)	Range P, P_s (%)
Bromodichloromethane	15.2–24.8	4.3	10.7–32.0	42–172
Bromoform	14.7–25.3	4.7	5.0–29.3	13–159
Bromomethane	11.7–28.3	7.6	3.4–24.5	D–144
Carbon tetrachloride	13.7–26.3	5.6	11.8–25.3	43–143
Chlorobenzene	14.4–25.6	5.0	10.2–27.4	38–150
Chloroethane	15.4–24.6	4.4	11.3–25.2	46–137
2-Chloroethylvinyl ether	12.0–28.0	8.3	4.5–35.5	14–186
Chloroform	15.0–25.0	4.5	12.4–24.0	49–133
Chloromethane	11.9–28.1	7.4	D–34.9	D–193
Dibromochloromethane	13.1–26.9	6.3	7.9–35.1	24–191
1,2-Dichlorobenzene	14.0–26.0	5.5	1.7–38.9	D–208
1,3-Dichlorobenzene	9.9–30.1	9.1	6.2–32.6	7–187
1,4-Dichlorobenzene	13.9–26.1	5.5	11.5–25.5	42–143
1,1-Dichloroethane	16.8–23.2	3.2	11.2–24.6	47–132
1,2-Dichloroethane	14.3–25.7	5.2	13.0–26.5	51–147
1,1-Dichloroethene	12.6–27.4	6.6	10.2–27.3	28–167
trans-1,2-Dichloroethene	12.8–27.2	6.4	11.4–27.1	38–155
1,2-Dichloropropane	14.8–25.2	5.2	10.1–29.9	44–156
cis-1,3-Dichloropropene	12.8–27.2	7.3	6.2–33.8	22–178
trans-1,3-Dichloropropene	12.8–27.2	7.3	6.2–33.8	22–178
Methylene chloride	15.5–24.5	4.0	7.0–27.6	25–162
1,1,2,2-Tetrachloroethane	9.8–30.2	9.2	6.6–31.8	8–184
Tetrachloroethene	14.0–26.0	5.4	8.1–29.6	26–162
1,1,1-Trichloroethane	14.2–25.8	4.9	10.8–24.8	41–138
1,1,2-Trichloroethane	15.7–24.3	3.9	9.6–25.4	39–136
Trichloroethene	15.4–24.6	4.2	9.2–26.6	35–146
Trichlorofluoromethane	13.3–26.7	6.0	7.4–28.1	21–156
Vinyl chloride	13.7–26.3	5.7	8.2–29.9	28–163

[a] Criteria were calculated assuming a QC check sample concentration of 20 μg/L.

Q—Concentration measured in QC check sample, in μg/L (Section 7.5.3).

s—Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).

$\bar{X}$—Average recovery for four recovery measurements, in μg/L (Section 8.2.4).

P, P_s—Percent recovery measured (Section 8.3.2, Section 8.4.2).

D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 601

Parameter	Accuracy, as recovery, X' (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S' (μg/L)
Bromodichloromethane	1.12C−1.02	$0.11\bar{X}+0.04$	$0.20\bar{X}+1.00$
Bromoform	0.96C−2.05	$0.12\bar{X}+0.58$	$0.21\bar{X}+2.41$
Bromomethane	0.76C−1.27	$0.28\bar{X}+0.27$	$0.36\bar{X}+0.94$
Carbon tetrachloride	0.98C−1.04	$0.15\bar{X}+0.38$	$0.20\bar{X}+0.39$

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 601—Continued

Parameter	Accuracy, as recovery, X' (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S' (μg/L)
Chlorobenzene	1.00C−1.23	$0.15\bar{X}-0.02$	$0.16\bar{X}+1.21$
Choroethane	0.99C−1.53	$0.14\bar{X}-0.13$	$0.17\bar{X}+0.63$
2-Chloroethylvinyl ether [a]	1.00C	$0.20\bar{X}$	$0.35\bar{X}$
Chloroform	0.93C−0.39	$0.13\bar{X}+0.15$	$0.19\bar{X}-0.02$
Chloromethane	0.77C+0.18	$0.28\bar{X}-0.31$	$0.52\bar{X}+1.31$
Dibromochloromethane	0.94C+2.72	$0.11\bar{X}+1.10$	$0.24\bar{X}+1.68$
1,2-Dichlorobenzene	0.93C+1.70	$0.20\bar{X}+0.97$	$0.13\bar{X}+6.13$
1,3-Dichlorobenzene	0.95C+0.43	$0.14\bar{X}+2.33$	$0.26\bar{X}+2.34$
1,4-Dichlorobenzene	0.93C−0.09	$0.15\bar{X}+0.29$	$0.20\bar{X}+0.41$
1,1-Dichloroethane	0.95C−1.08	$0.09\bar{X}+0.17$	$0.14\bar{X}+0.94$
1,2-Dichloroethane	1.04C−1.06	$0.11\bar{X}+0.70$	$0.15\bar{X}+0.94$
1,1-Dichloroethene	0.98C−0.87	$0.21\bar{X}-0.23$	$0.29\bar{X}-0.40$
trans-1,2-Dichloroethene	0.97C−0.16	$0.11\bar{X}+1.46$	$0.17\bar{X}+1.46$
1,2-Dichloropropane [a]	1.00C	$0.13\bar{X}$	$0.23\bar{X}$
cis-1,3-Dichloropropene [a]	1.00C	$0.18\bar{X}$	$0.32\bar{X}$
trans-1,3-Dichloropropene [a]	1.00C	$0.18\bar{X}$	$0.32\bar{X}$
Methylene chloride	0.91C−0.93	$0.11\bar{X}+0.33$	$0.21\bar{X}+1.43$
1,1,2,2-Tetrachloroethene	0.95C+0.19	$0.14\bar{X}+2.41$	$0.23\bar{X}+2.79$
Tetrachloroethene	0.94C+0.06	$0.14\bar{X}+0.38$	$0.18\bar{X}+2.21$
1,1,1-Trichloroethane	0.90C−0.16	$0.15\bar{X}+0.04$	$0.20\bar{X}+0.37$
1,1,2-Trichloroethane	0.86C+0.30	$0.13\bar{X}-0.14$	$0.19\bar{X}+0.67$
Trichloroethene	0.87C+0.48	$0.13\bar{X}-0.03$	$0.23\bar{X}+0.30$
Trichlorofluoromethane	0.89C−0.07	$0.15\bar{X}+0.67$	$0.26\bar{X}+0.91$
Vinyl chloride	0.97C−0.36	$0.13\bar{X}+0.65$	$0.27\bar{X}+0.40$

$\bar{X}'$=Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.

s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.

S'=Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.

C=True value for the concentration, in μg/L.

$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in μg/L.

[a] Estimates based upon the performance in a single laboratory.[10]

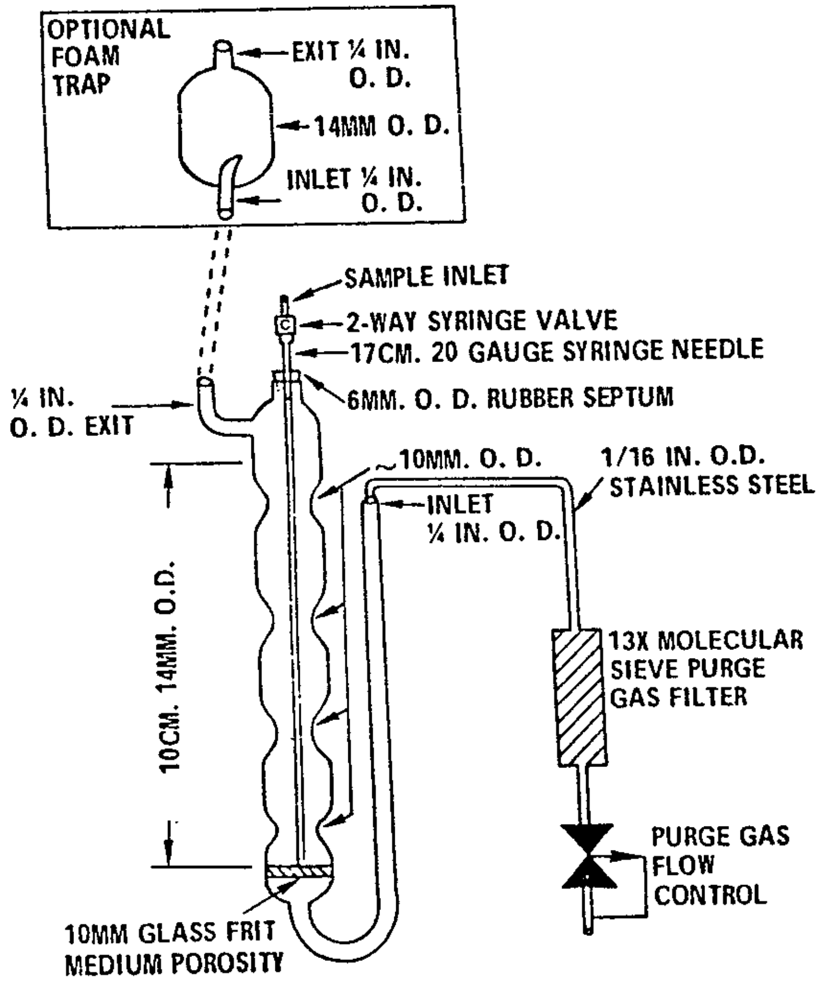

Figure 1. Purging device.

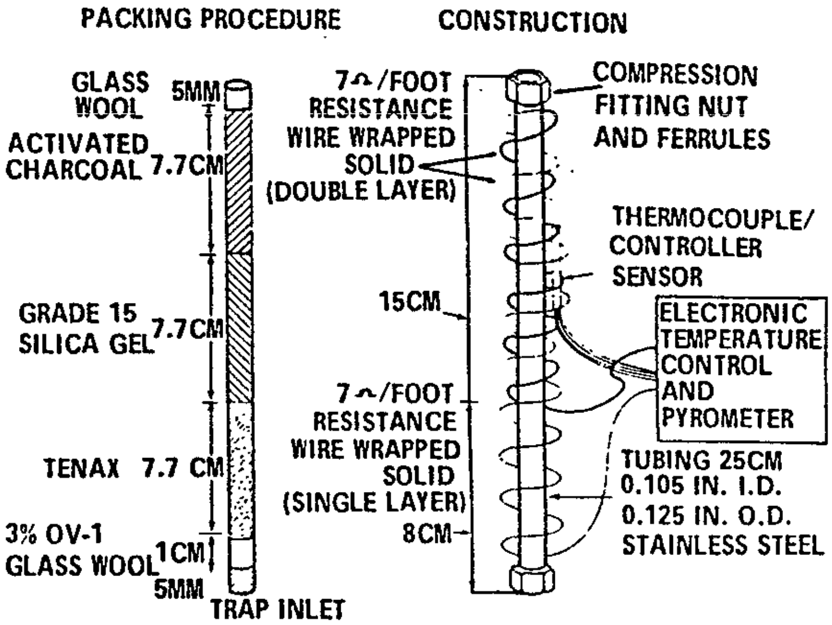

Figure 2. Trap packings and construction to include desorb capability

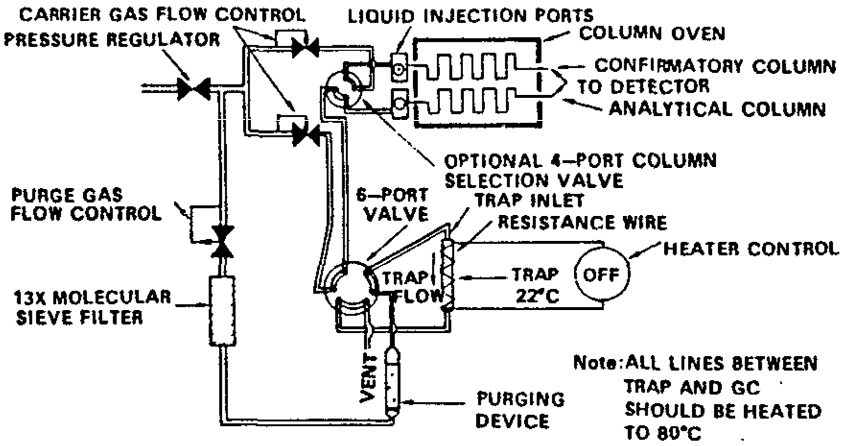

Figure 3. Purge and trap system-purge mode.

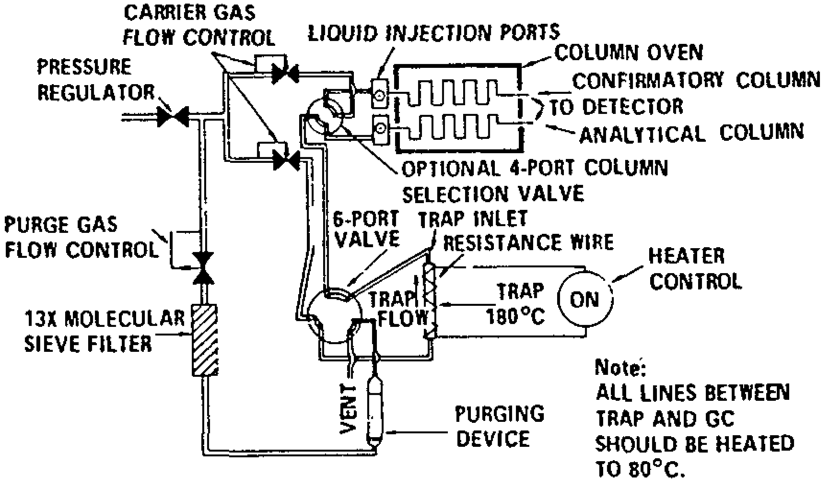

Figure 4. Purge and trap system - desorb mode.

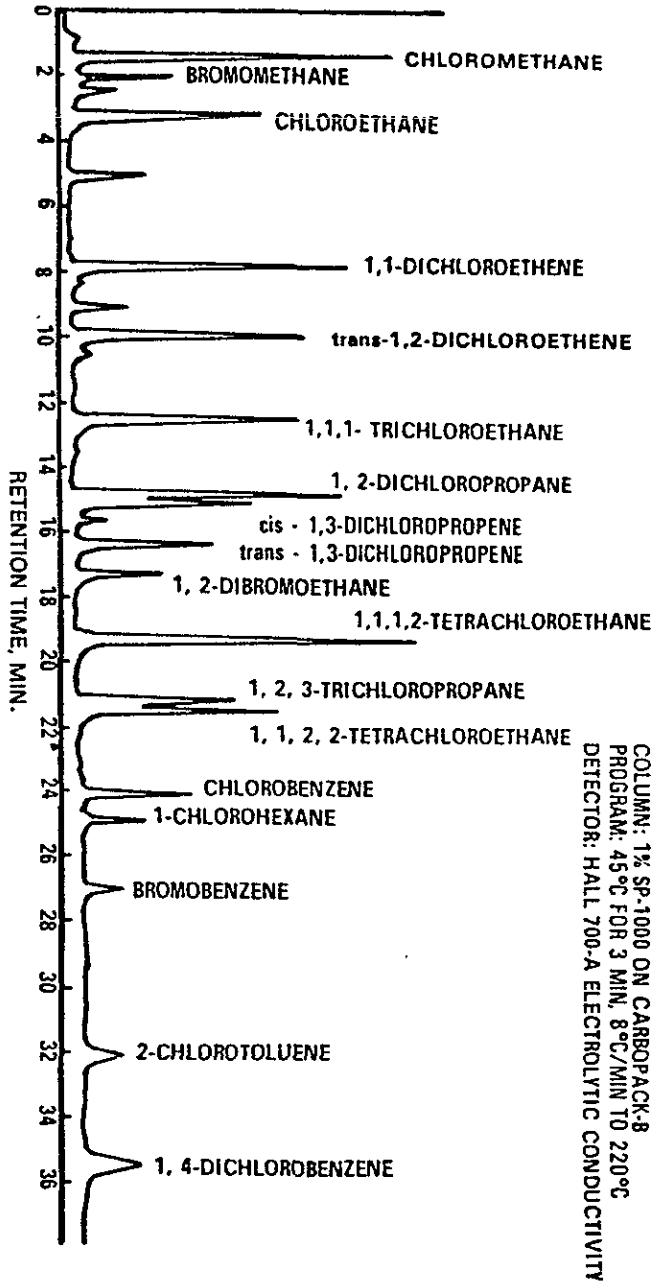

Figure 5. Gas chromatogram of purgeable halocarbons.

METHOD 602—PURGEABLE AROMATICS

1. Scope and Application

1.1 This method covers the determination of various purgeable aromatics. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No
Benzene	34030	71-43-2
Chlorobenzene	34301	108-90-7
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
Ethylbenzene	34371	100-41-4
Toluene	34010	108-88-3

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1) [1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The aromatics are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the aromatics are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the aromatics onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the aromatics which are then detected with a photoionization detector.[2, 3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high aromatic levels, it may be necessary to wash the purging device with a detergent solution, rinse it with distilled water, and then dry it in an oven at 105 °C between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene and 1,4-dichlorobenzene. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial]25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: A purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in.

5.2.2.1 The trap is packed with 1 cm of methyl silicone coated packing (Section 6.4.2) and 23 cm of 2,6-diphenylene oxide polymer (Section 6.4.1) as shown in Figure 2. This trap was used to develop the method performance statements in Section 12.

5.2.2.2 Alternatively, either of the two traps described in Method 601 may be used, although water vapor will preclude the measurement of low concentrations of benzene.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3, 4, and 5.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—6 ft long x 0.082 in. ID stainless steel or glass, packed with 5% SP-1200 and 1.75% Bentone-34 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—8 ft long x 0.1 in ID stainless steel or glass, packed with 5% 1,2,3-Tris(2-cyanoethoxy)propane on Chromosorb W-AW (60/80 mesh) or equivalent.

5.3.3 Detector—Photoionization detector (h-Nu Systems, Inc. Model PI-51-02 or equivalent). This type of detector has been proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-µL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.8 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Hydrochloric acid (1+1)—Add 50 mL of concentrated HCl (ACS) to 50 mL of reagent water.

6.4 Trap Materials:

6.4.1 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.4.2 Methyl silicone packing—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.5 Methanol—Pesticide quality or equivalent.

6.6 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids. Because of the toxicity of benzene and 1,4-dichlorobenzene, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.6.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.6.2 Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.6.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in μg/μL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source

6.6.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store at 4 °C and protect from light.

6.6.5 All standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.7 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary solution standards must be stored with zero headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.8 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards must be prepared fresh daily.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compound, α,α,α,-trifluorotoluene, recommended as a surrogate spiking compound in Section 8.7 has been used successfully as an internal standard.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.6 and 6.7. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μl of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

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

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard

C_s=Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is} vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The mimimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10 μg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 µg/L of each parameter by adding 200 µL of QC check sample concentrate to 100 mL of reagant water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 2 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8 2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 µL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A-B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 µg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T) ± 2.44(100 S'/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 10 µL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate compounds (e.g. α, α, α,-trifluorotoluene) that encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 6.6, add a volume to give 750 µg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume for a concentration of 15 mg/µL. Add 10 µL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis. If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2).

9. Sample Collection, Preservation, and Handling

9.1 The samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. EPA Method 330.4 or 330.5 may be used for measurement of residual chlorine.[3] Field test kits are available for this purpose.

9.2 Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding 1 + 1 HCl while stirring. Fill the sample bottle in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.[3]

10. Procedure

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Column 1 is shown in Figure 6. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the surrogate spiking solution (Section 8.7) and 10.0 µL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 12.0±0.1 min at ambient temperature.

10.7 After the 12-min purge time, disconnect the purging device from the trap. Dry the trap by maintaining a flow of 40 mL/min of dry purge gas through it for 6 min (Figure 4). If the purging device has no provision for bypassing the purger for this step, a dry purger should be inserted into the device to minimize moisture in the gas. Attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 5), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry and random retention time problems persist) instead of the initial program temperature of 50 °C.

10.8 While the trap is being desorbed into the gas chromatograph column, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2.

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

Equation 2

where:

- A_s = Response for the parameter to be measured.
- A_{is} = Response for the internal standard.
- C_{is} = Concentration of the internal standard.

11.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method has been demonstrated to be applicable for the concentration range from the MDL to 100 X MDL.[9] Direct aqueous injection techniques should be used to measure concentration levels above 1000 x MDL.

12.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 2.1 to 550 µg/L.[9] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

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4. "Carcinogens—Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

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7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15*, 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3. is two times the value 1.22 derived in this report.)

8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. March 1979.

9. "EPA Method Study 25, Method 602, Purgeable Aromatics," EPA 600/4-84-042, National Technical Information Service, PB84-196682, Springfield, Virginia 22161, May 1984.

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
Benzene	3.33	2.75	0.2
Toluene	5.75	4.25	0.2
Ethylbenzene	8.25	6.25	0.2
Chlorobenzene	9.17	8.02	0.2
1,4-Dichlorobenzene	16.8	16.2	0.3
1,3-Dichlorobenzene	18.2	15.0	0.4
1,2-Dichlorobenzene	25.9	19.4	0.4

Column 1 conditions: Supelcoport (100/120 mesh) coated with 5% SP-1200/1.75% Bentone-34 packed in a 6 ft x 0.085 in. ID stainless steel column with helium carrier gas at 36 mL/min flow rate. Column temperature held at 50 °C for 2 min then programmed at 6 °C/min to 90 °C for a final hold.

Column 2 conditions: Chromosorb W-AW (60/80 mesh) coated with 5% 1,2,3-Tris(2-cyanoethyoxy)propane packed in a 6 ft x 0.085 in. ID stainless steel column with helium carrier gas at 30 mL/min flow rate. Column temperature held at 40 °C for 2 min then programmed at 2 °C/min to 100 °C for a final hold.

TABLE 2.—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 602[a]

Parameter	Range for Q (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s (%)
Benzene	15.4–24.6	4.1	10.0–27.9	39–150
Chlorobenzene	16.1–23.9	3.5	12.7–25.4	55–135
1,2-Dichlorobenzene	13.6–26.4	5.8	10.6–27.6	37–154
1,3-Dichlorobenzene	14.5–25.5	5.0	12.8–25.5	50–141
1,4-Dichlorobenzene	13.9–26.1	5.5	11.6–25.5	42–143
Ethylbenzene	12.6–27.4	6.7	10.0–28.2	32–160
Toluene	15.5–24.5	4.0	11.2–27.7	46–148

Q = Concentration measured in QC check sample, in µg/L (Section 7.5.3).
s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P_s, P = Percent recovery measured (Section 8.3.2, Section 8.4.2).
[a] Criteria were calculated assuming a QC check sample concentration of 20 µg/L.

Note: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 602

Parameter	Accuracy, as recovery, X' (μg/L)	Single analyst precision, s' (μg/L)	Overall precision, S' (μg/L)
Benzene	$0.92C+0.57$	$0.09\bar{X}+0.59$	$0.21\bar{X}+0.56$
Chlorobenzene	$0.95C+0.02$	$0.09\bar{X}+0.23$	$0.17\bar{X}+0.10$
1,2-Dichlorobenzene	$0.93C+0.52$	$0.17\bar{X}-0.04$	$0.22\bar{X}+0.53$
1,3-Dichlorobenzene	$0.96C-0.05$	$0.15\bar{X}-0.10$	$0.19\bar{X}+0.09$
1,4-Dichlorobenzene	$0.93C-0.09$	$0.15\bar{X}+0.28$	$0.20\bar{X}+0.41$
Ethylbenzene	$0.94C+0.31$	$0.17\bar{X}+0.46$	$0.26\bar{X}+0.23$
Toluene	$0.94C+0.65$	$0.09\bar{X}+0.48$	$0.18\bar{X}+0.71$

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s' = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the Concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

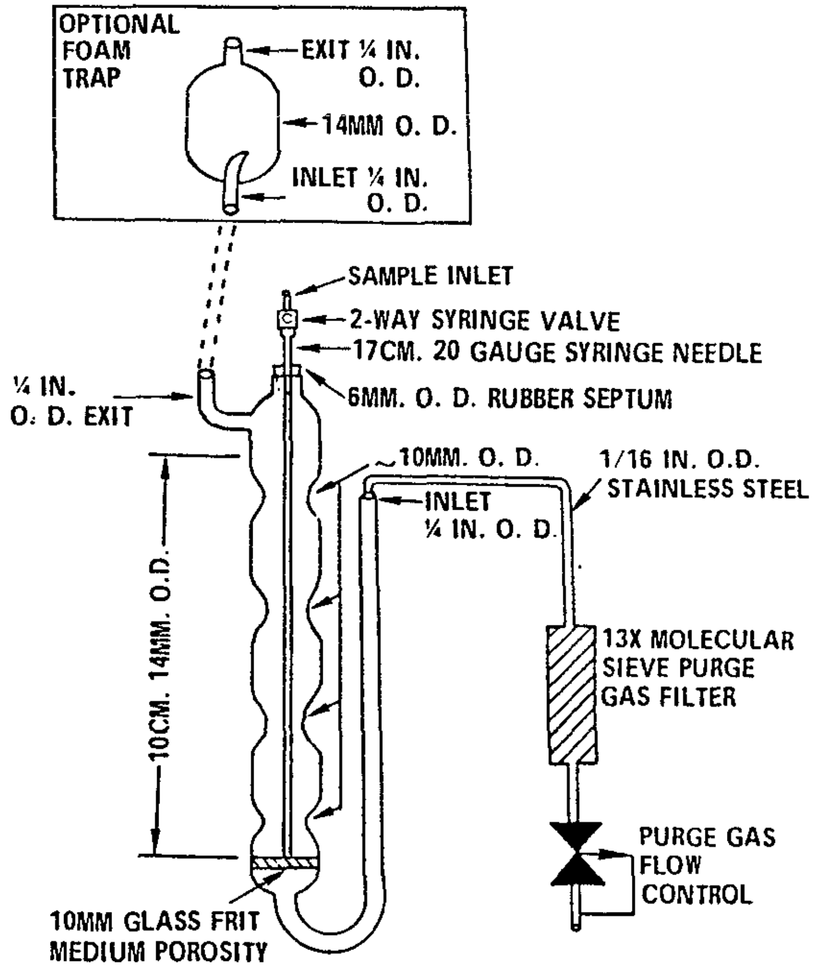

Figure 1. Purging device.

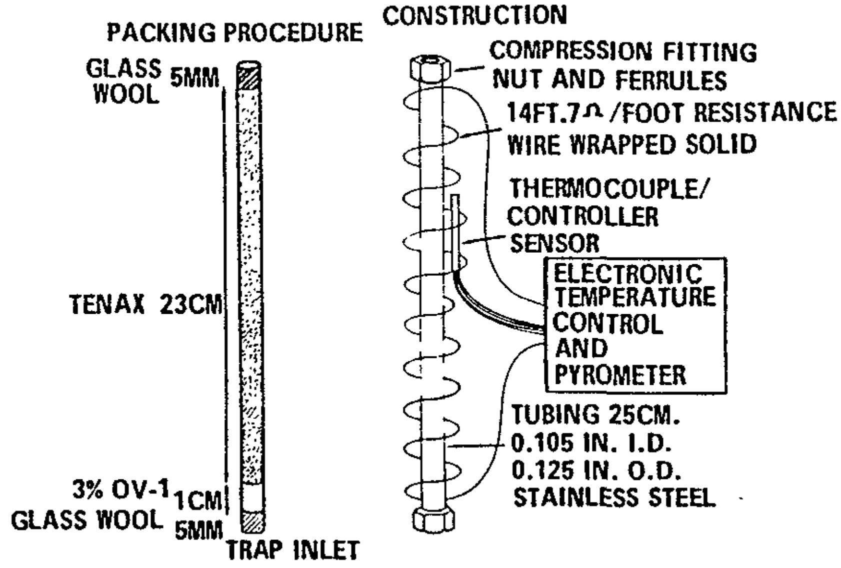

Figure 2. Trap packings and construction to include desorb capability.

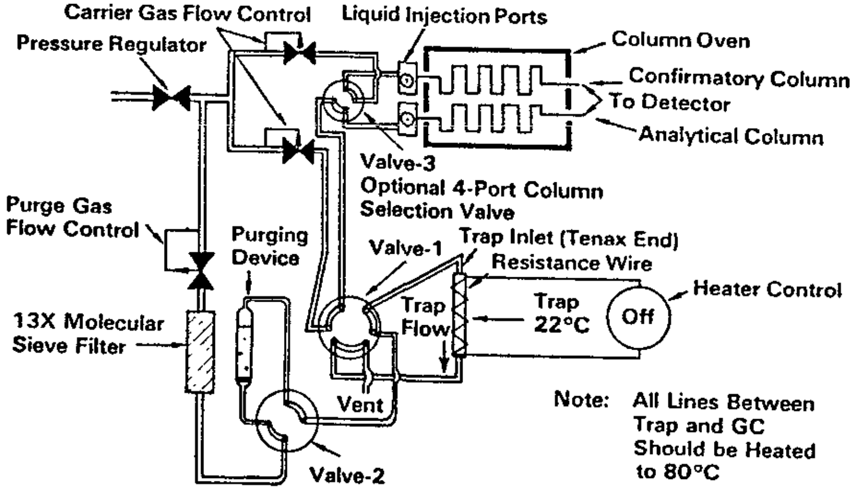

Figure 3. Purge and trap system - purge mode.

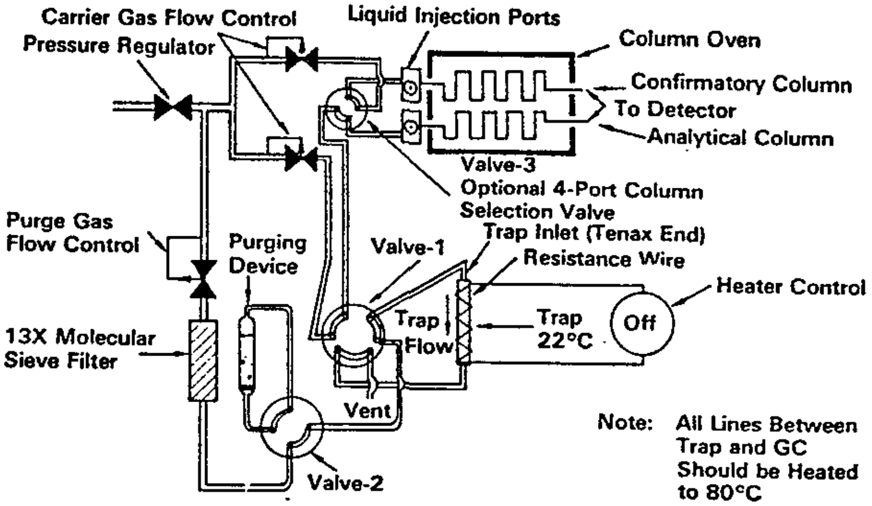

Figure 4. Purge and trap system-dry mode.

METHOD 624—PURGEABLES

1. Scope and Application

1.1 This method covers the determination of a number of purgeable organics. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No
Benzene	34030	71-43-2
Bromodichloromethane	32101	75-27-4
Bromoform	32104	75-25-2
Bromomethane	34413	74-83-9
Carbon tetrachloride	32102	56-23-5
Chlorobenzene	34301	108-90-7
Chloroethane	34311	75-00-3
2-Chloroethylvinyl ether	34576	110-75-8
Chloroform	32106	67-66-3
Chloromethane	34418	74-87-3
Dibromochloromethane	32105	124-48-1
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
1,1-Dichloroethane	34496	75-34-3
1,2-Dichloroethane	34531	107-06-2
1,1-Dichloroethane	34501	75-35-4
trans-1,2-Dichloroethane	34546	156-60-5
1,2-Dichloropropane	34541	78-87-5
cis-1,3-Dichloropropane	34704	10061-01-5
trans-1,3-Dichloropropane	34699	10061-02-6
Ethyl benzene	34371	100-41-4
Methylene chloride	34423	75-09-2
1,1,2,2-Tetrachloroethane	34516	79-34-5
Tetrachloroethene	34475	127-18-4
Toluene	34010	108-88-3
1,1,1-Trichloroethane	34506	71-55-6
1,1,2-Trichloroethene	34511	79-00-5
Trichloroethane	39180	79-01-6
Trichlorofluoromethane	34488	75-69-4
Vinyl chloride	39175	75-01-4

1.2 The method may be extended to screen samples for acrolein (STORET No. 34210, CAS No. 107-02-8) and acrylonitrile (STORET No. 34215, CAS No. 107-13-1), however, the preferred method for these two compounds in Method 603.

1.3 This is a purge and trap gas chromatographic/mass spectrometer (GC/MS) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1.

1.4 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 Any modification to this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5. Depending upon the nature of the modification and the extent of intended use, the applicant may be required to demonstrate that the modifications will produce equivalent results when applied to relevant wastewaters.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.[2,3]

3. Interferences

3.1 Impurities in the purge gas, organic compounds outgassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high pureeable levels, it may be necessary to wash the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105 ° C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this methmd. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4,6] for the information of the analyst.

4.2. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicane (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: A purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass though the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.2), 15 cm of 2,6-dyphenylene oxide polymer (Section 6.3.1), and 8 cm of silica gel (Section 6.3.3). The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber should be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 GC/MS system:

5.3.1 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

5.3.2 Column—6 ft long x 0.1 in ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 11.1.

5.3.3 Mass spectrometer—Capable of scanning from 20 to 260 amu every 7 s or less, utilizing 70 V (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the criteria in Table 2 when 50 ng of 4-bromofluorobenzene (BFB) is injected through the GC inlet.

5.3.4 GC/MS interface—Any GC to MS interface that gives acceptable calibration points at 50 ng or less per injection for each of the parameters of interest and achieves all acceptable performance criteria (Section 10) may be used. GC to MS interfaces constructed of all glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

5.3.5 Data system—A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for specific m/z (masses) and plotting such m/z abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

5.4 Syringes—5-mL, glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-µL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap materials:

6.3.1 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.2 Methyl silicone packing—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.3 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the compounds, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquids—Using a 100-µL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the four halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, and vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve in the methanol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in µg/µL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the four gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.6 Secondary dilution standards—Using stock solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7 Surrogate standard spiking solution—Select a minimum of three surrogate compounds from Table 3. Prepare stock standard solutions for each surrogate standard in methanol as described in Section 6.5. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 15 µg/mL in water. Store the solutions at 4 °C in Teflon-sealed glass containers with a minimum of headspace. The solutions should be checked frequently for stability. The addition of 10 µL of this solution of 5 mL of sample or standard is equivalent to a concentration of 30 µg/L of each surrogate standard.

6.8 BFB Standard—Prepare a 25 µg/mL solution of BFB in methanol.

6.9 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select three or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Some recommended internal standards are listed in Table 3.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 µL of one or more secondary dilution standards to 50, 250, or 500 mL of reagent water. A 25-µL syringe with a 0.006 in. ID needle should be used for this operation. One of the calibration standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system. These aqueous standards can be stored up to 24 h, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after 1 h.

7.3.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 µg/mL of each internal standard compound. The addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 µg/L.

7.3.3 Analyze each calibration standard according to Section 11, adding 10 µL of internal standard spiking solution directly to the syringe (Section 11.4). Tabulate the area response of the characteristic m/z against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

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

Equation 1

where:

A_s=Area of the characteristic m/z for the parameter to be measured.

A_{is}=Area of the characteristic m/z for the inernal standard.

C_{is}=Concentration of the internal standard.

C_s=Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<35% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve or RF must be verified on each working day by the measurement of a QC check sample.

7.4.1 Prepare the QC check sample as described in Section 8.2.2.

7.4.2 Analyze the QC check sample according to the method beginning in Section 10.

7.4.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 5. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, proceed according to Section 7.4.4.

NOTE: The large number of parameters in Table 5 present a substantial probability that one or more will not meet the calibration acceptance criteria when all parameters are analyzed.

7.4.4 Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve or RF must be prepared for that parameter according to Section 7.3.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a *quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 11.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 5% of all samples to monitor and evaluate laboratory data *quality. This procedure* is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses *is equivalent to 5% of* all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must spike all samples with surrogate standards to monitor continuing laboratory performance. This procedure is described in Section 8.5.

8.1.7 The *laboratory must maintain* performance records to document the quality of data that is generated. This procedure is described in Section 8.6.

8 2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate *is required containing* each parameter of interest at a concentration of 10 μg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate *must be* obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 *μg/L of each parameter* by adding 200 μL of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the *corresponding acceptance criteria* for precision and accuracy, respectively, found in Table 5. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range *for accuracy, the system performance* is unacceptable for that parameter.

NOTE: The large number of parameters in Table 5 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2 6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8 3 The laboratory must, on an ongoing basis, spike at least 5% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing 1 to 20 samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8 3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each *parameter. If necessary,* prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 μL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent *recovery (P) as 100(A-B)%/T,* where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 5. These acceptance criteria wer calculated to include an allowance for error in measurement of both the background *and spike concentrations*, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 μg/L, the analyst must use either the QC acceptance criteria in Table 5, or optional QC acceptance *criteria calculated for* the specific spike concentration. To calculate optional acceptance criteria for the recoveryof a parameter: (1) Calculate accuracy (X') using the equation in Table 6, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 6, substituting X' for $\bar{X}$; (3) calculate the *range for recovery at the* spike concentration as (100 X'/T) (±2.44(100 S'/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described *in Section 8.4.*

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required anlaysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 5 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed *with the* spiked sample.

8.4.1 Prepare the QC check standard by adding 10 μL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check *standard to* determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 5. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any *such parameter* falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As a quality control check, the laboratory must spike all samples with the surrogate standard spiking solutions as described in Section 11.4, and calculate the percent recovery of each surrogate compound.

8.6 As part of the QC program for the laboratory, method accuracy *for wastewater* samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ *and* $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter a regular basis (e.g. after each five to ten new accuracy measurements).

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use *with this method.* The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in *relevant performance* evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions.[3] Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined. Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding 1+1 HCl while stirring vigorously. Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample container as described in Section 9.2.

9.4 All samples must be analyzed within 14 days of collection.[3]

10. Daily GC/MS Performance Tests

10.1 At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria are achieved for BFB.[9] The performance test must be passed before any samples, blanks, or standards are analyzed, unless the instrument has met the DFTPP test described in Method 625 earlier in the day.[10]

10.2 These performance tests require the following instrumental parameters:

Electron Energy: 70 V (nominal)
Mass Range: 20 to 260 amu
Scan Time: To give at least 5 scans per peak but not to exceed 7 s per scan.

10.3 At the beginning of each day, inject 2 µL of BFB solution directly on the column. Alternatively, add 2 µL of BFB solution to 5.0 mL of reagent water or standard solution and analyze the solution according to section 11. Obtain a background-corrected mass spectrum of BFB and confirm that all the key m/z criteria in Table 2 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

11. Sample Purging and Gas Chromatography

11.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. An example of the separations achieved by this column is shown in Figure 5. Other packed columns or chromatographic conditions may be used if the requirements of Section 8.2 are met.

11.2 After achieving the key m/z abundance criteria in Section 10, calibrate the system daily as described in Section 7.

11.3 Adjust the purge gas (helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

11.4 Allow the sample to come to ambient temperature prior to introducing it into the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the surrogate spiking solution (Section 6.7) and 10.0 µL of the internal standard spiking solution (Section 7.3.2) through the valve bore, then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution.

11.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

11.6 Close both valves and purge the sample for 11.0±0.1 min at ambient temperature.

11.7 After the 11-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC cloumn must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if problems persist) instead of the initial program temperature of 45 °C.

11.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

11.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

11.10 If the response for any m/z exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

12. Qualitative Identification

12.1 Obtain EICPs for the primary m/z (Table 4) and at least two secondary masses for each parameter of interest. The following criteria must be met to make a qualitative identification:

12.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

12.1.2 The retention time must fall within ±30 s of the retention time of the authentic compound.

12.1.3 The relative peak heights of the three characteristic masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library.

12.2 Structural isomers that have very similar mass spectra and less than 30 s difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13. Calculations

13.1 When a parameter has been identified, the quantitation of that parameter should be based on the integrated abundance from the EICP of the primary characteristic m/z given in Table 4. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate.

Calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.3 and Equation 2.

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

Equation 2

where:
A_s=Area of the characteristic m/z for the parameter or surrogate standard to be measured.
A_{is}–Area of the characteristic m/z for the internal standard.
C_{is}–Concentration of the internal standard.

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[11] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method was tested by 15 laboratories using reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations over the range 5-600 µg/L [12] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix Linear equations to describe these relationships are presented in Table 5.

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TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)	Method detection limit (μg/L)
Chloromethane	2.3	nd
Bromomethane	3.1	nd
Vinyl chloride	3.8	nd
Chloroethane	4.6	nd
Methylene chloride	6.4	2.8
Trichlorofluoromethane	8.3	nd
1,1-Dichloroethene	9.0	2.8
1,1-Dichloroethane	10.1	4.7
trans-1,2 Dichloroethene	10.8	1.6
Chloroform	11.4	1.6
1,2-Dichloroethane	12.1	2.8
1,1,1-Trichloroethane	13.4	3.8
Carbon tetrachloride	13.7	2.8
Bromodichloromethane	14.3	2.2
1,2-Dichloroproane	15.7	6.0
cis-1,3-Dichloropropene	15.9	5.0
Trichloroethene	16.5	1.9
Benzene	17.0	4.4
Dibromochloromethane	17.1	3.1
1,1,2-Trichloroethane	17.2	5.0
trans-1,3 Dichloropropene	17.2	nd
2 Chloroethylvinyl ether	18.6	nd
Bromoform	19.8	4.7
1,1,2,2-Tetrachloroethane	22.1	6.9
Tetrachloroethene	22.2	4.1
Toluene	23.5	6.0
Chlorobenzene	24.6	6.0
Ethyl benzene	26.4	7.2
1,3-Dichlorobenzene	33.9	nd
1,2-Dichlorobenzene	35.0	nd
1,4-Dichlorobenzene	35.4	nd

Column conditions Carbopak B (60/80 mesh) coated with 1% SP-1000 packed in a 6 ft by 0.1 in. ID glass column with helium carrier gas at 30 mL/min flow rate. Column temperature held at 45°C for 3 min., then programmed at 8°C/min to 220°C and held for 15 min.

nd=not determined

TABLE 2.—BFB KEY M/Z ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
50	15 to 40% of mass 95.
75	30 to 60% of mass 95.
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95.
173	<2% of mass 174.
174	>50% of mass 95
175	5 to 9% of mass 174.
176	>95% but < 101% of mass 174
177	5 to 9% of mass 176.

TABLE 3.—SUGGESTED SURROGATE AND INTERNAL STANDARDS

Compound	Retention time (min)[*]	Primary m/z	Secondary masses
Benzene d 6	17.0	84	
4-Bromofluorobenzene	28.3	95	174, 176
1,2-Dichloroethane d 4	12.1	102	
1,4-Difluorobenzene	19.6	114	63, 88
Ethylbenzene d-5	26.4	111	
Ethylbenzene d-10	26.4	98	
Fluorobenzene	18.4	96	70
Pentafluorobenzene	23.5	168	
Bromochloromethane	9.3	128	49, 130, 51
2-Bromo 1-chloropropane	19.2	77	79, 156
1, 4-Dichlorobutane	25.8	55	90, 92

[*] For chromatographic conditions, see Table 1.

TABLE 4.—CHARACTERISTIC MASSES FOR PURGEABLE ORGANICS

Parameter	Primary	Secondary
Chloromethane	50	52
Bromomethane	94	96.
Vinyl chloride	62	64.
Chloroethane	64	66.
Methylene chloride	84	49, 51, and 86.
Trichlorofluoromethane	101	103.
1,1-Dichloroethene	96	61 and 98
1,1-Dichloroethane	63	65, 83, 85, 98, and 100
trans-1,2-Dichloroethene	96	61 and 98
Chloroform	83	85.
1,2-Dichloroethane	98	62, 64, and 100.
1,1,1-Trichloroethane	97	99, 117, and 119
Carbon tetrachloride	117	119 and 121
Bromodichloromethane	127	83, 85, and 129.
1,2-Dichloropropane	112	63, 65, and 114.
trans-1,3-Dichloropropene	75	77.
Trichloroethene	130	95, 97, and 132.
Benzene	78	
Dibromochloromethane	127	129, 208, and 206.
1,1,2-Trichloroethane	97	83, 85, 99, 132, and 134.
cis-1,3-Dichloropropene	75	77.
2-Chloroethylvinyl ether	106	63 and 65.
Bromoform	173	171, 175, 250, 252, 254, and 256
1,1,2,2-Tetrachloroethane	168	83, 85, 131, 133, and 166.
Tetrachloroethene	164	129, 131, and 166.
Toluene	92	91.
Chlorobenzene	112	114.
Ethyl benzene	106	91
1,3-Dichlorobenzene	146	148 and 113
1,2-Dichlorobenzene	146	148 and 113
1,4-Dichlorobenzene	146	148 and 113

TABLE 5.—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 624[*]

Parameter	Range for Q (μ/g/L)	Limit for s (μ/g/L)	Range for X̄ (μ/g/L)	Range for P, P$_s$ (%)
Benzene	12.8–27.2	6.9	15.2–26.0	37–151
Bromodichloromethane	13.1–26.9	6.4	10.1–28.0	35–155
Bromoform	14.2–25.8	5.4	11.4–31.1	45–169
Bromomethane	2.8–37.2	17.9	D–41.2	D–242
Carbon tetrachloride	14.6–25.4	5.2	17.2–23.5	70–140
Chlorobenzene	13.2–26.8	6.3	16.4–27.4	37–160
Chloroethane	7.6–32.4	11.4	8.4–40.4	14–230
2-Chloroethylvinyl ether	D–44.8	25.9	D–50.4	D–305
Chloroform	13.5–26.5	6.1	13.7–24.2	51–138
Chloromethane	D–40.8	19.8	D–45.9	D–273
Dibromochloromethane	13.5–26.5	6.1	13.8–26.6	53–149
1,2-Dichlorobenzene	12.6–27.4	7.1	11.8–34.7	18–190
1,3-Dichlorobenzene	14.6–25.4	5.5	17.0–28.8	59–156
1,4-Dichlorobenzene	12.6–27.4	7.1	11.8–34.7	18–190
1,1-Dichloroethane	14.5–25.5	5.1	14.2–28.5	59–155
1,2-Dichloroethane	13.6–26.4	6.0	14.3–27.4	49–155
1,1-Dichlorothene	10.1–29.9	9.1	3.7–42.3	D–234
trans-1,2-Dichloroethene	13.9–26.1	5.7	13.6–28.5	54–156
1,2-Dichloropropane	6.8–33.2	13.8	3.8–36.2	D–210
cis-1,3-Dichloropropene	4.8–35.2	15.8	1.0–39.0	D–227
trans-1,3-Dichloropropene	10.0–30.0	10.4	7.6–32.4	17–183
Ethyl benzene	11.8–28.2	7.5	17.4–26.7	37–162
Methylene chloride	12.1–27.9	7.4	D–41.0	D–221
1,1,2,2 Tetrachloroethane	12.1–27.9	7.4	13.5–27.2	46–157
Tetrachloroethene	14.7–25.3	5.0	17.0–26.6	64–148
Toluene	14.9–25.1	4.8	16.6–26.7	47–150
1,1,1-Trichloroethane	15.0–25.0	4.6	13.7–30.1	52–162
1,1,2-Trichloroethane	14.2–25.8	5.5	14.3–27.1	52–150
Trichloroethene	13.3–26.7	6.6	18.6–27.6	71–157
Trichlorofluoromethane	9.6–30.4	10.0	8.9–31.5	17–181
Vinyl chloride	0.8–39.2	20.0	D–43.5	D–251

Q=Concentration measured in QC check sample, in μg/L (Section 7.5.3).

s=Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).

X̄—Average recovery of four recovery measurements, in μg/L (Section 8.2.4)

P, P$_s$—Percent recovery measured, (Section 8.3.2, Section 8.4.2).

D=Detected; result must be greater than zero.

[*] Criteria were calculated assuming a QC check sample concentration of 20 μg/L.

NOTE: These criteria are based directly upon the method performance data in Table 6. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 6

TABLE 6.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 624

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S' (μg/L)
Benzene	$0.93C+2.00$	$0.26\bar{X}-1.74$	$0.25\bar{X}-1.33$
Bromodichloromethane	$1.03C-1.58$	$0.15\bar{X}+0.59$	$0.20\bar{X}+1.13$
Bromoform	$1.18C-2.35$	$0.12\bar{X}+0.36$	$0.17\bar{X}+1.38$
Bromomethane [a]	$1.00C$	$0.43\bar{X}$	$0.58\bar{X}$
Carbon tetrachloride	$1.10C-1.68$	$0.12\bar{X}+0.25$	$0.11\bar{X}+0.37$
Chlorobenzene	$0.98C+2.28$	$0.16\bar{X}\ \ 0.09$	$0.26\bar{X}-1.92$
Chloroethane	$1.18C+0.81$	$0.14\bar{X}+2.78$	$0.29\bar{X}+1.75$
2-Chloroethylvinyl ether [a]	$1.00C$	$0.62\bar{X}$	$0.84\bar{X}$
Chloroform	$0.93C+0.33$	$0.16\bar{X}+0.22$	$0.18\bar{X}+0.16$
Chloromethane	$1.03C+0.81$	$0.37\bar{X}+2.14$	$0.58\bar{X}+0.43$
Dibromochloromethane	$1.01C-0.03$	$0.17\bar{X}-0.18$	$0.17\bar{X}+0.49$
1,2-Dichlorobenzene [b]	$0.94C+4.47$	$0.22\bar{X}-1.45$	$0.30\bar{X}-1.20$
1,3-Dichlorobenzene	$1.06C+1.68$	$0.14\bar{X}-0.48$	$0.18\bar{X}-0.82$
1,4-Dichlorobenzene [b]	$0.94C+4.47$	$0.22\bar{X}-1.45$	$0.30\bar{X}-1.20$
1,1-Dichloroethane	$1.05C+0.36$	$0.13\bar{X}-0.05$	$0.16\bar{X}+0.47$
1,2-Dichloroethane	$1.02C+0.45$	$0.17\bar{X}-0.32$	$0.21\bar{X}-0.38$
1,1-Dichloroethene	$1.12C+0.61$	$0.17\bar{X}+1.06$	$0.43\bar{X}-0.22$
trans-1,2,-Dichloroethene	$1.05C+0.03$	$0.14\bar{X}+0.09$	$0.19\bar{X}+0.17$
1,2-Dichloropropane [a]	$1.00C$	$0.33\bar{X}$	$0.45\bar{X}$
cis-1,3-Dichloropropene [a]	$1.00C$	$0.38\bar{X}$	$0.52\bar{X}$
trans-1,3-Dichloropropene [a]	$1.00C$	$0.25\bar{X}$	$0.34\bar{X}$
Ethyl benzene	$0.98C+2.48$	$0.14\bar{X}+1.00$	$0.26\bar{X}-1.72$
Methylene chloride	$0.87C+1.88$	$0.15\bar{X}+1.07$	$0.32\bar{X}+4.00$
1,1,2,2-Tetrachloroethane	$0.93C+1.76$	$0.16\bar{X}+0.69$	$0.20\bar{X}+0.41$
Tetrachloroethene	$1.06C+0.60$	$0.13\bar{X}-0.18$	$0.16\bar{X}-0.45$
Toluene	$0.98C+2.03$	$0.15\bar{X}-0.71$	$0.22\bar{X}-1.71$
1,1,1-Trichloroethane	$1.06C+0.73$	$0.12\bar{X}-0.15$	$0.21\bar{X}-0.39$
1,1,2-Trichloroethane	$0.95C+1.71$	$0.14\bar{X}+0.02$	$0.18\bar{X}+0.00$
Trichloroethene	$1.04C+2.27$	$0.13\bar{X}+0.36$	$0.12\bar{X}+0.59$
Trichloroflouromethane	$0.99C+0.39$	$0.33\bar{X}-1.48$	$0.34\bar{X}-0.39$
Vinyl chloride	$1.00C$	$0.48\bar{X}$	$0.65\bar{X}$

$\bar{X}$ = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
S' = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

[a] Estimates based upon the performance in a single laboratory [13]

[b] Due to chromatographic resolution problems, performance statements for these isomers are based upon the sums of their concentrations.

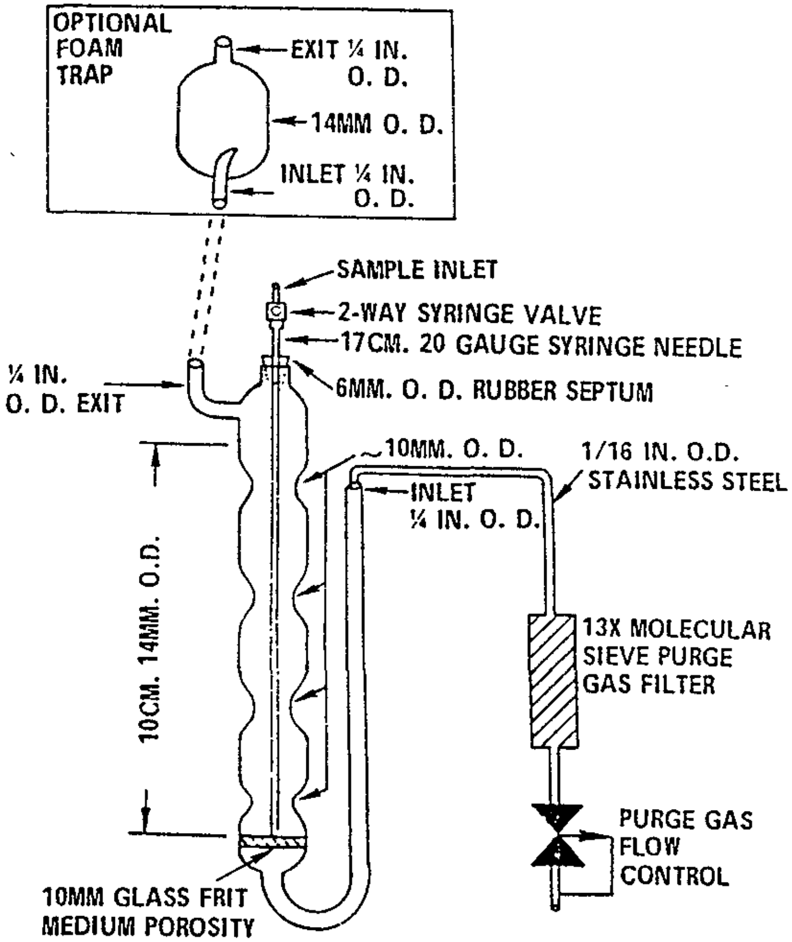


Figure 1. Purging device.

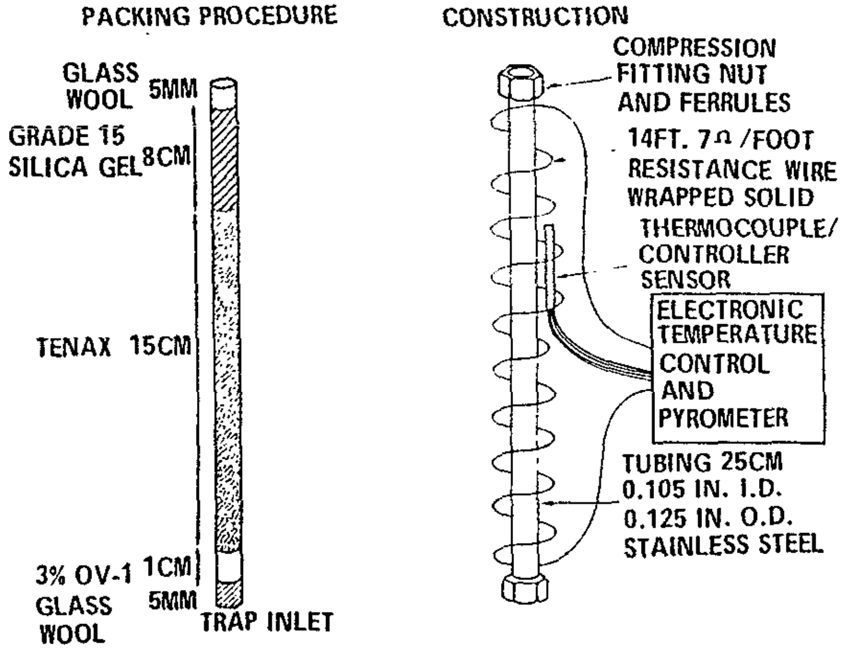
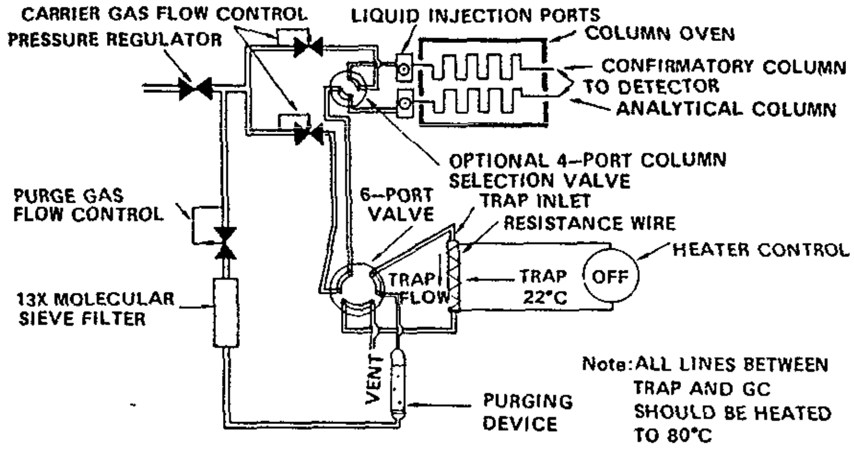
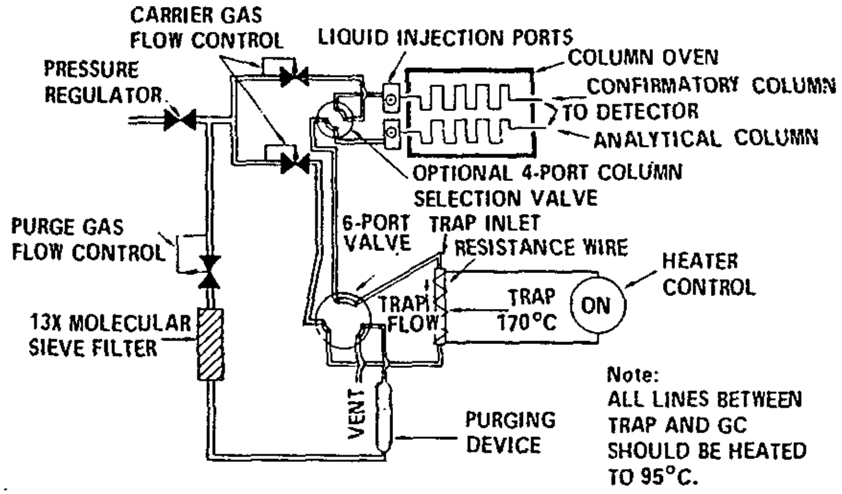


Figure 2. Trap packings and construction to include desorb capability.



Note: ALL LINES BETWEEN TRAP AND GC SHOULD BE HEATED TO 80°C

Figure 3. Purge and trap system - purge mode.



Note:
ALL LINES BETWEEN TRAP AND GC SHOULD BE HEATED TO 95°C.

Figure 4. Purge and trap system - desorb mode.

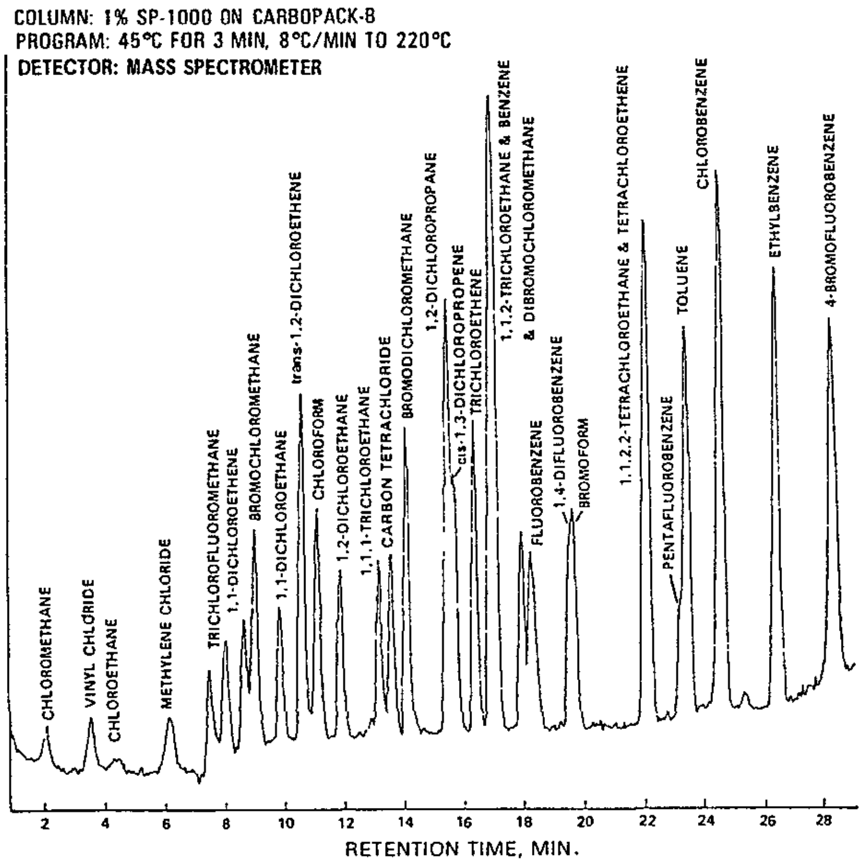


Figure 5. Gas chromatogram of volatile organics.

METHOD 625—BASE/NEUTRALS AND ACIDS

1. Scope and Application

1.1 This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. The parameters listed in Tables 1 and 2 may be qualitatively and quantitatively determined using this method.

1.2 The method may be extended to include the parameters listed in Table 3. Benzidine can be subject to oxidative losses during solvent concentration. Under the alkaline conditions of the extraction step, α-BHC, γ-BHC, endosulfan I and II, and endrin are subject to decomposition. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. The preferred method for each of these parameters is listed in Table 3.

1.3 This is a gas chromatographic/mass spectrometry (GC/MS) method[2, 14] applicable to the determination of the compounds listed in Tables 1, 2, and 3 in municipal and industrial discharges as provided under 40 CFR 136.1.

1.4 The method detection limit (MDL, defined in Section 16.1)[1] for each parameter is listed in Tables 4 and 5. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 Any modification to this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5. Depending upon the nature of the modification and the extent of intended use, the applicant may be required to demonstrate that the modifications will produce equivalent results when applied to relevant wastewaters.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1-L, is serially extracted with methylene chloride at a pH greater than 11 and again at a pH less than 2 using a separatory funnel or a continuous extractor.[2] The methylene chloride extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of three characteristic masses (m/z). Quantitative analysis is performed using internal standard techniques with a single characteristic m/z.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thmrough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled.

3.3 The base-neutral extraction may cause significantly reduced recovery of phenol, 2-methylphenol, and 2,4-dimethylphenol. The analyst must recognize that results obtained under these conditions are minimum concentrations.

3.4 The packed gas chromatographic columns recommended for the basic fraction may not exhibit sufficient resolution for certain isomeric pairs including the following: anthracene and phenanthrene; chry-

METHOD 3810

HEADSPACE

1.0 SCOPE AND APPLICATION

1.1 Method 3810 was formerly Method 5020 in the second edition of this manual.

1.2 Method 3810 is a static headspace technique for extracting volatile organic compounds from samples. It is a simple method that allows large numbers of samples to be screened in a relatively short period of time. It is ideal for screening samples prior to using the purge-and-trap method. Detection limits for this method may vary widely among samples because of the large variability and complicated matrices of waste samples. The method works best for compounds with boiling points of less than 125°C. The sensitivity of this method will depend on the equilibria of the various compounds between the vapor and dissolved phases.

1.3 Due to the variability of this method, this procedure is recommended for use only as a screening procedure for other, more accurate determinative methods (Methods 8010, 8015, 8020, 8030, and 8240).

2.0 SUMMARY OF METHOD

2.1 The sample is collected in sealed glass containers and allowed to equilibrate at 90°C. A sample of the headspace gas is withdrawn with a gas-tight syringe for screening analysis using the conditions specified in one of the GC or GC/MS determinative methods (8010, 8015, 8020, 8030, or 8240).

3.0 INTERFERENCES

3.1 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

3.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with reagent water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water. It may be necessary to wash out the syringe with detergent, rinse with distilled water, and dry in a 105°C oven between analyses.

3.3 Before processing any samples, the analyst should demonstrate daily through the analysis of an organic-free water or solvent blank that the entire analytical system is interference-free.

4.0 APPARATUS AND MATERIALS

4.1 Refer to the specific determinative method for appropriate apparatus and materials.

4.2 Vials: 125-mL Hypo-Vials (Pierce Chemical Co., #12995, or equivalent), four each.

4.3 Septa: Tuf-Bond (Pierce #12720 or equivalent).

4.4 Seals: Aluminum (Pierce #132141 or equivalent).

4.5 Crimper: Hand (Pierce #13212 or equivalent).

4.6 Syringe: 5-mL, gas-tight with shutoff valve and chromatographic needles.

4.7 Microsyringe: 250- or 500-uL.

4.8 Water bath: Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

5.0 REAGENTS

5.1 Refer to the specific determinative method and Method 8000 for preparation of calibration standards.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Gas chromatographic conditions and Calibration: Refer to the specific determinative method for GC operating conditions and to Method 8000, Section 7.4, for calibration procedures.

7.2 Sample preparation:

7.2.1 Place 10.0 g of a well-mixed waste sample into each of two separate 125-mL septum-seal vials.

7.2.2 Dose one sample vial through the septum with 200 uL of a 50 ng/uL calibration standard containing the compounds of interest. Label this "1-ppm spike."

7.2.3 Dose a separate (empty) 125-mL septum seal vial with 200 uL of the same 50 ng/uL calibration standard. Label this "1-ppm standard."

7.2.4 Place the sample, 1-ppm-spike, and 1-ppm-standard vials into a 90°C water bath for 1 hr. Store the remaining sample vial at 4.0°C for possible future analysis.

7.3 Sample analysis:

7.3.1 While maintaining the vials at 90°C, withdraw 2 mL of the headspace gas with a gas-tight syringe and analyze by direct injection into a GC. The GC should be operated using the same GC conditions listed in the method being screened (8010, 8015, 8020, 8030, or 8240).

7.3.2 Analyze the 1-ppm standard and adjust instrument sensitivity to give a minimum response of at least 2 times the background. Record retention times (RT) and peak areas of compounds of interest.

7.3.3 Analyze the 1-ppm spiked sample in the same manner. Record RTs and peak areas.

7.3.4 Analyze the undosed sample as in Paragraph 7.3.3.

7.3.5 Use the results obtained to determine if the sample requires dilution or methanolic extraction as indicated in Method 5030.

8.0 QUALITY CONTROL

8.1 Before processing any samples, the analyst should demonstrate through the analysis of a distilled water method blank that all glassware and reagents are interference-free. Each time a set of samples is extracted or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Standard quality assurance practices should be used with this method. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis. If the fortified waste samples do not indicate sufficient sensitivity to detect less than or equal to 1 ug/g of sample, then the sensitivity of the instrument should be increased.

9.0 METHOD PERFORMANCE

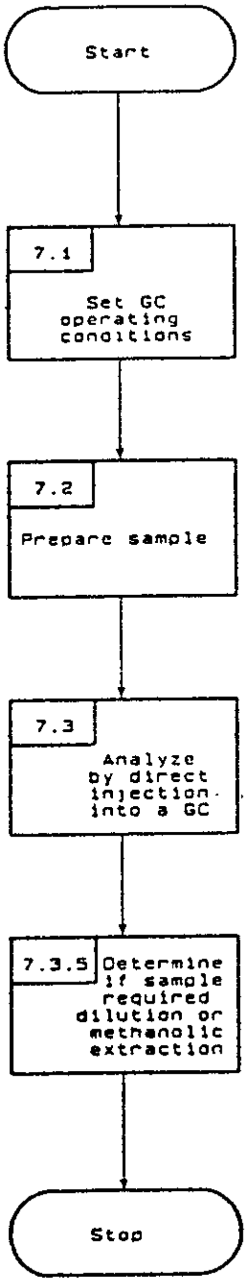
9.1 No data provided.

10.0 REFERENCES

1. Hachenberg, H. and A. Schmidt, Gas Chromatographic Headspace Analysis, Philadelphia: Hayden & Sons Inc., 1979.

2. Friant, S.L. and I.H. Suffet, "Interactive Effects of Temperature, Salt Concentration and pH on Headspace Analysis for Isolating Volatile Trace Organics in Aqueous Environmental Samples," Anal. Chem. 51, 2167-2172, 1979.

METHOD 3810
HEADSPACE METHOD



METHOD 8015

NONHALOGENATED VOLATILE ORGANICS

1.0 SCOPE AND APPLICATION

1.1 Method 8015 is used to determine the concentration of various nonhalogenated volatile organic compounds. Table 1 indicates the compounds that may be investigated by this method.

2.0 SUMMARY OF METHOD

2.1 Method 8015 provides gas chromatographic conditions for the detection of certain nonhalogenated volatile organic compounds. Samples may be analyzed using direct injection or purge-and-trap (Method 5030). Ground water samples must be analyzed by Method 5030. A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved by a flame ionization detector (FID).

2.2 If interferences are encountered, the method provides an optional gas chromatographic column that may be helpful in resolving the analytes from interferences that may occur and for analyte confirmation.

3.0 INTERFERENCES

3.1 Refer to Method 5030 and 8000.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 Gas Chromatograph - Analytical system complete with gas chromatograph suitable for on-column injections or purge-and-trap sample introduction and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

4.1.2 Columns

4.1.2.1 Column 1 - 8 ft x 0.1 in i.d. stainless steel or glass column packed with 1% SP-1000 on Carbopack-B 60/80 mesh or equivalent.

4.1.2.2 Column 2 - 6 ft x 0.1 in i.d. stainless steel or glass column packed with n-octane on Porasil-C 100/120 mesh (Durapak) or equivalent.

4.1.3 Detector - Flame ionization (FID).

4.2 Sample introduction apparatus - Refer to Method 5030 for the appropriate equipment for sample introduction purposes.

4.3 Syringes - A 5-mL Luerlok glass hypodermic and a 5-mL, gas-tight with shutoff valve.

4.4 Volumetric flasks - 10-, 50-, 100-, 500-, and 1,000-mL with a ground-glass stopper.

4.5 Microsyringes - 10- and 25-uL with a 0.006-in i.d. needle (Hamilton 702N or equivalent) and a 100-uL.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.

5.3 Stock standards - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standards in methanol using assayed liquids.

5.3.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

5.3.2 Using a 100-uL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.3.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter (ug/uL) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.4 Transfer the stock standard solution into a bottle with a Teflon lined screw-cap. Store, with minimal headspace, at -10°C to -20°C and protect from light.

5.3.5 Standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

5.4 Secondary dilution standards - Using stock standard solutions, prepare in methanol secondary dilution standards, as needed, that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Step 5.5 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace for volatiles and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.5 Calibration standards - Calibration standards at a minimum of five concentration levels are prepared in water from the secondary dilution of the stock standards. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Each standard should contain each analyte for detection by this method (e.g. some or all of the compounds listed in Table 1 may be included). In order to prepare accurate aqueous standard solutions, the following precautions must be observed:

5.5.1 Do not inject more than 20 uL of alcoholic standards into 100 mL of water.

5.5.2 Use a 25-uL Hamilton 702N microsyringe or equivalent (variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water).

5.5.3 Rapidly inject the alcoholic standard into the filled volumetric flask. Remove the needle as fast as possible after injection.

5.5.4 Mix aqueous standards by inverting the flask three times only.

5.5.5 Fill the sample syringe from the standard solution contained in the expanded area of the flask (do not use any solution contained in the neck of the flask).

5.5.6 Never use pipets to dilute or transfer samples or aqueous standards.

5.5.7 Aqueous standards are not stable and should be discarded after 1 hour, unless properly sealed and stored. The aqueous standards can be stored up to 24 hours, if held in sealed vials with zero headspace.

5.6 Internal standards (if internal standard calibration is used) - To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

5.6.1 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest as described in Step 5.5.

5.6.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Steps 5.3 and 5.4. It is recommended that the secondary dilution standard be prepared at a concentration of 15 ug/mL of each internal standard compound. The addition of 10 uL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 ug/L.

5.6.3 Analyze each calibration standard according to Section 7.0, adding 10 uL of internal standard spiking solution directly to the syringe.

5.7 Surrogate standards - The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and water blank with one or two surrogate compounds recommended to encompass the range of temperature program used in this method. From stock standard solutions prepared as in Step 5.3, add a volume to give 750 ug of each surrogate to 45 mL of water contained in a 50-mL volumetric flask, mix, and dilute to volume for a concentration of 15 ng/uL. Add 10 uL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. If the internal standard calibration procedure is used, the surrogate compounds may be added directly to the internal standard spiking solution (Step 5.6.2).

5.7 Methanol, CH_3OH. Pesticide quality or equivalent. Store away from other solvents.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this Chapter, Organic Analytes, Step 4.1.

7.0 PROCEDURE

7.1 Volatile compounds are introduced into the gas chromatograph either by direct injection or purge-and-trap (Method 5030). Method 5030 may be used directly on ground water samples or low-level contaminated soils and sediments. For medium-level soils or sediments, methanolic extraction, as described in Method 5030, may be necessary prior to purge-and-trap analysis.

7.2 Gas chromatography conditions (Recommended)

7.2.1 Column 1 - Set helium gas flow at 40 mL/min flow rate. Set column temperature at 45°C for 3 minutes; then program an 8°C/min temperature rise to 220°C and hold for 15 minutes.

7.2.2 Column 2 - Set helium gas flow at 40 mL/min flow rate. Set column temperature at 50°C for 3 minutes; then program a 6°C/min temperature rise to 170°C and hold for 4 minutes.

7.3 Calibration - Refer to Method 8000 for proper calibration techniques.

7.3.1 Calibration must take place using the same sample introduction method that will be used to analyze actual samples (see Step 7.4.1).

7.3.2 The procedure for internal or external calibration may be used. Refer to Method 8000 for a description of each of these procedures.

7.4 Gas chromatographic analysis

7.4.1 Introduce volatile compounds into the gas chromatograph using either Method 5030 (purge-and-trap method) or the direct injection method. If the internal standard calibration technique is used, add 10 uL of internal standard to the sample prior to purging.

7.4.1.1 Direct injection - In very limited applications (e.g. aqueous process wastes), direct injection of the sample into the GC system with a 10-uL syringe may be appropriate. One such application is for verification of the alcohol content of an aqueous sample prior to determining if the sample is ignitable (Methods 1010 or 1020). In this case, it is suggested that direct injection be used. The detection limit is very high (approximately 10,000 ug/L); therefore, it is only permitted when concentrations in excess of 10,000 ug/L are expected or for water-soluble compounds that do not purge. The system must be calibrated by direct injection (bypassing the purge-and-trap device).

7.4.2 Follow Step 7.6 in Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

7.4.3 Record the sample volume purged or injected and the resulting peak sizes (in area units or peak heights).

7.4.4 Calculation of concentration is covered in Step 7.8 of Method 8000.

7.4.5 If analytical interferences are suspected, or for the purpose of confirmation, analysis using the second GC column is recommended.

7.4.6 If the response for a peak is off-scale, prepare a dilution of the sample with water. The dilution must be performed on a second aliquot of the sample which has been properly sealed and stored prior to use.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 8000 for gas chromatographic procedures. Quality control to ensure the proper operation of the purge-and-trap device is covered in Method 5030.

8.2 Mandatory quality control to validate the GC system operation is found in Method 8000, Step 8.6.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if recovery is within limits (limits established by performing QC procedure outlined in Method 8000, Step 8.10).

8.3.1 If recovery is not within limits, the following is required:

- Check to be sure there are no errors in calculations, surrogate solutions, and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

9.0 METHOD PERFORMANCE

9.1 The accuracy and precision obtained will be determined by the sample matrix, sample introduction technique, and calibration procedures used.

9.2 Specific method performance information will be provided as it becomes available.

10.0 REFERENCES

1. Bellar, T.A., and J.J. Lichtenberg, Determining Volatile Organics at Microgram-per-Liter Levels by Gas Chromatography, J. Amer. Water Works Assoc., 66(12), pp. 739-744 (1974).

2. Bellar, T.A., and J.J. Lichtenberg, Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds, in Van Hall, ed., Measurement of Organic Pollutants in Water and Wastewater, ASTM STP 686, pp. 108-129, 1979.

3. Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters: Category 11 - Purgeables and Category 12 - Acrolein, Acrylonitrile, and Dichlorodifluoromethane, Report for EPA Contract 68-03-2635 (in preparation).

4. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

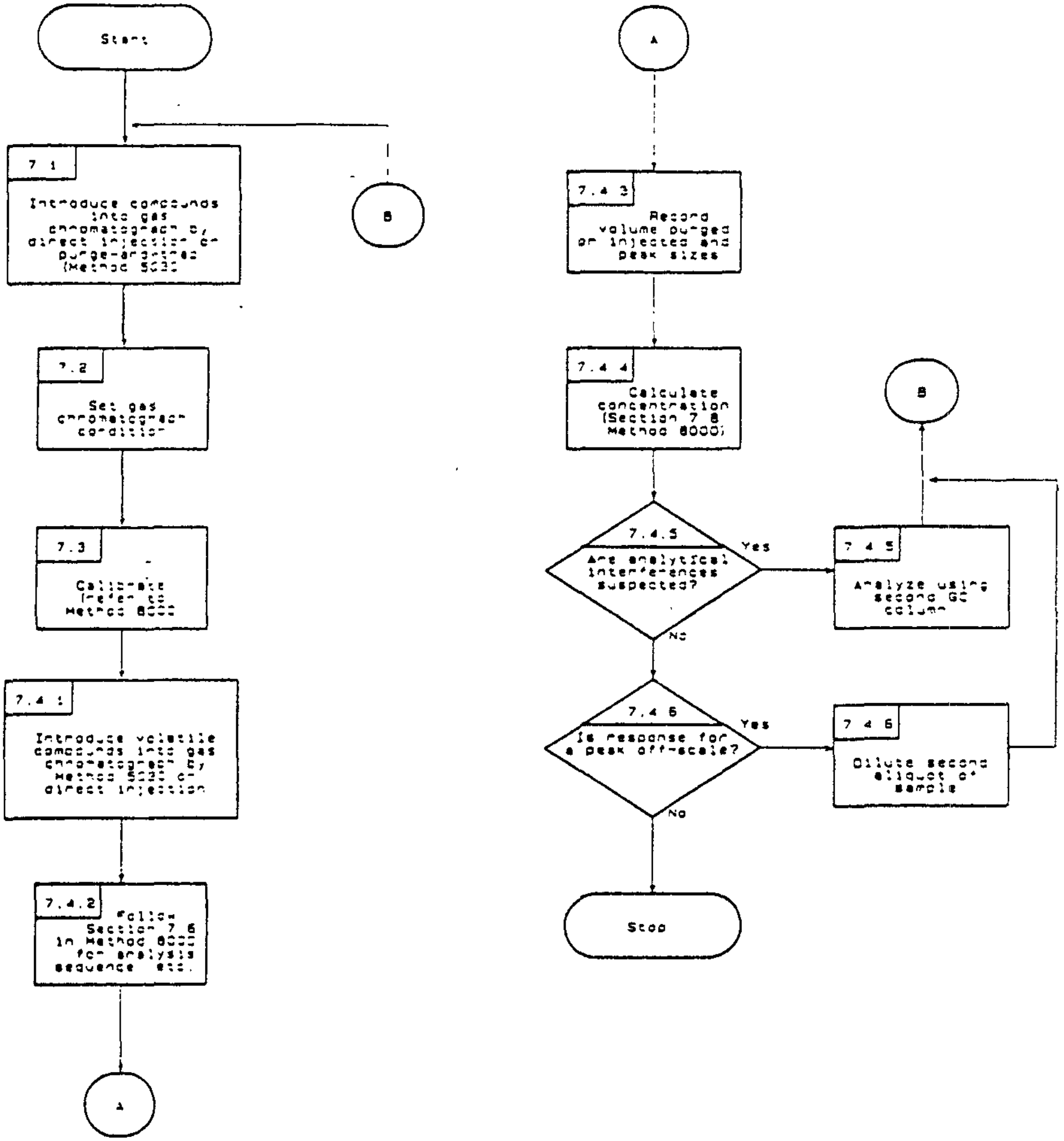
5. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

TABLE 1.
NONHALOGENATED VOLATILE ORGANICS

Diethyl ether
Ethanol
Methyl ethyl ketone (MEK)
Methyl isobutyl ketone (MIBK)

METHOD 8015

NONHALOGENATED VOLATILE ORGANICS



METHOD 8020

AROMATIC VOLATILE ORGANICS

1.0 SCOPE AND APPLICATION

1.1 Method 8020 is used to determine the concentration of various aromatic volatile organic compounds. Table 1 indicates compounds which may be determined by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit (PQL) for other matrices.

2.0 SUMMARY OF METHOD

2.1 Method 8020 provides chromatographic conditions for the detection of aromatic volatile compounds. Samples can be analyzed using direct injection or purge-and-trap (Method 5030). Ground water samples must be determined using Method 5030. A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved by a photo-ionization detector (PID).

2.2 If interferences are encountered, the method provides an optional gas chromatographic column that may be helpful in resolving the analytes from the interferences and for analyte confirmation.

3.0 INTERFERENCES

3.1 Refer to Method 5030 and 8000.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 **Gas Chromatograph:** Analytical system complete with gas chromatograph suitable for on-column injections or purge-and-trap sample introduction and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

TABLE 1. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS FOR AROMATIC VOLATILE ORGANICS

Compound	Retention time (min) Col. 1	Retention time (min) Col. 2	Method detection limit[a] (ug/L)
Benzene	3.33	2.75	0.2
Chlorobenzene	9.17	8.02	0.2
1,4-Dichlorobenzene	16.8	16.2	0.3
1,3-Dichlorobenzene	18.2	15.0	0.4
1,2-Dichlorobenzene	25.9	19.4	0.4
Ethyl Benzene	8.25	6.25	0.2
Toluene	5.75	4.25	0.2
Xylenes			

[a] Using purge-and-trap method (Method 5030).

TABLE 2. DETERMINATION OF PRACTICAL QUANTITATION LIMITS (PQL) FOR VARIOUS MATRICES[a]

Matrix	Factor[b]
Ground water	10
Low-level soil	10
Water miscible liquid waste	500
High-level soil and sludge	1250
Non-water miscible waste	1250

[a] Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b] PQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

4.1.2 Columns:

4.1.2.1 Column 1: 6-ft x 0.082-in I.D. #304 stainless steel or glass column packed with 5% SP-1200 and 1.75% Bentone-34 on 100/120 mesh Supelcort or equivalent.

4.1.2.2 Column 2: 8-ft x 0.1-in I.D. stainless steel or glass column packed with 5% 1,2,3-Tris(2-cyanoethoxy)propane on 60/80 mesh Chromosorb W-AW or equivalent.

4.1.3 **Detector:** Photoionization (PID) (h-Nu Systems, Inc. Model PI-51-02 or equivalent).

4.2 Sample introduction apparatus: Refer to Method 5030 for the appropriate equipment for sample introduction purposes.

4.3 Syringes: A 5-mL Luerlok glass hypodermic and a 5-mL, gas-tight with shutoff valve.

4.4 Volumetric flask: 10-, 50-, 100-, 500-, and 1,000-mL with a ground-glass stopper.

4.5 Microsyringe: 10- and 25-uL with a 0.006-in I.D. needle (Hamilton 702N or equivalent) and a 100-uL.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as a water in which an interferent is not observed at the method detection limit (MDL) of the parameters of interest.

5.2 Stock standards: Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standards in methanol using assayed liquids. Because of the toxicity of benzene and 1,4-dichlorobenzene, primary dilutions of these materials should be prepared in a hood.

5.2.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

5.2.2 Using a 100-uL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.2.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter (ug/uL) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.2.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at 4°C and protect from light.

5.2.5 All standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

5.3 Secondary dilution standards: Using stock standard solutions, prepare in methanol secondary dilution standards, as needed, that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Paragraph 5.4 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace for volatiles and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.4 Calibration standards: Calibration standards at a minimum of five concentration levels are prepared in reagent water from the secondary dilution of the stock standards. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Each standard should contain each analyte for detection by this method (e.g., some or all of the compounds listed in Table 1 may be included). In order to prepare accurate aqueous standard solutions, the following precautions must be observed.

5.4.1 Do not inject more than 20 uL of alcoholic standards into 100 mL of reagent water.

5.4.2 Use a 25-uL Hamilton 702N microsyringe or equivalent (variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water).

5.4.3 Rapidly inject the alcoholic standard into the filled volumetric flask. Remove the needle as fast as possible after injection.

5.4.4 Mix aqueous standards by inverting the flask three times only.

5.4.5 Fill the sample syringe from the standard solution contained in the expanded area of the flask (do not use any solution contained in the neck of the flask).

5.4.6 Never use pipets to dilute or transfer samples or aqueous standards.

5.4.7 Aqueous standards are not stable and should be discarded after 1 hr, unless properly sealed and stored. The aqueous standards can be stored up to 24 hr, if held in sealed vials with zero headspace.

5.5 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compound, alpha,alpha,alpha-trifluorotoluene recommended for use as a surrogate spiking compound (Paragraph 5.6) has been used successfully as an internal standards.

5.5.1 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest as described in Section 5.4.

5.5.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 5.2 and 5.3. It is recommended that the secondary dilution standard be prepared at a concentration of 15 ug/mL of each internal standard compound. The addition of 10 uL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 ug/L.

5.5.3 Analyze each calibration standard according to Section 7.0, adding 10 uL of internal standard spiking solution directly to the syringe.

5.6 Surrogate standards: The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate compounds (e.g, alpha,alpha,alpha-trifluorotoluene) recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 5.2, add a volume to give 750 ug of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix, and dilute to volume for a concentration of 15 ng/uL. Add 10 uL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. If the internal standard calibration procedure is used, the surrogate compounds may be added directly to the internal standard spiking solution (Paragraph 5.5.2).

5.7 Methanol: pesticide quality or equivalent. Store away from other solvents.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Volatile compounds are introduced into the gas chromatograph either by direct injection or purge-and-trap (Method 5030). Method 5030 may be used directly on ground water samples or low-level contaminated soils and sediments. For medium-level soils or sediments, methanolic extraction, as described in Method 5030, may be necessary prior to purge-and-trap analysis.

7.2 Gas chromatography conditions (Recommended):

7.2.1 **Column 1:** Set helium gas flow at 36 mL/min flow rate. The temperature program sequences are as follows: For lower boiling compounds, operate at 50°C isothermal for 2 min; then program at 6°C/min to 90°C and hold until all compounds have eluted. For higher boiling range of compounds, operate at 50°C isothermal for 2 min; then program at 3°C/min to 110°C and hold until all compounds have eluted. Column 1 provides outstanding separations for a wide variety of aromatic hydrocarbons. Column 1 should be used as the primary analytical column because of its unique ability to resolve para-, meta-, and ortho-aromatic isomers.

7.2.2 **Column 2:** Set helium gas flow at 30 mL/min flow rate. The temperature program sequence is as follows: 40°C isothermal for 2 min; then 2°C/min to 100°C and hold until all compounds have eluted. Column 2, an extremely high-polarity column, has been used for a number of years to resolve aromatic hydrocarbons from alkanes in complex samples. However, because resolution between some of the aromatics is not as efficient as with Column 1, Column 2 should be used as a confirmatory column.

7.3 Calibration: Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 Calibration must take place using the same sample introduction method that will be used to analyze actual samples (see Section 7.4.1).

7.3.2 The procedure for internal or external calibration may be used. Refer to Method 8000 for a description of each of these procedures.

7.4 Gas chromatographic analysis:

7.4.1 Introduce volatile compounds into the gas chromatograph using either Method 5030 (purge-and-trap method) or the direct injection method. If the internal standard calibration technique is used, add 10 uL of internal standard to the sample prior to purging.

7.4.1.1 Direct injection: In very limited applications (e.g., aqueous process wastes), direct injection of the sample into the GC system with a 10 uL syringe may be appropriate. The detection limit is very high (approximately 10,000 ug/L); therefore, it is only permitted when concentrations in excess of 10,000 ug/L are expected or for water-soluble compounds that do not purge. The system must be calibrated by direct injection (bypassing the purge-and-trap device).

7.4.2 Follow Section 7.6 of Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

7.4.3 Table 1 summarizes the estimated retention times and detection limits for a number of organic compounds analyzable using this method. An example of the separation achieved by Column 1 is shown in Figure 1. Figure 2 shows an example of the separation achieved using Column 2.

7.4.4 Record the sample volume purged or injected and the resulting peak sizes (in area units or peak heights).

7.4.5 Calculation of concentration is covered in Section 7.8 of Method 8000.

7.4.6 If analytical interferences are suspected, or for the purpose of confirmation, analysis using the second GC column is recommended.

7.4.7 If the response for a peak is off-scale, prepare a dilution of the sample with reagent water. The dilution must be performed on a second aliquot of the sample which has been properly sealed and stored prior to use.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 8000 for gas chromatographic procedures. Quality control to ensure the proper operation of the purge-and-trap device is covered in Method 5030.

8.2 Mandatory quality control to validate the GC system operation is found in Method 8000, Section 8.6.

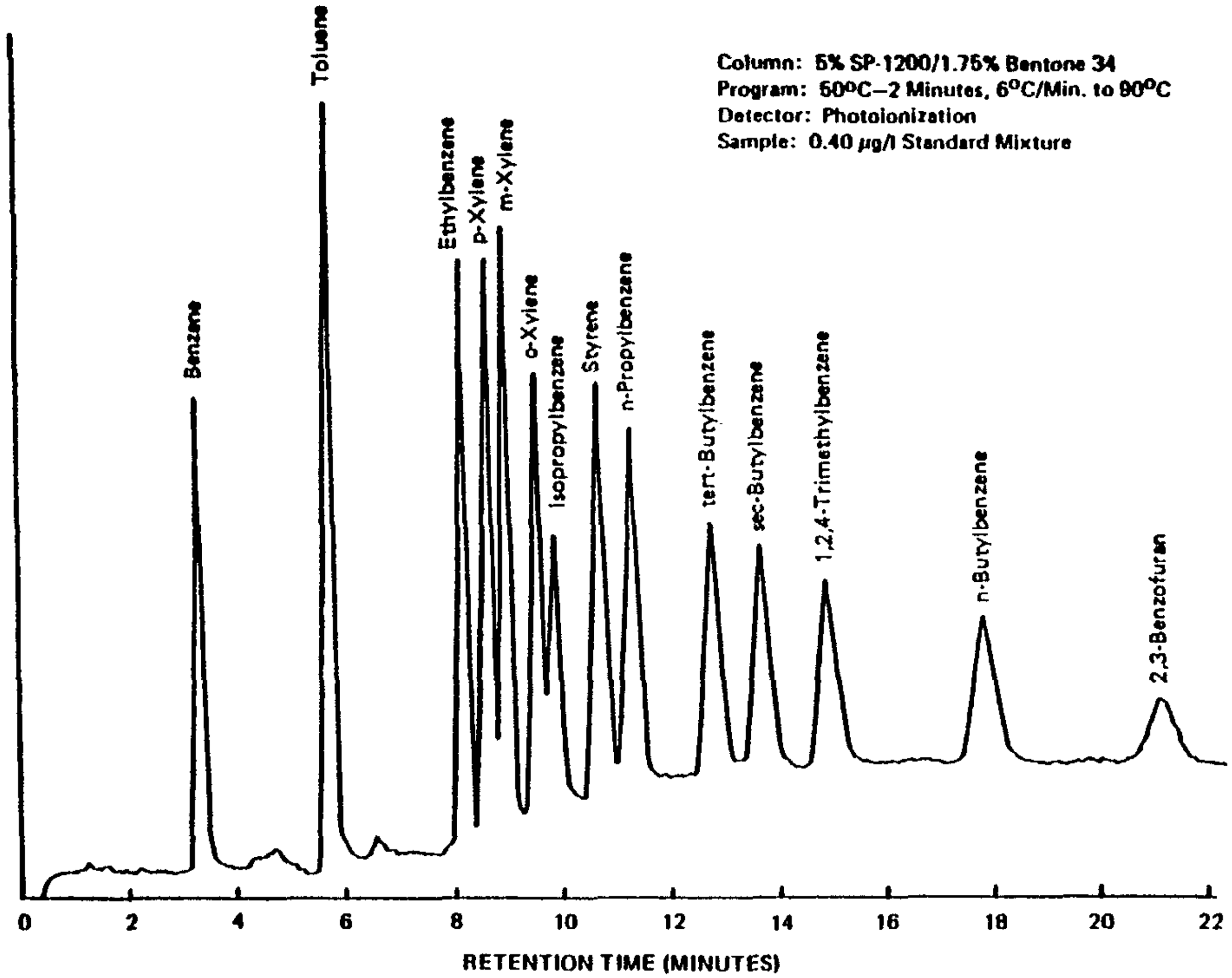
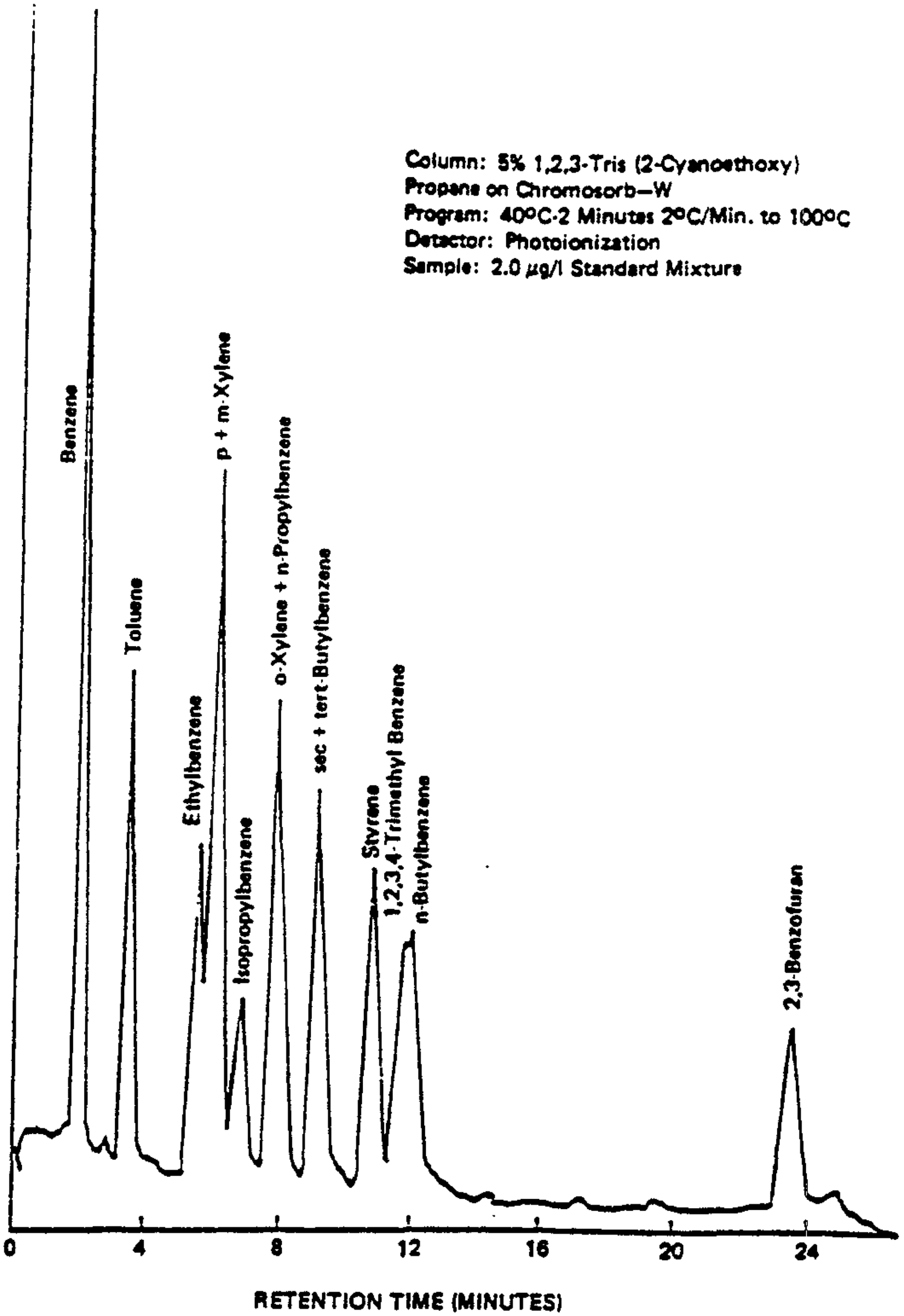


Figure 1. Chromatogram of aromatic volatile organics (column 1 conditions).



Column: 5% 1,2,3-Tris (2-Cyanoethoxy)
Propane on Chromosorb–W
Program: 40°C-2 Minutes 2°C/Min. to 100°C
Detector: Photoionization
Sample: 2.0 μg/l Standard Mixture

Figure 2. Chromatogram of aromatic volatile organics (column 2 conditions).

8.2.1 The quality control check sample concentrate (Method 8000, Section 8.6) should contain each parameter of interest at a concentration of 10 ug/mL in methanol.

8.2.2 Table 3 indicates the calibration and QC acceptance criteria for this method. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables should be used to evaluate a laboratory's ability to perform and generate acceptable data by this method.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if recovery is within limits (limits established by performing QC procedure outlined in Method 8000, Section 8.10).

8.3.1 If recovery is not within limits, the following is required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

9.0 METHOD PERFORMANCE

9.1 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 2.1-500 ug/L. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 4.

9.2 The accuracy and precision obtained will be determined by the sample matrix, sample introduction technique, and by the calibration procedure used.

10.0 REFERENCES

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2. Bellar, T.A., and J.J. Lichtenberg, Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds, in Van Hall (ed.), Measurement of Organic Pollutants in Water and Wastewater, ASTM STP 686, pp. 108-129, 1979.

3. Dowty, B.J., S.R. Antoine, and J.L. Laseter, "Quantitative and Qualitative Analysis of Purgeable Organics by High Resolution Gas Chromatography and Flame Ionization Detection," in Van Hall, ed., Measurement of Organic Pollutants in Water and Wastewater. ASTM STP 686, pp. 24-35, 1979.

4. Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 11 - Purgeables and Category 12 - Acrolein, Acrylonitrile, and Dichlorodifluoromethane. Report for EPA Contract 68-03-2635 (in preparation).

5. "EPA Method Validation Study 24, Method 602 (Purgeable Aromatics)," Report for EPA Contract 68-03-2856 (in preparation).

6. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

7. Provost, L.P., and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.

TABLE 3. CALIBRATION AND QC ACCEPTANCE CRITERIA[a]

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Benzene	15.4-24.6	4.1	10.0-27.9	39-150
Chlorobenzene	16.1-23.9	3.5	12.7-25.4	55-135
1,2-Dichlorobenzene	13.6-26.4	5.8	10.6-27.6	37-154
1,3-Dichlorobenzene	14.5-25.5	5.0	12.8-25.5	50-141
1,4-Dichlorobenzene	13.9-26.1	5.5	11.6-25.5	42-143
Ethylbenzene	12.6-27.4	6.7	10.0-28.2	32-160
Toluene	15.5-24.5	4.0	11.2-27.7	46-148

Q = Concentration measured in QC check sample, in ug/L.

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

[a] Criteria are from 40 CFR Part 136 for Method 602 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 1.

TABLE 4. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION

Parameter	Accuracy, as recovery, x' (ug/L)	Single analyst precision, s_r' (ug/L)	Overall precision, S' (ug/L)
Benzene	0.92C+0.57	$0.09\bar{X}+0.59$	$0.21\bar{X}+0.56$
Chlorobenzene	0.95C+0.02	$0.09\bar{X}+0.23$	$0.17\bar{X}+0.10$
1,2-Dichlorobenzene	0.93C+0.52	$0.17\bar{X}-0.04$	$0.22\bar{X}+0.53$
1,3-Dichlorobenzene	0.96C-0.04	$0.15\bar{X}-0.10$	$0.19\bar{X}+0.09$
1,4-Dichlorobenzene	0.93C-0.09	$0.15\bar{X}+0.28$	$0.20\bar{X}+0.41$
Ethylbenzene	0.94C+0.31	$0.17\bar{X}+0.46$	$0.26\bar{X}+0.23$
Toluene	0.94C+0.65	$0.09\bar{X}+0.48$	$0.18\bar{X}-0.71$

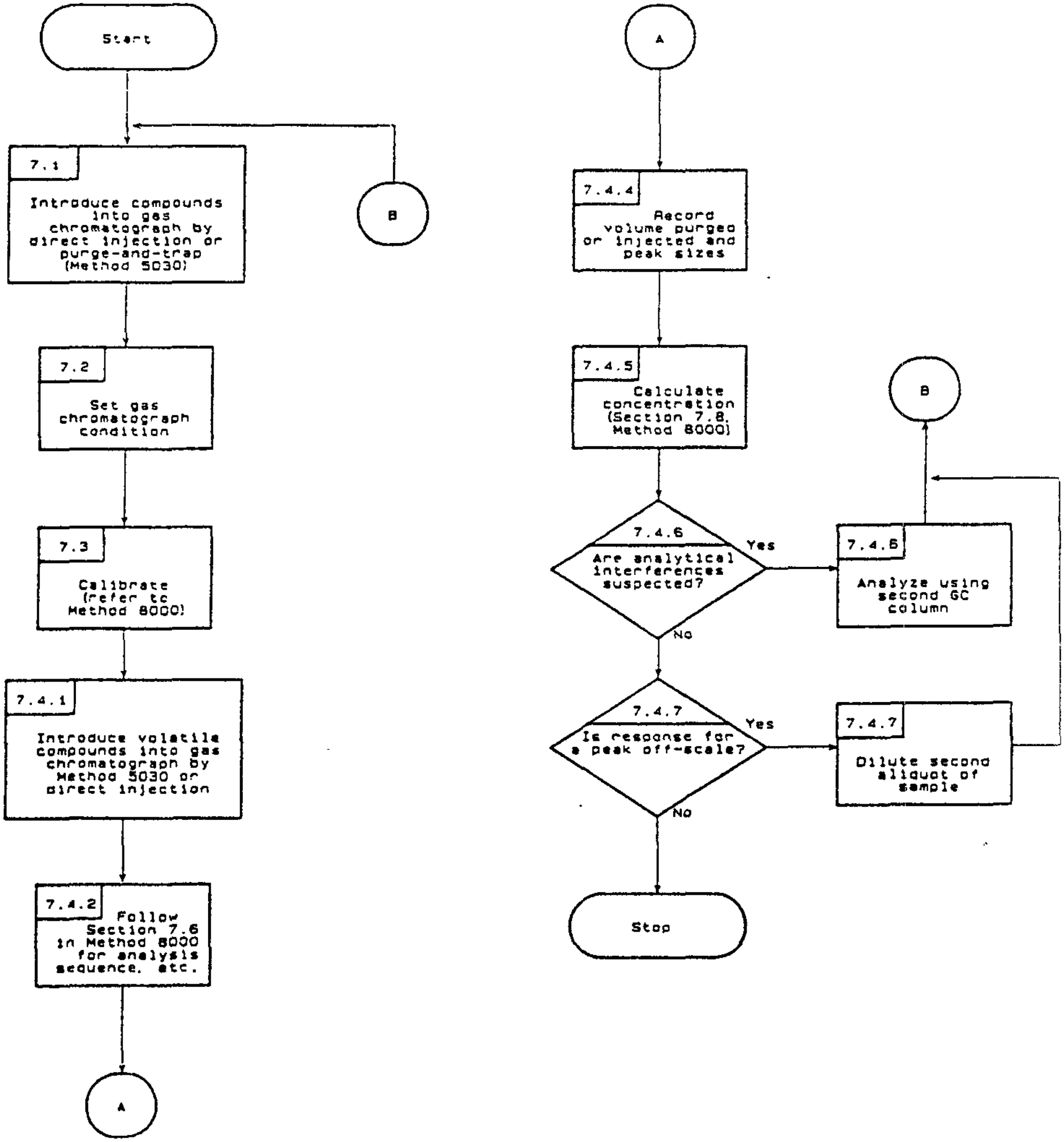
x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of $\bar{X}$, in ug/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in ug/L.

C = True value for the concentration, in ug/L.

$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in ug/L.


Start
7.1
Introduce compounds into gas chromatograph by direct injection or purge-and-trap (Method 5030)
B
7.2
Set gas chromatograph condition
7.3
Calibrate (refer to Method 8000)
7.4.1
Introduce volatile compounds into gas chromatograph by Method 5030 or direct injection
7.4.2
Follow Section 7.6 in Method 8000 for analysis sequence, etc.
A
A
7.4.4
Record volume purged or injected and peak sizes
7.4.5
Calculate concentration (Section 7.8, Method 8000)
7.4.6
Are analytical interferences suspected?
Yes
No
7.4.6
Analyze using second GC column
B
7.4.7
Is response for a peak off-scale?
Yes
No
7.4.7
Dilute second aliquot of sample
Stop

METHOD 5030

PURGE-AND-TRAP

1.0 SCOPE AND APPLICATION

1.1 This method describes sample preparation and extraction for the analysis of volatile organics by a purge-and-trap procedure. The gas chromatographic determinative steps are found in Methods 8010, 8015, 8021, and 8030. Although applicable to Method 8240, the purge-and-trap procedure is already incorporated into Method 8240.

1.2 Method 5030 can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a GC column packed with graphitized carbon lightly coated with a carbowax. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system.

1.4 This method also describes the preparation of water-miscible liquids, solids, wastes, and soil/sediments for analysis by the purge-and-trap procedure.

2.0 SUMMARY OF METHOD

2.1 The purge-and-trap process - An inert gas is bubbled through the solution at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a specially designed purging chamber. It is then analyzed by purge-and-trap GC following the normal water method.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic coating, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A field reagent blank prepared from reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-level and low-level samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination; therefore, frequent bake-out and purging of the entire system may be required.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents.

4.0 APPARATUS AND MATERIALS

4.1 Microsyringes - 10-uL, 25-uL, 100-uL, 250-uL, 500-uL, and 1,000 uL: These syringes should be equipped with a 20-gauge (0.006-in i.d.) needle having a length sufficient to extend from the sample inlet to within 1 cm of the glass frit in the purging device. The needle length will depend upon the dimensions of the purging device employed.

4.2 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.3 Syringe - 5-mL, gas-tight with shutoff valve.

4.4 Balance - Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g.

4.5 Glass scintillation vials - 20-mL, with Teflon lined screw-caps or glass culture tubes with Teflon lined screw-caps.

4.6 Volumetric flasks - 10-mL and 100-mL, class A with ground-glass stoppers.

4.7 Vials - 2-mL, for GC autosampler.

4.8 Spatula - Stainless steel.

4.9 Disposable pipets - Pasteur.

4.10 Purge-and-trap device - The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. Several complete devices are commercially available.

4.10.1 The recommended purging chamber is designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3-mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria. Alternate sample purge devices may be used, provided equivalent performance is demonstrated.

4.10.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap must contain the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet to extend the life of the trap (see Figures 2 and 3). If it is not necessary to analyze for dichlorodifluoromethane or other fluorocarbons of similar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

4.10.3 The desorber should be capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode. The desorber design illustrated in Figures 2 and 3 meet these criteria.

4.10.4 The purge-and-trap device may be assembled as a separate unit or may be coupled to a gas chromatograph, as shown in Figures 4 and 5.

4.10.5 Trap Packing Materials

4.10.5.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.10.5.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.10.5.3 Silica gel - 35/60 mesh, Davison, grade 15 or equivalent.

4.10.5.4 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26 lot #M-2649, by crushing through 26 mesh screen.

4.11 Heater or heated oil bath - Capable of maintaining the purging chamber to within 1°C over a temperature range from ambient to 100°C.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.

5.3 Methanol, CH_3OH. Pesticide quality or equivalent. Store away from other solvents.

5.4 Reagent Tetraglyme - Reagent tetraglyme is defined as tetraglyme in which interference is not observed at the method detection limit of compounds of interest.

5.4.1 Tetraglyme (tetraethylene glycol dimethylether, Aldrich #17, 240-5 or equivalent), $C_8H_{18}O_5$. Purify by treatment at reduced pressure in a rotary evaporator. The tetraglyme should have a peroxide content of less than 5 ppm as indicated by EM Quant Test Strips (available from Scientific Products Co., Catalog No. P1126-8 or equivalent).

<u>CAUTION</u>: Glycol ethers are suspected carcinogens. All solvent handling should be done in a hood while using proper protective equipment to minimize exposure to liquid and vapor.

Peroxides may be removed by passing the tetraglyme through a column of activated alumina. The tetraglyme is placed in a round bottom flask equipped with a standard taper joint, and the flask is affixed to a rotary evaporator. The flask is immersed in a water bath at 90-100°C and vacuum is maintained at < 10 mm Hg for at least two hours using a two-stage mechanical pump. The vacuum system is equipped with an all-glass trap, which is maintained in a dry ice/methanol bath. Cool the tetraglyme to ambient temperature and add 0.1 mg/mL of 2,6-di-tert-butyl-4-methyl-phenol to prevent peroxide formation. Store the tetraglyme in a tightly sealed screw-cap bottle in an area that is not contaminated by solvent vapors.

5.4.2 In order to demonstrate that all interfering volatiles have been removed from the tetraglyme, a water/tetraglyme blank must be analyzed.

5.5 Polyethylene glycol, $H(OCH_2CH_2)_nOH$. Free of interferences at the detection limit of the analytes.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to the introductory material to this Chapter, Organic Analytes, Step 4.1.

7.0 PROCEDURE

7.1 Initial calibration - Prior to using this introduction technique for any GC method, the system must be calibrated. General calibration procedures are discussed in Method 8000, Step 7.4, while the specific determinative methods and Method 3500 give details on preparation of standards.

7.1.1 Assemble a purge-and-trap device that meets the specification in Step 4.10. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, condition the trap daily for 10 minutes while backflushing at 180°C with the column at 220°C.

7.1.2 Connect the purge-and-trap device to a gas chromatograph.

7.1.3 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device. (Use freshly prepared stock solutions when preparing the calibration standards for the initial calibration.) Add 5.0 mL of water to the purging device. The water is added to the purging device using a 5-mL glass syringe fitted with a 15-cm 20-gauge needle. The needle is inserted through the sample inlet shown in Figure 1. The internal diameter of the 14-gauge needle that forms the sample inlet will permit insertion of the 20-gauge needle. Next, using a 10-uL or 25-uL microsyringe equipped with a long needle (Step 4.1), take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards. Add the aliquot of calibration solution directly to the reagent water in the purging device by inserting the needle through the sample inlet. When discharging the contents of the micro-syringe, be sure that the end of the syringe needle is well beneath the surface of the reagent water. Similarly, add 10 uL of the internal standard solution. Close the 2-way syringe valve at the sample inlet.

7.1.4 Carry out the purge-and-trap analysis procedure using the specific conditions given in Table 1.

7.1.5 Calculate response factors or calibration factors for each analyte of interest using the procedure described in Method 8000, Step 7.4.

7.1.6 The average RF must be calculated for each compound. A system performance check should be made before this calibration curve is used. If the purge-and-trap procedure is used with Method 8010, the following five compounds are checked for a minimum average response factor: chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-tetrachloroethane; and chlorobenzene. The minimum acceptable average RF for these compounds should be 0.300 (0.250 for bromoform). These compounds typically have RFs of 0.4-0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

1. Chloromethane - This compound is the most likely compound to be lost if the purge flow is too fast.

2. Bromoform - This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

3. Tetrachloroethane and 1,1-dichloroethane - These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2 On-going calibration - Refer to Method 8000, Steps 7.4.2.3 and 7.4.3.4 for details on continuing calibration.

7.3 Sample preparation

7.3.1 Water samples

7.3.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Two screening techniques that can be utilized are: the use of an automated headspace sampler (modified Method 3810), interfaced to a gas chromatograph (GC), equipped with a photo ionization detector (PID), in series with an electrolytic conductivity detector (ECD); and extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC with a FID and/or an ECD.

7.3.1.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.3.1.3 Assemble the purge-and-trap device. The operating conditions for the GC are given in Section 7.0 of the specific determinative method to be employed.

7.3.1.4 Daily GC calibration criteria must be met (Method 8000, Step 7.4) before analyzing samples.

7.3.1.5 Adjust the purge gas flow rate (nitrogen or helium) to that shown in Table 1, on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

7.3.1.6 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20-mL syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.3.1.7 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

7.3.1.7.1 Dilutions may be made in volumetric flasks (10-mL to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

7.3.1.7.2 Calculate the approximate volume of water to be added to the volumetric flask selected and add slightly less than this quantity of water to the flask.

7.3.1.7.3 Inject the proper aliquot of samples from the syringe prepared in Step 7.3.1.5 into the flask. Aliquots of less than 1-mL are not recommended. Dilute the sample to the mark with reagent water. Cap the flask, invert, and shake three times. Repeat the above procedure for additional dilutions.

7.3.1.7.4 Fill a 5-mL syringe with the diluted sample as in Step 7.3.1.5.

7.3.1.8 Add 10.0 uL of surrogate spiking solution (found in each determinative method, Section 5.0) and, if applicable, 10 uL of internal standard spiking solution through the valve bore of the syringe; then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. Matrix spiking solutions, if indicated, should be added (10 uL) to the sample at this time.

7.3.1.9 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

7.3.1.10 Close both valves and purge the sample for the time and at the temperature specified in Table 1.

7.3.1.11 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program and GC data acquisition. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180°C while backflushing the trap with inert gas between 20 and 60 mL/min for the time specified in Table 1.

7.3.1.12 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5-mL flushes of water (or methanol followed by water) to avoid carryover of pollutant compounds into subsequent analyses.

7.3.1.13 After desorbing the sample, recondition the trap by returning the purge-and-trap device to the purge mode. Wait 15 seconds; then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180°C for Methods 8010 and 8020, and 210°C for Methods 8015 and 8030. Trap temperatures up to 220°C may be employed; however, the higher temperature will shorten the useful life of the trap. After approximately 7 minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

7.3.1.14 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated response from a compound, this analysis must be followed by a blank water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.3.1.15 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to Method 8000 and the specific determinative method for details on calculating analyte response.

7.3.2 Water-miscible liquids

7.3.2.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with water.

7.3.2.2 Initial and serial dilutions can be prepared by pipetting 2 mL of the sample to a 100-mL volumetric flask and diluting to volume with water. Transfer immediately to a 5-mL gas-tight syringe.

7.3.2.3 Alternatively, prepare dilutions directly in a 5-mL syringe filled with water by adding at least 20 uL, but not more than 100-uL of liquid sample. The sample is ready for addition of surrogate and, if applicable, internal and matrix spiking standards.

7.3.3 Sediment/soil and waste samples - It is highly recommende that all samples of this type be screened prior to the purge-and-trap GC analysis. These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system, and require extensive cleanup and instrument downtime. See Step 7.3.1.1 for recommended screening techniques. Use the screening data to determine whether to use the low-level method (0.005-1 mg/kg) or the high-level method (> 1 mg/kg).

7.3.3.1 Low-level method - This is designed for samples containing individual purgeable compounds of < 1 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-level method is based on purging a heated sediment/soil sample mixed with water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples.

7.3.3.1.1 Use a 5-g sample if the expected concentration is < 0.1 mg/kg or a 1-g sample for expected concentrations between 0.1 and 1 mg/kg.

7.3.3.1.2 The GC system should be set up as in Section 7.0 of the specific determinative method. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature for Methods 8010 and 8020.

7.3.3.1.3 Remove the plunger from a 5-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 uL each of surrogate spiking solution and internal standard solution to the syringe through the valve. (Surrogate spiking solution and internal standard solution may be mixed together.) Matrix spiking solutions, if indicated, should be added (10 uL) to the sample at this time.

7.3.3.1.4 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Step 7.3.3.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

7.3.3.1.5 In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination. Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\% \text{ moisture} = \frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100$$

7.3.3.1.6 Add the spiked water to the purge device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system.

<u>NOTE</u>: Prior to the attachment of the purge device, Steps 7.3.3.1.4 and 7.3.3.1.6 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

7.3.3.1.7 Heat the sample to 40°C ± 1°C (Methods 8010 and 8020) or to 85°C ± 2°C (Methods 8015 and 8030) and purge the sample for the time shown in Table 1.

7.3.3.1.8 Proceed with the analysis as outlined in Steps 7.3.1.11-7.3.1.15. Use 5 mL of the same water as in the reagent blank. If saturated peaks occurred or would occur if a 1-g sample were analyzed, the high-level method must be followed.

7.3.3.2 High-level method - The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. Wastes (i.e. petroleum and coke wastes) that are insoluble in methanol are diluted with reagent tetraglyme or possibly polyethylene glycol (PEG). An aliquot of the extract is added to water containing surrogate and, if applicable, internal and matrix spiking standards. This is purged at the temperatures indicated in Table 1. All samples with an expected concentration of > 1.0 mg/kg should be analyzed by this method.

7.3.3.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and solid wastes that are insoluble in methanol, weigh 4 g (wet weight) of sample into a tared 20-mL vial. Use a top-loading balance. Note and record the actual weight to 0.1 gram and determine the percent moisture of the sample using the procedure in Step 7.3.3.1.5. For waste that is soluble in methanol, tetraglyme, or PEG, weigh 1 g (wet weight) into a tared scintillation vial or culture tube or a 10-mL volumetric flask. (If a vial or tube is used, it must be calibrated prior to use. Pipet 10.0 mL of solvent into the vial and mark the bottom of the meniscus. Discard this solvent.)

7.3.3.2.2 Quickly add 9.0 mL of appropriate solvent; then add 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 minutes.

<u>NOTE</u>: Steps 7.3.3.2.1 and 7.3.3.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.3.3.2.3 Pipet approximately 1 mL of the extract to a GC vial for storage, using a disposable pipet. The remainder may be disposed of. Transfer approximately 1 mL of appropriate solvent to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at 4°C in the dark, prior to analysis.

7.3.3.2.4 The GC system should be set up as in Section 7.0 of the specific determinative method. This should be done prior to the addition of the solvent extract to water.

7.3.3.2.5 Table 2 can be used to determine the volume of solvent extract to add to the 5 mL of water for analysis. If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-level analysis to determine the appropriate volume. If the sample was submitted as a high-level sample, start with 100 uL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.3.3.2.6 Remove the plunger from a 5.0-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10 uL of internal standard solution. Also add the volume of solvent extract determined in Step 7.3.3.2.5 and a volume of extraction or dissolution solvent to total 100 uL (excluding solvent in standards).

7.3.3.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/solvent sample into the purging chamber.

7.3.3.2.8 Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100 uL of solvent to simulate the sample conditions.

7.3.3.2.9 For a matrix spike in the high-level sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution and 1.0 mL of matrix spike solution.

Add a 100-uL aliquot of this extract to 5 mL of water for purging (as per Step 7.3.3.2.6).

7.4 Sample analysis

7.4.1 The samples prepared by this method may be analyzed by Methods 8010, 8015, 8020, 8021, and 8030. Refer to these methods for appropriate analysis conditions.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 3500 for sample preparation procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of a calibration blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a reagent blank should be processed as a safe-guard against chronic laboratory contamination. The blanks should be carried through all stages of the sample preparation and measurement.

8.3 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Spiked samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis. If the spiked samples do not indicate sufficient sensitivity to detect < 1 ug/g of the analytes in the sample, then the sensitivity of the instrument should be increased, or the sample should be subjected to additional cleanup.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

2. Gebhart, J.E.; Lucas, S.V.; Naber, S.J.; Berry, A.M.; Danison, T.H.; Burkholder, H.M. "Validation of SW-846 Methods 8010, 8015, and 8020"; U.S. Environmental Protection Agency. Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268, July 1987, Contract No. 68-03-1760.

3. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

4. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

TABLE 1.
PURGE-AND-TRAP OPERATING PARAMETERS

	Analysis Method				
	8010	8015	8020	8021	8030
Purge gas	Nitrogen or Helium	Nitrogen or Helium	Nitrogen or Helium	Nitrogen or Helium	Nitrogen or Helium
Purge gas flow rate (mL/min)	40	20	40	25-40	20
Purge time (min)	11.0 ± 0.1	15.0 ± 0.1	12.0 ± 0.1	11.0 ± 0.1	15.0 ± 0.1
Purge temperature (°C)	Ambient	85 ± 2	Ambient	Ambient	85 ± 2
Desorb temperature (°C)	180	180	180	180	180
Backflush inert gas flow (mL/min)	20-60	20-60	20-60	6	20-60
Desorb time (min)	4	1.5	4	4 ± 0.1	1.5

TABLE 2.

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH-LEVEL SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract[a]
500-10,000 ug/kg	100 uL
1,000-20,000 ug/kg	50 uL
5,000-100,000 ug/kg	10 uL
25,000-500,000 ug/kg	100 uL of 1/50 dilution [b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a volume of 100 uL added to the syringe.

[b] Dilute an aliquot of the methanol extract and then take 100 uL for analysis.

FIGURE 1.
PURGING CHAMBER

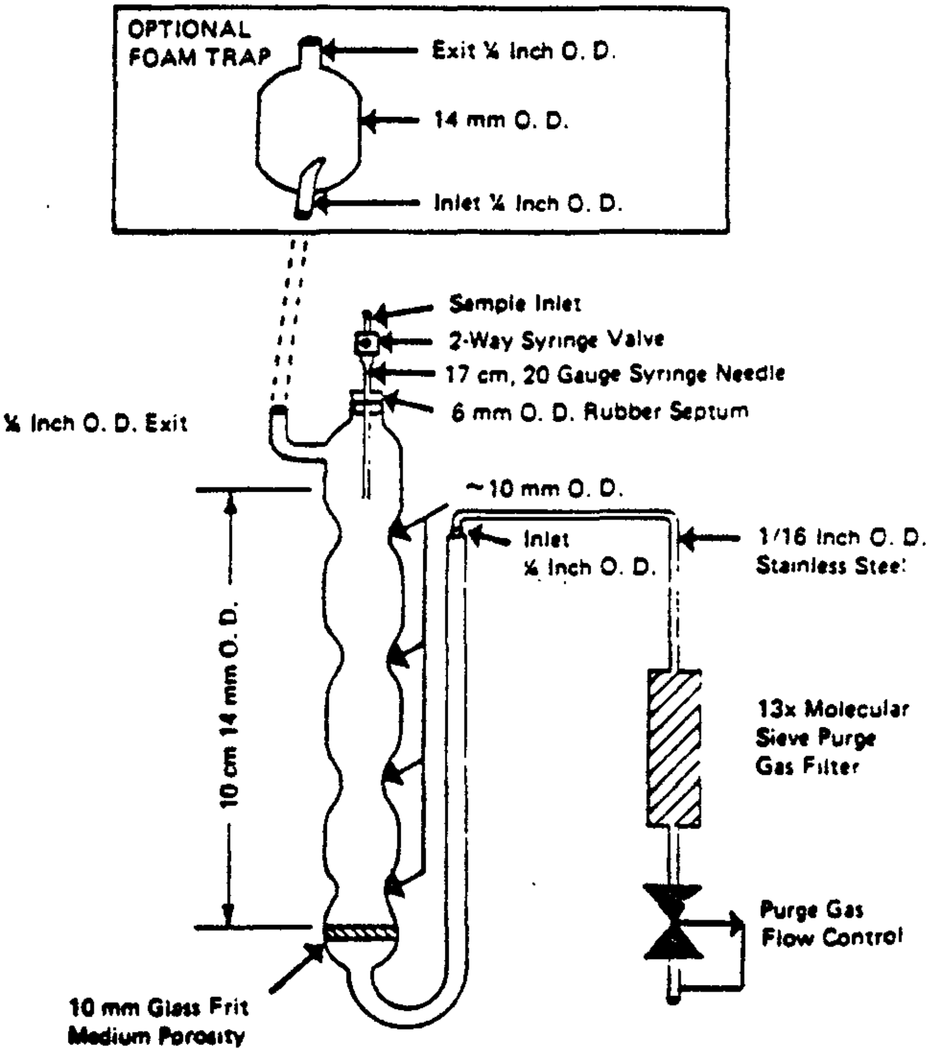


FIGURE 2.
TRAP PACKING MATERIALS AND CONSTRUCTION FOR METHOD 8010

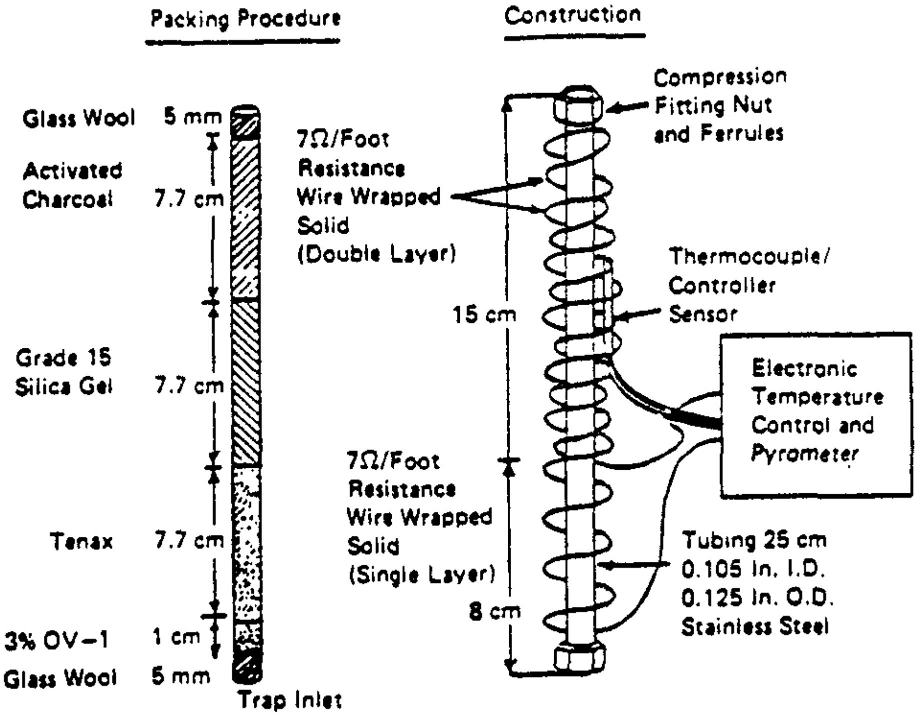


FIGURE 3.
TRAP PACKING MATERIALS AND CONSTRUCTION FOR METHODS 8020 AND 8030

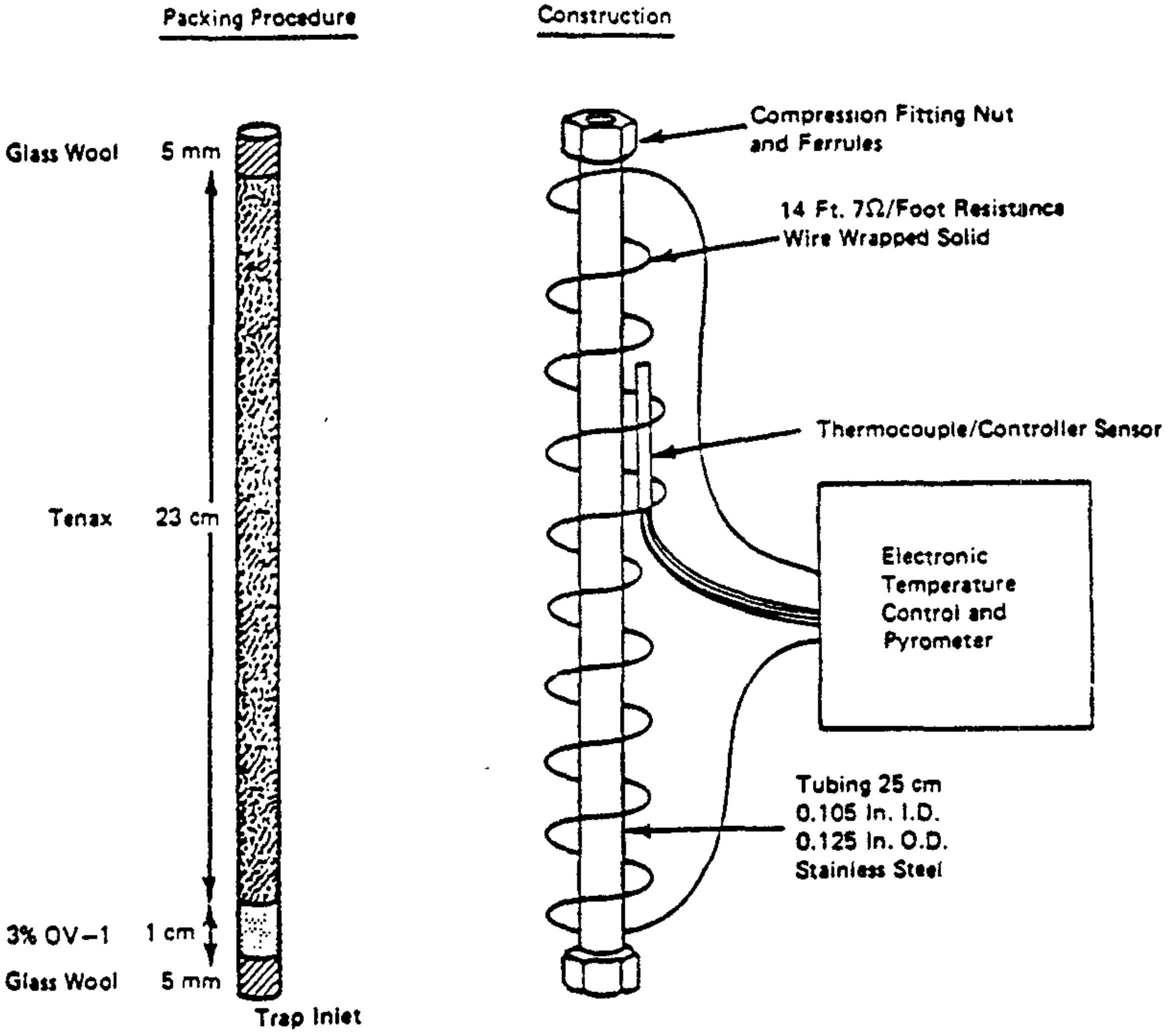
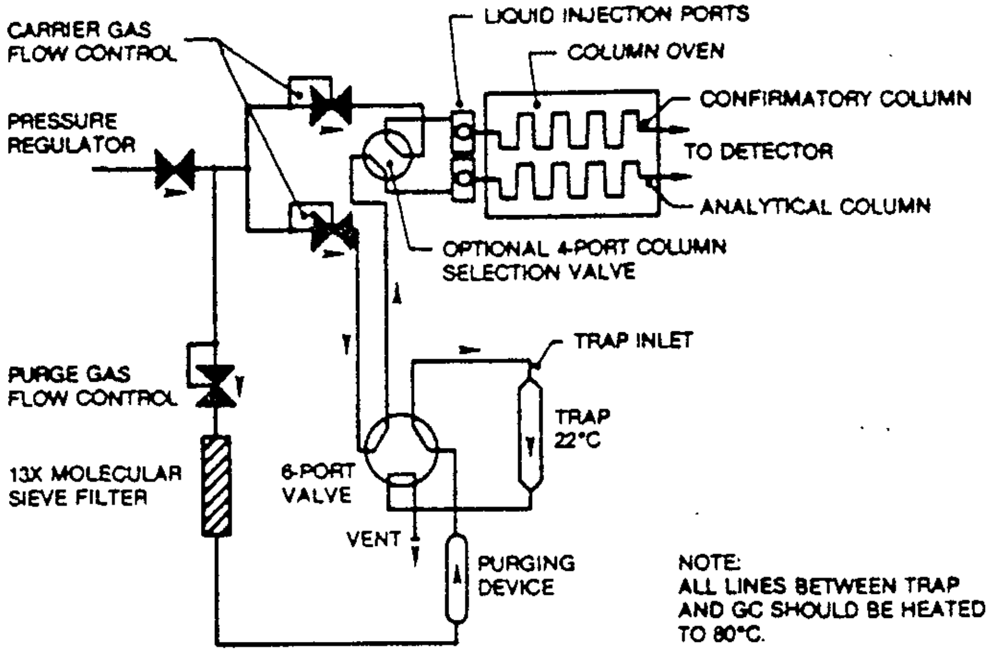
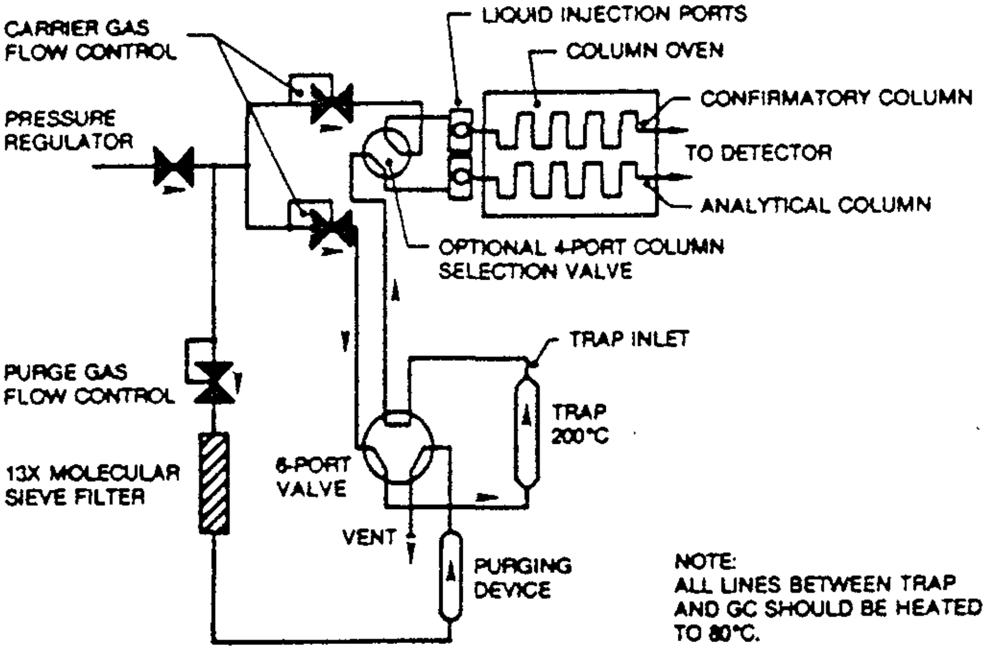


FIGURE 4.
PURGE-AND-TRAP SYSTEM, PURGE-SORB MODE,
FOR METHODS 8010, 8020, AND 8030

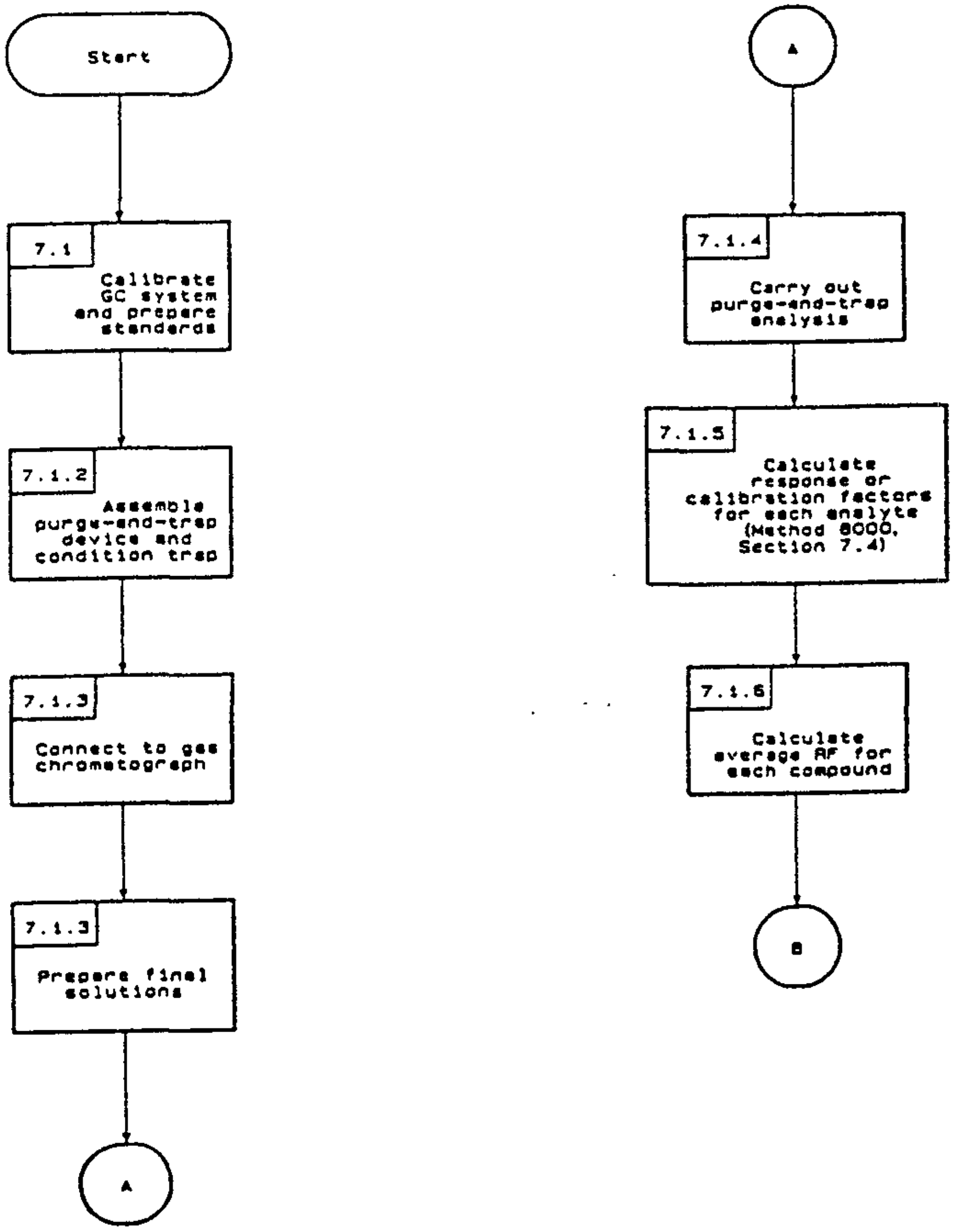


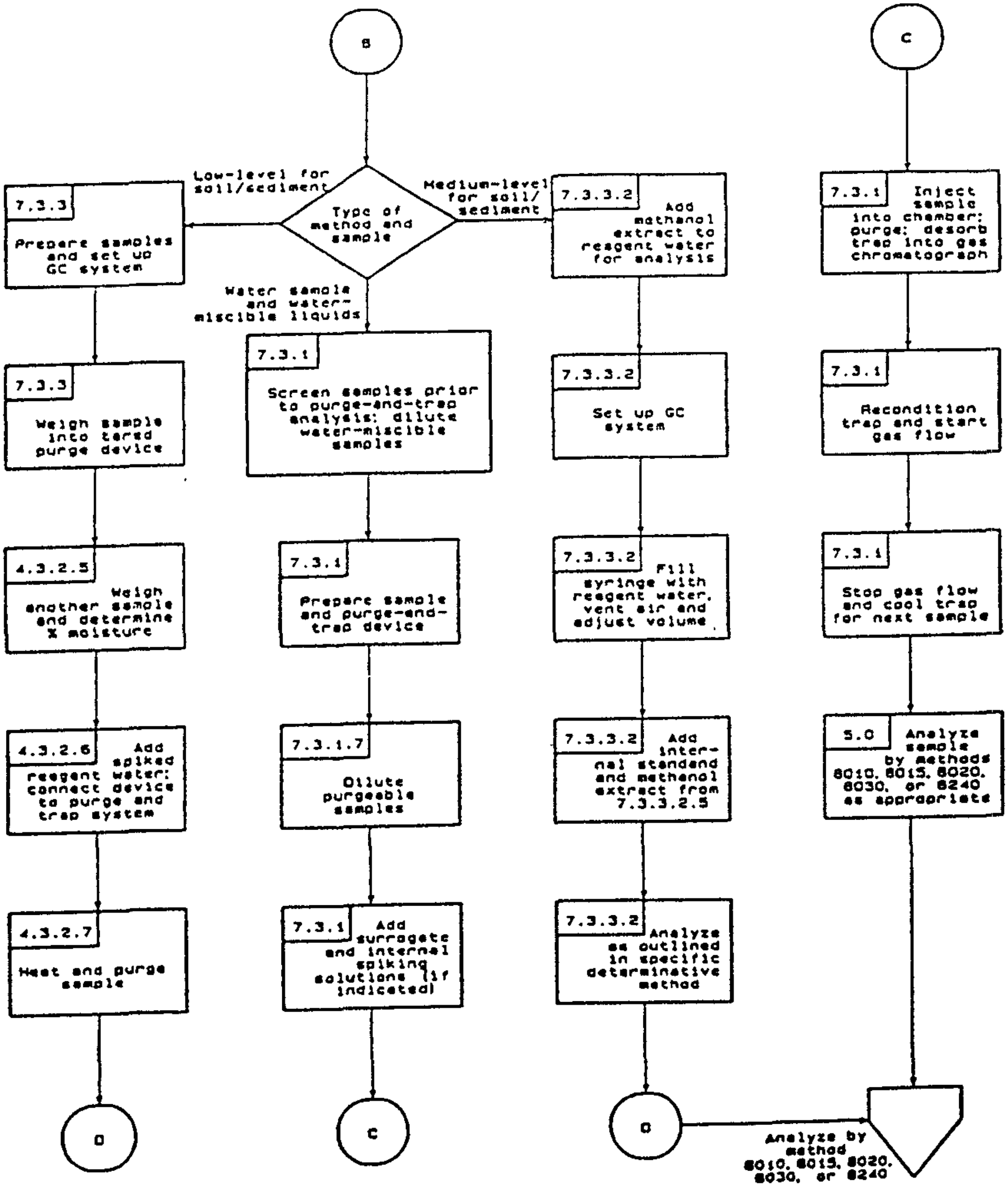
NOTE:
ALL LINES BETWEEN TRAP
AND GC SHOULD BE HEATED
TO 80°C.

FIGURE 5.
PURGE-AND-TRAP SYSTEM, DESORB MODE,
FOR METHODS 8010, 8020, AND 8030



METHOD 5030
PURGE-AND-TRAP




B
Low-level for soil/sediment
Type of method and sample
Medium-level for soil/sediment
Water sample and water-miscible liquids
7.3.3 Prepare samples and set up GC system
7.3.3 Weigh sample into tared purge device
4.3.2.5 Weigh another sample and determine % moisture
4.3.2.6 Add spiked reagent water; connect device to purge and trap system
4.3.2.7 Heat and purge sample
D
7.3.1 Screen samples prior to purge-and-trap analysis: dilute water-miscible samples
7.3.1 Prepare sample and purge-and-trap device
7.3.1.7 Dilute purgeable samples
7.3.1 Add surrogate and internal spiking solutions (if indicated)
C
7.3.3.2 Add methanol extract to reagent water for analysis
7.3.3.2 Set up GC system
7.3.3.2 Fill syringe with reagent water, vent air and adjust volume
7.3.3.2 Add internal standard and methanol extract from 7.3.3.2.5
7.3.3.2 Analyze as outlined in specific determinative method
D
Analyze by method 8010, 8015, 8020, 8030, or 8240
C
7.3.1 Inject sample into chamber; purge; desorb trap into gas chromatograph
7.3.1 Recondition trap and start gas flow
7.3.1 Stop gas flow and cool trap for next sample
5.0 Analyze sample by methods 8010, 8015, 8020, 8030, or 8240 as appropriate

HEALTH AND SAFETY PLAN

INTRODUCTION

- All persons potentially exposed to site-specific contaminants during excavation sampling, and/or remediation of any contaminated soil are subject to this health and safety plan.
- This health and safety program shall be available to employees at the excavation site, to their designated representatives, regulatory officials, and shall be provided to all subcontractors involved in the project. The program shall be designed to identify, evaluate, and control safety and health hazards, and provide for emergency response.
- All personnel shall be trained in the health and safety requirements of this health and safety plan in accordance with 29 CFR 1910.1200.
- All personnel required to wear respirators during work tasks shall be properly trained in the use of respirators, and shall have undergone qualitative fit testing.
- The site-specific plan is written to specify the objectives, job tasks, training, and personnel requirements of the site operations.

RESPONSIBILITIES OF KEY PROJECT MANAGER

The CEC Project Manager will ensure that all requirements of this health and safety plan are implemented and followed by all persons involved in the project.

SITE-SPECIFIC HEALTH AND SAFETY INFORMATION

A site-specific health and safety plan has been developed and is included in this section as Attachment 7-A. This plan will be available for all employees, their designated representatives, and regulatory personnel. This site safety and health plan includes:

1. Site History
2. Names of key health and safety personnel
3. Hazardous Assessment
4. A safety and health risk analysis for each phase of the operation
5. Project Organization

6. Employee training requirements

7. Work plan review

8. PPE requirements

9. Review of Site Safety Rules

10. Medical surveillance requirements

11. Emergency Response

12. Monitoring requirements

13. Site control measures

14. Decontamination procedures

15. Contingency plan for safe and effective emergency response

SITE CHARACTERIZATION AND ANALYSIS

1. Site Evaluations A preliminary site evaluation shall be performed prior to site entry by a trained person to aid in the selection of appropriate employee protection methods prior to site entry. During site entry, a more detailed evaluation of the site-specific hazards shall be performed to further aid in the selection of appropriate engineering controls and PPE requirements. All immediately dangerous to life or health (IDLH) conditions shall be identified during the site evaluation. Such conditions are not expected for this job.

2. Site Information The following information shall be obtained prior to allowing employees to enter the site:

    a. Location and approximate size of site

    b. Description of job tasks and duration of planned activity

    c. Site topography and accessibility by air and roads

    d. Pathways for hazardous substance dispersion

    e. Hazardous substances and health hazards involved or expected at the site and their chemical and physical properties.

3. Initial Site Entry: Personal Protective Equipment (PPE)
PPE shall be provided and used during initial site entry according to the following guidelines:

   a. Based upon the results of the preliminary site evaluation, the appropriate PPE shall be selected by CEC and used during initial site entry to provide protection to a level of exposure below the PEL for known or suspected hazardous substances and health hazards and will provide protection against other known and suspected hazards identified during the preliminary site evaluation.

   b. Once the hazards of the site have been positively identified, the appropriate PPE shall be selected by CEC and used.

Initial Site Entry: Monitoring

If there is a potential for IDLH conditions, air monitoring will be conducted using a combustible gas indicator, an oxygen deficiency meter, toxic substance indicators, etc. as well as noting visual observations for IDLH conditions.

Communication of Site Hazards

   1. Risks associated with the site-specific hazards shall be identified and communicated to employees.

   2. Relevant information concerning the physical, chemical, toxicologic properties of each substance known or expected to be present at the site shall be communicated to employees prior to the start of the job.

SITE CONTROL

A site control plan for preventing contamination of employees will be developed. This plan includes a site map, site work zones, standard operating procedures or safe work practices, and identification of nearest medical assistance.

1. Safe Work Practices

   a. At least one copy of this plan shall be available on-site.

   b. Contaminated protective equipment (respirators, boots, etc.) shall not be removed from the regulated area until it has been cleaned, or properly packaged and labeled.

c. Legible and understandable precautionary labels shall be affixed to containers of contaminated scrap, waste, and clothing.

d. Removal of contaminated soil from protective clothing or equipment by blowing, shaking, or any other means that disperses contaminants into the air is prohibited.

e. No food or beverages shall be present or consumed in the regulated area.

f. No tobacco products shall be present or used, and cosmetics shall not be applied in the regulated area.

g. Contaminated materials shall be stored in tightly closed containers.

h. Emergency equipment shall be located outside the storage areas in readily accessible locations.

i. Unauthorized visitors shall not be allowed into the regulated areas.

j. Visual contact shall be maintained among personnel at all times and workers must observe each other for signs of toxic exposure, including:

   - Changes in complexion and skin discoloration
   - Changes in coordination
   - Changes in demeanor
   - Excessive salivation and pupillary response
   - Changes in speech pattern

k. Employees shall inform each other of nonvisual effects of toxic exposure such as:

   - Headaches
   - Dizziness
   - Nausea
   - Blurred vision
   - Cramps
   - Irritation of eyes, skin, or respiratory tract

2. First Aid At least one person qualified to perform first aid will be present on site at all times during work activity. This person will have earned a certificate in first-aid training from the American Red Cross or will have received equivalent training.

3. Transportation To Emergency Treatment A vehicle will be available at all times for use in transporting personnel to the hospital. Hospital routes shall be discussed prior to on site activity.

4. Contingency Planning Prior to commencement of on site activities, field personnel will review safety considerations with the Site Safety Officer. The Site Safety Officer is responsible for adherence to the designated safety precautions and assumes the role of CERTIFIED's on-site coordinator with the client in an emergency response situation.

POTENTIAL HAZARDS

The potential hazards associated with hazardous waste site investigation include 1) accidents; 2) contact, inhalation or ingestion of hazardous materials; 3) explosion; and, 4) fire.

1. Accidents Accidents must be handled on a case by case basis. Minor cuts, bruises, muscle pulls, etc., will still allow the injured person to undergo reasonable normal decontamination procedures prior to receiving direct first aid. More serious injuries may not permit complete decontamination procedures to be undertaken, particularly if the nature of the injury is such that the victim should not be moved. The nature and degree of surface contamination at a site is generally low enough that emergency vehicles could reach the victim on-site without undue hazard. However, in the event that access on-site is limited, accident victims may be transported by CERTIFIED personnel trained for this response.

2. Contact and/or Ingestion of Hazardous Materials Properly prescribed and maintained protective clothing and adherence to established safety procedures are designed to minimize these hazards. However, it is still a possibility that contact or ingestion of materials may occur. One possibility for contamination is the puncture of a buried drum of liquid during drilling operations which might cause the random distribution of the drum contents. Standard first aid procedures should be followed. The drilling rig will have a tank of water which may be useful in some circumstances, particularly to flush off any exposed skin areas. Eye wash bottles will also be maintained at the site in case of emergencies. In cases of ingestion or other than minor contact with known substances, the Poison Control Center and local hospital should be contacted and the victim brought there immediately for further treatment and observation.

3. Explosion The drilling crew should be keenly aware of combustible gas meter readings and withdraw at an indication of imminently hazardous conditions. The detection of such conditions shall be reported to local agencies for potential execution of the evacuation plan should the situation be assessed as warranting such response.

4. Fire The combustible gas meter will also warn of imminent fire hazards at borings. The greatest fire hazard at the site should be recognized as handling the methanol used for decontamination. No smoking or open flames are allowed in this area. Carbon dioxide fire extinguishers will be kept at the drilling rig, and the decontamination area/field office. The Fire Department, previously informed of site activities, will be called as needed.

5. Rig Safety CERTIFIED has incorporated the National Drilling Federation's (NDF/DCDMA/NDCA) "Drilling Safety Guide" as our mechanical hazards and rig safety guide. This booklet is required reading for all field personnel and is included as Appendix A.

6. Heat Stress Heat stress may be a concern depending on the ambient temperature and the type of PPE used. Measures to help control heat stress include:

   a. Adequate liquids to replace lost body fluids must be provided.

   b. A work regimen providing adequate rest periods for cooling down.

   c. Cooling vests may be worn beneath protective garments.

   d. All breaks are to be taken in a shaded rest area.

   e. Employees shall remove impermeable protective clothing during rest periods.

   f. Employees shall not be assigned other tasks during rest periods.

   Signs and symptoms of heat stress:

   a. Heat rash due to heat or humid air.

   b. Heat cramps caused by excessive sweating; symptoms include muscle spasms, and pain in the hands, feet, and abdomen.

   c. Heat exhaustion caused by increased stress on various body organs due to cardiovascular insufficiency or dehydration; symptoms include pale, cool, moist skin, heavy sweating, dizziness, nausea, fainting.

d. Heat stroke is the most serious because temperature regulation fails and the body temperature can rise to critical levels. Immediate action must be taken to cool the body before serious injury occurs; symptoms include red, hot dry skin, lack of or reduced perspiration, nausea, dizziness and confusion, strong, rapid pulse, coma.

EVACUATION RESPONSE LEVELS

Evacuation responses will occur at three levels: (1) withdraw from immediate work area (100+ feet upwind); (2) site evacuation; (3) evacuation of surrounding area. Anticipated conditions which might require these responses are described below:

1. Withdrawal Up-Wind (100 or more feet)

    a. Sensing ambient air conditions as containing greater contaminant concentrations than guidelines allow for the type of respiratory protection being worn. The work party may return upon donning greater respiratory protection and/or assessing the situation as transient or past.

    b. Breach in protective clothing or minor accident. The party may return when tear or other malfunction is repaired and first aid or decontamination has been administered.

    c. Respirator malfunctions and must be replaced.

2. Site Evacuation

    a. Sensing ambient air conditions as containing explosive and persistent levels of combustible gas or excessive levels of toxic gases, i.e., in excess of 100 ppm or 10% of LEL.

    b. Fire or major accident

    c. Imminent explosion or explosion

3. Surrounding Area Evacuation Persistent, unsuppressible release of toxic or explosive vapors from test pits and borings, (e.g., possible pressure release of punctured underground storage tank or drums). Air quality should be monitored at several distances downwind to assess danger to surrounding area before initiating this response.

EVACUATION PROCESSES

1. Withdrawal Upwind The field crew will note general wind directions while on-site. (A simple wind sock may be set up near the drilling site for visual determinations). Upon noting the conditions warranting movement away from the test pit or bore hole, the crew will move upwind a distance of approximately 100 feet or further as indicated by the PI meter. Donning SCBA and a safety harness and line, the Site Safety Officer or a member of the crew may return to the test pit or bore hole to determine if the condition noted was transient or persistent. If persistent, then an alarm should be raised to notify on-site personnel of the situation and the need to leave the site or don SCBA. An attempt should be made to plug the source to decrease emissions only if greater respiratory protection is donned. The Site Safety Officer and client will be notified of hazardous conditions. Considering access to the site, if this situation occurs the crew may be instructed to evacuate the site rather than move upwind.

2. Site Evacuation Upon determination of conditions warranting site evacuation, the work party will proceed upwind of the bore hole and notify the security force, Site Safety Officer and the field office of site conditions. If the decontamination area is upwind and greater than 500 feet from the bore hole, the crew will pass quickly through decontamination to remove contaminated outer suits. If the hazard is toxic gas, respirators will be retained. The crew will proceed to the field office to assess the situation. There the respirators may be removed (if the PI meter indicates an acceptable condition). As more facts are determined from the field crew, these will be relayed to the appropriate agencies.

3. Evacuation of Surrounding Area When the Site Manager determines that conditions warrant evacuation of downwind residences and commercial operations, the local agencies will be notified and assistance requested. Designated on site personnel will initiate evacuation of the immediate off-site area without delay.

HAZARDOUS WASTE SITE TRAINING

CERTIFIED employees must complete 40-hours of health and safety training prior to commencing work at sites where hazardous materials may be present. Each employee will be provided with 8-hours of followup training on an annual basis. The 40-hour and 8-hour

Hazardous Waste Site Training Programs provided by CERTIFIED meet or exceed Federal OSHA requirements as presented in Federal Register Volume 51, Number 244 dated December 19, 1987. The training programs include instruction in the following topics:

a. Hazard Assessment

b. Levels of Protection

c. Limits and Use of Air Purifying Respirators

d. Federal OSHA Regulations

e. Toxicology

d. Emergency Response and Spill Contingencies

e. Site Control

f. Decontamination

g. Biological Monitoring Program

h. Air Monitoring Instrumentation

i. Review of General Chemical & Mechanical Dangers

j. Documentation and Record Keeping

In addition to instruction in the above, each trainee will receive certification for the American Red Cross in First Aid and Cardiopulmonary Resuscitation (CPR). The First Aid certification is repeated at 3-year intervals and each employee is certified in CPR on any annual basis.

MEDICAL SURVEILLANCE Shall be in accordance with 29 CFR 1910.1200

A medical surveillance program shall be developed for:

1. All employees who are or may be exposed to hazardous substances at or above the PEL, without regard to respirators, for 30 days or more per year.

2. All employees who wear a respirator for 30 days or more per year.

3. All employees who may have been exposed in an emergency situation to hazardous substances at concentrations above the PEL shall be allowed to have a medical examination or consultation.

   The frequency of these employer-financed medical examinations shall be:

4. Prior to assignment and annually thereafter.

5. Upon termination of employment or reassignment.

6. As soon as possible, upon notification by an employee that he has developed signs or symptoms indicating possible overexposure to hazardous substances or health hazards.

7. More frequently if recommended by a physician.

Records and the content of medical examinations shall be in accordance with 29 CFR 1910.

1. Health Monitoring Program All personnel engaged in field activities must participate in a Health Monitoring Program. This program consists of an initial medical examination to establish the employee's general health profile and provides important baseline laboratory data for comparative study. The scope of the initial comprehensive physical examination and laboratory testing routine is detailed in Table 1. Follow-up examinations are completed for all personnel enrolled in the health monitoring program on an annual basis, or more frequently if project assignments warrant testing following specific field activities.

2. Biological Monitoring Program In addition to a physical examination and routine laboratory work, Biological Monitoring Program is required. This program is designed to measure the concentration of specific toxic chemicals and their metabolites in the body. Concentrations of halogenated volatiles and aromatic and halogenated solvents present in the blood are determined as well as levels of halogenated and aromatic metabolites in the urine. The specific parameters of the Biological Monitoring Program are shown in Table 2.

TABLE 1
MEDICAL EXAMINATION

Physical Examination

- Medical history survey
- Medical examination
- Vision: near and distance vision, color vision
- Hearing: audiometry
- Respiratory: spirometry

Lab Studies

- Hematology
  - Complete blood count
  - Hemoglobin
  - Hematocrit
  - Sedimentation rate
  - Red blood cell morphology

- Urinalysis

- Blood Chemistry

SMAC 25:

- Uric Acid
- Iron, Total
- Protein, Total
- Albumin
- Globulin
- A/G Ratio
- Cholesterol
- Triglycerides
- Total Bilirubin
- Alkaline Phosphatase
- Lead concentration
- Glucose
- Carbon Dioxide
- BUN
- Electrolytes
- Creatinine
- Sodium
- Potassium
- Carbon Dioxide
- Calcium
- Phosphorous

TABLE 2
BIOLOGICAL MONITORING PROGRAM

PARAMETER

- Ketone Solvents
  - Methyl Ethyl Ketone
  - Methyl Isobutyl Ketone
  - Methyl n-Butyl Ketone

- Aromatic Solvents
  - Benzene
  - Toluene
  - Ethylbenzene
  - Xylenes
  - Styrene

- Halogenated Volatiles
  - Carbon Tetrachloride
  - Chloroform
  - Dibromochloropropane
  - o-Dichlorobenzene
  - p-Dichlorobenzene
  - Dichloropropene
  - Ethylene Dibromide
  - Ethylene Dichloride
  - Methylene Chloride
  - Perchloroethylene
  - Trichloroethylene
  - 1,1,1 Trichloroethane

- Petroleum Distillates
  - Kerosene
  - Mineral Spirits
  - Hexane
  - Cyclohexane

- Aromatic Metabolites
  - Hippuric Acid
  - Mandelic Acid
  - Phenylglyoxylic Acid
  - Meth-Hippuric Acid
  - Phenol
  - o-Cresol

- Halogenated Metabolites
  - Trichloroethanol
  - Trichloroacetic Acid

REVIEW OF GENERAL CHEMICAL & MECHANICAL DANGERS

A set of standard on-site safety practices will be enforced during site activities to reduce the risks associated with handling contaminated materials and dangers inherent with working near heavy machinery. These safety practices are divided into three categories: personal precautions, rig safety, and general procedures and operations.

PERSONAL PRECAUTIONS

Any practice which increases the probability of hand-to-mouth transfer and ingestion of contaminated material will be prohibited in any area designated contaminated. Prohibited activities include eating, drinking, chewing gum or tobacco, and smoking. Hands and face will be thoroughly washed upon leaving the work area and before eating, drinking or any other activities.

Any excess facial hair which interferes with proper fit of the mask-to-face seal will be prohibited on personnel required to wear respirator protection. (While respirators are not typically required, work will be staged to upgrade to Level "C" protection requiring the use of respirators, if needed).

Unnecessary contact with contaminated or suspected contaminated surfaces will be avoided. Workers will be instructed to avoid walking through puddles, mud, or other discolored surfaces; kneeling on the ground; and leaning, sitting, or placing equipment on drums, containers, vehicles or the ground.

Medicine and alcohol can increase adverse effects from exposure to toxic chemicals. Therefore, prescribed medication will not be taken by personnel during field activities unless authorized by a physician. Also, alcoholic beverage intake will not be tolerated immediately before or during field work.

The effects of heat stress in all personnel will be monitored by the Health and Safety Officer. Appropriate measures will be taken to remove any potential victim of heat stress from the work area, provide cooling to the body and provide plenty of liquids to replace body fluids. Personal protective equipment, including the use of cool vests, will be made available, and work/rest regimes will be established as needed. Use of personal protective equipment includes hard hats, gloves, boots, and coveralls is site specific and will be directed by site project manager and safety officer.

GENERAL PROCEDURES AND OPERATIONS

Entrance and exit to the site will be planned and emergency escape routes will be determined. Before drilling begins a working phone will be located and the most expeditious route to a hospital established. Site-specific hazards will be discussed and the clients safety requirements will be adopted in addition to CERTIFIED policies. Personnel will practice any unfamiliar procedures prior to performing them in the field. The number of personnel and pieces of equipment in the work area will be minimized to the extent that it compromises the effectiveness of site operations. Procedures for leaving a contaminated work area will be established prior to going on site. Work areas and decontamination procedures will be established based on-site conditions.

1. Levels of Protection The level of personnel protective equipment required shall be determined by the type and levels of waste or spill material present at the site where project personnel may be exposed. In situations where the types of waste or spill material on-site are unknown or the hazards are not clearly established or the situation changes during on site activities, the Site Safety Officer must make a reasonable determination of the level of protection that will assure the safety of field personnel until the potential hazards have been determined precisely through monitoring, sampling, informational assessment, or other reliable methods. Once the hazards have been determined, protective levels commensurate with the hazards shall be employed. Protection levels will be continuously evaluated to reflect any new information acquired.

   The levels of protection utilized by CERTIFIED are presented below:

   Level A - Level A protection must be selected when the Site Safety Officer makes a reasonable determination that the highest available level of both respiratory and skin and eye contact protection is needed. It should be noted that while Level A provides maximum available protection, it does not protect against all possible hazards. Consideration of the heat stress that can arise from wearing Level A protection should also enter into the sub task leaders decision. (Comfort is not a decision factor, but heat stress will influence work rate, scheduling, and other work practices.)

   Level B - The Site Safety Officer must select Level B protection when the highest level of respiratory protection is needed, but hazardous material exposure to the few unprotected areas of the body (i.e., the back of the neck) or permeation/breakthrough of chemicals through protective clothing and gloves is unlikely.

   Level C - The Site Safety Officer may select Level C when the required level of respiratory protection is known, or reasonably assumed to be, not greater than the level of protection afforded by full face air purifying respirators; and hazardous materials exposure to the few unprotected areas of the body (i.e., the back of the neck) is unlikely. Level C may require carrying an emergency escape respirator. (A half mask respirator is acceptable when airborne contaminate level are lower than TWA action levels.

Level D - Level D is the basic work uniform. Investigators and response personnel must not be permitted to work in civilian clothes. An emergency escape respirator may be required.

2. Fit testing of safety equipment will be an important part of establishing adequate respiratory and dermal protection. Fit testing will be accomplished prior to site explorations and each individual will be assigned a fitted respirator for the duration of the project. These will be tagged for identification.

   It should be recognized that most situations require a different combination of respiratory and dermal protective gear, e.g., where no splash protection is required but a high respiratory hazard is present. The Site Safety Officer may elect a modification of the above.

ENGINEERING CONTROLS AND PERSONAL PROTECTIVE EQUIPMENT

Engineering controls shall be instituted to reduce and maintain employee exposure within permissible exposure limits whenever feasible. This will be accomplished by the use of dust suppression techniques, air dilution, or other work practice controls, coupled with protective clothing that is appropriate for the level of exposure. Whenever engineering controls are not feasible, PPE shall be used.

Personal Protective Equipment shall be selected according to site characterization and analysis information, job tasks, site hazards, intended use, and duration of potential employee exposures.

When respirator protection is required the following should be instituted:

1. Respiratory Protection

   a. Only properly cleaned and maintained NIOSH/MSHA approved respirators shall be used on-site.

   b. Respirator selection, as well as decisions regarding upgrading and downgrading, shall be made by CEC.

   c. Used air purifying cartridges or canisters shall be replaced at the end of each shift or when load up (an increase in breathing resistance) or breakthrough occurs.

d. Only employees who are qualified to wear respirators and have had qualitative fit tests, and semiannual fit tests thereafter, shall be allowed to work in atmospheres where respirators are required.

e. Contact lenses shall not be worn on-site.

f. Excessive facial hear (beards) prohibits proper face fit and effectiveness of respirators. Persons required to wear full-face or half-face respirators must not have beards, wide mustaches, goatees, or extended sideburns. All personnel wearing full-face or half-face respirators shall be required to be clean shaven prior to each day's shift.

2. <u>Exposure Records</u> (See attached monitoring forms)

The records will include at least the following information:

a. Date of measurement.

b. Description of operation being monitored.

c. Sampling and analytical methods, evidence of accuracy.

d. Number, duration, and results of samples collected.

e. Type of protective devices worn.

f. Name, social security number, and exposure levels of employee's represented.

If the exposure level was below the action level and no sampling was done, evidence to prove the lack of exposure will be recorded.

Monitoring equipment will be protected as much as possible from contamination by draping, masking or otherwise covering as much of the instruments as possible with plastic without hindering the operation of the unit. The OVA meter, for example, can be placed in a clear plastic bag which allows reading of the scale and operation of the knobs. The OVA sensor can be partially wrapped, keeping the sensor tip and discharge port clear.

The contaminated equipment will be taken from the drop area and the protective coverings removed and disposed of in the appropriate containers. Any dirt or obvious contamination will be brushed or wiped with a disposable paper wipe and the used wipers discarded. The units will then be taken inside in a clean plastic tub, wiped off with damp disposable wipes and dried. The units will be checked, standardized and recharged as necessary for the next day's operation. They will then be covered with new protective coverings.

3. Respirators

a. Respirator Fit Tests (See attached fit test record)

A summary of all test results for respirator fit tests will be maintained for 3 years. The record will include:

- Name of the test subject.
- Date of testing.
- Name of test conductor.
- Respirator selection, manufacturer, model, size, NIOSH approval number.
- Testing protocol.

Fit testing is to be repeated at least every 6 months at a minimum.

QUALITATIVE FIT TEST PROTOCOL

1. Fitting will be done using Irritant Smoke.

2. The employee will put on the test respirator, secure the straps and perform a positive and negative pressure check for leaks.

3. If there are no leaks he will step under the fit test tent and a cloud of smoke will be blown around the mask using stannic chloride tubes, after the test subject has been instructed to close his eyes.

4. The tester will then be asked to:

   a. Breathe deeply.

   b. Turn their head side to side, and up and down while inhaling deep at the end of each directional movement.

   c. Run in place.

   d. Breathe normally.

5. The employee will then be asked to read the rainbow passage (see below) while the edges of the mask are being heavily smoked. (Note if a half mask respirator is being tested chemical goggles will be worn).

   <u>RAINBOW PASSAGE:</u>

   "When the sunlight strikes raindrops in the air, they act like a prism and form a rainbow. The rainbow is a division of white light into many beautiful colors. These take the shape of a long round arch, with its path high above, and its two ends apparently beyond the horizon. There is, according to legend, a boiling pot of gold at one end. People look, but no one ever finds it. When a man looks for something beyond his reach, his friends say he is looking for the pot of gold at the end of the rainbow."

6. After testing the mask will be removed before existing the tent to ensure the employees sensitivity to the smoke.

7. Testing will not be done if there is any facial hair (even one days growth in the masks sealing surfaces).

8. Repeat test will be scheduled once every 6 months or :

- if a loss of 20 or more pounds is realized
- any significant scaring beneath the masks sealing surfaces
- cosmetic surgery
- or any other condition that would affect the masks ability to seal

Respirators will be decontaminated daily. Taken from the drop area, the masks will be disassembled, the cartridges set aside and the rest placed in a cleansing solution. (Parts will be precoded, e.g., #1 on all parts of mask #1). After an appropriate time within the solution, the parts will be removed and rinsed off with tap water. The old cartridges will be marked to indicate length of usage and will be discarded into the contaminated trash container for disposal when considered spent. In the morning the masks will be re-assembled and new cartridges installed if appropriate. Personnel will inspect their own masks to be sure of proper readjustment of straps for proper fit.

1. Employers can change HEPA filters whenever the breathing resistance increases. Additional cartridges will be available in the clean room at any of the asbestos removal sites.

2. Employees who are feeling some irritation around their respirator may notify their supervisor so that they can exit the site to wash their face and respirators.

MONITORING

Air monitoring shall be used to identify and quantify airborne levels of hazardous substances in order to determine the appropriate level of employee protection required on-site.

The type and frequency of air and personal monitoring shall be designated by CEC based on the hazards identified at the site and the job tasks. CEC will be responsible for maintaining records of all monitoring.

As a minimum, the need for monitoring shall be evaluated when:

- Work begins on a different section of the site.
- A different type of operation is initiated.
- Contaminants other than those previously identified are being handled.
- Employees are handling leaking drums or containers.
- High risk employees (those closest to the source of containment generation) shall receive personal monitoring to characterize their exposure.

HANDLING DRUMS AND CONTAINERS

Drums and containers used during clean-up shall meet the appropriate DOT, OSHA, and EPA regulations for the wastes they contain.

DECONTAMINATION

A decontamination procedure shall be developed, communicated to employees, and implemented before any employees or equipment may enter contaminated areas. Decontamination shall be performed in areas that will minimized the exposure of uncontaminated employees/equipment to contaminated employees/equipment.

All equipment and solvents used for decontamination shall be collected and decontaminated or disposed of properly.

All employees leaving a contaminated area shall be properly decontaminated; all clothing and equipment leaving a contaminated area shall be properly disposed of or decontaminated.

CEC shall monitor the site decontamination procedures to establish their effectiveness.

1. Personal Protective Equipment Decontamination

   a. PPE (clothing and equipment) shall be decontaminated, cleaned, laundered, maintained, or replaced as needed to maintain their effectiveness.

   b. All reusable PPE shall be decontaminated and reconditioned in a designed area within the contamination reduction zone.

   c. If protective clothing is removed from the contamination reduction zone for cleaning or laundering, it shall be bagged and appropriately labeled.

   d. All disposable protective clothing shall be bagged and labeled to identify potential contaminants. These bags shall be treated as hazardous waste.

PERSONNEL DECONTAMINATION PROCEDURE

A decontamination procedure will be carried out by all personnel leaving hazardous waste sites. Under no circumstances (except emergency evacuation) will personnel be allowed to leave the site prior to decontamination. CEC will monitor the site decontamination procedure to evaluate their effectiveness. Procedures for removal of protective clothing are as follows:

- Drop tools, monitors, samples and trash at designated drop stations. These will be plastic containers or drop sheets.
- Step into designated shuffle pit area and scuff feet to remove gross amounts of dirt from outer boots. If necessary, wash boots down with clear water in designated wash pit area.
- Remove tape from boots and remove boots. Discard in drum container.
- Remove outer gloves and place in container.
- Remove hard hat and respirator and hang in the designated area.
- Remove coveralls and discard in container.
- Remove inner gloves and discard in container.
- If the site required utilization of a decontamination trailer, all personnel would also shower before leaving the site at the end of the work day.

Note: Disposable items (coverall, inner gloves, and over boots) will be changed on a daily basis unless there is reason for changing sooner. Dual respirator canisters will be changed twice weekly unless more frequent changes are deemed appropriate by site surveillance data or personnel assessment.

A water hose and/or designated wash area will be available for wash down and cleaning purposes.

Equipment Decontamination Equipment to be decontaminated during the project may include: (1) drilling rig and tools; (2) sample containers; (3) monitoring equipment; and (4) respirators.

All decontamination will be done by personnel in protective gear appropriate for the level of decontamination, determined by the Site Safety Officer. The decontamination work tasks will be split or rotated among support and work crews. Decontamination procedures within the trailer (if used) should take place only after other personnel have cleared the "hot area", moved to the clean area and the door between the two areas closed.

Miscellaneous tools and samplers will be dropped into a plastic pail, tub or other container. They will be brushed off and rinsed (outside, if possible) and transferred into a second pail to be carried to further decontamination stations. They will be washed with a trisodium phosphate or detergent solution, rinsed with acetone or methanol, rinsed with a trisodium phosphate or detergent solution and finally rinsed with clean water.

SITE SPECIFIC
HEALTH AND SAFETY PLAN
FOR
BERKELEY FARM'S TELEGRAPH AVENUE PROJECT

I. Site: Berkeley Farms Lot, 23555 Saklan Avenue, Hayward, California.

II. Key Personnel and Project Assignments

PROJECT ASSIGNMENT	NAME/AGENCY	PHONE
Principal Investigator	Marcus Bass/ CEC	(707) 745-0171
Geologist	Scott Parker/ CEC	(707) 745-0171
Project Manager and Certified Industrial Hygienist (Site Safety Officer	Michael T. Noble/ CEC	(707) 745-0171
Other		

Attachment 7-A

III. WORK PLAN

A. Objectives Determine nature and extent of subsurface contamination.

B. Project description Soil/waste sampling during placement of monitoring wells and development of remediation plan.

C. Field activities: job tasks

1. Four sample bore holes to a 15 foot depth and placement of four monitoring wells including sample analysis every 5-feet to depth.

2. Sampling of any soil thought to be contaminated that is encountered during the excavation.

3. Stock piling of soil water thought to be contaminated, until it can be treated on site or removed for disposal.

D. Duration Five days.

E. Personnel requirements Field geologist and project manager, drilling container.

IV. JOB HAZARD ANALYSIS

SUBSTANCE	OSHA PEL	ACGIH TLV	NIOSH REL
Waste Oil	5 mg/m^3	5 mg/m^3 10 mg/m^3 (STEL)	5 mg/m^3

Toxicological hazards of wastes (including local and systemic health effects) In general, oils have a low order of toxicity. The principal health hazard of oils in dermatitis. The best protection is to avoid unnecessary skin contact with the oil, refraining from wearing oil-soaked clothing, washing the oils from the body with soap and warm water.

Physical hazards associated with site activities Slip, trip and fall hazards; safety hazards associated with construction equipment; excessive noise.

V. SITE CHARACTERIZATION AND ANALYSIS

A. Site Information

1. Location of site 23555 Saklan Avenue, Hayward, California

2. Approximate size of site About 210 ' by 210'

3. Site topography Flat lot with a concrete pad on the parcel used by Berkeley Farms. The entire site including the site leased by Quality Tow is fenced.

4. Site accessibility All areas of the site are readily accessible. The property is bound by Saklan road on the east and Middle Lane on the South.

5. Pathways for hazardous substance dispersion The hazardous substance is a low volatile waste oil which is not likely to dissipate into the air. The primary path for dispersion is and has been through the soil and groundwater aquifers.

6. Anticipated weather conditions (temperature, humidity) and potential for heat/cold stress Mild weather conditions. If work is performed during the spring months there is a possibility of rain.

7. Past use of site The site was used as fueling lot by the previous owners and Berkeley Farms. Aerial photos showed the lot at one time appeared to have a small orchard on it in 1953.

8. Current use of site Currently the site is shared by a leaser, Quality Tow.

B. Description of on-site wastes (based on preliminary site evaluation)

1. Location The groundwater and soil on the entire site appears to be contaminated with diesel. The site's water well has over two feet of diesel floating on it.

2. Physical state of wastes Liquid, moving through the soil and groundwater.

3. Chemical characteristics of wastes Diesel

4. Range of concentration found to date Soil up to 24,000 ppm. Product in he well is 100 percent diesel No. 2

C. Site Map

1. Site control and security measures The site will remain fenced off during the installation of monitoring wells and sample bore holes. Only authorized personnel will be allowed in the contaminated areas. Proposed sample bore holes along Saklan Road will be placed after regulator approval.

2. Attached is an area map indicating:
   - The location of the site
   - The route to the nearest emergency room

VI. PERSONAL PROTECTIVE EQUIPMENT (PPE)

A. Level of protection required for this visit (see the next page for descriptions):

A________ B________ C________ D____X____

B. Respiratory protection Half-mask dual cartridge equipped with organic vapor cartridge. Only required if the airborne concentrations of contamination are above the action levels.

C. Protective clothing Level D protection to include hard hats, safety shoes, and coveralls as necessary.

D. Action levels regarding limitations in tasks assigned, PPE requirements and/or withdrawal from the site Withdrawal from the site if detected organics in the breathing zone exceeds 20 ppm (constant).

VII. LIST PPE FOR EACH JOB CLASSIFICATION AT THE SITE IF DIFFERENT LEVELS OF PROTECTION ARE SPECIFIED.

TASK	NAME	RESP	CLOTH	GLOVES	BOOTS	OTHER

All persons at the site who will be working within the contaminated area shall use the same Personal Protective Equipment, as outlined in paragraph VI.

Key:
B = Butyl
C = Covers
F = Fireman's
K = Kaysams
L = Latex
N = Neoprene
S - Saranex
T = Tyvek
V = Viton

VIII. EXPOSURE MONITORING PLAN

A. Frequency and types of air, personnel, and environmental monitoring Air should be continually monitored during the sampling process and every 30 minutes while excavating in areas suspected to be contaminated. Monitoring will be performed using an organic vapor analyzer.

B. Methodology Monitor down wind in the breathing zone of the field samples.

C. Equipment calibration procedures Equipment will be calibrated according to the manufactures directions.

IX. DECONTAMINATION PROCEDURES

A. PPE Leave the sampling area. Remove protective clothing (coveralls, gloves) inside out. Lastly, remove respirator. All non reusable clothing will be disposed of into the available receptacle. Respirators will be cleaned and placed into their storage container.

X. EMERGENCY RESPONSE

A. Decontamination procedures for personnel injured or exposed in the work zone Assist the injured or exposed worker out of the sampling area. If possible, carefully remove his PPE, and remove your own, according to standard decontamination procedures administer CPR/first aid as needed. Call for medical help immediately. CEC will have a portable phone on site.

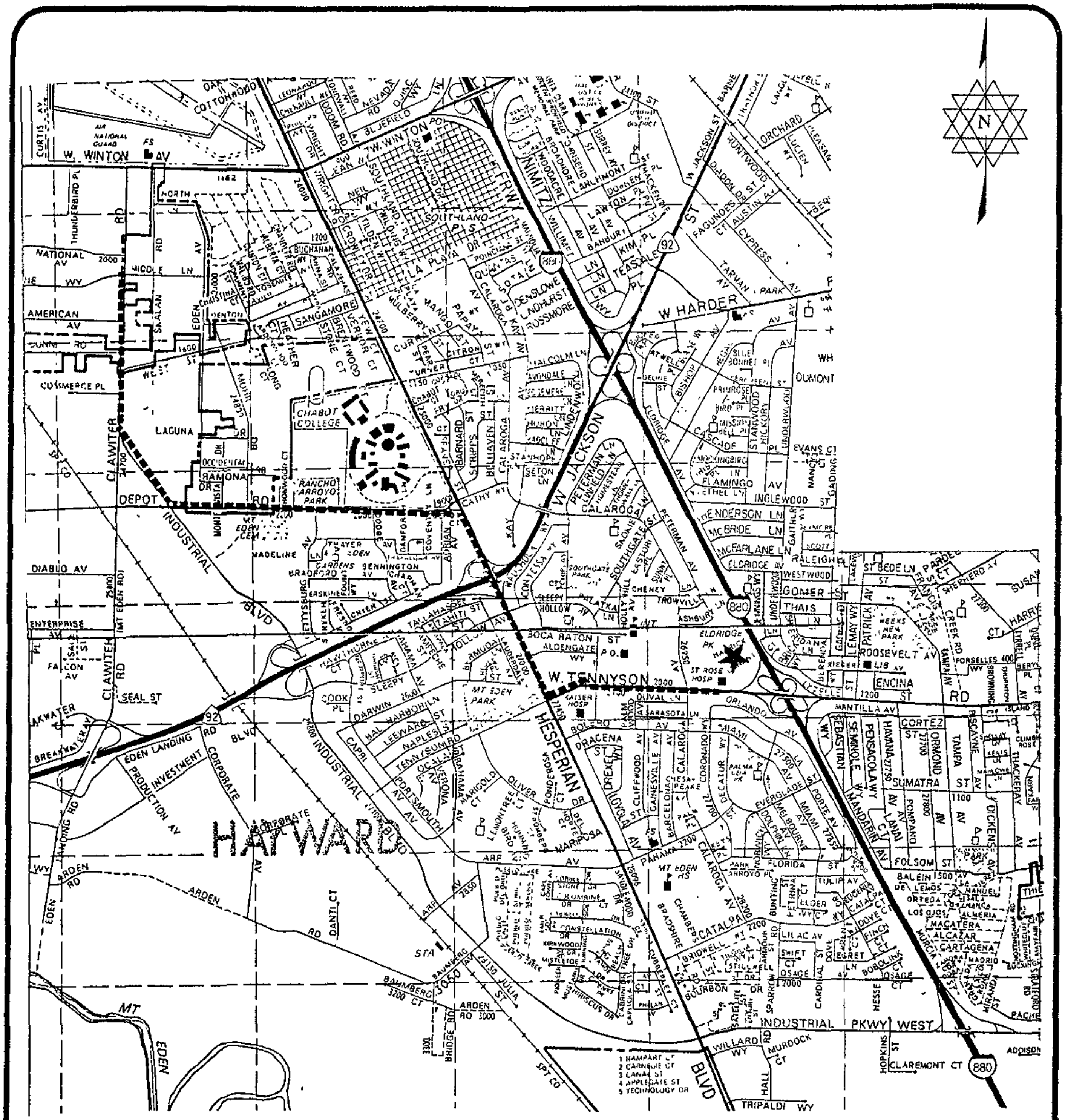
B. Emergency response plan

Personnel roles, lines of authority, communications The on-site CEC representative will have final authority on site health and safety methods concerning sampling.

C. Telephone numbers of emergency agencies, key contractor and responsible party.

	NAME/AGENCY	TELEPHONE
Ambulance	Oakland	911
Hospital	St. Rose Hospital	(415) 782-6200
Police Department	Hayward	911
Fire Department	Hayward	911
Project Manager	Michael T. Noble	(707) 745-0171
Alameda County Health Department	Thomas Peacock	(415) 271-4320
Health/Safety Coordinator	Michael T. Noble	(707) 745-0171
CA Dept. Health Services	DHS	(415) 540-2122
US EPA	Emergency Spills in California	(415) 974-8131
Federal OSHA	OSHA	(800) 648-1003
CHEMTREC	CHEMTREC	(800) 424-9300

Directions to hospital (See area map for route to St. Rose Hospital.) From site, go right on Middle then turn left on Clawiter, stay left as Clawiter and Industrial merge (5 blocks) follow industrial south and turn left on Depot, go to Hesperian and turn right, until reach W. Tenyson Road where you will turn left and go 2-3 blocks, turn left of Calaroga Avenue. The hospital will be on the right side of the street 27200 Calaroga Avenue, Hayward.



CERTIFIED

FIGURE 4

HOSPITAL ROUTE

TRAINING

- "Tail-gate" safety meetings on the health and safety considerations of the job and the necessary PPE are to be conducted at the beginning of the job, whenever new employees arrive at the job site, and whenever the job task changes such that new hazards are encountered.

- Employees will be trained on the requirements of the general and site-specific health and safety plans, the names of key health and safety personnel, site-specific health and safety hazards, site-specific safe work practices, PPE requirements, safe use of engineering controls and equipment on site, and medical surveillance requirements.

- Documentation of employee health and safety training will be accomplished by using the form entitle "Documenting Site-Specific Employee Health and Safety Training for Hazardous Waste Site Operations," included as Attachment 1 to this Section. This form shall be completed following all formal and informal health and safety discussions and copies shall be maintained by CEC as part of the sampling record of the site.

DOCUMENTING SITE-SPECIFIC EMPLOYEE HEALTH AND SAFETY TRAINING FOR HAZARDOUS WASTE SITE OPERATIONS

Site____

Name, title, agency, and phone number of person conducting health training:

Name:__________________ Agency:__________________

Title: __________________ Phone:__________________

TRAINING SUBJECTS DISCUSSED (initial topics discussed):

______ Requirements of the general site-specific health and safety plans.

______ Names of key health and safety personnel.

______ Site-specific safe work practices.

______ Personal Protective Equipment (PPE) requirements.

______ Safe use of engineering controls and equipment on site.

______ Medical surveillance requirements.

Specific Topic Covered:____

Signature of person conducting health and safety training:__________________

Signature of employees in attendance

Section 8
Attachment 1

EMERGENCY PROCEDURES

The following emergency response plan will be implemented to handle unanticipated on-site emergencies prior to start up of hazardous waste operations. All emergency incidents will be dealt with in a manner that minimizes adverse health risks to workers.

A. Emergency first air procedures:
Employee injury: Remove the employee from the contaminated zone and conduct decontamination procedures, first aid, and preparation for transport at a safe distance from the work site.

Eye exposure: Wash eyes with large amounts of potable water for at least 15 minutes; lift the upper and lower lids occasionally. Obtain medical attention. Contact lenses must not be worn when working at the site.

Skin Exposure: Flush the contaminated skin with water for at least 15 minutes. Remove contaminated clothing. Obtain medical attention immediately when exposed to concentrated solids or liquids.

If paramedic/rescue services are required, they will provide transportation to the hospital. For less serious circumstances, the project manager for CEC will provide transportation.

B. Emergency telephone numbers are given in the Site Specific Health and Safety Plan.

C. CEC will document the emergency situation. It will include:

- A description of the incident (including the date and time) that necessitated emergency response procedures, and complete an accident/incident investigation or critique of the incident.
- The date, time, and names of all persons/agencies that were notified and their responses.
- The resolution of the incident (including its duration) and the method/corrective action involved.

On-Site First Aid

All CERTIFIED personnel engaged in field activities will have available at the job site the following health and safety items at all times.

- First aid Kit

- Half Mask respirator
- Organic vapor or other appropriate cartridges
- Hard Hat
- Safety Glasses
- Hearing protection devices
- Protective gloves
- Chemical resistant coveralls (coated Tyvek)
- Rubber boots with steel toes
- Eye wash (portable)
- Cool vest devices
- On-site direct reading instruments for heat stress and chemical exposure

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Pacific Gas and Electric Company

Mission Division
24300 Clawiter Road
Hayward, CA 94545
415/783-2380

R L Larson
Manager

February 26, 1990

Certified Environmental Consulting
140 W. Industrial Way
Benicia, CA 94510

Attention: Mike Noble

Thank you for notifying us of your intent to work in the vicinity of our underground facilities. Information about their locations is, or will be, provided by surface markings at the work site.

In our marking for you, we naturally will exercise due care to make these markings as complete and accurate as reasonably possible. As you can appreciate, the nature of underground construction and installations prohibit any guarantee as to the absolute accuracy of surface markings. The precise location of underground facilities can only be determined by you through careful probing or hand digging in compliance with Article 6 of the Cal/OSHA Construction Safety Orders, which states in part:

1. Prior to opening an excavation, the employer shall determine to the extent possible whether underground installations such as sewer, fuel, water, electric lines, telecommunications, etc., will be encountered and, if so, where such underground installations are located.

2. When the excavation work approaches the approximate crossing or parallel location of such an underground installation and danger of accidental contact or disturbance is possible, the exact location shall be determined by appropriate means before proceeding. When it is uncovered, adequate protection shall be provided for the existing installation.

3. All known owners of underground facilities in the area involved shall be advised of proposed work at least 48 working hours prior to the start of excavation work.

Any further information you may desire may be obtained by contacting our Mapping Department at (415) 784-3224.

Thank you for your cooperation.

Sincerely,

Jess Brown

Jess Brown
Mapping Supervisor

<u>Codes</u>

blue – water
orange - communications
RED – electric
green - sewer/storm
yellow - gas/chemical

⑬

ALAMEDA COUNTY
HEALTH CARE SERVICES
AGENCY
DAVID J. KEARS, Agency Director

DEPARTMENT OF ENVIRONMENTAL HEALTH
Hazardous Materials Program
80 Swan Way, Rm. 200
Oakland, CA 94621
(415)

October 13, 1989

Mr. John Subuttec
Berkeley Land Co.
4550 San Pablo Ave.
Emeryville, CA 94608

Re: Unauthorized release from underground storage tank, 23555 Saklan Ave., Hayward

Dear Mr. Subuttec:

As you know, in June, 1988, your underground storage tank was removed from the above location. Analytical results of two soil samples taken from the tank pit indicate a hydrocarbon level of 2076 ppm and 24144 ppm. These are above the threshold level that the Regional Water Quality Control Board (RWQCB) considers to be evidence of an unauthorized release requiring further investigation. An unauthorized release report has been filed with this office; you must now initiate further investigation and/or cleanup activities at this site.

A preliminary assessment should be conducted to determine the extent of soil and groundwater contamination that has resulted from the leaking tank system. The information gathered by this investigation will be used to assess the need for additional actions at the site. The preliminary assessment should be designed to provide all of the information in the format shown in the attachment at the end of this letter. This format is based on RWQCB guidelines. You should be prepared to install one monitoring well, if you can verify the direction of groundwater flow in the immediate vicinity of the site, and three wells if you cannot.

Until cleanup is complete, you will need to submit reports to this office and to the RWQCB every three months (or at a more frequent interval, if specified at any time by either agency). These reports should include information pertaining to further investigative results; the methods and costs of cleanup actions implemented to date; and the method and location of disposal of any contaminated material.

Soils contaminated at hazardous waste concentrations should be transported by a licensed hazardous waste hauler and disposed of or treated at a facility approved by the California Department of

WORK PLAN REQUIREMENTS FOR AN INITIAL SUBSURFACE INVESTIGATION

This outline should be followed by professional engineering or geologic consultants in preparing work plans to be submitted to the RWQCB and local agencies. Work plans must be signed by a California-registered engineer or geologist.

This outline should be referred to in context with the "Regional Board Staff Recommendations for Initial Evaluation and Investigation of Underground Tanks" (June 2, 1988).

PROPOSAL FORMAT

I. Introduction
   - A. State the scope of work
   - B. Items are omitted that have already been received by this Department

II. Site Description
   - A. Describe the hydrogeologic setting of the site vicinity
   - B. Prepare a vicinity map (including wells located on-site or on adjoining lots, as well as any nearby streams

III. Plan for Determining Extent of Soil Contamination
   - A. Describe method for determining the extent of contamination within the excavation
   - B. Describe sampling methods and procedures to be used
     1. If a soil gas survey is planned, then:
        - identify number of boreholes, locations, sampling depths, etc.;
        - identify subcontractors, if any;
        - identify analytical methods;
        - provide a quality assurance plan for field testing.
     2. If soil borings are to be used to determine the extent of soil contamination, then:
        - identify number, location (mapped), and depth of the proposed borings;
        - describe the soil classification system, soil sampling method, and rationale;
        - describe the drilling method for the borings, including decontamination procedures;
        - explain how borings will be abandoned.
   - C. Describe how clean and contaminated soil will be differentiated, and describe how excavated soil will be stored and disposed of. If on-site soil aeration is to be used, then describe:
     1. The volume and rate of aeration/turning;
     2. The method of containment and cover;
     3. Wet-weather contingency plans;
     4. Results of consultation with the Bay Area Air Quality Management District.

Other on-site treatments (such as bioremediation) require permits issued by the RWQCB. Off-site storage or treatment also requires RWQCB permits.

D. Describe security measures planned for the excavated hole and contaminated soil

IV. Plan for Characterizing Groundwater Contamination

Construction and placement of wells should adhere to the requirements of the "Regional Board Staff Recommendations for Initial Evaluation and Investigation of Underground Tanks."

A. Explain the proposed locations of monitoring wells (including construction diagrams), and prepare a map to scale

B. Describe the method of monitoring well construction and associated decontamination procedures

1. Expected depth and diameter of monitoring wells.
2. Date of expected drilling.
3. Locations of soil borings and sample collection method.
4. Casing type, diameter, screen interval, and pack and slot sizing technique.
5. Depth and type of seal.
6. Development method and criteria for determining adequate development.
7. Plans for disposal of cuttings and development water.
8. Surveying plans for wells (requirements include surveying to established benchmark to 0.01 foot).

C. Groundwater sampling plans

1. Water level measurement procedure.
2. Well purging procedures and disposal protocol.
3. Sample collection and analysis procedures.
4. Quality assurance plan.
5. Chain-of-custody procedures.

V. Prepare a Site Safety Plan

Health Services. Soils contaminated below the hazardous waste threshold may be managed as nonhazardous, but are still subject to the RWQCB's waste discharge requirements. In no case may stockpiled soil with any detectable level of contamination be used to backfill the hole.

Your work plan must be submitted to this office by **November 16, 1989**. Copies of the proposal should also be sent to the RWQCB (attention: Lester Feldman). You may implement remedial actions before approval of the work plan, but final concurrence by this office will depend on the extent to which the work done meets the requirements described in this letter.

You will need to submit an additional deposit of $333 to cover costs that the Division of Hazardous Materials incurs during remediation oversight. If you have any questions about this letter or about remediation requirements established by the RWQCB, please contact the undersigned, at 271-4320.

Sincerely,

Thomas F. Peacock

Thomas F. Peacock, Senior HMS
Hazardous Materials Division

TP:tp

enclosure

cc: Howard Hatayama, DOHS
Lester Feldman, San Francisco Bay RWQCB
Gil Jensen, District Attorney, Alameda County Consumer and Environmental Protection Agency
Rafat Shahid, Assistant Agency Director, Environmental Health

ALAMEDA COUNTY
HEALTH CARE SERVICES
AGENCY
DAVID J. KEARS, Director

Telephone Number: (415)

Michael T. Noble, Vice President
Certified Environmental Consulting
140 West Industrial Way
Benicia CA 94510-1016

Re: Site Search Request for Hayward Facilities

March 12, 1990

Dear Mr. Noble:

An evaluation was done of the documents pertaining to the above properties in this office. This search included checks for underground tank records, hazardous waste generator inspections, hazardous material releases, emergency responses, and other records.

Below is a summary of the documents available from files maintained by this office:

Address	Hazardous Waste Generator Inspection	Underground Tank Insp/ Permit	Prop. 65 Report
22950 Clawiter	X		
22991 Clawiter	X		X
(Has also filed a Business Plan)			
23724 Saklan	X	X	
1490 W. Winton	X		

No file information was available for the following sites:

23001 Skywest Dr.
1525 W. Winton Av.
Krueger & Sons, Saklan Rd.
Alhambra Water, Saklan Rd.

This statement is limited to information available to this department. Other information may be available from other agencies or parties. The City of Hayward Fire Department has primary jurisdiction over underground tanks as well as business plans for hazardous materials, and enforces its own storage ordinance.

Please contact Pam Evans at 271-4320 with any questions you may have.

Sincerely,

Edgar B Howell

Edgar B. Howell III, Chief
Hazardous Materials Division

EBH:PJE

c: Rafat Shahid, Assistant Agency Director
County of Alameda Environmental Health Department

SITES LISTED ON EPA'S CERCLIS OR THE REGIONAL WATER QUALITY CONTROL BOARD'S FUEL LIST

American Pipe Processing	29901 Industrial
Bay Ford Tractors	975 Industrial
Clarkes Bld Materials	23040 Clawiter
Continental White Cap	24493 Clawiter
Drewry Photo Corp	27105 Industrial
EKC Tech	1739 Sabre Avenue
Equity Property Development Corp.	24668 Hesprian
Exxon	26115 Hesperian
Farrer Properties	944 West Winton
Foodmaker Inc.	2395 American Avenue
Hayward Corp Yard	3050 Winton
Jackson St. Lumber Co.	73 W. Jackson Street
J. C. Penny Trucking	2001 Chabot
Laura Scudders	27751 Industrial
Nature's Farm	2702 McCome Ave
Rotten Robbie	27814 Hesparian Blvd
Imiser Freght	2340 Industrial
Smothers Pacific	Chabot Road
Swift Adhesive and Coatings	2074 National Ave
Texaco	23990 Hesparian
Thrifty Oil	20200 Hespiran
Thermos Tuscon Inc.	2342 American
Toscano Baking Co	2227 National Ave
Unocal	28250 Hesperian
Western Drums	21301 Cloud Way

CERTIFIED ENVIRONMENTAL CONSULTING, INC.

February 23, 1990

Alameda County
Department of Environmental Health
Attention: Pam Evans
Hazardous Materials Program
80 Swan Way, Room 200
Oakland, CA 94621

Dear Ms. Evans:

I would like to review the following case files for Tank Permits/Closure, or contamination reports:

1. Hayward Airport, Winton Avenue, Hayward
2. United States Marine Corps, Airport
3. Air National Guard, Airport
4. Chevron, Winton Avenue, Hayward
5. Oliver de Silva Clawiter, Hayward
6. Columbus Salome Distributor, Clairter, Hayward
7. Krueger & Sons, Sakland Road, Hayward
8. Alhambra Water, Sakland Road, Hayward
9. Trident Truck Lines C., Clairter or Sakland, Hayward

If possible, I would like to review these files on Thursday, March 1, 1990.

Sincerely,

Michael T. Noble, CIH
Vice President

MTN:cak

Consulting in Environmental Engineering • Industrial Hygiene • Asbestos • Hydrogeology

140 West Industrial Way • Benicia, California • 94510-1016 • (707) 745-0171 • FAX (707) 745-0163

⑤ 1. Hayward Airport, 23001 Skywest Drive, Hayward
⑨ 2. United States Marine Corps, 1525 Winton, Hayward ✓
⑥ 3. Air National Guard, 1525 Winton, Hayward
⑧ 4. Chevron, 1490 West Winton Avenue, Hayward
⑦ 5. Oliver de Silva, 22991 Clawiter, Hayward
④ 6. Columbus Salome Distributor, 22950 Clawiter, Hayward
① 7. Krueger & Sons, Saklan Road, Hayward (415) 895-2050
② 8. Alhambra Water, Saklan Road, Hayward (800) 772-3440 ✓
③ 9. Trident Truck Lines C., 23724 Saklan, Hayward

22-Hewitt

Site: INTERLOCKING ROOF/CALIF. TERR.
Location: 500 KING ST
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: HYSTER COMPANY
Location: 47132 KATO RD.
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: HOWARD'S BACKHOE RENTAL
Location: 41875 OSGOOD RD.
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: HERITAGE VILLAGE
Location: 38050 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: GRADE WAY CONSTRUCTION
Location: 438012 OSGOOD RD.
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: GM-FREMONT
Location: ADJACENT TO NUMMI
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: FREMONT UNIFIED SCHOOL DISTR.
Location: 38442 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: FREMONT UNIFIED SCHOOL DIST.
Location: WAREHOUSE
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: FREMONT UNIFIED SCHOOL DIST.
Location: 43770 GRIMMER BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: ROTTEN ROBBIE/FREMONT SHOPPING
Location: 40575 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: FREMONT HUB
Location: 39201 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: FEE CONSTRUCTION
Location: 42000 OSGOOD RD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: EXXON
Location: 4995 MOWRY AVE
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: EXXON
Location: 39990 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: ECONO/KAYO
Location: 41100 ROBERTS AVE
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: CITY OF FREMONT: CORP. YARD
Location: 37350 SEQUOIA RD.
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: CITY OF FREMONT FIRE STN. #1
Location: 4200 MOWRY ST
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: CHEVRON
Location: 5300 MOWRY AVE.
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: CHEVRON
Location: 2020 DRISCOLL
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: CENTRAL CHEVROLET
Location: 4949 THORNTON AVE
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: CBI SERVICES INC.
Location: 41777 BOYCE RD.
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: CATALINA TRUCKING
Location: 185 KING AVENUE
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: CALIFORNIA LIFETILE
Location: 45111 INDUSTRIAL DR
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: BORDEN INDUSTRIAL
Location: 41100 BOYCE RD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: BEACON
Location: 47700 WARM SPRINGS BLVD.
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: BEACON
Location: 42245 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: BEACON
Location: 41100 WASHINGTON BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: BEACON
Location: 40500 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: BAY AREA CONCRETES, INC.
Location: 43055 OSGOOD RD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: AUTOWEST MITSUBISHI
Location: 37156 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: ARCO
Location: 43500 GRIMMER
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: ARCO
Location: 40055 BLACOW RD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: ARCO
Location: 35900 FREMONT BLVD
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: 7-ELEVEN
Location: 3868 DELAWARE STREET
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: 7-ELEVEN
Location: NILES CYN/MISSION BV
City: FREMONT
Source: WRCB Problem: TANK LEAK

Site: AMCHEM PRODUCTS, INC.
Location: 37899 NILES BOULEVARD
City: FREMONT Zip: 94536
Source: DHS5

Site: PACIFIC CEMENT & AGGREGATES
Location: 35171 SEQUOIA ROAD
City: FREMONT Zip: 94536
Source: DHS1

IMPACT CITY: * HAYWARD

Site: OAKLAND FENCE CO
Location: 1580 W WINTON AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: BEACON
Location: 29705 MISSION BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: UNKNOWN
Location: 1015 A ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: TEXACO
Location: 528 JACKSON ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: GOODYEAR TIRE & RUBBER CO
Location: 1051 A ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: BAY FORD TRACTORS
Location: 975 INDUSTRIAL PKWY W
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HAYWARD CORPORATION YARD
Location: 3050 WINTON AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MUNSON,WARREN
Location: 21011 MONTGOMERY AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: J.C. PENNY
Location: 21105 CABOT BLVD.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SMISER FRIEGHT
Location: 2340 INDUSTRIAL PARKWAY W
City: HAYWARD
Source: WRCB Problem: TANK LEAK

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* *Indicates incorporated city*

Site: LAURA SCUDDERS
Location: 27751 INDUSTRIAL BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 2492 WHIPPLE AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ARCO
Location: 365 JACKSON ST.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: YELLOW FREIGHT SYSTEM
Location: 25555 CLAWITER
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: WILMAC METALS
Location: 529 C ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: WEYERHAEUSER COMPANY
Location: 3495 BREAKWATER CT
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: WESTERN DRUMS, INC.
Location: 21301 CLOUD WAY
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: WALKER'S CONCRETE
Location: 1844 W WINTON AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: VIC HUBBARD
Location: 411 W A ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: VALLEY PET SUPPLY
Location: 30845 HUNTWOOD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: VALLEY PET SUPPLY
Location: 1200 ZEPHYR AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: UNOCAL
Location: 3500 BREAKWATER AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: UNOCAL
Location: 2701 EAST AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: UNOCAL
Location: 25995 MISSION BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: UNOCAL
Location: TENNYSON & HUNTWOOD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: UNITED CAN COMPANY
Location: UNKNOWN
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: TOSCANO BAKING COMPANY
Location: 2227 NATIONAL AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: THRIFTY OIL
Location: 25225 MISSION BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: THRIFTY OIL
Location: 207 A ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: THRIFTY OIL
Location: 22200 HESPERIAN BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SUPER STRUCTURES/WENDLAND TRKG
Location: 24200 CLAWITER RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SUNSET HIGH SCHOOL
Location: 22100 PRINCETON
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SOUTHERN PACIFIC
Location: CABOT RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SHELL
Location: 27501 LOYOLA
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SHELL
Location: 2408 WHIPPLE RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SHELL
Location: 138 JACKSON
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SHELL
Location: 1097 W TENNYSON
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SERVOMATION
Location: 2331 TRIPALDI WAY
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: SEARS AUTOMOTIVE CENTER
Location: 660 W WINTON
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: S & J DETAIL
Location: 352 A ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: RUNNELS INDUSTRIES
Location: 3590 ENTERPRISE AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ROTTEN ROBBIE
Location: 720 W TENNYSON
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ROTTEN ROBBIE
Location: 27814 HESPARIAN BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ROHMN & HAAS CALIFORNIA, INC.
Location: 25500 WHITESELL ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: REYNOLDS ALMNM-S.F. CAN PLANT
Location: 2425 WHIPPLE RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: REGIONAL OCCUPATIONAL CENTER
Location: 26316 HESPERIAN BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: REGAL
Location: 193 WINTON AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: REDCO
Location: 1975 NATIONAL AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: RED TOP ELECTRIC
Location: 24967 HUNTWOOD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: PRECISION METALS
Location: 3402 ENTERPRISE AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: PG&E
Location: 24300 CLAWITER RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: PESTANA PROPERTY
Location: 29234 MISSION BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: PACIFIC DISTRIBUTION CENTER
Location: 21001 CHABOT RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: OLIVER DE SILVA
Location: 22991 CLAWITER RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: NORPAK
Location: 20550 CORSAIR BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: NATURE'S FARM
Location: 2707 MCCONE AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 525 W. A ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 494 ROUSSEAU ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 486 A ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 404 W HARDER RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 210 W JACKSON ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 1974 W TENNYSON
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 1109 W TENNYSON RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: COTTER WAY
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MILLER'S ALUMINUM
Location: 25362 CYPRESS AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MERVYN'S DEPT. STORE
Location: 22301 FOOTHILL BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MCDONALD CONSTRUCTION
Location: 3500 ENTERPRISE AVE.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MC CULLOUGH CHEVROLET-HAYWARD
Location: 22645 WATKINS ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: MAURY COX VANS
Location: 25700 MISSION BLVD.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: LEW'S DIESEL REPAIR
Location: 29318 PACIFIC ST.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: LAWRENCE DAIRY
Location: 23555 SAKLAN AVE.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: KAYO
Location: 438 W TENNYSON ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: STONETREE TOWNHOUSES/JAMES PRP
Location: 811 D ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: J.R. TRUCKING
Location: 2001 CHABOT ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: J&R WAREHOUSE
Location: 31281 WIEGMAN DR.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: INTERN'L WINDOW
Location: 30526 SAN ANTONIO
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HUTCH'S CAR WASH
Location: 1367 A ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HORMEL CO.
Location: 30611 SAN ANTONIO ST.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HOLY SEPULCHRE CEMETERY
Location: 26320 MISSION BLVD.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HOLLAND OIL
Location: 789 FLETCHER LN
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HERRINGER PROPERTY
Location: 22701 WATKINS AVE.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HERRICK CORP.
Location: 25450 CLAWITER RD.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HAYWARD UNIFIED SCHOOL DIST.
Location: 2440 AMADOR
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: HAYWARD DODGE, INC.
Location: 24773 MISSION BLVD.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: GRUNOW PROPERTY
Location: 19483 WESTERN BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: GI TRUCKING
Location: 30542 SAN ANTONIO
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: G.I. TRUCKING
Location: 22300 FOOTHILL BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: FUJIIS PLANT OUTLET
Location: 24949 SOTO RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: FOODMAKER INC.
Location: 2395 AMERICAN AVE.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: FIRE FAB INC.
Location: 23315 CONNECTICUT
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: FARRER PROPERTY
Location: 944 W WINTON
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: FAIRBANKS SCALES
Location: 3494 INVESTMENT BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: EXXON
Location: 26115 HESPERIAN BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: DUNCAN & SON PETROLEUM
Location: 29303 PACIFICA ST.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: DREWRY PHOTCOLOR CORPORATION
Location: 27105 INDUSTRIAL BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: DINO'S FURNITURE
Location: 21564 MISSION BLVD.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: DIAMOND BATHURST
Location: 22302 HATHWAY AVE.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: DEXTER CORP.-MIDLAND DIVISION
Location: 31500 HAYMAN ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CRESCENT TRUCK LINES
Location: 2480 WHIPPLE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: COREMARK INC.
Location: 31300 MEDALLION ST.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CONTINENTAL WHITE CAP, INC.
Location: 22493 CLAWITER RD.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CON-WAY EXPRESS
Location: 2200 CLAREMONT CT
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CLEMENTINA LTD
Location: 31823 HAYMAN ST.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CITGO
Location: 660 W WINTON AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CHEVRON
Location: 24086 MISSION BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CHEVRON
Location: 21995 FOOTHILL BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

* *Indicates incorporated city*

Calif Office of Planning & Research HW Site List 6/89

Site: CHEVRON
Location: 2ND & B ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CHAPEL OF THE CHIMES MEM. PARK
Location: 32992 MISSION BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CHABOT COLLEGE
Location: 25555 HESPERIAN BLVD.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CASSARO PROPERTY
Location: 593 W HARDER
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CALIFORNIA AIR NATIONAL GUARD
Location: 1525 W WINTON AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CALIF. STATE UNIV. HAYWARD
Location: MISSION BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: CAL TRANS PROPERTY
Location: 25030 MISSION BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: C.C. ENDOWMENT BOARD
Location: 1609 WHIPPLE RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: BEECHCRAFT WEST
Location: 19990 SKYWEST DRIVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: BEACON
Location: 392 W HARDER RD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: BAY CITY AUTO AUCTION
Location: INDUSTRIAL WAY
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: AT&T
Location: 100 ORCHARD AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ARCO
Location: 17601 HESPERIAN BLVD
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: AMERICAN PIPE PROCESSING
Location: 29901 INDUSTRIAL PKWY
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ALLIED SYSCO, INCORPORATED
Location: 30977 SAN ANTONIO STREET
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ALLGOOD INDUSTRIES
Location: 3466 ENTERPRISE AVE
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ALHAMBRA HAYWARD
Location: 22990 CLAWITER
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ALAMEDA COUNTY BUILDING MAINT.
Location: 951 TURNER COURT
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ADN CORPORATION
Location: 29001 HOPKINS STREET
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: ABC SERVICES
Location: 31845 HAYMAN ST
City: HAYWARD
Source: WRCB Problem: TANK LEAK

Site: A & J ELECTRIC CABLE COMPANY
Location: 30608 SAN ANTONIO ST.
City: HAYWARD
Source: WRCB Problem: TANK LEAK

IMPACT CITY: * LIVERMORE

Site: DE PAOLI EQUIPMENT
Location: 4001 N VASCO RD
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: FIRE STATION #1
Location: 4550 EAST AVE
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: MILL SPRINGS PARK APARTMENTS
Location: RAILROAD AVE/S.L&S.P
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: DUPERLY PROPERTY
Location: 10057 TESLA RD.
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: UNOCAL
Location: 900 S LIVERMORE AVE
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: TEXACO
Location: 930 SPRINGTOWN
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: TEXACO
Location: 1175 CATALINA DR
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: SHELL
Location: 1155 PORTOLA AVE
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: SANDIA NATIONAL LABORATORIES
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: PACIFIC BELL
Location: 2324 2ND ST
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 4707 1ST ST
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: LIVERMORE DISPOSAL
Location: 5175 S. FRONT RD.
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: LAWRENCE LIVERMORE LAB
Location: 7000 EAST AVE
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: LAWRENCE LIVER. NL-SW CORN
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: LAWRENCE LIVER. NL-BLDG 40
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: MOBIL
Location: 2008 1ST ST
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: CHEVRON
Location: 4904 S. FRONT
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: CHEVRON
Location: 1925 BARCELONA ST.
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: CHEVRON
Location: 1334 W 1ST ST
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: CALTRANS
Location: 6153 S FRONT ST
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: ARCO
Location: 899 RINCON AVE
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: RICHMOND TANK CAR
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: INTEL LIVERMORE FAB. PLANT II
Location: 250 N MINES RD
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: HEXCEL COMPST. MTRL. MFG. PLT.
Location: TREVANO RD.
City: LIVERMORE
Source: WRCB Problem: TANK LEAK

Site: HEXEL CORPORATION
Location: 10 TREVARNO ROAD
City: LIVERMORE Zip: 94550
Source: DHSI

IMPACT CITY: * NEWARK

Site: SHELL
Location: 6005 JARVIS
City: NEWARK
Source: WRCB Problem: TANK LEAK

* *Indicates incorporated city*

38 files

FILE CHECK-OUT FORM FOR UNDERGROUND FUEL LEAK CASES

17 files

Today's Date: 2-5-90 Name: Noble, M.T

Representing: BERKLEY FARMS Phone: 745-0171

Please follow these instructions:

1- Check in the fuel leak case list to make sure that each of your sites is a case.

2- Please list cases **SORTED BY COUNTY, THEN IN ALPHABETICAL ORDER BY SITENAME.**

3- This sheet is our receipt for the files we give you. Please fill out the top completely. We will keep this list.

4- Please **RETURN THE FILES IN ALPHABETICAL ORDER** for refiling. THANK YOU!

5- Maximum 10 files per person.

6- The files cannot leave the building.

7- If there is a line for the xerox machine, you must limit your xeroxing to 10 pages per time.

Sitename Address County

Herrick Corp, 25450 Clawiter Rd, Hayward/Alameda
Oakland Fence Co., 1570 W. Winton, Hayward/Alameda
✓ PCI 23520 Clawiter Rd, Hayward/Alameda
PG&E 24000 Clawiter Rd, Hayward/Alameda
Sears Automotive 160 W Winton, Hayward/Alameda
Super Structures/Windland Trucking, 24200 Clawiter Rd, Hayward, Alameda
✓ Yellow Freight 2555 Clawiter Rd, Hayward Alameda

FILE CHECK-OUT FORM FOR UNDERGROUND FUEL LEAK CASES

Today's Date: 2-5-90 Name: Noble, M.T.

Representing: Berkley Farms Phone: 745-0171

Please follow these instructions:

1- Check in the fuel leak case list to make sure that each of your sites is a case.

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Sitename	Address	County
Caly Air National Guard, Hayward	1525 Winton, Hayward	Alameda
Alhambra Water,	Oakland Road, Hayward	"
Chevron	1490 West Winton Ave, Hayward	"
Columbus Salami Dist.	22930 Clawiter, Hayward	"
Hayward Airport	23001 Skywest Dr, Hayward	"
Krueger & Sons	Oakland Rd, Hayward	"
Olive de Silva,	22991 Clawiter, Hayward	"
Trident Truck Lines,	23724 Oakland, Hayward	"
U. S. Marine Corp,	1525 Winton, Hayward	"
Clarke Building Materials	23040 Clawiter Rd. Hayward	"
Herrick Corp,	25-450 Clawiter Rd, Hayward	"
Oakland Fence Co,	1580 W Winton, Haywad, Caly	
PC1	23520 Clawiter Rd, Hayward, Caly	
[illegible]	24000 Clawiter Rd, Hayward, Caly	

P

SOIL VAPOR MONITORING RESULTS

Date: 2/27 & 3/2
Job number: 90-57-237
Client: Berkeley Land Co.
Location/Address: 23555 Saklan Rd
Hayward, Calif

Phone #:

Project mgr M.T. Noble
Samples collected by Noble & Cornils
Sample map attached [✓ Y]/N

Assesement for underground utilities: [✓ Y]/N, explain USA/PGE identified sewer, cable and gas lines on Saklan + Middle Roads

Probe number	Depth (ft)	Vacuum (Inches of Hg)	OVM reading (ppm)	Comments
VP-1	7½	18"	22	probe pulled
	7½		18	and reinserted
	10		21	after cleaning
	12½		25.6	and removing clogged
	15		16.8	Clay [illegible] from
VP-2	5½		22	the probe
	7½		20	
	10		21	
	12		26	
	15		17	
VP-3	7½		18	
VP-4	7½		22	
	10		12	
VP-5	7½		22	
	10		9	
	12½		7	
VP-6	7½		22	
	10		42	
	12½		12	
VP-7	7½		18	
	10		10	
	12		9	
	15		7	

CERTIFIED

SOIL VAPOR MONITORING RESULTS

Date: 2/27 & 3/2
Job number:
Client: Berkley Land Co.
Location/Address: 23555 Saklan Rd
Hayward

Phone #:

Project mgr Noble
Samples collected by Noble & Cornils
Sample map attached [✓ Y]/N

Assesment for underground utilities [✓ Y]/N, explain

Probe number	Depth (ft)	Vacuum (Inches of Hg)	OVM reading (ppm)	Comments
VP-8	7½		83	
	10		185	
	12½		220	
	15		112	

CERTIFIED

CERTIFIED *Environmental Consulting, Inc.*

Consultants in Environmental Engineering and Hydrogeology
140 West Industrial Way, Benicia, CA, 94510-1016
Ofc. (707) 745-0171 (800) 447-0171 Fax. (707) 745-0163

Chain of Custody Record and Analysis Request Form

Collected By: Cornils
Job Number:
Facility or Generator: Berkeley Farms (Hayward)
CEC Project Manager: Noble

Sample Information

Sample Number	Lab Identification Number	Type	Depth	Sample Date	Sample Time	Analysis Requested
BC-C227-A10		Soil	10.0'	2-27-90	1607	TPH (G&D), BTX&E
BC-C301-B13		Soil	13.6'	3-1-90	—	" "
BC-C301-B15		Soil	15.5'	3-1-90	—	" "
BC-C301-W1		Water	—	3-1-90	—	" "
BC-C301-W2		Water	—	3-1-90	—	" "
BC-C301-W3	- Product	Water	—	3-1-90	—	" "

Suspected Waste Constituents/Remarks: Diesel

Sample Allocation

Name of Lab: Superior Laboratory
Address: 825 Arnold Ave, Suite 2
Martinez, CA
Telephone: 415-229-1512
Signature:

(check)

☐ Sample received intact
☐ Sample damaged or missing (describe below)

Chain of Possession

Relinquished By:

	Signature	Date	Time
1)		3-2-90	1250
2)			
3)			
4)			

Received By:

	Signature	Date	Time
1)		3-2-90	1250
2)			
3)			
4)			

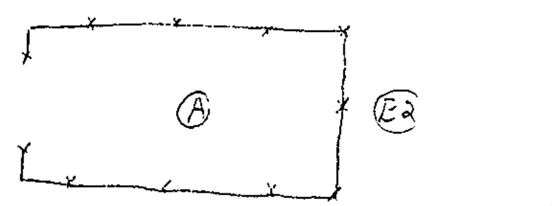
CERTIFIED
ENVIRONMENTAL CONSULTING, INC.

LOG OF EXPLORATORY BORING

PROJECT No.
CLIENT Berkeley Farms
LOCATION Hayward
LOGGED BY [illegible]
DRILLER NA
DRILLING METHOD
Hand Auger
HOLE DIA. ~6"
INSTALLATION DATA NA

BORING No. A
SHEET 1 OF 2
DATE 2/27/90
3/1/90

FIELD LOCATION OF BORING



WATER LEVEL					
TIME					
DATE					

DEPTH	GRAPHIC LOG	BLOW / FT	VAPOR CONCENTRATION	SAMPLE TYPE AND DEPTH	SOIL GROUP SYMBOL (U.S.C.S.)	DESCRIPTION

FORM # CECF18

SUPERIOR ANALYTICAL LABORATORY, INC.
1555 BURKE, UNIT 1 • SAN FRANCISCO, CA 94124 • PHONE (415) 647-2081

CERTIFICATE OF ANALYSIS

LABORATORY NO.: 80597
CLIENT: Certified Environmental Consulting,
CLIENT JOB NO.: BERKELY FARM

DATE RECEIVED: 03/02/90
DATE REPORTED: 03/09/90

ANALYSIS FOR TOTAL PETROLEUM HYDROCARBONS
by Modified EPA SW-846 Method 8015

LAB #	Sample Identification	Concentration (mg/kg)(mg/L*) Gasoline Range	Diesel Range
1	BC-0227-A120	ND<10	40
2	BC-0301-B163	ND<10	40
3	BC-0301-B186	ND<10	550
4	BC-0301-W1	ND<1*	110*
5	BC-0301-W2	ND<1*	5*
6	BC-0301-W3 Product sample	ND<1%	100%

mg/kg - parts per million (ppm)

Method Detection Limit for Gasoline and Diesel in Soil: 10 mg/kg
Method Detection Limit for Gasoline and Diesel in Water: 1 mg/L

QAQC Summary:

Daily Standard run at 200mg/L: RPD Gasoline = 2.5%
RPD Diesel = 6%
MS/MSD Average Recovery = 130%: Duplicate RPD = 9%

Edward R. Morales
Laboratory Manager

SUPERIOR ANALYTICAL LABORATORY, INC.

1555 BURKE, UNIT 1 • SAN FRANCISCO, CA 94124 • PHONE (415) 647-2081

CERTIFICATE OF ANALYSIS

LABORATORY NO.: 80597	DATE RECEIVED: 03/02/90
CLIENT: Certified Environmental Consulting,	DATE REPORTED: 03/09/90
CLIENT JOB NO.: BERKELY FARM	

ANALYSIS FOR BENZENE, TOLUENE, ETHYL BENZENE & XYLENES by EPA SW-846 Methods 5030 and 8020

LAB #	Sample Identification	Concentration(ug/kg)(ug/L)* Benzene	Toluene	Ethyl Benzene	Xylenes
1	BC-0227-A120	ND<3	15	6	13
2	BC-0301-B163	ND<3	ND<3	ND<3	ND<3
3	BC-0301-B186	ND<3	ND<3	4	10
4	BC-0301-W1	ND<0.3*	ND<0.3*	4.4*	15*
5	BC-0301-W2	0.4*	14*	ND<0.3*	0.6*

ug/kg - parts per billion (ppb)

Method Detection Limit in Soil: 3 ug/kg
Method Detection Limit in Water: 0.3 ug/L

QAQC Summary:

Daily Standard run at 20ug/L: RPD = <15%
MS/MSD Average Recovery = 104%: Duplicate RPD = 1%

Edward R. Morales
Laboratory Manager

MT#80597

CERTIFIED Environmental Consulting, Inc.

Consultants in Environmental Engineering and Hydrogeology
140 West Industrial Way, Benicia, CA, 94510-1016
Ofc. (707) 745-0171 (800) 447-0171 Fax. (707) 745-0163

Chain of Custody Record and Analysis Request Form

Collected By: Cornils
Job Number:
Facility or Generator: Berkeley Farms (Hayward)
CEC Project Manager: Noble

Sample Information

Sample Number	Lab Identification Number	Type	Depth	Sample Date	Sample Time	Analysis Requested
BF-C227-A110		Soil	11.0'	2-27-90	1607	TPH (G & D), BTX & E
BF-C301-B136		Soil	13.6'	3-1-90	—	" "
BF-C301-B155		Soil	15.5'	3-1-90	—	" "
BF-C301-W1		Water	—	3-1-90	—	" "
BF-C301-W2		Water	—	3-1-90	—	" "
BF-C301-W3	**Product**	Water	—	3-1-90	—	"

Suspected Waste Constituents/Remarks: Diesel

Sample Allocation

Name of Lab: Superior Laboratory
Address: 825 Arnold Ave, Suite 2
Martinez CA
Telephone: 415-229-1512
Signature:

(check)

☐ Sample received intact
☐ Sample damaged or missing (describe below)

Chain of Possession

Relinquished By:

	Signature	Date	Time
1)		3-2-90	1250
2)			
3)			
4)			

Received By:

	Signature	Date	Time
1)		3-2-90	1250
2)			
3)			
4)			

KAPREALIAN ENGINEERING, INC.
Consulting Engineers
P. O. BOX 913
BENICIA, CA 94510
(415) 676-9100 (707) 746-6915

KEI-J88-1110
December 6, 1988

Paradiso Construction
P.O. Box 6397
Oakland, CA 94603

Attention: Mr. Paul Paradiso

RECEIVED
[illegible] 1988
PARADISO
CONSTRUCTION CO

RE: Stockpiled Soil Sampling for
Berkeley Farms
23555 Saklan
Hayward, California

Dear Mr. Paradiso:

This letter report summarizes the results of the stockpiled soil sampling and laboratory analyses for the referenced site. The soil analyses were conducted to comply with the County Health Department requirements for proper disposal of contaminated soil.

On November 7, 1988, soil samples from approximately 130 cubic yards of stockpiled soil at the referenced site were collected to determine proper disposal of the stockpile. Two composite soil samples (designated as Comp 1 and Comp 2) were taken. Each composite sample consisted of four individual grab samples taken at various locations and depths ranging from one to two feet. The samples were collected in 2" by 4", clean brass tubes, which were then sealed with aluminum foil, plastic caps and tape, and placed in a cooled ice chest for subsequent delivery to a certified laboratory for analysis. All samples were taken to Sequoia Analytical Laboratory in Redwood City, California and were accompanied by properly executed Chain of Custody documentation. Sample locations are as shown on the attached Site Plan.

The composite samples were analyzed to determine concentrations of total petroleum hydrocarbons (TPH) as gasoline using EPA method 5030 or 3810 in conjunction with modified 8015, TPH as diesel using EPA method 3550 in conjunction with modified 8015, benzene, toluene, xylenes and ethylbenzene (BTX&E) using EPA methods 5030 and 8020. The results of the soil analyses showed concentrations of TPH as gasoline as 57 ppm for Comp 1 and 1.4 ppm for Comp 2. TPH as diesel concentrations are 140 ppm for Comp 1 and 9.0 ppm for Comp 2. Copies of the laboratory analyses, and the Chain of Custody forms are attached to this report.

The TPH levels in the soil exceed the action limits set by the Regional Water Quality Control Board (RWQCB).

Should you have any questions on this report, please do not hesitate to contact me at (707) 746-6915.

Sincerely,

Kaprealian Engineering, Inc.

Gary S. Johnson

Gary S. Johnson
Registered Geologist

License No. 4315
Exp. Date 6/30/90

Attachments: Laboratory Results
Chain of Custody

SEQUOIA ANALYTICAL
680 Chesapeake Drive • Redwood City, CA 94063
(415) 364-9222 • FAX (415) 364-9233

Kaprealian Engineering, Inc.
P.O. Box 913
Benicia, CA 94510
Attn: Mardo Kaprealian, P.E.

Date Sampled: 11/07/88
Date Received: 11/08/88
Date Analyzed: 11/20/88
Date Reported: 11/28/88

Project: Berkeley Farms, Hayward

TOTAL PETROLEUM FUEL HYDROCARBONS WITH BTEX DISTINCTION

Sample Number	Sample Description Soil	Low to Medium Boiling Point Hydrocarbons ppm	Benzene ppm	Toluene ppm	Ethyl Benzene ppm	Xylenes ppm
8110906	Composite 1	57	N.D.	N.D.	N.D.	N.D.
8110907	Composite 2	1.4	N.D.	N.D.	N.D.	N.D.
Detection Limits:		1.0	0.05	0.1	0.1	0.1

Method of Analysis: EPA 5030 or 3810/8015/8020

Analytes reported as N.D. were not present above the stated limit of detection.

SEQUOIA ANALYTICAL

Arthur G. Burton
Laboratory Director

SEQUOIA ANALYTICAL
680 Chesapeake Drive • Redwood City, CA 94063
(415) 364-9222 • FAX (415) 364-9233

Kaprealian Engineering, Inc.
P.O. Box 913
Benicia, CA 94510
Attn: Mardo Kaprealian, P.E.

Date Sampled: 11/07/88
Date Received: 11/08/88
Date Analyzed: 11/21/88
Date Reported: 11/28/88

Project: Berkeley Farms, Hayward

TOTAL PETROLEUM HYDROCARBONS

Sample Number	Sample Description Soil	Detection Limit ppm	High Boiling Point Hydrocarbons ppm
8110906	Composite 1	1.0	140
8110907	Composite 2	1.0	9.0

Method of Analysis: EPA 3550/8015

Analytes reported as N.D. were not present above the stated limit of detection.

SEQUOIA ANALYTICAL LABORATORY

Arthur G. Burton
Laboratory Director