ARNESON TIMBER SITE

REMOVAL ACTION WORK PLAN

JUNE 15, 2000

PREPARED BY: LAFSER & ASSOCIATES, INC.

FRED A. LAFSER
PRESIDENT

APPROVED BY: ARNE ARNESON, PRES.
ARNESON TIMBER COMPANY

DATE: 6-15-00

ARNESON TIMBER SITE
REMOVAL ACTION WORK PLAN

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REGISTRY

ARNESON TIMBER SITE

REMOVAL ACTION WORK PLAN

March 30, 1999

Revised June 15, 2000

Prepared by:

Lafser and Associates, Inc.

Fred A. Lafser, President

ARNESON TIMBER COMPANY
Crawford County, Missouri
REMOVAL ACTION WORK PLAN

INTRODUCTION

Site Name and Location

The Arneson Timber company site is located near the top of a ridge in an unincorporated wooded area between Cuba and Steelville, Missouri (figure 1: site location map). It lies along Lucky Clover Road near the intersection of Missouri 19. The legal description of the property in NE 1/4 of SW 1/4 of section 20, T38N, R[illegible] of Crawford County, Missouri. The exact size of the site is [illegible].07 acres.

Site Description

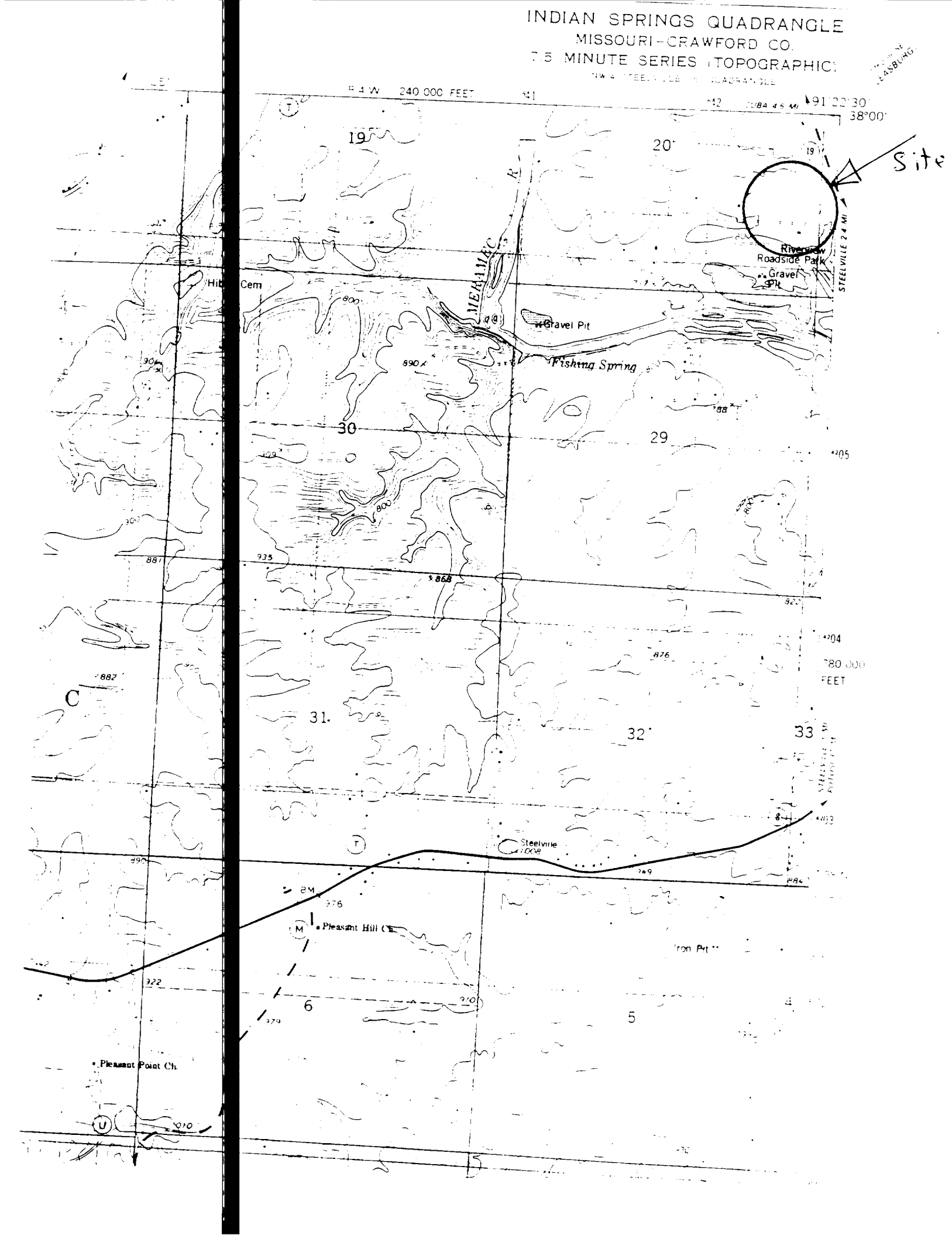
A log cabin once used as an office is the only building on site, several concrete foundations, including a former drip pad are on site. The surface soil is a stony loam 8 to 15 inches deep and classified within the Hobson-Coulston-Clarksville series. The subsoil is a well developed red clay containing iron enrichment derived from the underlying dolomite and sandstone formation. This clay is uniformly distributed throughout the site and is 24" to 48" deep providing a good base liner for the treatment project.

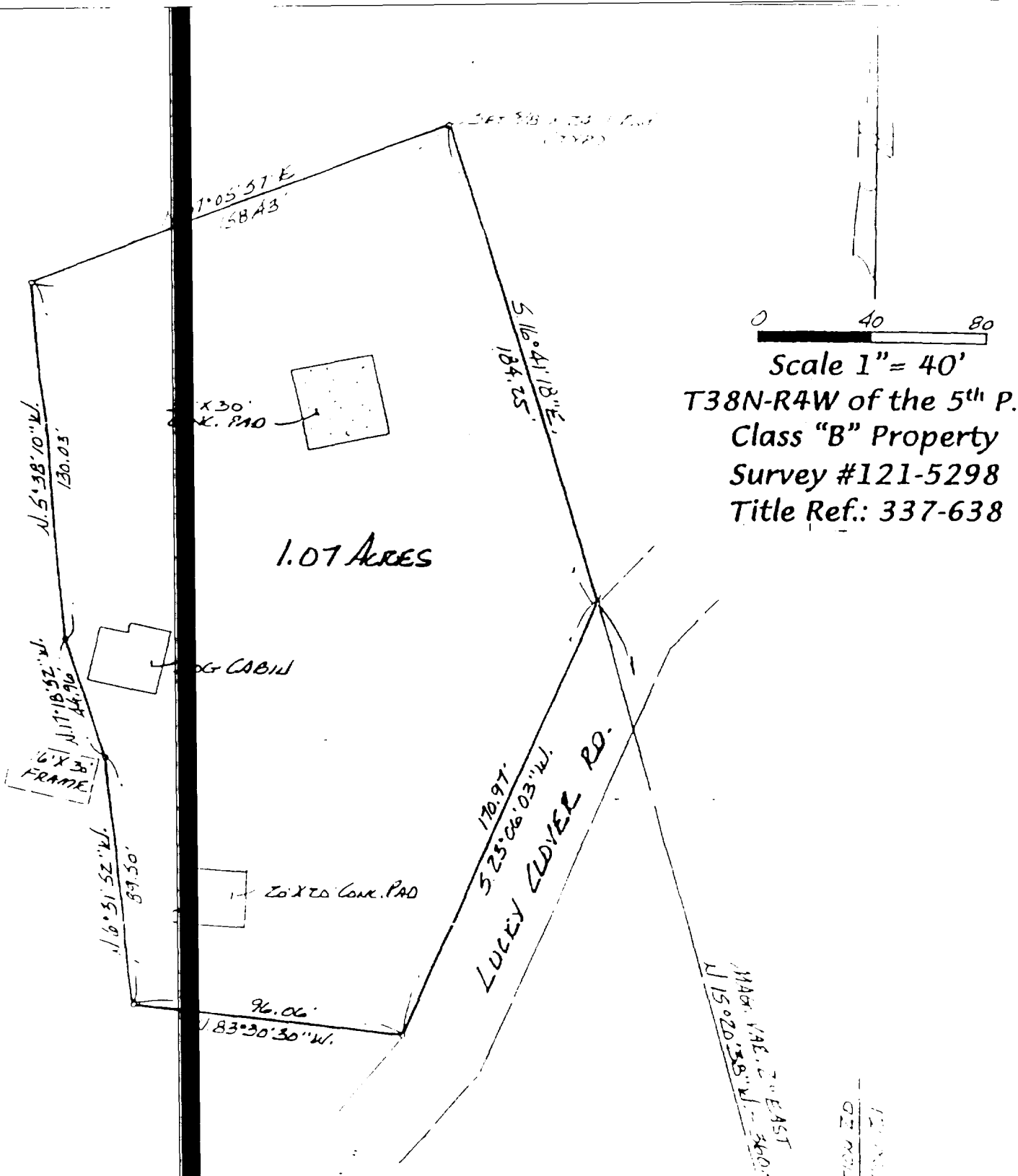
The bedrock statigraphy consists of a series of dolomite and sandstone formations of Ordovician-age overlying similar formations of Cambrian-age. Sandstone outcrops are typical surface features on site. These series of dolomite to sandstone layers overlay approximately 800 feet of water bearing formations.

The site lies in a karst area in which the underlying aquifer is approximately 160-180 feet deep. The Meramec River is 1500 feet south (down slope) from the site.

Site History and Background

In 1978, Arneson Timber Company purchased and began operating a timber cutting and pentachlorophenol (PCP) preservation business at the site. In January 1983, the company moved to an industrial park development in Steelville, Missouri. As part of the move, an


INDIAN SPRINGS QUADRANGLE
MISSOURI-CRAWFORD CO.
7.5 MINUTE SERIES (TOPOGRAPHIC)
91°22'30"
38°00'
Site
Riverview
Roadside Park
Gravel Pit
Gravel Pit
Fishing Spring
Cem
19
20
30
29
31
32
33
6
5
Steelville
Pleasant Hill Ch
Pleasant Point Ch
STEELVILLE 2.4 MI
240 000 FEET



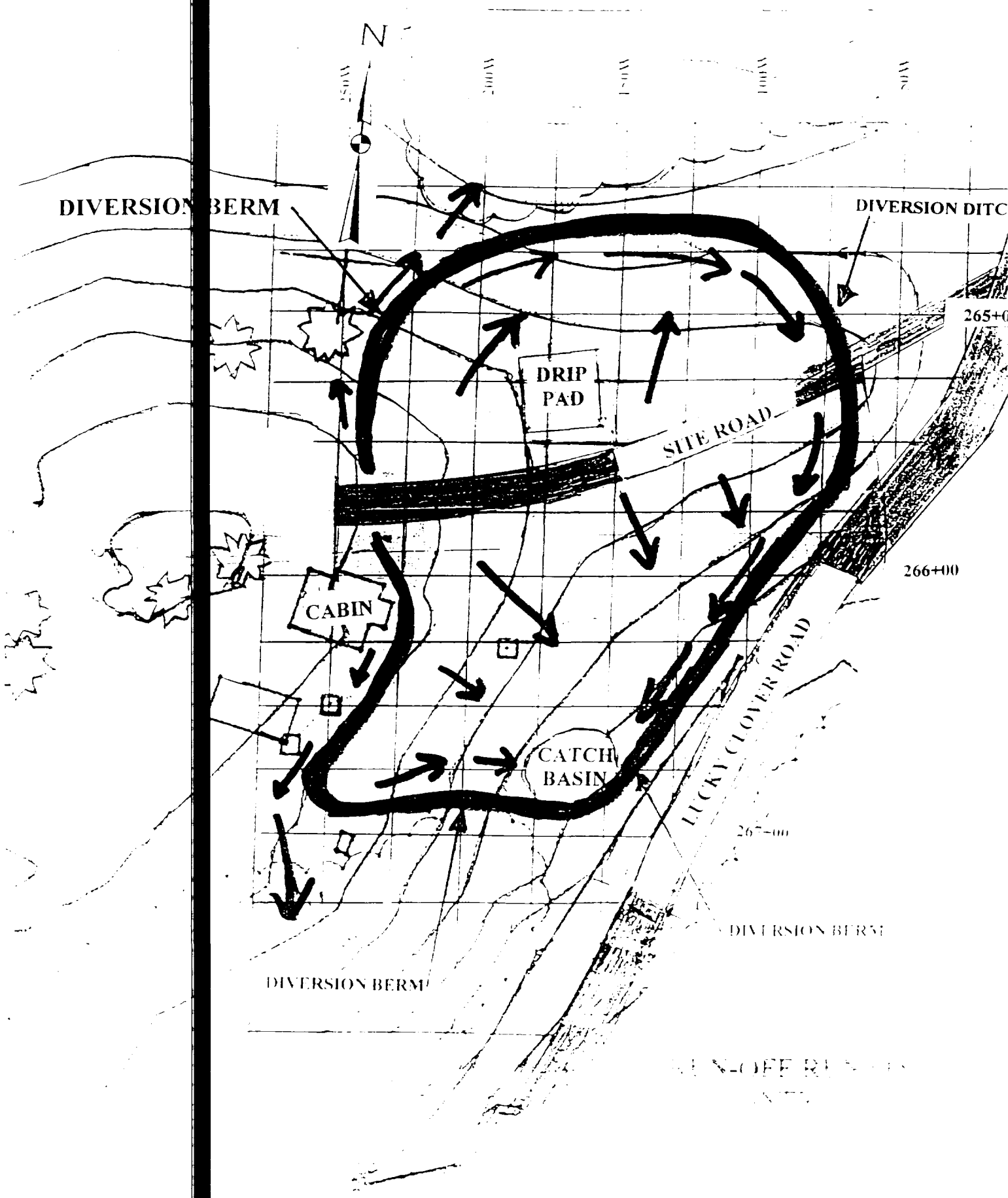
TRACT DESCRIPTION

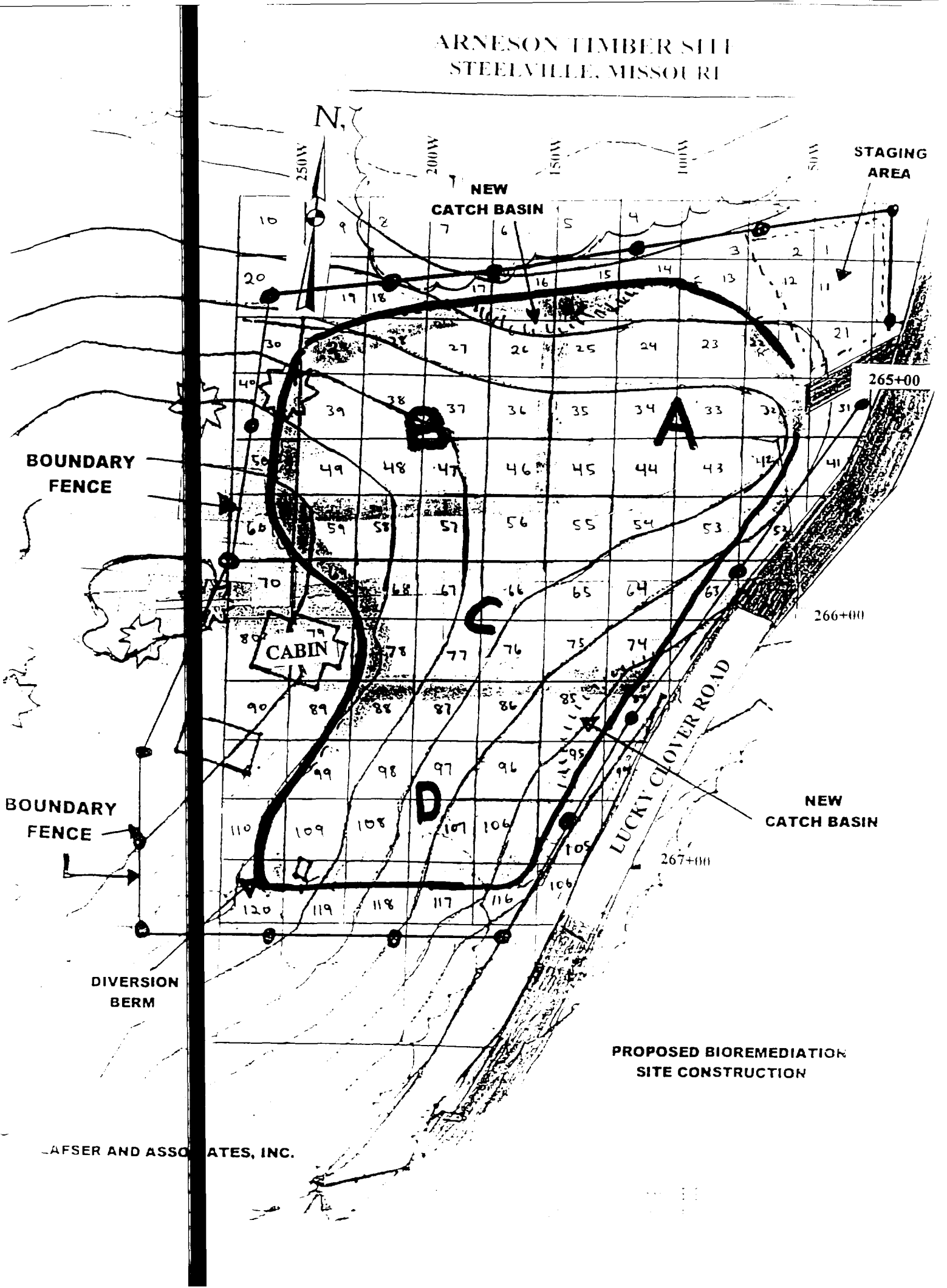
Part of the NE ¼ of the SE ¼ of Section 20, T38N-R4W of the 5th P.M. in Crawford County, Missouri more fully described as follows:

Commencing at the SE Corner of said ¼ ¼ Section; thence N.15°20'38"W. 360.42 feet to the point of beginning; thence S.23°06'03"W. 170.97 feet; thence N.83°30'30"W. 96.06 feet; thence N.6°31'52"W. 89.50 feet; thence N.17°18'52"W. 44.90 feet; thence N.15°38'10"W. 130.03 feet; thence N.67°05'57"E. 158.43 feet; thence S.16°41'18"E. 184.25 feet to the point of beginning. CONTAINING 1.07 acres.

SURVEYORS DECLARATION

This is to declare that I, Mark A. Mueller have during the month of November 1998 by order of Charles N. Leezy executed a survey of the tract of land platted hereon in accordance with the current Missouri Minimum Standards for Property


DIVERSION BERM
DIVERSION DITC
265+0
DRIP PAD
SITE ROAD
CABIN
266+00
CATCH BASIN
LUCKY CLOVER ROAD
267+00
DIVERSION BERM
DIVERSION BERM


ARNESON TIMBER SITE
STEELVILLE, MISSOURI
N
250W
200W
150W
100W
50W
STAGING AREA
NEW CATCH BASIN
265+00
266+00
267+00
BOUNDARY FENCE
BOUNDARY FENCE
CABIN
A
B
C
D
LUCKY CLOVER ROAD
NEW CATCH BASIN
DIVERSION BERM
PROPOSED BIOREMEDIATION SITE CONSTRUCTION
AFSER AND ASSOCIATES, INC.

empty [redacted],000-gallon capacity, above-ground storage tank containing PCP wa[redacted] cut apart and residual sludge and diesel fuel was spread on the [redacted]ground within a containment basin. The containment basin consis[redacted]ed of an open trench-type structure in which the PCP sludge was pl[redacted]ced. For the initial cleanup conducted by Arneson Timber the sa[redacted]e year, the diesel fuel and penta in the containment basin was pu[redacted] in 55-gallon metal drums along with some visibly contam[redacted]nated soil. After the concentrated sludges in the contai[redacted]ment basin were removed, the basin was filled with soil contam[redacted]nated with PCP drippage from treated lumber which was not regula[redacted]ed as a hazardous waste at the time. The area was then graded[redacted] to allow runoff rather than percolation.

The Mi[redacted]souri Department of Natural Resources (MDNR) performed an inspec[redacted]ion cleanup in 1983 and found an oily layer visible on water [redacted]bout two feet below the surface near an area of surficial oil co[redacted]amination. The MDNR requested Arneson Timber to provide furthe[redacted] cleanup of the site. Additional cleanup activities includ[redacted] disposal of two additional 55-gallon drums of contam[redacted]nated soil collected from the catch basin area.

Site A[redacted]essments

On Sep[redacted]mber 20-22, 1983, a CERCLA Preliminary Assessment (PA) was co[redacted]ucted by the Ecology and Environment, Inc., Field Investi[redacted]ation Team (FIT), under contract with EPA. This assessm[redacted]nt included the sampling of five off-site residential water w[redacted]ells. The closest well, located at the David Parker reside[redacted]e, is within 300 feet and down gradient of the containment basin. [redacted]No evidence of contamination due to PCP was found in any of the [redacted]ampled wells.

A site [redacted]nvestigation was conducted by the E & E (FIT) on July 24 and 25, [redacted]1986 and August 4-7, 1986. A geophysical survey, conduct[redacted]d on July 24 and 25, failed to confirm the presence of a tank [redacted] a large metal object alleged to have been buried on site. [redacted]e surface soil samples indicated concentrations ranging form 6.[redacted] to 340 ug/g PCP. The investigation also revealed that off-sit[redacted] migration may have occurred, as surface stream sediment samples[redacted]ielded 2.4 and 5.6 ug/g PCP in intermittent channels adjacen[redacted] to the site. These levels are below the Missouri Departm[redacted]t of Health "any use" determination for this site, which is 9.1 u[redacted]/g PCP. Data for subsurface soil samples indicated PCP concentr[redacted]tions ranging from non-detect to 4.4 ug/g PCP. FIT also collecte[redacted] water samples from the on-site well and the five nearest down gra[redacted]ient private residential wells. All water samples were

non-de[redacted]ect for PCP.

On Jun[redacted]3, 1991, the E & E Technical Assistance Team (TAT) was tasked[redacted]y EPA's Region VII Emergency Planning and Response (EP&RP) Branch[redacted]o conduct a Site Assessment in an effort to further charac[redacted]rize the extent of contamination on site, to determine whethe[redacted]a removal was justified. In addition, that investigation was to[redacted]ollect additional data on concentrations of contaminants in the[redacted]oil and ground water caused by pentachlorophenol's potent[redacted]l contaminants or byproducts, specifically, dioxins and furans[redacted]

The ma[redacted]er of dioxin-contaminated soil was addressed to determine whethe[redacted]the sum of the dioxin and furan isomers detected exceeded 1 ppb [redacted]3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8,-TCDD) equiva[redacted]nts. PCP concentrations in soil above 5 mg/kg (as determi[redacted]ed by EPA Method #8250) or U.S. EPA contract Lab Program (CLP) [redacted]atement of Work (SOW) for Organic Analysis Dioxins and furans[redacted]eported as 2,3,7,8-TCDD equivalents greater than 1 ppb in soi[redacted](as determined by EPA Method #82806) or U.S. EPA Contract Lab Pro[redacted]ram (CLP) Statement of Work (SOW) for Organic Analysis) were t[redacted]be considered significant. Dioxins and furans, reported as 2,3[redacted],8-TCDD equivalents greater than 1 ug/kg in soil (as determi[redacted]ed by EPA Method # 8280) were to be considered signifi[redacted]ant.

The sa[redacted]led area during the 1991 TAT Site Assessment included the former[redacted]ontainment basin where PCP sludges were disposed and the area i[redacted]ediately surrounding it and where there was evidence of soil di[redacted]turbance. A background surface soil sample was collected 20 feet[redacted]west of Highway 19, approximately 800 feet east of the former[redacted]CP catch basin.

The res[redacted]lts of 95 percent upper confidence limit (95) UCLP samplin[redacted]for four 0-to-2-inch soil sample sections of approxi[redacted]ately 5,000 square feet each ranged from non detect to 217 ppm of[redacted]CP. Composite 10-aliquot soil samples in drainage pathway[redacted]showed PCP concentrations ranging from non detect to 34 ppm. S[redacted]bsurface sampling results ranged from non detect to 710 ppm in[redacted]samples collected from 1 to 9.5 feet deep. The eight soil sa[redacted]ples (both surficial and at depth) submitted for 2,3,7,8-TCDD eq[redacted]valent analysis had concentrations from 1.2 to 10 ppb. Duplica[redacted]e soil samples suggested a correlation for PCP and 2,3,7,7[redacted]CDD-equivalent concentrations, as the two highest concent[redacted]ations of PCP, 620 and 710 ppm, showed the highest 2,3,7,8[redacted]CDD equivalents, 10 and 6.5 ppb, respectively. Some

duplicate samples, however, were non detect for PCP, but showed between 2.7 and 3.2 ppb TCDD-equivalents.

In May 1996, the Ecology & Environment, Inc. (E&E) Superfund Technical Assessment and Response Team (START) was tasked by the U.S. Environmental Protection Agency (EPA) Region VII Emergency Response and Removal (ER&R Program under Technical Direction Document (TDD) S07-9603-013 to conduct sampling for pentachlorophenol (PCP) in soil at the Arneson Timber site in Steelville, Missouri. The sampling activities were coordinated with the Missouri Highway and Transportation Department (MH&TD), which was considering realignments and improvements of Missouri Highway 19 and of Lucky Clover Road that could result in construction of the new roadways through portions of the Arneson Timber site. START was tasked to conduct comprehensive surface and subsurface sampling at the site concurrent with surveying activities by the MH&TD. The primary objective was to characterize the areal and vertical extent of PCP contamination to allow for EPA and the MH&TD to calculate volumetric estimates of contaminated soil and to allow for development of options for handling the contaminated soil. In addition to sampling for PCP, a limited number of samples were analyzed for 2,3,7,8-tetrachlorodibenzo-p-dioxin equivalencies (TCDD-Equivalents).

Surveyors established a grid with the starting point at 265+00, 0 on the centerline of the then proposed realigned Highway 19. Point 265+00 designated the north-south coordinate, and 0 designates the east-west coordinate. North-south reference points on the map are 100 feet apart, with the point 100 feet due south of the starting point, being shown as 266+00, for example, and the point midway between that point and the starting point being 265+50. All grids surveyed, as well as subsequent measurements that START made for laying out additional sampling points, were measured from the starting point. The sampling nodes established by the MH&TD survey crew were 50 feet apart. Each sampling point was first measured south (or north) from the starting point and then west, because of the starting point's location northeast of the area of known contamination. As an example, the point 150 feet south and 100 feet west of the starting point was designated 166+50, 100 west. This same grid will be employed for activities in this removal action work plan. START conducted sampling activities on May 28-30, 1996, collecting 69 samples, which ranged from surface samples (0 to 2 inches) to as deep as 9 feet. Seventy-five samples were screened at three different detection levels (2.5,5, & 50 ppm PCP) using EnSys Penta RISC™ kits (some of the 69 samples were screened more than once). Results of

this sampling effort are the most useful for this RAWP and are exhibited on the following pages.

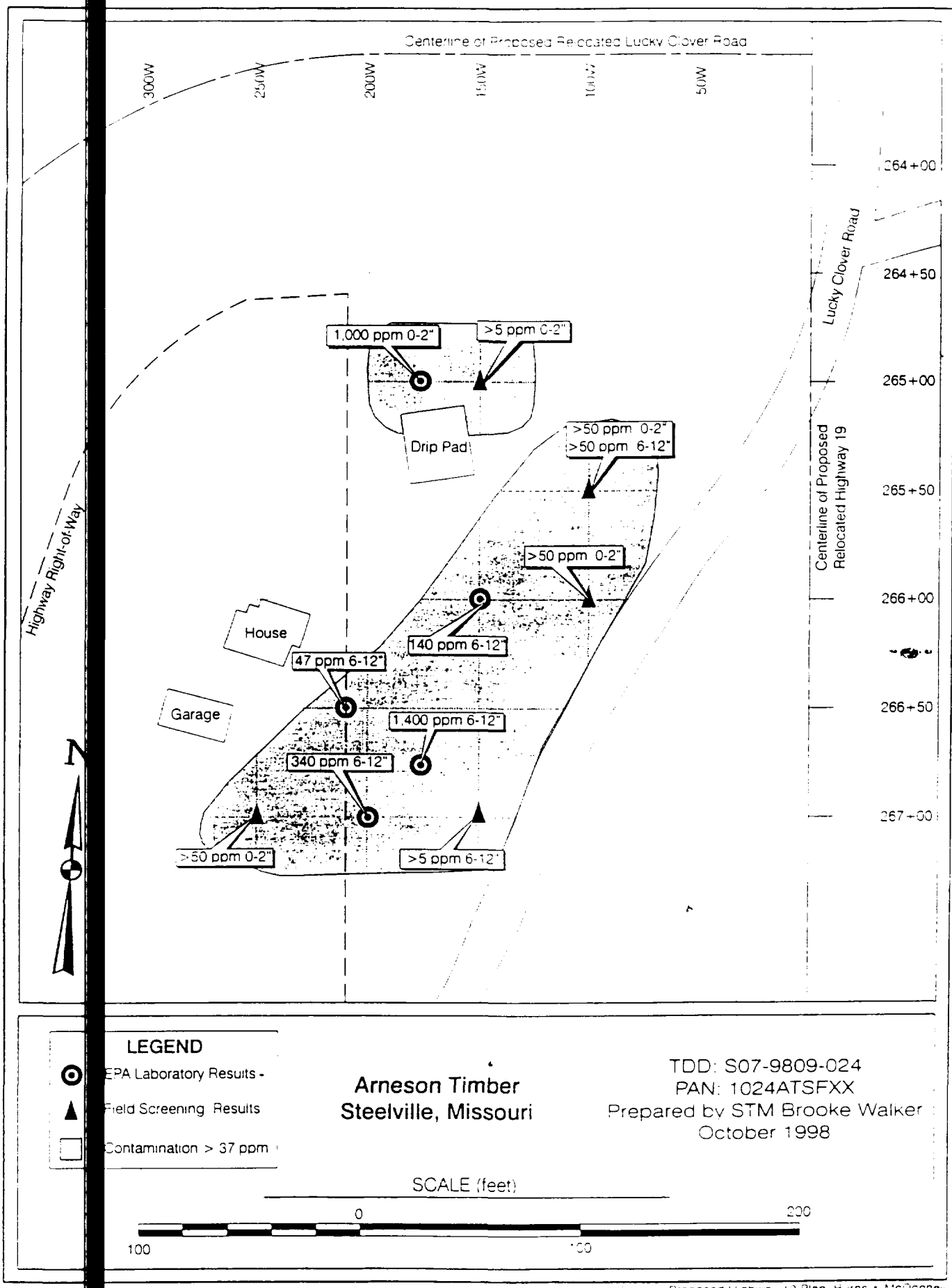


Figure 1: Estimated Area of Contamination At Industrial Action Level of 37 Parts Per Million PCP

Table 1

ARNESON TIMBER SITE
Steelville, Missouri

SUMMARY OF PENTACHLOROPHENOL RESULTS

May 1996 Sampling by EPA START

Table 1

ARNESON TIMBER SITE, STEELVILLE, MISSOURI
SUMMARY OF PENTACHLOROPHENOL RESULTS
TDD #: S07-9603-013/PAN #: 0126ATSFXX

EnSys Screening Number	Lab Analysis Number	Location & Depth	EnSys ≥.5 ppm	EnSys ≥5 ppm	EnSys ≥50 ppm	Lab Data, If Analyzed In mg/kg
6-1	N/A	265 + 00, 200W 6-12 inches	No	No	No	N/A
6-2	N/A	265 + 00, 100W 6-12 inches	No	No	No	N/A
6-3	N/A	W. End of Burned-Out Building	No	No	Yes	N/A
7-1	N/A	266 + 00, 150W 0-2 inches	No	No	No	N/A
7-2	DUI1R 004	265 + 50, 200W 0-2 inches	Yes	Yes	No	25
7-3	DUI1R 002	265 + 00, 175W 6-12 inches	Yes	Yes	Yes	4.5
8-1	N/A	265 + 00, 150W 6-12 inches	No	Yes	No	N/A
8-2	DUI1R 001	265 + 00, 175W 0-2 inches	Yes	Yes	Yes	1,000
8-3	DUI1R 005	265 + 00, 200W 0-2 inches	Yes	No	Yes	94
9-1	DUI1R 025	266 + 50, 175W 6-12 inches	Yes	No	No	2

Key

Shaded Boxes = Indicate PCP Concentration Exceeds Potential Action Level of 5 PPM.
N/A = Sample Not Screened or Analyzed in Laboratory

Table 1

ARNESON TIMBER SITE, STEELVILLE, MISSOURI
SUMMARY OF PENTACHLOROPHENOL RESULTS
TDD #: S07-9603-013/PAN #: 0126ATSFXX

EnSys Screening Number	Lab Analysis Number	Location & Depth	EnSys ≥.5 ppm	EnSys ≥5 ppm	EnSys ≥50 ppm	Lab Data, If Analyzed In mg/kg
9 2	N/A	266 + 00, 175W 6-12 inches	No	No	No	N/A
9 3	DU11R 003	265 + 00, 175W 2-3 feet	Yes	No	No	< 87
9 4 Retest of 6 3	N/A	W. End of Burned-Out Building	No	No	No	N/A
10 1	N/A	266 + 00, 200W 6 12 inches	No	No	No	N/A
10 2	N/A	265 + 00, 150W 2-3 feet	No	No	No	N/A
10 3	DU11R-031	266 + 00, 150W 6-12 inches	Yes	**Yes**	**Yes**	**140**
10 4	N/A	266 + 50, 150W 6-12 inches	No	No	No	N/A
11 1	N/A	265 + 50, 100W 0 2 inches	**Yes**	**Yes**	**Yes**	N/A
11 2	DU11R 011	265 + 50, 150W 0 2 inches	Yes	No	No	< 1 1
11 3	N/A	267 + 00, 150W 6 12 inches	No	**Yes**	No	N/A

Key

Shaded Boxes — Indicate PCP Concentration Exceeds Potential Action Level of 5 PPM.

N/A — Sample Not Submitted for Analysis

Table 1

ARNESON TIMBER SITE, STEELVILLE, MISSOURI
SUMMARY OF PENTACHLOROPHENOL RESULTS
TDD #: S07-9603-013/PAN #: 0126ATSFXX

EnSys Screening Number	Lab Analysis Number	Location & Depth	EnSys ≥.5 ppm	EnSys ≥5 ppm	EnSys ≥50 ppm	Lab Data, If Analyzed In mg/kg
11 4	DU11R 012	266 + 75, 175W 6-12 inches	Yes	Yes	Yes	1,400
12 1	N/A	264 + 75, 175W 6-12 inches	No	No	No	N/A
12 2	N/A	266 + 25, 125W 6-12 inches	No	No	No	N/A
12 3	DU11R 006	266 + 50, 200W 6-12 inches	Yes	Yes	Yes	47 ppm
12 4	N/A	267 + 00, 250W 0-2 inches	No	No	Yes	N/A
13 1	N/A	266 + 00, 250W 0-2 inches	Yes	No	No	N/A
13 2	DU11R 032	265 + 00, 250 0-2 inches	Yes	Yes	No	98
13 3	DU11R 013	265 + 50, 250W 0-2 inches	Yes	No	No	3.2
13 4	DU11R 015	265 + 50, 250W 0-2 inches	No	No	No	1.1
14 1	DU11R 014	267 + 00, 100W 0-2 inches	Yes	No	No	5.9

Shaded Boxes — Indicate PCP Concentration Exceeds Potential Action Level of 5 PPM.
N/A — Sample Not Screened or Analyzed in Laboratory

ARNESON TIMBER SITE, STEELVILLE, MISSOURI
SUMMARY OF PENTACHLOROPHENOL RESULTS
TDD #: S07-9603-013/PAN #: 0126ATSFXX

EnSys Screening Number	Lab Analysis Number	Location & Depth	EnSys ≥.5 ppm	EnSys ≥5 ppm	EnSys ≥50 ppm	Lab Data, If Analyzed In mg/kg
14-2	N/A	264 + 75, 100W 6-12 inches	No	No	No	N/A
14-3	N/A	264 + 75, 200W 6-12 inches	Yes	No	No	N/A
15-1	DU11R 016	265 + 25, 125W 6-12 inches	Yes	Yes	Lost	12
15-2	N/A	266 + 00, 100W 0-2 inches	Yes	Yes	Yes	N/A
15-3	DU11R 017	266 + 50, 100W 0-2 inches	Yes	Yes	No	< 82
15-4	DU11R 007	267 + 00, 200W 0-2 inches	Yes	Yes	Yes	11
16-1	DU11R 018	267 + 00, 150W 2-3 feet	Yes	Yes	No	25
16-2	DU11R 008	267 + 00, 200W 6-12 inches	Yes	Yes	Yes	340
16-3	DU11R 019	266 + 50, 225W 6-12 inches	No	No	No	< 37
16-4	DU11R 033	267 + 00, 100W 6-12 inches	No	No	No	< 94

Key:

Shaded Boxes Indicate PCP Concentration Exceeds Potential Action Level of 5 PPM.

ARNESON TIMBER SITE, STEELVILLE, MISSOURI
SUMMARY OF PENTACHLOROPHENOL RESULTS
TDD #: S07-9603-013/PAN #: 0126ATSFXX

EnSys Screening Number	Lab Analysis Number	Location & Depth	EnSys ≥.5 ppm	EnSys ≥5 ppm	EnSys ≥50 ppm	Lab Data, If Analyzed In mg/kg
17-1	DU11R 020	266 + 50, 200W 3-4 feet	Yes	Yes	No	< 96
17-2	DU11R-021	267 + 00, 150W 5-6 feet	Yes	No	No	< 1
17-3	DU11R 009	266 + 00, 200W 2-3 feet	No	No	No	< 88
17-4	N/A	265 + 50, 100W 6-12 inches	No	No	Yes	N/A
18-1	N/A	267 + 00, 200W 5-6 feet	No	No	No	N/A
18-2	N/A	266 + 75, 225W 3-4 feet	No	No	No	N/A
18-3	N/A	267 + 00, 130W 5-6 feet	No	No	No	N/A
18-4	N/A	265 + 50, 50W 6-12 inches	No	No	No	N/A
19-1	DU11R 022	266 + 62, 162W 9-10 feet	No	No	No	< 9
19-2	N/A	267 + 00, 130W 3-4 feet	No	No	No	N/A

Key:

[illegible] Exceeds Potential Action Level of 5 PPM

Table 1

ARNESON TIMBER SITE, STEELVILLE, MISSOURI
SUMMARY OF PENTACHLOROPHENOL RESULTS
TDD #: S07-9603-013/PAN #: 0126ATSFXX

EnSys Screening Number	Lab Analysis Number	Location & Depth	EnSys ≥.5 ppm	EnSys ≥5 ppm	EnSys ≥50 ppm	Lab Data, If Analyzed In mg/kg
19-3	N/A	265 + 00, 225W 6-12 inches	No	No	No	N/A
19-4	DU11R-023	266 + 50, 125W 6-12 inches	Yes	Yes	No	8.9
20-1	DU11R-010	265 + 50, 200W 6-12 inches	Yes	No	No	1.9
20-1 Dupe	DU11R-010	265 + 50, 200W 6-12 inches	Yes	No	No	1.9
20-3	DU11R-024	265 + 00, 250W 6-12 inches	No	No	No	< .91
20-3 Dupe	DU11R-024	265 + 00, 250W 6-12 inches	No	No	Lost	< .91
Not Screened	DU11R-026	266 + 75, 225W 6-7 feet	N/A	N/A	N/A	< .91
Not Screened	DU11R-027	264 + 75, 175W 2-3 feet	N/A	N/A	N/A	< .86
Not Screened	DU11R-028	267 + 00, 250W 6-12 inches	N/A	N/A	N/A	1.1

Key

Shaded Boxes - Indicate PCP Concentration Exceeds Potential Action Level of 5 PPM.

N/A - Sample Not Screened or Analyzed in Laboratory

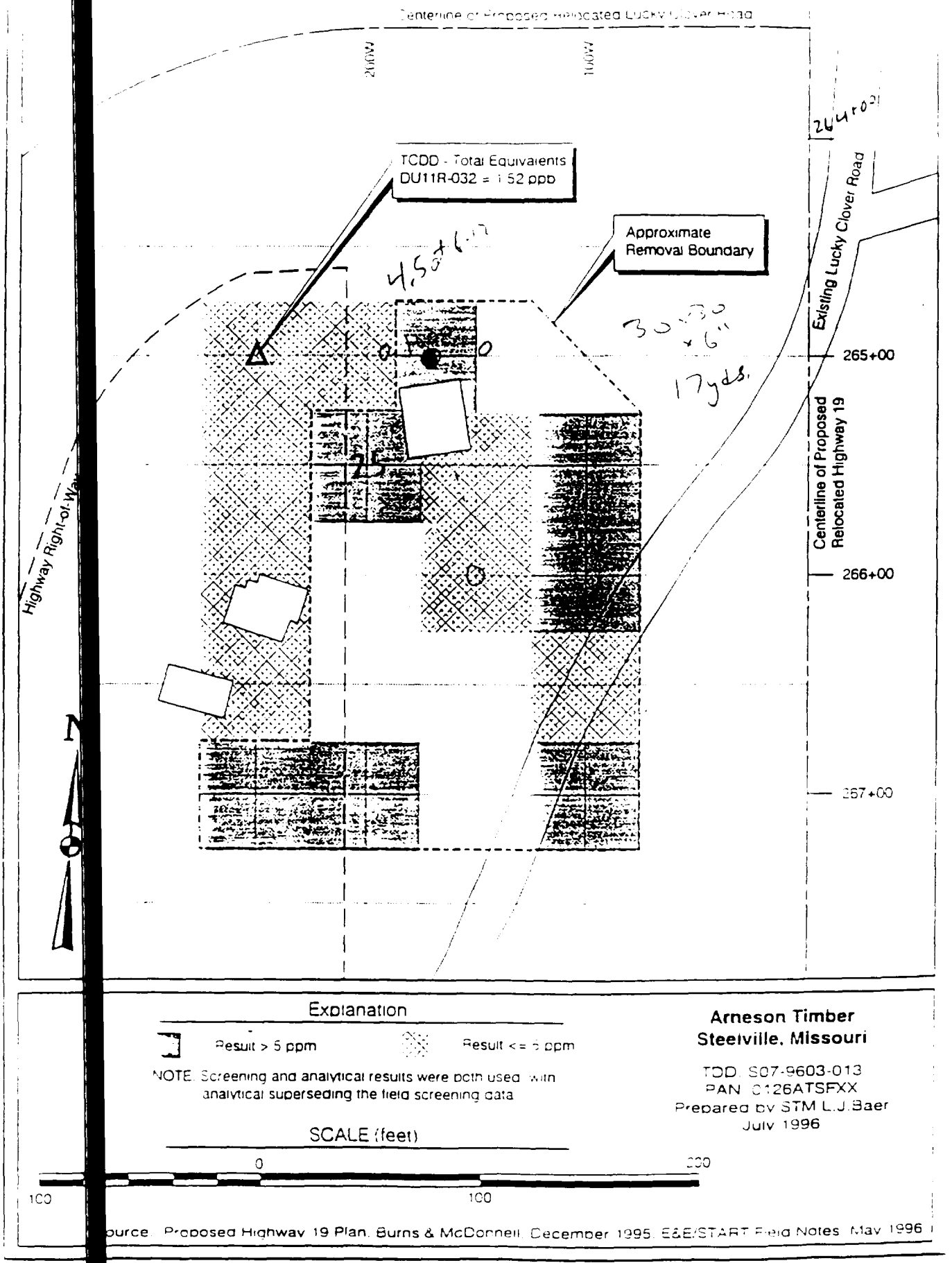
Table 1

ARNESON TIMBER SITE, STEELVILLE, MISSOURI
SUMMARY OF PENTACHLOROPHENOL RESULTS
TDD #: S07-9603-013/PAN #: 0126ATSFXX

EnSys Screening Number	Lab Analysis Number	Location & Depth	EnSys ≥.5 ppm	EnSys ≥5 ppm	EnSys ≥50 ppm	Lab Data, If Analyzed In mg/kg
Not Screened	DU11R-029	Riverview Park Background 0-2 inches	N/A	N/A	N/A	< 93
Not Screened	DU11R-030	Riverview Park Background 2-3 feet	N/A	N/A	N/A	1 8
Not Screened	DU11R-036	Rinsate	N/A	N/A	N/A	Nondetect

Key

Shaded Boxes - Indicate PCP Concentration Exceeds Potential Action Level of 5 PPM.



-TS_FIGS.CDR

Figure 3: SURFACE (0-2") SAMPLE RESULTS

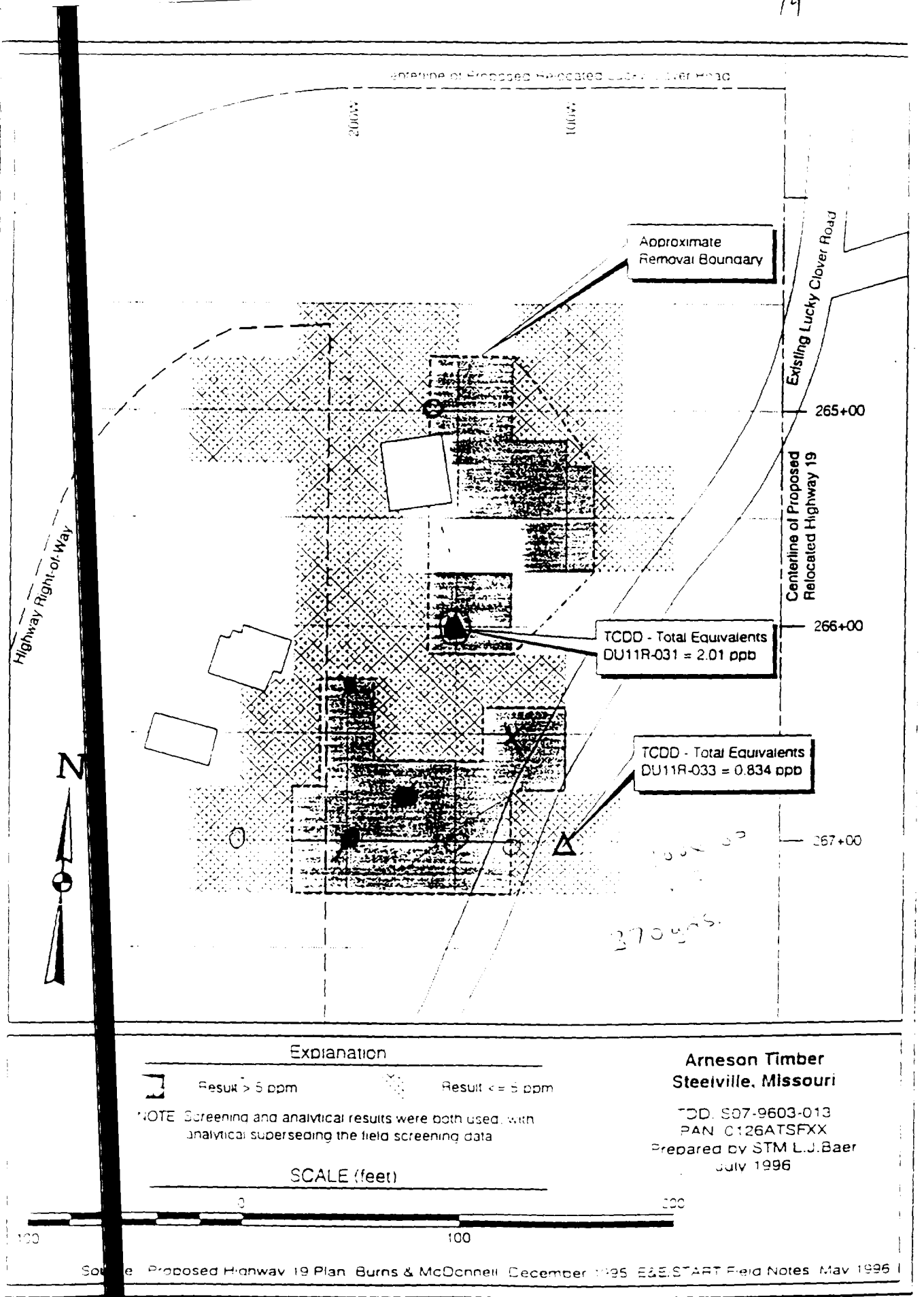


Figure 4: NEAR-SURFACE (6-12") SAMPLE RESULTS

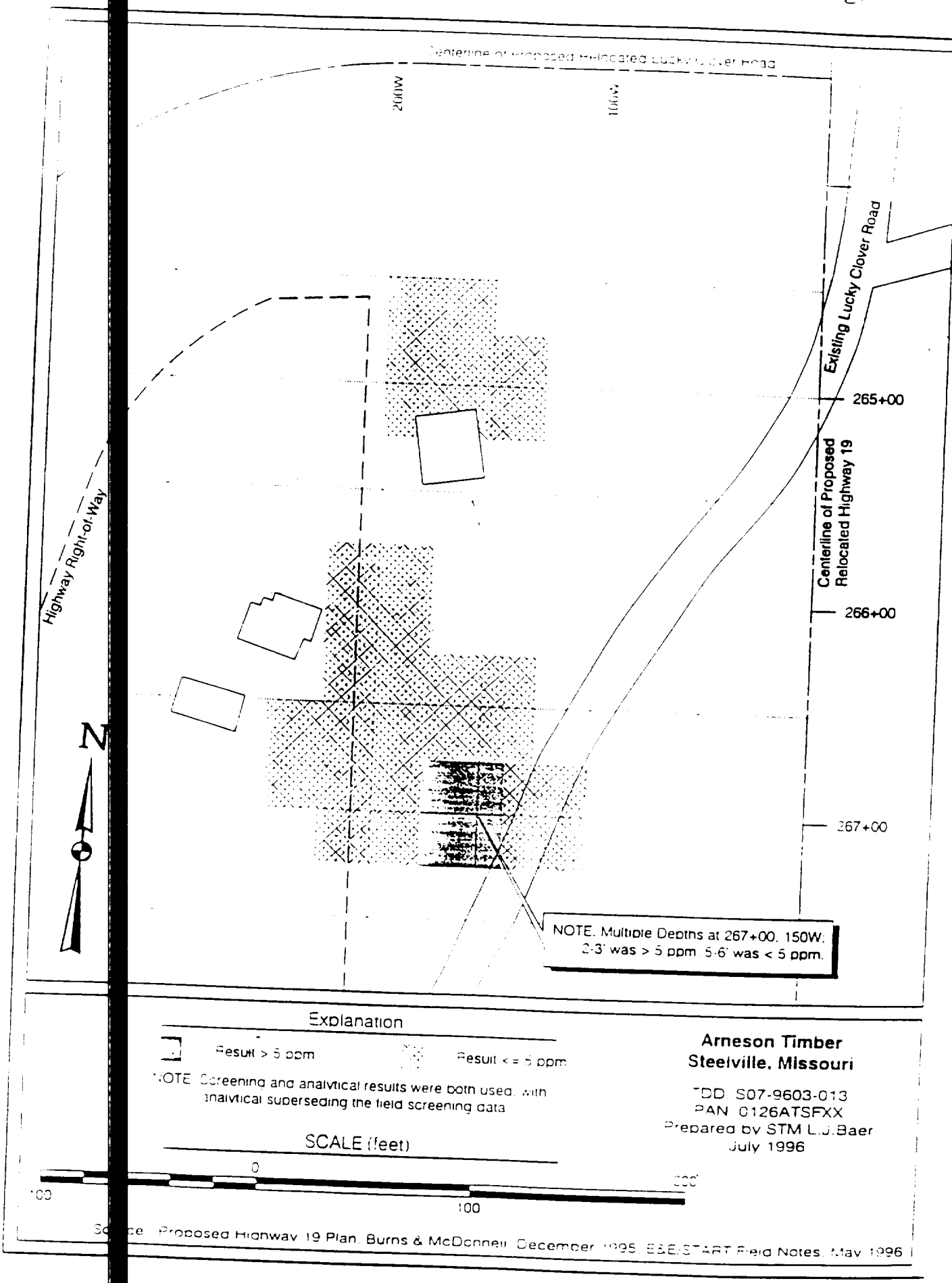


Figure 5: SUBSURFACE (>12") SAMPLE RESULTS

Site Organization

Survey markers still exist from the 1996 EPA START project site work and the same grid will be employed for this RAWP. To encompass a larger area to the North, the starting point will be point 264+00, OW. This is a point immediately north of the site access road as it intersects Lucky Clover Road. The reference points 264, 265, 266 are north-south points, 100 feet apart. Points between reference points are designated, as an example 265+75' to indicate a point 75' south of the reference point 265. The east-west locations are measured from the starting point moving to the west. A point 250' south of the starting point and 150' west is identified as 266 50', 150 w. Survey markers are still in place at most of the 50' intervals.

A grid numbering system is established to assist with the management of the site. The grid consists of cells 25' X 25' and are numbered 1 through 120, as shown on the "Proposed Bioremediation Site Construction Map". For reference example, point 265+00, 150 W is the south east corner of Cell 66. The site is also divided into four Sampling Areas, A,B,C, AND D.

Removal Strategy

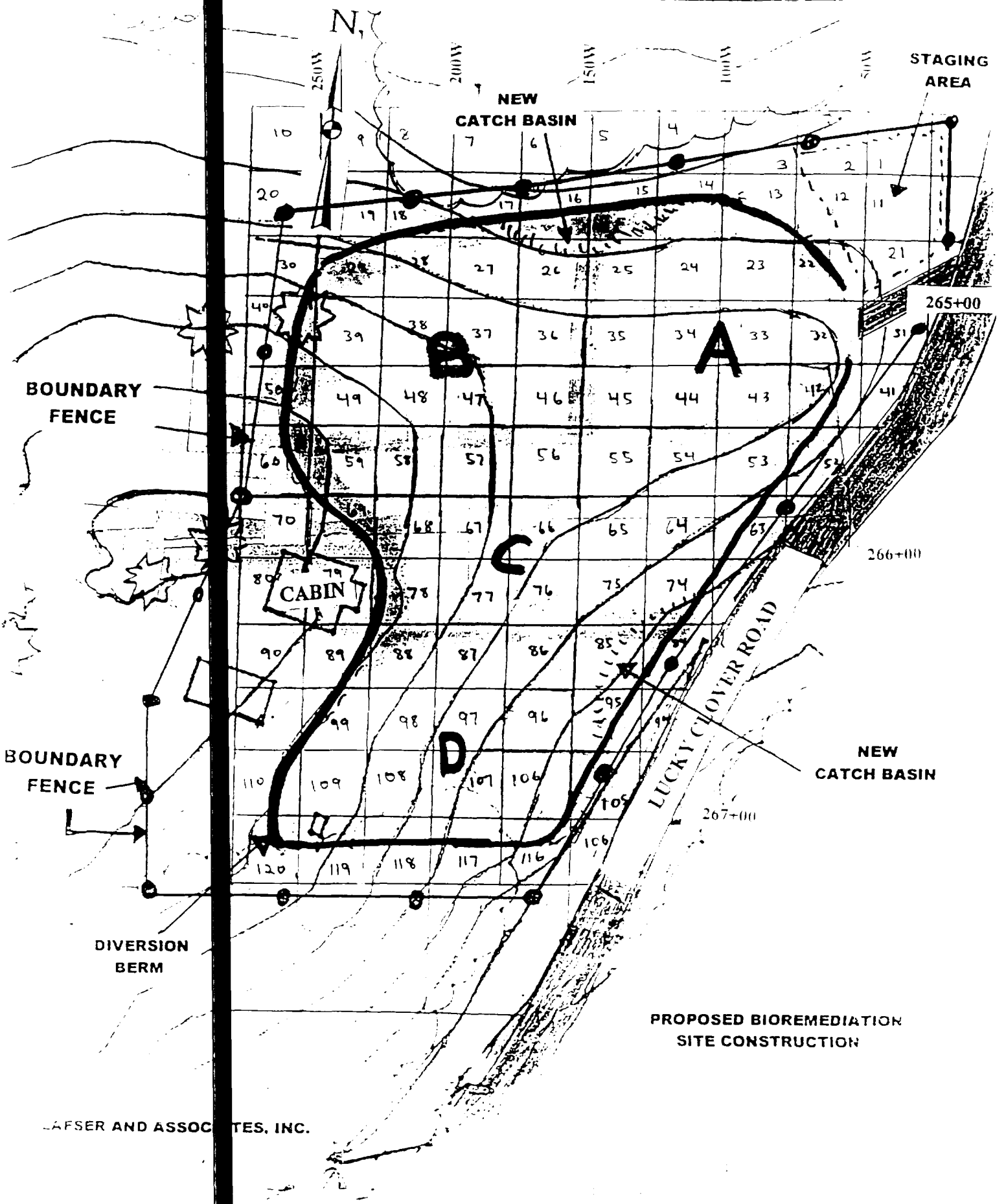
Site office and clean zone (staging area) will be established in the extreme north east corner of the site. No contamination has been detected in this area. A transition zone and decontamination area will be established adjacent to the staging area. There are two known areas of elevated PCP concentrations:

- the location immediately north of the former drip pad, near point 265+00;
- the location of the former catch basin, near point 267+00, 165 W; 175 W, which likely contains contamination too deep for biological activity (greater than one foot).

A diversion ditch and /or berm will be constructed north of the drip pad to insure that any run-off water from contamination north of the site ridge road will eventually flow to the site north catch basin. The north catch basin will be constructed approximately 75 feet north east of the drip pad. All material north of the site ridge road will drain to the north catch basin.

A berm will be constructed along the west boundary of the contamination to prevent run-on. A berm/diversion ditch will be

ARNESON TIMBER SITE
STEELVILLE, MISSOURI



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constructed from the site access road to the south along the eastern boundary of the site.

A south catch basin, approximately 4 to 6 feet deep and 30 feet x 30 feet will be constructed in the southeast corner of the site at the lowest elevation, approximately 165 feet southwest of the entrance gate.

The first activity was the breaking up of the concrete drip pad at point 2[illegible]5 +25, 175 W in March, 2000. This was accomplished with a jack hammer attachment to a backhoe. The broken concrete and gravel were stockpiled on site. This material will be tested and used on site as fill or further broken up and incorporated into the treatment unit. Sampling and testing revealed PCP contaminated soil under the drip pad at levels of 0 to 66 mg/kg. Since the site of the catch basin is the only location with contamination above 9.1 ppm below 1 foot, this area will be excavated further until test results confirm levels of PCP below action levels. Once the gross contamination is removed, field sampling will be performed to determine if the soils remaining are above the 9.1 ppm or 37.7 ppm action levels. Areas field tested as clean will be sampled per the site QASP and sent to the laboratory for confirmation. The contaminated soil will be stockpiled north of the catch basin location near point 2[illegible]6 +25, 150 W.

The next phase of activity will be to develop a portion of the site of approximately 35,000 square feet into a landfarm unit for bio-remediation of the remaining PCP.

The approximate boundaries of the unit are shown on the Proposed Bioremediation Site Construction Map. Clean soils will be used to develop an up-gradient water diversion berm to prevent water "run-on" which could encourage erosion or other water management problems. A down-gradient berm will also be established with clean soil to control "run-off" from the site. At the two low points in the site the berms will create the north and south catch basins. If the removal activities extend more than 3-4 feet below current surface, clean soil will be used to fill the hole. The intent is to engineer and create run-off storage capacity to contain a two-year storm event and to reduce the need for discharge monitoring. A secondary purpose of the detention structure is to create a reservoir of water for land farm irrigation. Straw bales will be placed up gradient of the catch basin to capture silt erosion.

The concrete pad was destroyed in March, 2000. The top layer was stockpiled on site. the pad was poured on top of an older concrete pad and gravel fill. There was no visible contamination and surface residue samples confirmed non-detectable levels of PCP. This top material was segregated and an application will be submitted to MDNR for permission to use the material on site as fill. Alternatively, the material will be further crushed and integrated into the treatment unit along with the lower pad and gravel.

The next phase will be to spread the stockpiled PCP contaminated soils evenly throughout the land farm unit using a small bulldozer with a ripper. The site will be graded during which time the soils will be homogenized. Final passes will create small wind rows along topographic contours to minimize erosion.

Once established, the land farm unit will be monitored and managed according to the Site Biotreatment Plan. Further, the heavy equipment will be decontaminated and moved from the site once the site is developed.

Project Management

Respondent Mr. Charles Leezy is responsible for providing site access, perimeter fencing and placement of the site on the state register.

Respondent Arneson Timber Company, President-Arne Arneson, is responsible for contracting services required to implement the RAWP and for providing the necessary financial resources.

The Project Manager will be Fred Lafser,of Lafser And Associates, Inc. He will report directly to Mr. Arneson. Due to the small nature of this site, he will also serve as Quality Assurance Officer and Site Safety Office. David Schau, of Ecosafe, Inc., will provide backup services.

The Construction Supervisor will be Mr. K.R. Wilson of K.R. Wilson Contracting, Inc.

P.O. Box 517 South Service Road
Sullivan, Mo 63080
(573) 468-3005

He will report to the Project Manager. The construction personnel will be employees of Mr. K.R. Wilson and will report directly to him.

Project Schedule

Weather permitting, the removal action will commence as soon as possible, and no later than sixty days after EPA approval of the RAWP. The initial phase will be the removal of the concrete structures and sampling of the exposed area.

A period of forecasted dry weather will trigger site mobilization. It is anticipated that all site construction activities and concrete removal will be completed within seven days, assuming good weather and the absence of unforeseen conditions.

The Bioremediation Plan is supported by a limited front-end study. The land farm will be constructed within sixty days of EPA approval of the RAWP. This will allow a full season of warm months (May through October) for optimal biological activity.

If this schedule is delayed and commencement of construction activities occurs in September, or later, the soils would then be exposed to wind and weather throughout the winter with minimal biological activity. If this occurs, construction should be delayed until the following spring.

Project Schedule

February 4, 1999	Request placement of site on State Registry
February 9, 1999	Effective date of the Order
March , 1999	Feasibility Study (BRFS)
March , 1999	Submittal of draft RAWP
March , 1999	Preliminary comments on RAWP from EPA; In depth review to follow
May 19	Fence erected to control access
May 5, 999	Approval to landfill obtained
May 12 1999	Land ban rule effective
June 2 1999	Site placed on State Registry
August 1999	Submit BRFS to EPA
Decemb , 1999	Submit final RAWP to MDNR and EPA
March , 2000	Begin site construction; removal of concrete drip pad, test
April , 2000	Obtain results of soil tests under pad
June 1 2000	Submit revised RAWP
July 1 2000	Complete construction of land farm, begin management and monitoring of land farm
August , 2000	Submit post construction inspection and report
August 5, 2000	Notify EPA of final inspection readiness

Septem	er 15, 2000	Submit removal action completion report
Octobe	15, 2000	Conduct baseline sampling
Octobe	2001	Anticipated completion and closure

Site P eparation Activities

Site s curity is primarily a three-strand barbed-wire fence constr cted by Mr. Leezy around the perimeter of the site. The fence ncompasses at least a 100 foot buffer around the 1.07 acre site, xcept where it follows the right of way of Lucky Clover Road. A gate with a lock is installed. The fence will have the signag required by the ORDER: A larger area to the northeast of the si e is included for a support area.

The bo ndary of the contaminated zones as identified by EPA START sampli g effort in May 1996, are delineated with wood posts with barbed wire. The grid will be established with wood stakes on the perime er with white paint and the point coordinates labeled. The transi ion and decontamination zone will be delineated with wood stakes painted blue. Plastic tape will delineate the boundaries.

Site c eaning and grubbing will not be necessary if project start- p is before June 1, 1999. After that time, vegetative growth will need to be managed. Any vegetation cut within the site b undary will be chipped on site and used to augment the biolog cal treatment.

On the "Proposed Bioremediation Site Construction Map" the 25' X 25' ce ls are labeled from 1 to 120 starting in the north east corner f the grid. Excavation will only occur in the area of the old ca ch basin, in cells 96,97,106 and 107. We estimate 220 cubic ards will be excavated before a "clean" bottem is encoun ered, or a maximum four feet depth is reached. This materi l will be spread in the lightly contaminated area in approx mately 19 cells in the north and east portion of the site. The ma erial will be spread in six inch or smaller lifts. The remain er of the site will be ripped, disked and redistributed withou significant translocation.

When c lls 96, 97, 106 and 107 are tested to be below action levels clean material wil be used to fill the hole. The borrow area i the west edge of the site.

The Wa hington University study recommends a 50 to 100% ratio of additi e manure and sawdust. Due to the handling and mixing challe ges, the contractor has requested that this material be

incorporated over a period of several months. It should rapidly decay so that the ultimate increase in soil mass is 5% to 10%. There is a distinct soil color and texture change at 12 inches from the gravelyloam to the red-brown clay. This will assist with the mixing and working of the top 12 inches of contaminated material.

Fugitive Dust and Air Monitoring

A flag or windsock will be employed to monitor wind direction. A portable water source will be available with a spray mist capability to control fugitive dust during construction. The project manager will determine the need for and direct the use of the water spray. If soil moisture and wind conditions are such that the water spray is ineffective, the project will be shut down immediately until the conditions are corrected. Personal high volume air samplers will be employed on one employee and at a perimeter down wind location.

Sampling Criteria for Excavation

Excavation activities are proposed for the area of the former catch basin. All visible stained soils will be excavated from the area and distributed in the landfarm. It is anticipated that the area will less than 50' x 50'. Six (6) random aliquots will be composited as an extended surface sample (0-6 inches). The sample will be homogenized and split into four quarters. One sample will be sent to the laboratory for testing and one sample split will be provided to EPA. The others will be a back-up samples and will be returned to the sampling location if not needed.

If the test indicates contamination above 37.7 mg/kg, the excavation with placement of material in other cells will be continued for and the sampling process repeated. This will continue until the cleanup goal is achieved. The respondent may choose to continue institutional controls if the clearance testing is below 37.7 mg/kg but greater than 9.1 mg/kg and cap the area. The respondent may choose to remove additional material to acheive levels below 9.1 mg/kg.

DIOXIN EQUIVALENTS

Dioxin equivalents have been detected at three locations. In the North west corner of cell 39, 1.5 ppb was detected at 0-2". The top 12 inches of this area will be moved and distributed to cells 37,38 and 47 for biotreatment.It is below the 10 ppb action level for industrial sites.

Levels of 2 ppb were detected in the southeast corner of the cell 66, also below the action level of 10 ppb. This material will be treated in place. The other area of detection was at Lucky Clover road in the southeast corner of cell 104. This level was 0.8 ppb and is below the any use action level of 1.0 ppb. No action is required or proposed. No dioxin testing is proposed until final clearance.

Air Monitoring

Air monitoring will be limited due to the short term nature of soil disturbance activities. Since the PCP is bound in oil and to soil, particulates will be monitored using a personal high-volume air sampler. An action level based on the OSHA, PEL (Permissible Exposure Limits) for Penta (0.5 micrograms per cubic meter) is provided to provide a threshold particulate level to trigger engineering controls (water spray) and a higher level of respiratory protection. Since the Dioxin Equivalents are below the 10 ppb industrial level and site activity will be relatively brief (less than 20 days), a level of concern for Dioxin equivalents will not be established.

The personal air monitors will be located on the perimeter of the site and on the employee with greatest exposure risk. The nearest potential receptor from the site is the residence 100 yards to the south, upway form the prevailing winds and buffered by a forested area. Pentachlorophenol/OSHA Method 39 will be employed. Equipment and testing will be provided by Data/Analysis Technologies, Inc.

Sampling and Analytical Procedures

Field sampling will be in a manner consistent with the "Superfund Program Representative Sampling Guidance:, Volume I: Soil, December 1995. Laboratory testing for soils will be by Enviroanalytics, Inc. in St. Louis, Missouri, using Method 8270C for PCP. Laboratory testing for air samples will be by Data/Analysis technologies, Inc. using Method 39. The EPA Methods and QA/QC Manuals follow.

Control of Fugitive Dust

Fugitive dust will first be minimized by assessing the adequacy of moisture levels in soils prior to start of construction. If soils are dry, and/or wind speeds raise concerns, earth disturbing activities will cease until water is applied to the site to reduce fugitive dust. If the water does not reduce particulate levels adequately, the activity will be shut down until the water controls are adequate or the wind velocity is reduced.

Transportation

(not applicable)

Environmental Regulatory Requirements

Missouri DNR and EPA will be notified and provided with copies of this RAWP. A NPDES operating Permit has been applied for. MDNR advised that no construction permit is required. To our knowledge, there are no other environmental regulatory permits required.

Establishment and Closure of Bioremediation Cell

The establishment of the landfarm cell was detailed previously. Landfarming has been chosen in order to simplify and enhance aeration and management due to the smallness of the site (less than one acre). Once the site has been documented and approved as meeting treatment objectives through the collection and testing of groundwater and soil samples, a final grading will be conducted to eliminate the site berms and effectively blend the site into the topography of the surrounding land. The site will be planted with appropriate perennial vegetation to control erosion and meet the desires of the landowner. A seed mix of a variety of grasses and legumes is anticipated.

QUALITY ASSURANCE PROJECT PLAN
for the
ARNESON TIMBER SITE,

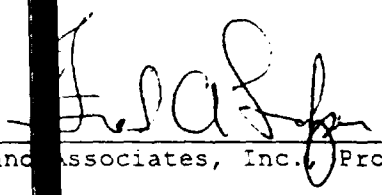
Steelville, Missouri

Lafser and Associates, Inc.

December 1, 1999

Revised, June 15, 2000

APPROVED:



Lafser and Associates, Inc., Project Manager — 6-15-00
Date

EPA Region VII Site Manager — Date

Site Description

The Arneson Timber Company site is located near the top of a ridge in an unincorporated wooded area between Cuba and Steelville, Missouri (figure 1: Site location map). It is bounded on the south-southeast by Lucky Clover Road, approximately 100 yards west-southwest of the intersection of that road and Missouri Route 19. The property is within the NE 1/4 of SW 1/4 of Section 20, T38N R4W of Crawford County, Missouri. The size of the site is 1.07 acres.

One building, a log cabin that once was used as an office building and various cement foundations (including a drip pad), are on the site. The surface soil is a stony loam 8 to 15 inches deep that is classified within the Hobson-Coulstone-Clarksville series. The subsoil is a well-developed red clay containing iron enrichment derived from the underlying dolomite and sandstone formation. Sandstone outcrops are typical surface features on the site. The site is in a karst area with the underlying aquifer approximately 160-180 feet below ground surface. The Meramec River is 1,500 feet south (down gradient) of the site.

Site History

In 1978, Arneson Timber Company purchased and took over the operation of a timber cutting and pentachlorophenol (PCP) preservation business at the site. In January 1983, the company moved to an industrial park development in Steelville, Missouri. As part of the move, an empty 9,000-gallon capacity, above-ground storage tank containing PCP was cut apart and residual sludge and diesel fuel was spread on the ground within a containment basin. The containment basin consisted of an open trench-type structure in which the Phenol-containing sludge was placed. For the initial cleanup conducted by Arneson Timber the same year, the diesel fuel and penta in the containment basin was placed in nine, 55-gallon metal drums along with some visibly contaminated soil. After the concentrated sludges in the containment basin were removed, the basin was filled with soil contaminated with PCP drippage from treated lumber which was not regulated as a hazardous waste at the time. The area was then graded to allow runoff rather than percolation.

The Missouri Department of Natural Resources (MDNR) performed an inspection of the cleanup in 1983 and found an oily layer visible on water about two feet below the surface near an area of surficial soil contamination. The MDNR requested Arneson Timber to provide further cleanup of the site. Additional cleanup

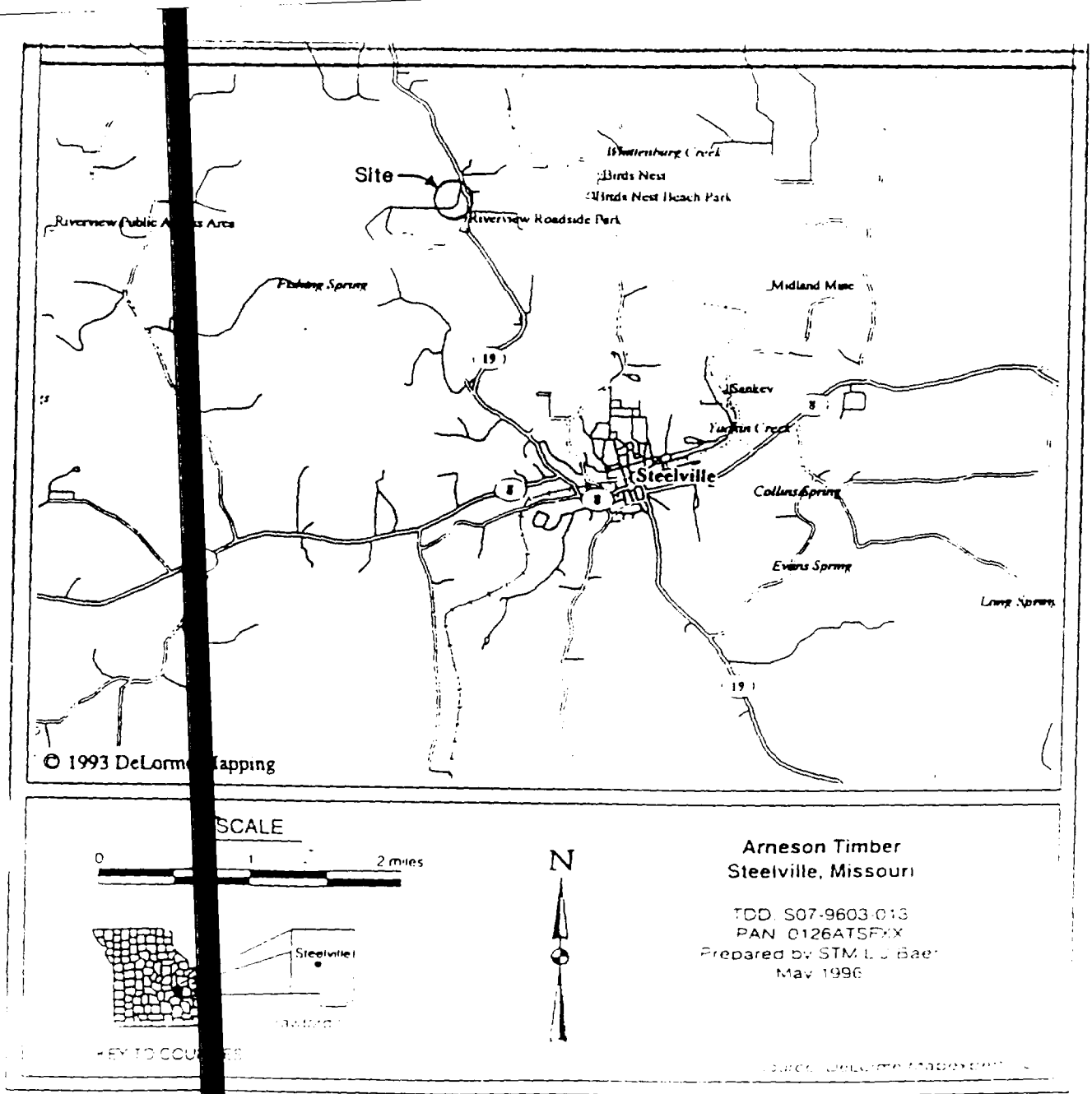
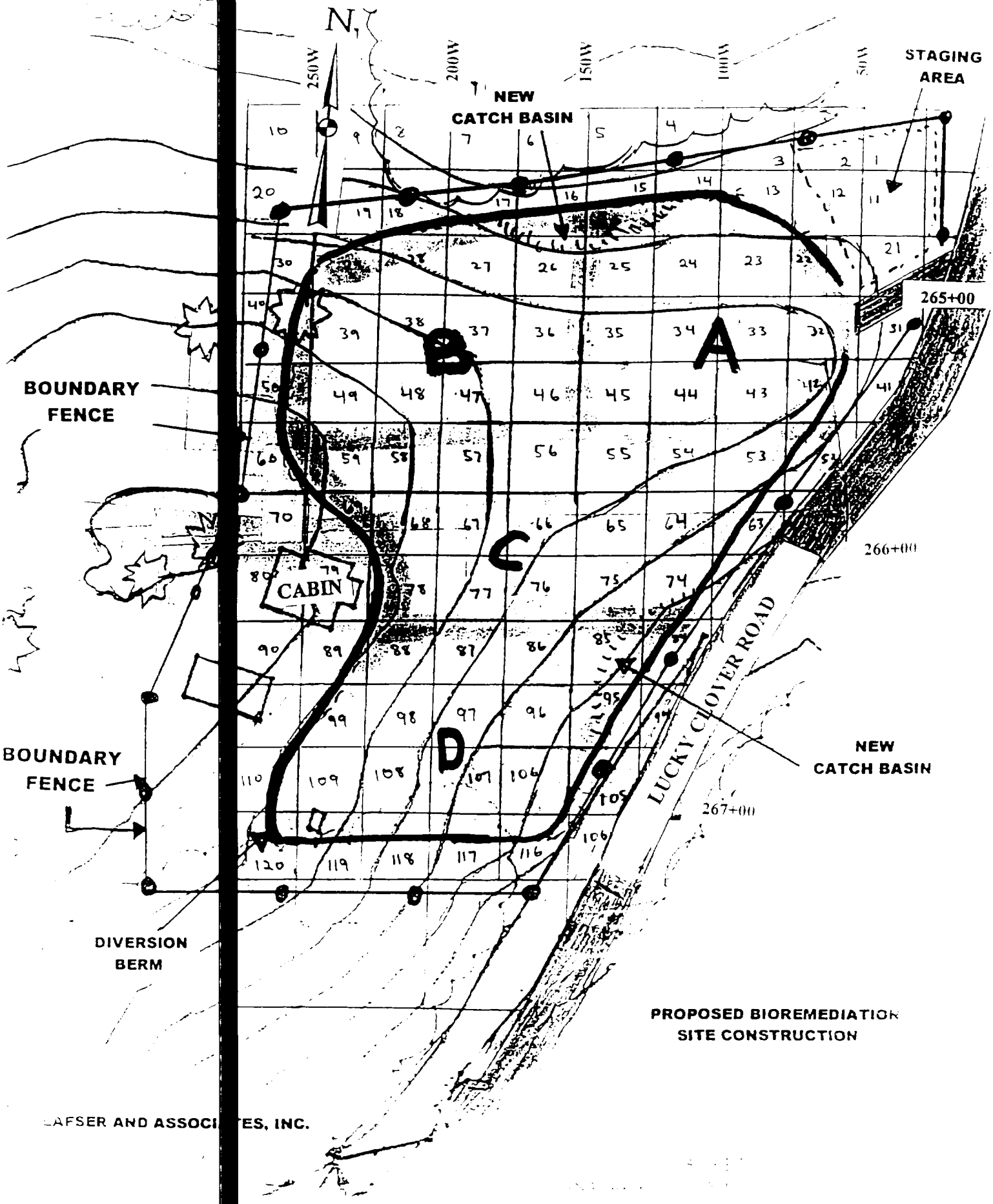


Figure 1: SITE LOCATION MAP

ARNESON TIMBER SITE
STEELVILLE, MISSOURI



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activities included disposal of two additional 55-gallon drums of contaminated soil collected from the basin area.

Objectives and Scope of Work

The primary objectives of this effort are to:

1) conduct biotreatment of contaminated soils to appropriate action levels

and

2) determine the areal and vertical extent of PCP contaminated soils at the site.

Collection and testing of surface and subsurface soil samples for laboratory analysis of PCP will be employed to monitor progress and completion of remedial actions.

Concentrations of Interest

An action level of 9.1 milligrams per kilogram (mg/kg) for PCP has been established for soils in residential settings by EPA and 37.7 ppm of PCP as an acceptable level for sites such as this with institutional controls. An action level of 1 ppb for dioxin Toxicity Equivalency Factors is in effect as part of the F032-contaminated soil analytical requirements based on the Resource Conservation and Recovery Act (RCRA) Land Disposal Restriction. F032 designates hazardous waste from wood processing using chlorophenolic compounds. A level of 10 ppb dioxin equivalents has been established for sites such as this with institutional controls. Since all test results are below this level, dioxin equivalents will only be tested when the site or portions of the site are brought for declaration of the 1 ppb any-use level. All areas testing above 9.1 mg/kg PCP and 1ppb dioxin equivalents are included in the fenced zone.

Required Detection Limits

The standard detection limits for the analytical methods are described in Section 6, Testing Methods, and Section 7, Laboratory Q/A-Q/C, for pentachlorophenol(F032)-contaminated wastes. The laboratory confirmation results will be the determining factor in establishment of clean boundaries and areas of contamination. The SOP (Standard Operating Procedure) for dioxin testing will be provided to EPA prior to testing if it is determined to be

necessary.

Laboratory sample results will be used to confirm levels of PCP in soils on site. Laboratory sample data will be used for evaluation and ultimate decision-making purposes.

PROPOSED FIELD ACTIVITIES

Sampling Rationale and Locations

For the area to be excavated and relocated(cells 96,97,106 and 107), at least six extended surface sample aliquots will be collected within the composite zone. The zone will be determined in the field. Each aliquot will be from a depth of 0 to 6 inches and homogenized. Sufficient sample will be collected to allow for submitting a split to the EPA on-scene coordinator. The composite sample will be transported to the laboratory for analysis.

Each extended surface soil aliquot/sample (0" - 6") will be collected into a new aluminum pie pan with a stainless steel spoon, homogenized thoroughly with the spoon, and field screened for PCP. Each sampled location will be marked on the grid.

Select grab samples from grid nodes beyond the identified area of contamination may be collected at the discretion of the project manager wherever it appears warranted. Emphasis will be on establishing clean zones.

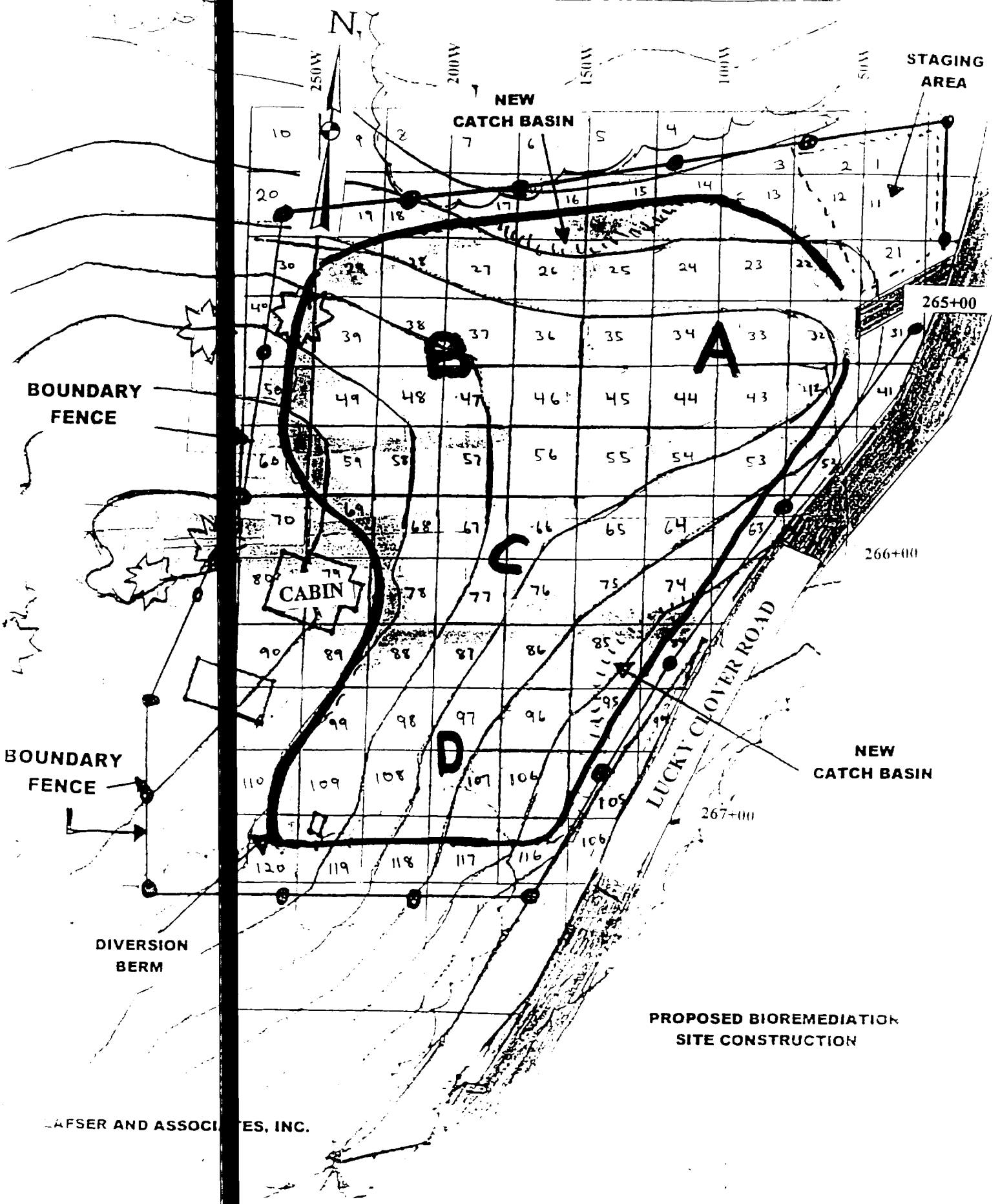
Since the contaminated soil is not homogeneous, and the site preparation (with additives and mixing) will take several months, baseline sampling is not proposed until October, 2000.

After completion, the landfarm will be divided into 4 composite zones. At least 6 aliquots at depth 0" to 12" will be taken in each zone and analyzed by the laboratory. This will establish the base concentration levels at the site. This sampling will be repeated annually to monitor progress.

Subsurface Soil Sampling/Screening

The only area of the site known to be contaminated at depth greater than 12 inches has been sampled previously. Further sampling using a Geoprobe hydraulic sampling apparatus will not be conducted. Extended surface composite sampling (0"-6") using a hand auger will be conducted after each removal "lift" with

ARNESON TIMBER SITE
STEELVILLE, MISSOURI



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efforts concentrated on determining the extent of subsurface contamination to reach a "clean level". At-depth sampling if determined to be necessary, will normally begin at an interval of 6 inches to 1 foot using the split-spoon hand auger. The selected points will be at the discretion of the project manager, and located on a site map in the field Sampling at the selected nodes and composite zones will progress vertically to a depth where the testing results indicate the soil is not contaminated, or four feet as required by the Administrative Order.

Quality Control Samples Needed

Approximately 15 QC samples will be required to verify the validity of analytical results and to assess whether the samples were contaminated as a result of improper decontamination procedures, the use of contaminated containers or preservatives, and/or the introduction of contaminants during transportation of the samples to the laboratory. Field QC samples will include field-trip blanks, background samples, and replicates, as appropriate. Laboratory QC samples will include duplicates, spikes and performance evaluation (PE) samples, as appropriate. All pertinent SOPs and guidance documents referenced in this QASP will be followed to ensure that the QA objectives are met. Laboratory quality control procedures will be performed in accordance with the SOPs for the applicable analytical methods. Laboratory quality control elements are included in the analytical SOPs for Environmetrics, Inc, laboratory.

Decontamination Procedures

Prior to the initiation of sampling and between subsurface samples, reusable accessories that come in contact with the sampled media will be washed with an Alconox/water solution (or equivalent) and rinsed with tap water. Disposable acetate sleeves will be used inside soil collection tubes where applicable, preventing the necessity for decontamination of the tubes between subsurface samples. Dedicated stainless steel spoons and aluminum pie pans will be used for the collection of all soil samples.

Sampling personnel will change outer gloves between composite sample locations to minimize the possibility of cross-contamination. Personal decontamination procedures will be addressed in the site safety plan.

Sample Containers, Preservation, and Holding Times

Soil samples will be placed into 8-ounce glass jars with Teflon-lined lids. All samples will be packaged and preserved according to Region VII EPA SOPs #AYE and #2130.5A: "Field Chain-of Custody for Environmental Samples" and "Identification, Documentation, and Tracking of Samples," respectively. The maximum holding time for PCPs is 14 days until extraction and 40 days after extraction until analysis.

Field Documentation, Sample Shipment, and Chain-of-Custody

Field documentation and chain-of-custody will be in accordance with Region VII EPA SOPs #AYE and #2130.3B: "Field Chain-of-Custody for Environmental Samples" and "Identification, Documentation, and Tracking of Samples", respectively. Samples will be conveyed by the site personnel to the laboratory. In the event that shipment becomes necessary, transportation procedures will meet U. S. Department of Transportation regulations.

For each sample to be submitted for laboratory analysis, the date, time of collection, depth, and location will be recorded on a field sheet. Other pertinent information describing the physical nature of the sampled material will be included as well.

Requested Analysis

Analysis for samples submitted to the laboratory will be determined by the project manager and will be specified in an Analytical Services Request (ASR) form, which will be completed prior to submittal of the samples. All samples will be tested for PCP using Method 8270C. Field sheets and sample labels will be produced by the site manager prior to mobilization. All samples will be tested for PCP using Method 8270C.

Personnel Requirements

The project manager, Fred Lafser, Lafser and Associates, Inc., will serve as the team leader and will be responsible for overall coordination of field activities for the project. He will also be responsible for periodic updates to the EPA regional on-site manager, Tom Curry. The sampling team will consist of two to three members, who will be responsible for sample collection and

screeni g, field documentation, and arranging for transportation and sub ttal of confirmation samples to the laboratory. Certifi tes of Training will be submitted prior to commencement of site ctivity. (See following pages) Medical monitoring will be incl ded for any team member expected to exceed the OSHA require nts of 29CFR 1910.120(f) Medical Surveillance.

Equipme Requirements

Pe sonal Protective Equipment (PPE)

Level D PE, as described in EPA's Standard Operating Safety Guides ll be utilized by the field teams during sample collecti n. Protection for sampling may be upgraded to Level C PPE if e soil conditions are dry and fugitive dust indicate control are inadequate as the target compounds are non-volatile and have a high affinity for soil. Level D PPE will be employed for all ther non-sampling activities (i.e., field screening, sample nagement, documentation, excavation, etc.). Required PPE will be temized in the site safety plan (SSP).

Dec ntamination Equipment

No speci lized decontamination equipment will be required to complete he tasks described in the QASP. PPE will be kept to a minimum nd will be rendered useless, double-bagged, and disposed of in a ntrolled container, if no apparent staining from on-site wastes e sts. Otherwise, contaminated PPE will be stored in a containe for proper disposal.

Sampling Equipment

All samp s will be collected using equipment and methods describe in the SSP. All sampling equipment will be itemized in the SSP.

Schedule

The exac schedule of sampling activities will be determined by the sche le of the project manager, contractor, the EPA on-site manager, nd by existing and forecasted weather conditions. Saturate soil conditions could increase the difficulty of using

excavating equipment and sampling for PCP analysis because of the difficulty of homogenizing soil samples without heating and drying. The project manager or his designee will convey the samples to the laboratory upon completion of collection, preservation, and finalization of pertinent documentation.

Site Access

Access for personnel to perform the described sampling activities has been arranged by EPA and the land owner, Mr. Charles Leezy.

Public and Media Inquiries

All public and media inquiries will be referred to the project manager and/or the EPA Region VII Office of External Programs.

Compliance Solutions

"Today's Training - Tomorrow's Solution"

10515 E. 40th Ave, Suite 116, Denver Colorado 80239 Phone: 800 711 2706

Lafser & Associates, Inc
99000072

Certificate of Completion

This is to certify that

Fred Lafser

has successfully completed the classroom requirements for

8 Hour HAZWOPER Refresher

29 CFR 1910.120(e)

Presented

Friday, March 31, 2000

Compliance Solutions Occupational Trainers, Inc.

Certificate Number: 23885

Neval Gupta
Vice President

Larry Erwin
National Training Manager

Compliance Solutions

"Today's Training....Tomorrow's Solution"

Certificate of Completion

This is to certify that

Fred Lafser

has completed the classroom requirements for

8 Hour Site Supervisor

29 CFR 1910.120

Presented

Tuesday, March 09, 1999

Compliance Solutions Occupational Trainers, Inc.

Certificate Number: 13817

Neval Gupta
Vice President

George Morrison
Instructor

Compliance Solutions

"Today's Training....Tomorrow's Solution"

Certificate of Completion

This is to certify that

Fred Lafser

has completed the classroom requirements for

8 Hour HAZWOPER Refresher

29 CFR 1910.120(e)

Presented

Thursday, April 01, 1999

Compliance Solutions Occupational Trainers, Inc.

Certificate Number: 14335

Neval Gupta
Vice President

George Morrison
Instructor

Compliance Solutions

"Today's Training - Tomorrow's Safety"

Certificate of Completion

This is to certify that

Thomad Heintz

has completed the classroom requirements for

40 Hour HAZWOPER

29 CFR 1910.120(e)

Presented

Thursday, April 01, 1999

Compliance Solutions Occupational Trainers, Inc.

Certificate Number: 14643

Neval Gupta
Vice President

George Morrison
Instructor

Compliance Solutions

"Today's Training - Tomorrow's Solutions"

Certificate of Completion

This is to certify that

Jerry Butyenek

has completed the classroom requirements for

40 Hour HAZWOPER

29 CFR 1910.120(e)

Presented

Thursday, April 01, 1999

Compliance Solutions Occupational Trainers, Inc.

Certificate Number: 14642

Neval Gupta
Vice President

George Morrison
Instructor

SAFETY TECHNOLOGIES, INC.

DATE 4/2/00

COMPANY K.R. Wilson Contracting REFIT DATE 4/2/00

EMPLOYEE Thomas W Heintz

MEDICAL EVALUATION ✓ YES ___ NO

BRAND Sundstrom MODEL Blue 2000 SIZE M

SAR - 1/2 _____ PAPR _____ SCBA _____ APR - 1/2 ✓
FULL FACE _____ FULL FACE _____

FIT TEST METHOD	SMOKE	ISOAMYL ACETATE	PORTA COUNT
BREATHE NORMAL	✓	___	___
BREATHE DEEP	✓	___	___
HEAD SIDE/SIDE, UP/DOWN	✓	___	___
RAINBOW PASSAGE	✓	___	___
KNEE BENDS/JOG/SANDBLASTER (test subject should not perform this exercise if no medical evaluation)	N/A	___	___
BREATHE NORMAL	___	___	___

PASS ✓ FAIL _____ FIT FACTOR _____

*WAS SUBJECT TESTED WITH ALL FACIAL EQUIPMENT NORMALLY WORN ON JOB SITE? ✓ YES _____ NO

*DOES IT APPEAR THAT TEST SUBJECT HAS BEEN TRAINED IN PROPER DONNING OF RESPIRATOR? ✓ YES _____ NO

**NOTE ANY CONDITIONS THAT MIGHT INVALIDATE TEST eg; NOT FOLLOWING PROCEDURES, LACK OF COOPERATION, SIGNS OF CLAUSTROPHOBIA:

_____

_____

I have read & understand the above and have received & verified all information.

Signed Todd A Litt Date 4/2/00

300 Manchester Rd • St. Louis, MO 63144 • 314-961-7075 • Fax 314-961-9166

SPIROMETRY RE[illegible]ORT
PB100 SW Rev [illegible]

TEST DATE: 04/02/99
TIME: 09:34 [illegible]

Patient Name: [illegible]
Med ID: 497520763 | Age: [illegible] | Height (in): 66 | Weight (lbs): [illegible] | Sex: Male | Race Correction: No | Smoker: Yes
Barometric Pressure (mmHg): 759 | Temp (deg F): 65 | BTPS Correction: [illegible] | Sensor: PS200 | Dsp Code: None
Cal Date: 04/01/99
PreMed Time: 09:35 AM

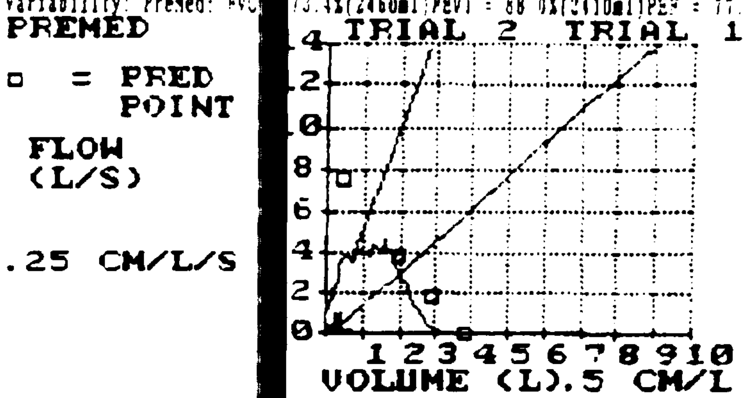
FVC TEST DATA - Clinical Format

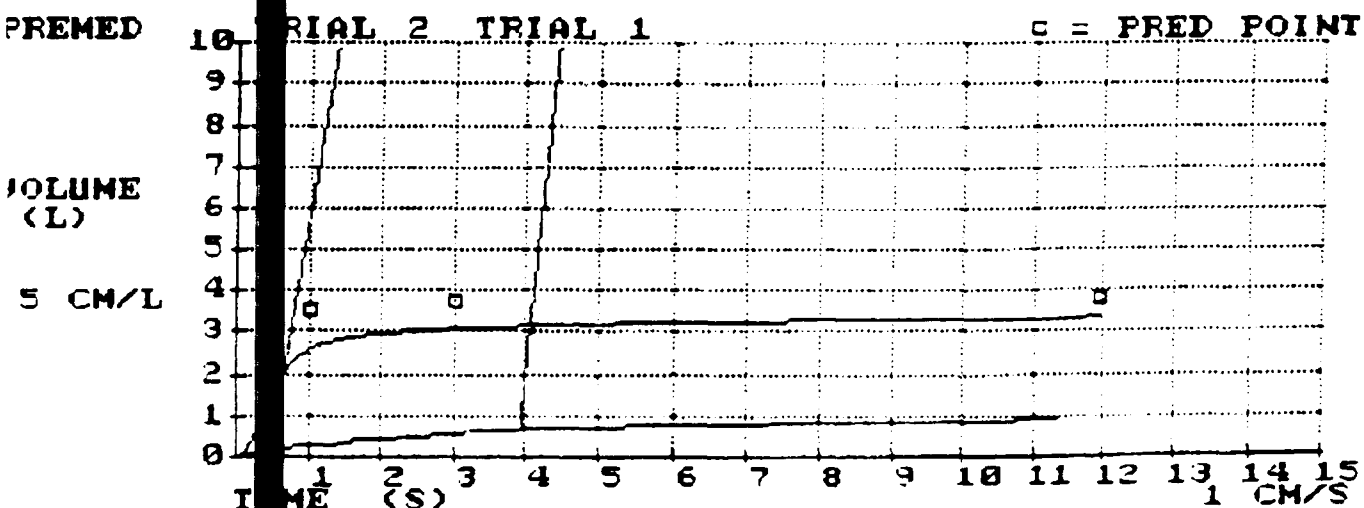
BEST TEST SUMMARY

Polgar Pediatric Predicted Normals

Measurement		PreMed	Pred	%Pred	PostMed	%Pred	%Change
FVC	(L)	[illegible]	3.92	88%			
FEV1	(L)	[illegible]74	3.33	77%			
%FEV1	(%)	[illegible]79	92.89	88%			
FEF25%-75%	(L/S)	[illegible]90	3.86	75%			
PEF	(L/S)	[illegible]62	7.56	61%			
FEV3	(L)	[illegible]10	3.75	83%			
FET	(S)	[illegible]51					

Variability: PreMed: FVC [illegible] 73.4%(2460ml)FEV1 = 88.0%(2410ml)PEF = 77.3%





nterpretations

EMED: Testing indicate[illegible]ild obstruction

omments:

SAFETY TECHNOLOGIES, INC.

DATE 4/2/99

REFIT DATE 4/2/00

COMPANY K.R. Wilson Contracting

EMPLOYEE JERRY BUTYENEK

MEDICAL EVALUATION ✓ YES ___ NO

BRAND Survivair MODEL Blue 2000 SIZE M

SAR 1/2 _____ PAPR _____ SCBA _____ APR - 1/2 ✓
FULL FACE _____ FULL FACE _____

FIT TEST METHOD	SMOKE	ISOAMYL ACETATE	PORTA COUNT
BREATHE NORMAL	✓	___	___
BREATHE DEEP	✓	___	___
HEAD SIDE/SIDE, UP/DOWN	✓	___	___
RAINBOW PASSAGE	✓	___	___
KNEE BENDS/JOG/SANDBLASTER (test subject should not perform this exercise if no medical evaluation)	N/A	___	___
BREATHE NORMAL	___	___	___

PASS ✓ FAIL _____ FIT FACTOR _____

*WAS SUBJECT TESTED WITH ALL FACIAL EQUIPMENT NORMALLY WORN ON JOB SITE? ✓ YES _____ NO

*DOES IT APPEAR THAT TEST SUBJECT HAS BEEN TRAINED IN PROPER DONNING OF RESPIRATOR? ✓ YES _____ NO

**NOTE ANY CONDITIONS THAT MIGHT INVALIDATE TEST eg: NOT FOLLOWING PROCEDURES, LACK OF COOPERATION, SIGNS OF CLAUSTROPHOBIA:

I have read & understand the above and have received & verified all information.

Signed Todd A Gatt Date 4/2/99

500 Manchester Rd • St. Louis, MO 63144 • 314-961-7075 • Fax 314-961-9166

SPIRCMETRY PORT

TEST DATE: 04/02/99
9:26 AM

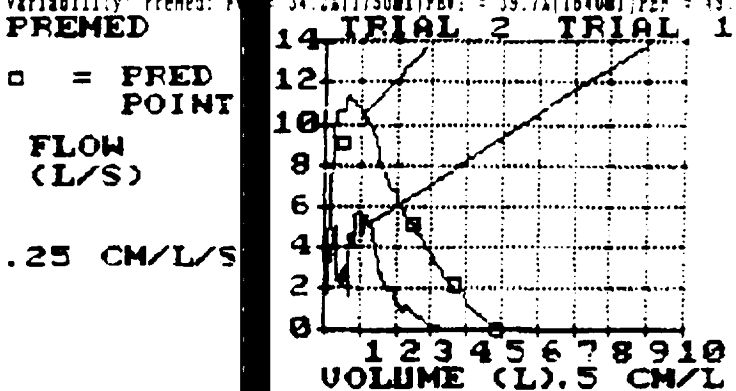
Patient Name: [illegible]
Patient ID: 500 | Age 32 | Height 58 | Weight 220 | Sex: Male | Race Correction: No | Smoker: Yes
metric Pressure (m) 760 | Temp (deg F): 63 | BTPS Correction 1.122 | Sensor: FS200 | Insp Code: None
Cal Date: 04/11/9

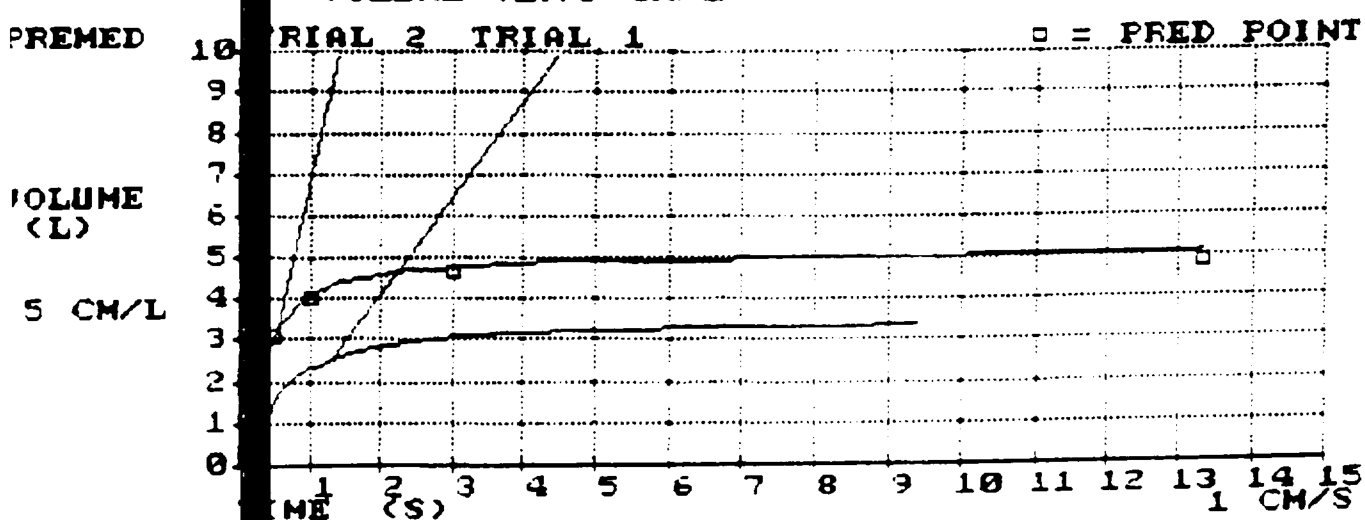
PreMed Time 09:29 AM

TEST DATA Clinic Format | BEST TEST SUMMARY | Knudson 83 Adult Predicted Normals

easurement		Med	Pred	%Pred	PostMed	%Pred	%Change
FVC	(L)	.12	4.84	106%			
FEV1	(L)	4.13	4.04	102%			
%FEV1	(%)	0.65	84.05	96%			
FEF25%-75%	(L/S)	3.89	4.32	90%			
PEF	(L/S)	.28	9.12	124%			
FEV3	(L)	.82	4.64	104%			
FET	(S)	.14					

Variability: PreMed: F = 34.2%(1750ml)FEV1 = 39.7%(1640ml);PEF = 49.6%





nterpretation:

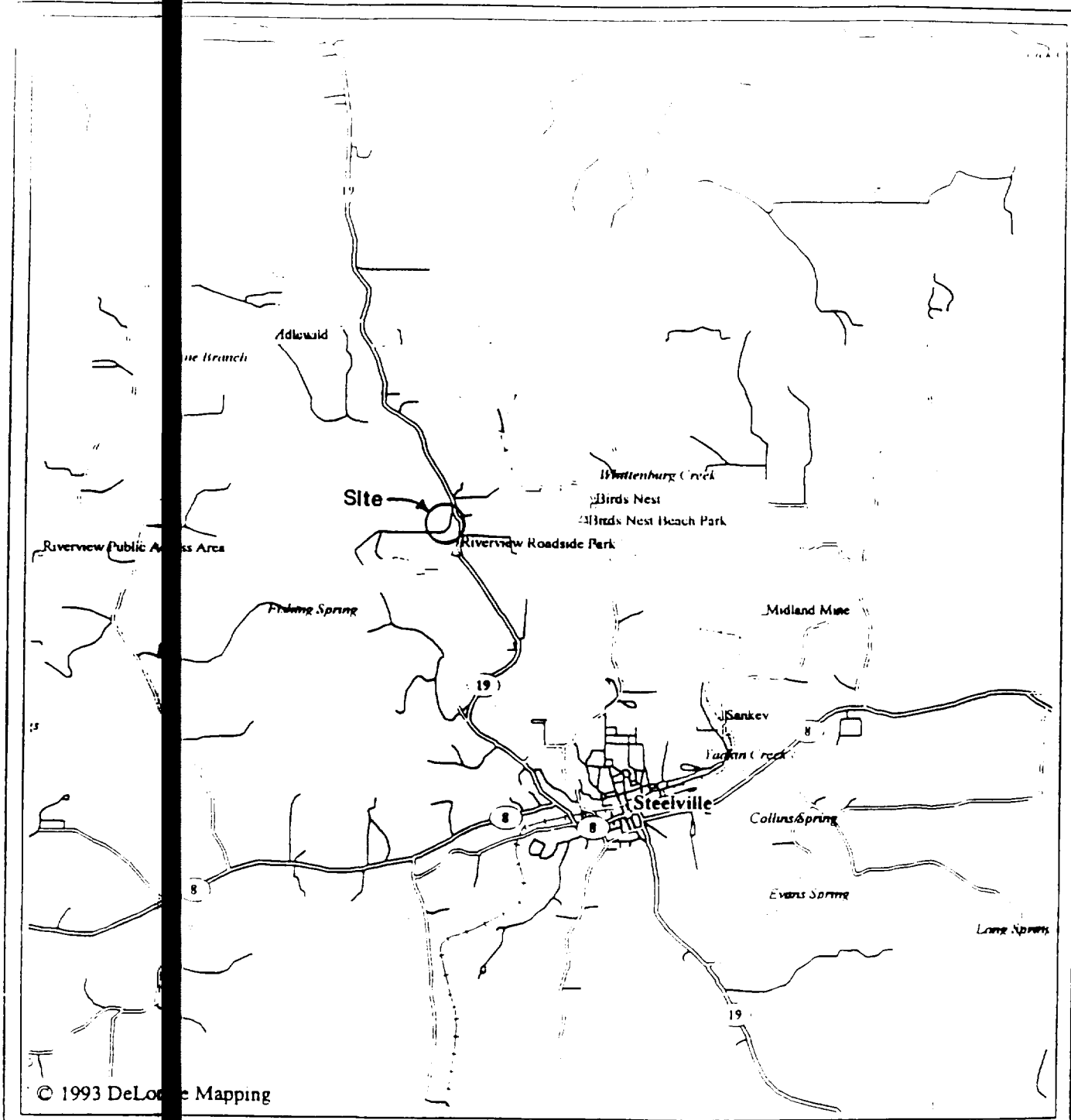
ng Age: 32 years
EMED: Testing indica normal spirometry.

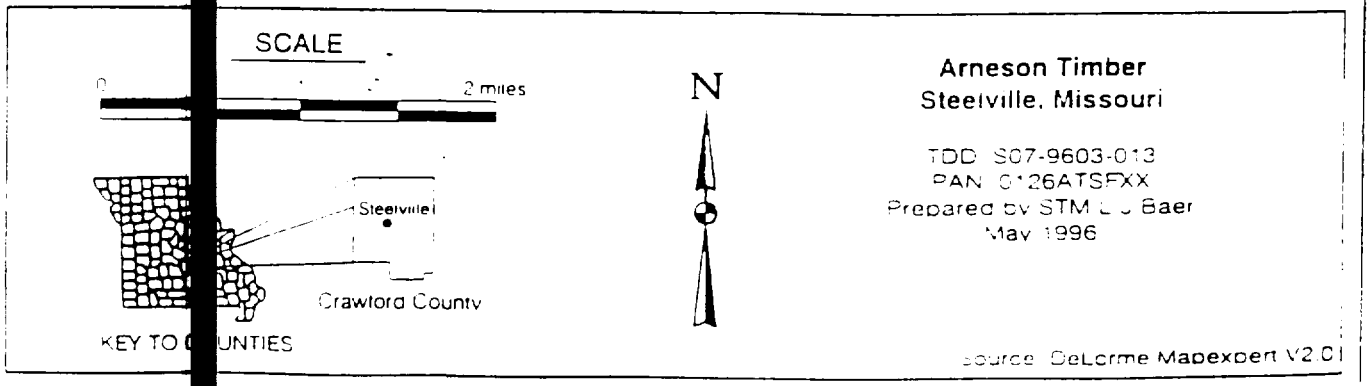
omments:

ATTACHMENTS

A. Figure 1 Site Location Map

B. Figure 2 Site Map with Sampling Grid

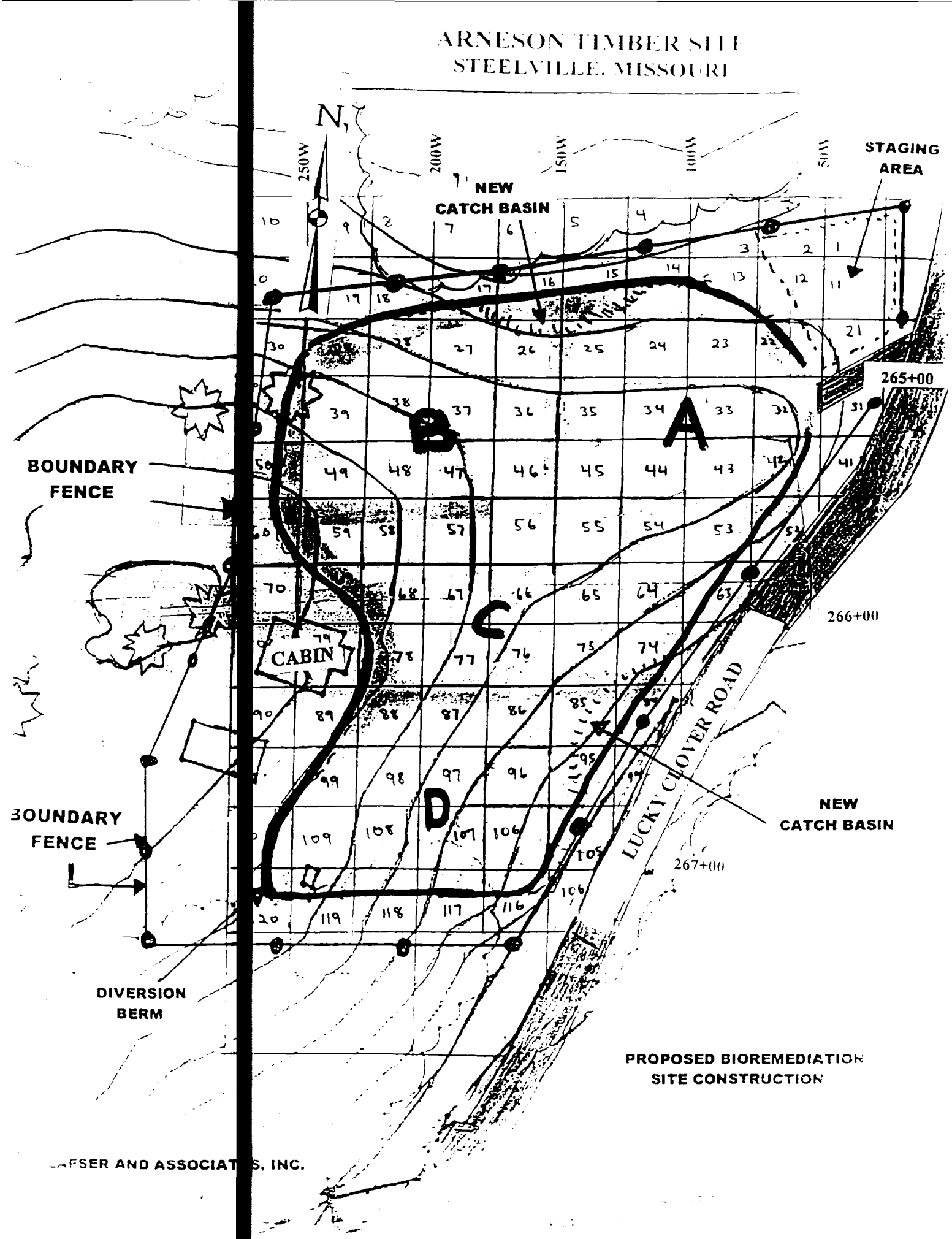




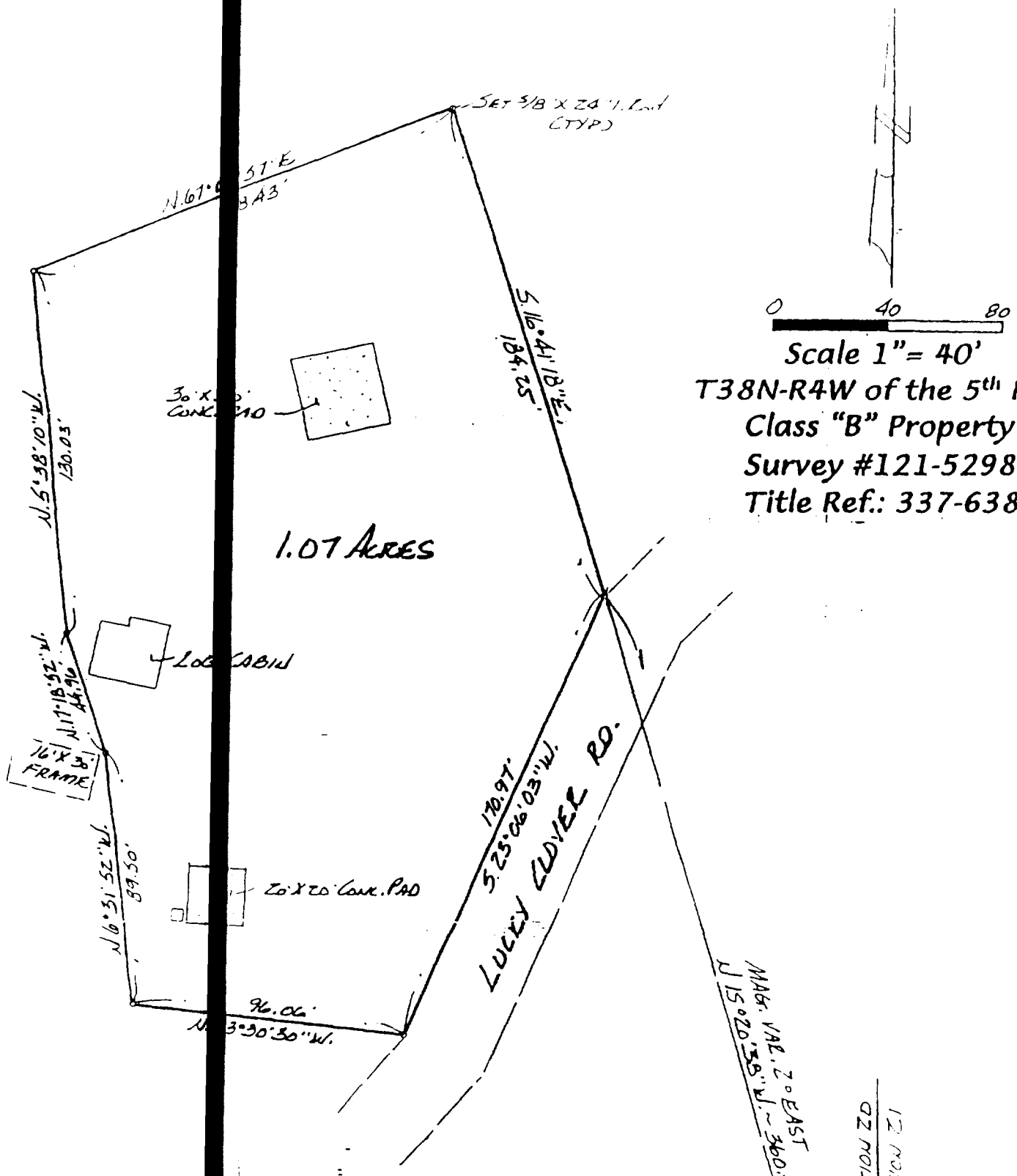
ATSSTLOC.CDR

Figure 1: SITE LOCATION MAP

ARNESON TIMBER SITE
STEELVILLE, MISSOURI



-AESER AND ASSOCIATES, INC.



Scale 1"= 40'

T38N-R4W of the 5th P.M.

Class "B" Property

Survey #121-5298

Title Ref.: 337-638

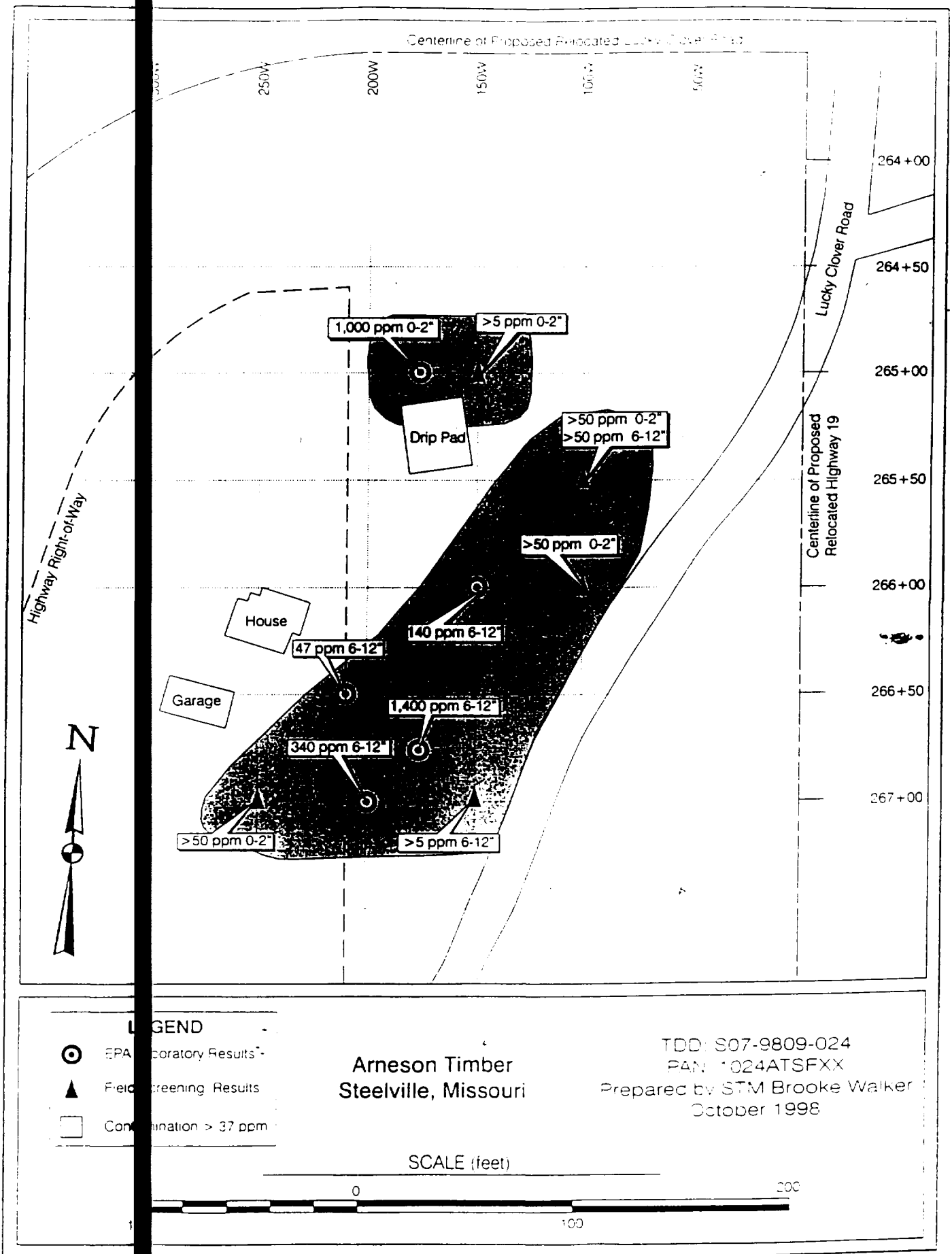
TRACT DESCRIPTION

Part of the NE ¼ of th SE ¼ of Section 20, T38N-R4W of the 5th P.M. in Crawford County, Missouri mor ully described as follows:

Commencing at the Corner of said ¼ ¼ Section; thence N.15°20'38"W. 360.42 feet to the point of be nning; thence S.23°06'03"W. 170.97 feet; thence N.83°30'30"W. 96.06 t; thence N.6°31'52"W. 89.50 feet; thence N.17°18'52"W. 44.96 feet; thence N.5 8'10"W. 130.03 feet; thence N.67°05'57"E. 158.43 feet; thence S.16°41'18"E. 184.25 feet to the point of beginning. CONTAINING 1.07 acres.

SURVEYORS DECLARATION

This is to declare th I, Mark A. Mueller have during the month of November, 1998 by order of Che s N. Leezy executed a survey of the tract of land platted hereon in accordance th the current Missouri Minimum Standards for Property



ATPCPCDR

Source: Proposed Highway 19 Plan, Burns & McDonnell, December 1995; E&E/START Field Notes, May 1996

Figure 1: Estimated Area of Contamination At
Industrial Action Level of 37 Parts Per Million PCB

SITE SPECIFIC HEALTH AND SAFETY PLAN

For

ARNESON TIMBER SITE
Project Title

STEELVILLE, MISSOURI
Location

Prepared by: FRED A. LAFSER Date JUNE 14, 2000

Approved by: _____ Date _____
Regional or Divisional
Health and Safety Manager

Adopted by: _____ Date _____
Program Manager

TABLE OF CONTENTS

TABLE OF CONTENTS

LIST OF ATTACHMENTS

SITE SAFETY PLAN

[Site Name] ARNESON TIMBER SITE

GENERAL INFORMATION:

Project Number:

Client Name: ARNESON TIMBER CO

Site Name & Location:

ARNESON TIMBER SITE–STEELVILLE, MISSOURI

Client's Tel. #:

314-692-9999

Project Manager: FRED A. LAFSER

Site Tel. #:

314-570-6969

SITE DESCRIPTION AND FEATURES: (Include approximate size, accessibility and unusual features; attach site map)

The site is 17 acres and is along Lucky Clover Rd. approximately 1/4 mile south of its intersection with MO Route 19 in Crawford Co. MO. A gravel site road proceeds west from Lucky Clover Rd. along a high ridge. To the North a concrete drip pad exists. South west of the drip pad is a log cabin. Contamination occurs from the cabin and drip pad to the south east proceeding down hill through a cleared area to the location of a foormer catch basin, which is immediately north of a forested area and west of Lucky Clover. The undeveloped land is vegetated, with surface soil of stoney loam, subsoil of red clay with underlying dolomite and sandstone formations.

BACKGROUND/SITE HISTORY: (Summarize below)

The site was used for pentachlorophenol (PCP) wood treating beginning in 1972. Pressure treated lumber was moved from a tank located east of the log cabin to the drip pad. In 1978 the site was purchased by Ameson Timber Company and a concrete drip pad was constructed on top of the former dnppage area. In 1982 the company closed the operation, removed the treatment tank and the associated sludge which had been drained to the catch basin. Contaminated soil from the area between the treatment tank and the drip pad was pushed down hill to fill in the cleaned out catch basin. Hot spots are under and north of the drip pad and in the area of the catch basin. The balance of the site is lightly contaminated and has been extensively characterized.

SITE MAP

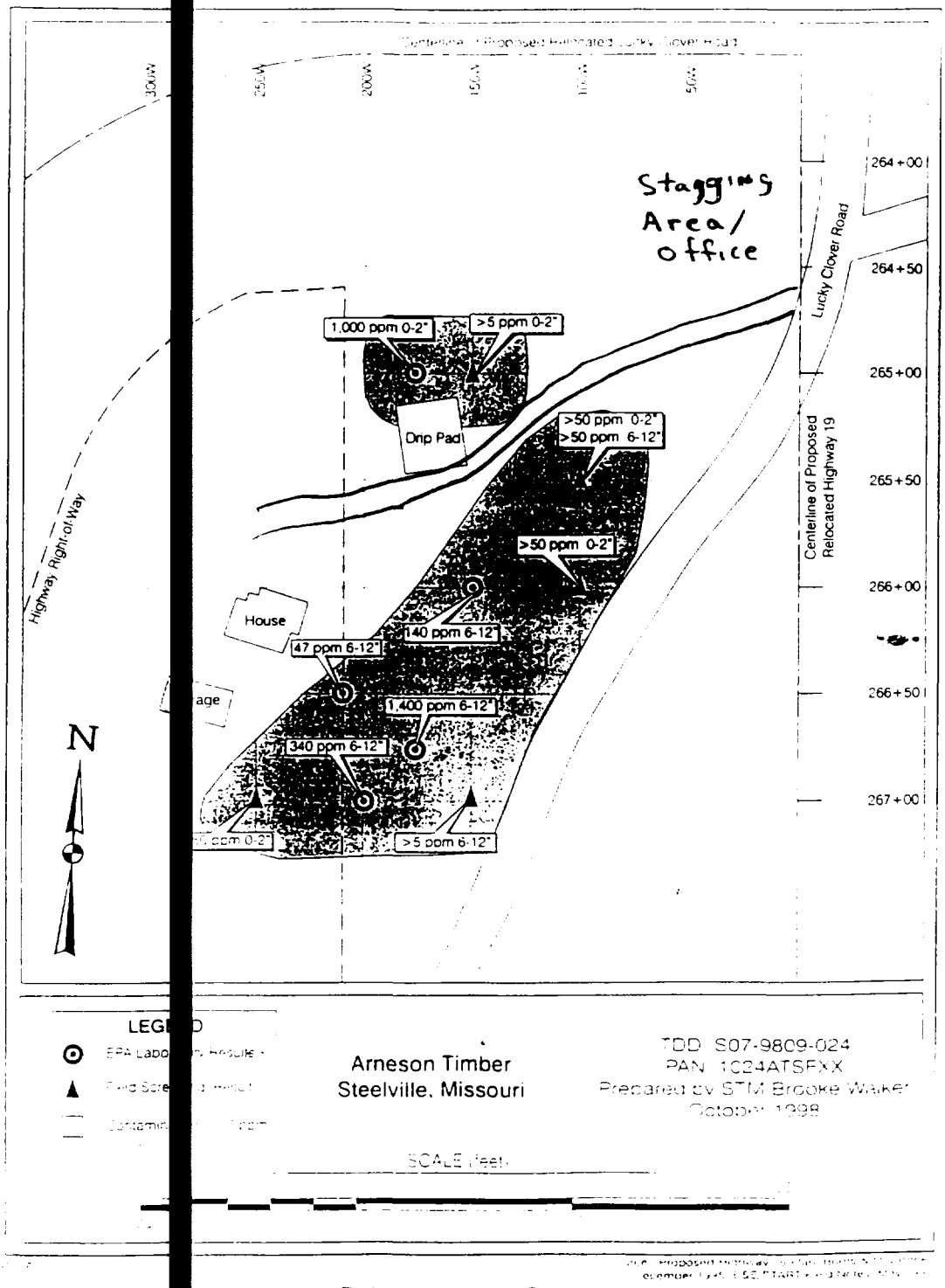


Figure 1: Estimated Area of Contamination At Industrial Action Level of 37 Parts Per Million PCP

SCOPE OF WORK/PLANNED SITE ACTIVITIES: (Summarize below)

Work on site will include surface soil sampling and excavation of contaminated soils. Concrete will be broken up and stored on site for later use or loaded for off-site disposal. Remaining contamination will be incorporated into a biological landfarm treatment unit. The unit will be periodically irregated and aerated by plowing and discing. Nutrients will be added as necessary. Periodic sampling will be conducted for a period of one to five years.

SITE PERSONNEL:*

Name	Title	Company	Activity
Fred A. Lafser	Site Manager	Lafser and Associates, Inc.	Management
Arne Arneson	Owner representative	Arneson Timber Co.	Management
Ken Wilson	Construction Manager	K. R. Wilson Co.	Management
Jerry Butyene	Equipment Operator	K. R. Wilson Co.	Earth Moving
Tom Heintz	Sampler/equipment Op.	K.R. Wilson Co.	Sampling/ Eq. Operating
Dave Schau	Alternate Safety Officer	Ecosafe, Inc.	Asst. Manager/ safety

*Note that all personnel arriving or departing the site should log in and out with the Site Manager and read and sign this health and safety plan.

WASTE CHARACTERISTICS:

Waste Types (Check all that apply)

( ) Liquid	( ) Sludge	( ) Unknown
(X) Solid	( ) Gas	

Waste Characteristics: (Check all that apply)

( ) Corrosive	( ) Flammable	( ) Radioactive
(X) Toxic	( ) Volatile	( ) Reactive
( ) Inert	( ) Carcinogenic	( ) Unknown
( ) Asphyxiant	( ) Other _____	

Hazardous Materials Summary: (Check all that apply)

Chemicals

( ) Acids	( ) Metals	(X) Phenolic Cpds.
( ) Caustics	( ) Pesticides	( ) Paints
( ) Halogen	( ) PCB's	( ) Solvents
( ) Other: _____		

Oils/Fuels

(X) Fuel (Diesel) Oil	( ) AVGAS	( ) MOGAS
( ) Gasoline		
( ) Other: _____		

Sludges

( ) Metal sludges	( ) Oily sludges	( ) Septic sludges
( ) Other: _____		

Solids

( ) Asbestos	( ) Land refuse	( ) Tailings
( ) Other: _____		

NOTES: _____

HAZARD D TERMINATION

Task Name: Excavation

Potential Hazards: Check all that apply to either existing conditions or are a result of site operations)

( ) Rotating Mach ery	( ) Projectiles	( ) Confined Space
( ) Heat Stress	(X) Physical Exertion	( ) Biological
( ) Cold Stress	(X) Noise (>85 dBA)	( ) Electrical (utilities)
(X) Heavy Equipm t	(X) Vehicle Traffic	(X) Chemical Exposure
(X) Intrusive Acti (underline)	( ) Fire/Explosion (underline)	( ) Other (List)
- Drilling	- Flam. Materials	_____
- Soil Gas apor Surv.	- Low-lying Areas	_____
- Cone Pe rom. Surv.	- Fuel Lines	_____
- Samplin		

Control or Protecti Measures: (Check all that apply)

(X) Tailgate Meeti s	(X) PPE, Level D	(X) Safe Work Practices
(X) Operator Train g	(X) Site Control	(X) Decontamination
(X) Engineering C rols: Water mist dust control		
( ) SOP's: _____		
( ) Other: _____		

PERSONAL ROTECTIVE EQUIPMENT (PPE)

Initial levels of pro tion have been assigned per work task. Levels may be upgraded or downgraded depending on moni ring data and site conditions, as determined by the onsite health and safety coordinator.

LEVEL OF PROT TION:	( ) A	( ) C	( ) Modified
	( ) B	(X) D	D
RESPIRATOR:			
(Level C and above if de rmined)	( ) SCBA, Airline	( ) Purif. Resp.	( ) Escape Mask
	(X) OV/AG/Dust Cart.	( ) Other Cart. _____	
PROTECTIVE CL THING:	( ) Encap. Suit	(X) Tyvek If determined	( ) PE Tyvek
	( ) Saranex	( ) Splash Suit	( ) Other _____
HEAD/EYE/EAR:	( ) Hard Hat	(X) Safety Glasses	( ) Goggles
	( ) Splash Shield	(X) Ear Plugs/Muffs	( ) Other
GLOVES: (outer)	(X) Nitrile	( ) Neoprene	( ) PVC
(inner)	( ) Latex	( ) Vinyl	( ) Other _____
FOOTWEAR:	( ) Steel-toed Leather	( ) Overboots	
	(X) Steel-toed Rubber	( ) Other _____	

Modifications Permitted: _____

Task Name: Sampling

Potential Hazards: (Check all that apply to either existing conditions or are a result of site operations)

( ) Rotating Machinery	( ) Projectiles	( ) Confined Space
( ) Heat Stress	( ) Physical Exertion	( ) Biological
( ) Cold Stress	( ) Noise (>85 dBA)	( ) Electrical (utilities)
( ) Heavy Equipment	(X) Vehicle Traffic	(X) Chemical Exposure
(X) Intrusive Activities (underline)	( ) Fire/Explosion (underline)	( ) Other (List)
- Drilling	- Flam. Materials	_____
- Soil Gas/Vapor Surv.	- Low-lying Areas	_____
- Cone Penetrom. Surv.	- Fuel Lines	_____
- Sampling		

Control or Protective Measures: (Check all that apply)

(X) Tailgate Meeting	(X) PPE, Level D	(X) Safe Work Practices
(X) Operator Training	(X) Site Control	(X) Decontamination
( ) Engineering Controls: _____		
( ) SOP's: _____		
( ) Other: _____		

PERSONAL PROTECTIVE EQUIPMENT (PPE)

Initial levels of protection have been assigned per work task. Levels may be upgraded or downgraded depending on monitoring data and site conditions, as determined by the onsite health and safety coordinator.

LEVEL OF PROTECTION:	( ) A	( ) C	(X) Modified D
	( ) B	( ) D	
RESPIRATOR: (Level C and above)	( ) SCBA, Airline	( ) Purif. Resp.	( ) Escape Mask
	( ) OV/AG/Dust Cart.	( ) Other Cart. _____	
PROTECTIVE CLOTHING:	( ) Encap. Suit	( ) Tyvek	( ) PE Tyvek
	( ) Saranex	( ) Splash Suit	( ) Other _____
HEAD/EYE/EAR:	( ) Hard Hat	(X) Safety Glasses	( ) Goggles
	( ) Splash Shield	( ) Ear Plugs/Muffs	( ) Other
GLOVES: (outer)	(X) Nitrile	( ) Neoprene	( ) PVC
(inner)	( ) Latex	( ) Vinyl	( ) Other _____
FOOTWEAR:	( ) Steel-toed Leather	( ) Overboots	
	(X) Steel-toed Rubber	( ) Other _____	

Modifications Permitted: _____

Modifications Permitted: _____

Task Name:

Potential Hazards: (Check all that apply to either existing conditions or are a result of site operations)

( ) Rotating Machinery	( ) Projectiles	( ) Confined Space
( ) Heat Stress	( ) Physical Exertion	( ) Biological
( ) Cold Stress	( ) Noise (>85 dBA)	( ) Electrical (utilities)
( ) Heavy Equipment	( ) Vehicle Traffic	( ) Chemical Exposure
( ) Intrusive Activities (underline)	( ) Fire/Explosion (underline)	( ) Other (List)
- Drilling	- Flam. Materials	_____
- Soil Gas Vapor Surv.	- Low-lying Areas	_____
- Cone Pen. Chrom. Surv.	- Fuel Lines	_____
- Sampling		

Control or Protective Measures: (Check all that apply)

( ) Tailgate Meeting	( ) PPE, Level___	( ) Safe Work Practices
( ) Operator Training	( ) Site Control	( ) Decontamination

( ) Engineering Controls: _____

( ) SOP's: _____

( ) Other: _____

PERSONAL PROTECTIVE EQUIPMENT (PPE)

Initial levels of protection have been assigned per work task. Levels may be upgraded or downgraded depending on monitoring data and site conditions, as determined by the onsite health and safety coordinator.

LEVEL OF PROTECTION:	( ) A	( ) C	( ) Modified
	( ) B	( ) D	D

RESPIRATOR:

(Level C and above)	( ) SCBA, Airline	( ) Purif. Resp.	( ) Escape Mask
	( ) OV/AG/Dust Cart.	( ) Other Cart. _____	

PROTECTIVE CLOTHING:	( ) Encap. Suit	( ) Tyvek	( ) PE Tyvek
	( ) Saranex	( ) Splash Suit	( ) Other _____

HEAD/EYE/EAR:	( ) Hard Hat	( ) Safety Glasses	( ) Goggles
	( ) Splash Shield	( ) Ear Plugs/Muffs	( ) Other

GLOVES: (outer)	( ) Nitrile	( ) Neoprene	( ) PVC
(inner)	( ) Latex	( ) Vinyl	( ) Other _____

FOOTWEAR:	( ) Steel-toed Leather	( ) Overboots
	( ) Steel-toed Rubber	( ) Other _____

Modifications Permitted: _____

Modifications Per[redacted]ted: _____

Task Name:

Potential Hazards: (Check all that apply to either existing conditions or are a result of site operations)

( ) Rotating Mach[redacted]ery	( ) Projectiles	( ) Confined Space
( ) Heat Stress	( ) Physical Exertion	( ) Biological
( ) Cold Stress	( ) Noise (>85 dBA)	( ) Electrical (utilities)
( ) Heavy Equipm[redacted]t	( ) Vehicle Traffic	( ) Chemical Exposure
( ) Intrusive Acti[redacted] (underline)	( ) Fire/Explosion (underline)	( ) Other (List)
- Drilling	- Flam. Materials	_____
- Soil Gas [redacted]apor Surv.	- Low-lying Areas	_____
- Cone Pe[redacted]trom. Surv.	- Fuel Lines	_____
- Samplin[redacted]		

Control or Protecti[redacted] Measures: (Check all that apply)

( ) Tailgate Meeti[redacted]s	( ) PPE, Level___	( ) Safe Work Practices
( ) Operator Train[redacted]g	( ) Site Control	( ) Decontamination

( ) Engineering C[redacted]rols: _____
( ) SOP's: _____
( ) Other: _____

PERSONAL [redacted]ROTECTIVE EQUIPMENT (PPE)

Initial levels of pro[redacted]tion have been assigned per work task. Levels may be upgraded or downgraded depending on moni[redacted]ring data and site conditions, as determined by the onsite health and safety coordinator.

LEVEL OF PROT[redacted]TION:	( ) A	( ) C	( ) Modified
	( ) B	( ) D	D
RESPIRATOR:			
(Level C and above	( ) SCBA, Airline	( ) Purif. Resp.	( ) Escape Mask
	OV/AG/Dust Cart.	( ) Other Cart. _____	
PROTECTIVE CL[redacted]THING:	( ) Encap. Suit	( ) Tyvek	( ) PE Tyvek
	( ) Saranex	( ) Splash Suit	( ) Other _____
HEAD/EYE/EAR:	( ) Hard Hat	( ) Safety Glasses	( ) Goggles
	( ) Splash Shield	( ) Ear Plugs/Muffs	( ) Other
GLOVES: (outer)	( ) Nitrile	( ) Neoprene	( ) PVC
(inner)	( ) Latex	( ) Vinyl	( ) Other _____
FOOTWEAR:	( ) Steel-toed Leather	( ) Overboots	
	( ) Steel-toed Rubber	( ) Other _____	

Modifications Perm[redacted]ed: _____

See att[illegible]hed MSDS

TABLE 1

CHEMICAL HAZARD ASSESSMENT OF IDENTIFIED KEY COMPOUNDS

Exposure A[illegible]ssment		Exposure Limits		Physical Data		Health Effects	
Substance Name	Max. Conc./Media* ppm	TLV ppm	IDLH ppm	IP eV	LEL %	Exposure Route**	Target Organ***

* Abbrev's: A = A[illegible]D = Drum, F = Flyash, GW = Ground Water, L = Lagoon/Pit, LF = Landfill, S = Soil, SL = Sludge SW = Surface W[illegible], T = Tailings, TK = Tank, U = Unknown, WW = Wastewater, N./A = Not Available, N/E = Not Established

** Inh = Inhalation, [illegible] = Ingestion, Abs = Skin absorption, Con = Skin or eye contact

*** 1 = Skin, 2 = Eye[illegible] = Resp, 4 = CNS, 5 = Liv, 6 = Kid, 7 = Bio, 8 = Bla, 9 = Lung, 10 = Pros, 11 = GI, 12 = Hrt, C = Carcinogen

MATERIAL SAFETY DATA SHEET

CORPORATE RESEARCH & DEVELOPMENT
120 ERIE BOULEVARD
SCHENECTADY, N.Y. 12305

MATERIALS INFORMATION SERVICES

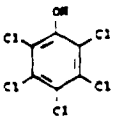
NO. 517

PENTACHLOROPHENOL

DATE October 1983

SECTION I. MATERIAL IDENTIFICATION

MATERIAL NAME: PENTACHLOROPHENOL
DESCRIPTION: Wood preservative and fungicide, algacide in cooling towers.
OTHER DESIGNATIONS: PCP, Phenchlorol, Penta, CAS #000 087 865, C_6Cl_5OH
MANUFACTURER: Available from several suppliers.



SECTION II. INGREDIENTS AND HAZARDS	%	HAZARD DATA
Pentachlorophenol (Purified)*		8-hr TWA 0.5 mg/m^3 (skin)**
Composition varies with each industrial batch. PCP is about 90% for commercial material with major impurities tetrachlorophenol and hydroxychlorodiphenyl ethers. Low level contamination by certain chlorinated dioxins and dibenzofurans may be responsible for some toxic effects of commercial PCP, such as chloroacne.		Human, Oral LDLo 29 mg/kg Rat, Oral LD_{50} 50 mg/kg Rat, Inhalation LD_{50} 1170 ug/kg Rat, Oral, 9D preg. TDLo 60 mg/kg TFX:TER
*Current OSHA PEL and ACGIH (1983) TLV. The ACGIH STEL is 1.5 mg/m^3. *2,3,7,8-TCDD is not a contaminate.		

SECTION III. PHYSICAL DATA

Boiling point, 1 atm deg C (decomposes) -	310	Specific gravity, 22/4C ------	1.978*
Vapor pressure, mm Hg @ 20C ----------	0.0001	Melting point, deg C ---------	191*
@ 100C ----------	0.12	Molecular weight ----------	266.35
Vapor density (Air=1) ----------	9.2	Log octanol/water partition coeff. ----------	5.01
Solubility in water, ppm @ 20C ---------	14		

Appearance and Odor: White flake, needle-like crystals or block form; pungent odor only when hot. (Technical grades dark grey to brown). Good warning properties (irritation of eyes and nose) at 1 mg/m^3.
*Purified PCP. Values for typical commercial products can be SG1.85 and m.p. 188-189C

SECTION IV. FIRE AND EXPLOSION DATA

Flash Point and Method	Autoignition Temp.	Flammability Limits in Air	Lower	Upper
None*		Not combustible*		

Extinguishing media: Use that appropriate for surrounding fire: Dry chemical, carbon dioxide, foam, water spray. Use water spray to cool fire-exposed containers. When involved in a fire, it emits toxic fumes.
Firefighters should wear self-contained breathing apparatus and full protective clothing.

*The neat material is not combustible. However, wood treating is preformed using 5% PCP soln. in petroleum solvents (mineral spirits, kerosine) which are combustible.

SECTION V. REACTIVITY DATA

This is a stable material in closed containers at room temperature under normal storage and handling conditions. It does not polymerize.
A weak acid which forms salts in alkaline solutions.
Solutions subjected to sunlight or ultraviolet light undergo photochemical degradation.
Thermal-oxidative degradation products include vapors of hydrogen chloride and chlorine-containing compounds.

GENERAL ELECTRIC

NO. 517

SECTION VI. HEALTH HAZARD INFORMATION

TLV 0.5 mg/m^3 (skin) (See Sect II)

Unacclimated individuals exposed to dust or sprays (conc. >1.0 mg/m^3) can experience irritation the upper respiratory tract (coughing & sneezing), eyes (tearing) and skin. Readily absorbed through the skin causing systemic injury. The risk of serious intoxication is increased in hot weather. Skin contact causes irritation, dermatitis, and acneform (believed mainly from impurities in PCP). Solutions as dilute as 1% can cause skin irritation upon prolonged or repeated contact. Symptoms from excessive exposure include anorexia, fever, profuse sweating, increased respiration, muscular weakness, hyperglycemia, weight loss and heart failure.

FIRST AID:

Eye Contact: Flush thoroughly with running water for 15 min. including under eyelids.

Skin Contact: Remove contaminated clothing! Wash affected area with soap and water.

Inhalation: Remove to fresh air.

Ingestion: Give several glasses of water to drink. Induce vomiting. Control rising body temp. with ice-cold towels or cold water bath.

Get prompt medical help for further treatment, observation and support after first aid.

SECTION VII. SPILL, LEAK, AND DISPOSAL PROCEDURES

Notify safety personnel. Provide ventilation. Clean-up workers need full protection against inhalation of vapors and contact with solid or solution. Collect solution spill with absorbent solid and place in metal container for disposal. Wash residue with soap and water. Prevent spills from entering sewers, streams, and open waters.

DISPOSAL: Waste material can be buried in an approved landfill or dissolved in a flammable solvent, and burned in an approved, high temperature (>600C) incinerator using an afterburner and scrubber for HCl abatement. Follow Federal, State, and Local regulations.

EPA(CWA) RQ is 0 lbs (40 CFR 117); EPA(RCRA) HW No. U242 (40CFR261).

AQUATIC TOXICI TLm 24 (fathead minnow): 0.33 mg/L

SECTION VIII. SPECIAL PROTECTION INFORMATION

Provide general and local exhaust ventilation to meet TLV requirements. When needed a chemical cartridge respirator with a dust, mist and fume filter and organic vapor cartridge can be used to 25 mg/m^3; a full facepiece is needed >2.5 mg/m^3. Approved, self-contained, positive pressure, breathing apparatus can be used to 150 mg/m^3.

Use impervious gloves, boots, protective clothing, etc. to avoid skin contact with liquid. Use chemical safety goggles with a faceshield if splashing is possible. Contaminated clothing to be removed and laundered before reuse. Showering after workshift with a complete change to street clothes is desirable. Use separated lockers for street clothes.

Eyewash station and washing facilities should be available near use areas.

Preplacement and periodic physical exams should emphasize cardiovasular system, eyes, upper respiratory tract, liver, kidneys and skin. Provide training to those working with PCP.

SECTION IX. SPECIAL PRECAUTIONS AND COMMENTS

Store in closed containers in a cool, dry, well-ventilated, low fire hazard area away from sources of heat and combustible materials. Protect containers from physical damage. Outside or detached storage preferred. Accumulated sludge at the bottom of dipping tanks may concentrate toxic impurities at much higher levels than original product.

Avoid inhalation of vapors, dust or fumes. Avoid eye or skin contact. Do not ingest.

Wash thoroughly after handling and follow good personal hygiene practices.

PCP can be embryotoxic and teratogenic in test animals. No safe exposure level for pregnant women is established at this time. (See AIHA Journal 43, pp 799-810, 1982).

DOT Classification: ORM-E I.D. No. NA2020 Label: POISON

DATA SOURCE(S) CODE: 2-14,16,20,23,27,31,34,37,38,46-49; DHHS(NIOSH) Publ. #83-106.

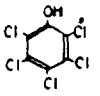
APPROVALS: MIS/CRD J. W. Nielsen

INDUST. HYGIENE/SAFETY [illegible] 9-8-83

MEDICAL REVIEW: 17 September 1983

GENERAL ELECTRIC

493 Pen chlorophenol



Uses: Antiseptic, fungicide, herbicide, preservative.

Properties: Mol wt 266.35; sp gr 1.978 (22/4); bp 310°C with decomposition; mp 190°C. White power or crystals; slightly sol in water; sol in dilute alkali, alcohol, acetone, ether, benzene, carbitol, cellosolve.

Hazardous Potent s:

A) Flammabili Vapor pressure 0.00011 (20).

B) Toxicity: TLV: 500 $\mu g/m^3$

TDL: orl-hmn LDL_0: 29 mg/kg; orl-rat LD_{50}: 27 mg/kg;
skn-rat LD_{50}: 105 mg/kg; ipr-rat LDL_0: 420 mg/kg;
scu-rat LD_{50}: 100 mg/kg; orl-rbt LDL_0: 70 mg/kg;
skn-rbt LDL_0: 40 mg/kg; scu-rbt LDL_0: 70 mg/kg

Symptoms: Conjunctivitis, skin burn, stictacne, furunculosis, pigmentation, cough and shortness of breath, nausea, vomiting, abdominal cramps, profuse sweating, fever, quick pulse, weakness of lower extremities and paresthesis, loss of weight, convulsions.

Handling & Stora Store in dry place. Wear rubber gloves, self-contained breathing apparatus, overalls.

Emergency Treat nt and Measures:

A) Hygienic Pr u-tions: Adequate ventilation and protectors. Keep workmen clean and sanitary.

B) Hygienic Tr t-ments (Firs id): Irrigate thoroughly the eyes with water. Wash contaminated areas of body with soap and water. Treat skin burns as usual.

Spills and Leakag Absorb the spills with paper towels or the like materials. Place in a hood to evaporate. Dispose by burning the towel.

Disposal and Was Treatment: Dissolve in a combustible solvent. Scatter the spray of the solution into the furnace with afterburner and alkali scrubber.

494 n-Pe tane $CH_3(CH_2)_3CH_3$

Uses: Manufacture of artificial ice, cryometer, for blow molding of plastics, insecticide.

Properties: Mol wt 72.15; sp gr 0.62638 (20); bp 36.07°C; mp −129.7°C; ref ind 1.35768 (20). Colorless liquid with pleasant odor; sol in hydrocarbons, oils, ether; very slightly sol in alcohol; sol in water.

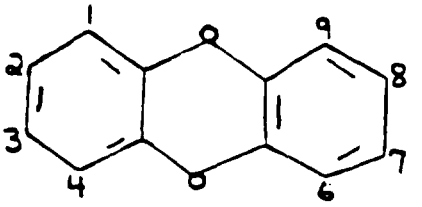
Hazardous Potent s:

A) Flammabili Flash point −40°C (closed cup); ignition temperature 309°C; flammable limits 1.4 − 8.0%; vapor density 2.48; vapor pressure 500 (24 − 34).

B) Toxicity: TLV: 1000 ppm 2950 mg/m^3; Carcinogenic.

TDL: ihl-hmn TCL_0: 130,000 ppm TFX: CNS

Poly[illegible]lorinated dibenzo-p-dioxins (PCDD) are a class of [illegible]pounds which are [illegible]esently receiving considerable attention regarding [illegible]eir environmental occu[illegible]nce and significance. The compounds have been found at trace levels in c[illegible]mercial products, such as chlorinated phenols. There are 75 possible PCDD [illegible]somers ranging from monochloro to octachloro species. The parent comp[illegible]nd and ring numbering system is shown below.



dibenzo-p-dioxin

Of p[illegible]ticular environmental concern is the 2,3,7,8-tetrachloro isomer whic[illegible] is commonly referred to as "TCDD" or simply "dioxin." There are 22 poss[illegible]le tetrachloro-dibenzo-p-dioxin isomers. However, of that group, the [illegible]3,7,8 isomer is believed to be the most toxic. In fact, 2,3,7,8-TCDD is o[illegible] of the most toxic chemicals known to man. Acute (lethal) oral toxi[illegible]ty is generally expressed in terms of amount per kilogram of body weig[illegible] required to kill 50 percent of a population of specified laboratory anim[illegible]s such as rats, mice, or hamsters. This value is called LD_{50} and is g[illegible]erally expressed in units of milligrams per kilogram (mg/kg) of body [illegible]eight. Table 1 gives LD_{50} values for common poisons and pesticides.

TABLE 1. Lethal Oral Toxicity Data

Compound	LD_{50} (mg/kg)
STRYCHNINE[1]	5 rats
ARSENIC TOXIDE[1]	138 rats
DDT[2]	113-250 rats
POTASSIUM CYANIDE[1]	2 dogs
2,3,7,8-TCDD[3]	0.002 guinea pigs 0.284 mice
2,3,7-TCDD[3]	>3,000 mice

[1]The [illegible]erck Index - 8th Edition
[2]Per[illegible]stent Pesticides in the Environment - 2nd Edition
[3]MIE[illegible]S/IARC Working Group Report

Hen[illegible], the lethal dosage is several orders of magnitude lower than clas[illegible]cal poisons such as strychnine and cyanide. Other PCDD isomers 1,2[illegible], pentachlorodibenzo-p-dioxin have been demonstrated to have similar

toxic[redacted]. The most highly toxic PCDD's have chlorine atoms in at least three [redacted] the four lateral ring positions (2,3,7, and 8) with at least one unsub[redacted]tuted ring position. Toxic effects in humans include the following:

1. Ch[redacted]racne 2,3,7,8-TCDD is an active skin irritant capable of inducing form lesions. Chloracne may appear weeks or months after initial exposure.

2. He[redacted]toxicity 2,3,7,8-TCDD causes liver damage in laboratory animals and man.

3. Me[redacted]bolism disorders

4. Ca[redacted]iovascular disorders

5. Ur[redacted]ary tract disorders

6. Re[redacted]iratory tract disorders

7. Pa[redacted]reatic disorders

8. Pe[redacted]pheral neuritis

9. Lo[redacted]r extremi[redacted]/ weakness

10. S[redacted]sorial impairments (sight, hearing, smell, taste)

11. P[redacted]chiatric problems

Also 2[redacted],7,8-TCDD can produce or induce benign or malignant tumers in living [redacted]nimals. It is known to be embryotoxic and is capable of producing birth [redacted]normalities in test animals.

In sum[redacted]ry, 2,3,7,8,-TCDD is an extremely potent toxicant which should be handle[redacted]with the utmost caution. However, it has been handled for years with o[redacted] known injury in many analytical and biological laboratories. It should [redacted]e emphasized that all incidences, where human health effects have been c[redacted]arly demonstrated, have involved exposure to parts per million (milli[redacted]ams/kilo of sample) quantities of TCDD.

Material Safety Data [redacted]eet

[redacted]uired under USDL Safety [redacted] Health Regulations
[redacted]ipyard Employment (29 [redacted]R 1915)

U.S. Department of Labor
Occupational Safety and Health Administration

OMB No. 1218-0074
Expiration Date 05/31/86

PREPARED 1/10/86

Section I

Manufacturer's Name: ALCONOX, IN[redacted].

Emergency Telephone Number: (212) 473-1300

Address (Number, Street, City, S[redacted]. and ZIP Code): 215 PARK AV[redacted]NUE SOUTH

NEW YORK, N[redacted]Y. 10003

Chemical Name and Synonyms: N.A.

Trade Name and Synonyms: ALCONOX

C6300-1, C6301[redacted], C6301-2,-3,-4,-5

Chemical Family: ANIONIC DETERGENT

Formula: N.A.

Section II - Hazardous Ingre[redacted]nts D51-.502

Paints, Preservatives, and So[redacted]nts	%	TLV (Units)	Alloys and Metallic Coatings	%	TLV (Units)
Pigments: NONE			Base Metal: NONE		
Catalyst: NONE			Alloys: NONE		
Vehicle: NONE			Metallic Coatings: NONE		
Solvents: NONE			Filler Metal Plus Coating or Core Flux: NONE		
[redacted]dditives: NONE			Others: NONE		
[redacted]rs: NONE					

[redacted]zardous Mixtures of Othe[redacted]quids, Solids or Gases

	%	TLV (Units)
NONE		

Section III - Physical Data

Boiling Point (°F)	N.A.	Specific Gravity (H_2O=1)	N.A.
Vapor Pressure (mm Hg.)	N.A.	Percent Volatile by Volume (%)	N.A.
Vapor Density (AIR=1)	N.A.	Evaporation Rate (_____=1)	N.A.

Solubility in Water: APPR[redacted]IABLE

Appearance and Odor: WHIT[redacted] POWDER INTERSPERSED WITH CREAM COLORED FLAKES - ODORLESS

Section IV - Fire and Explos[redacted] Hazard Data

Flash Point (Method Used)	Flammable Limits	Lel	Uel
[redacted]NONE	N.A.	N.A.	N.A.

Extinguishing Media: WATE[redacted], CO_2, DRY CHEMICAL, FOAM, SAND/EARTH

[redacted]l Fire Fighting Procedure: FOR [redacted]IRES INVOLVING THIS MATERIAL, DO NOT ENTER WITHOUT PROT[redacted]CTIVE EQUIPMENT AND SELF CONTAINED BREATHING APPARATUS

Unusual Fire and Explosion H[redacted]rds: NONE

Section V - Health Hazard Data

Threshold Limit Value

NO DATA AVAILABLE - TREAT AS NUISANCE DUST

Effects of Overexposure

PROLONGED EXPOSURE TO DUST MAY IRRITATE MUCOUS MEMBRANES

Emergency First Aid Procedures

EYE - FLUSH WITH PLENTY OF WATER FOR 15 MINUTES, SKIN-FLUSH WITH PLENTY OF WATER. INGESTION - DRINK LARGE QUANTITIES OF WATER TO DILUTE MATERIAL. GET MEDICAL ATTENTION FOR DISCOMFORT.

Section VI - Reactivity Data

Stability	Unstable		Conditions to Avoid	NONE
	Stable			

Incompatability (Materials to Avoid) AVOID STRONG ACIDS

Hazardous Decomposition Products MAY RELEASE CO_2 GAS ON BURNING

Hazardous Polymerization	May Occur		Conditions to Avoid	NONE
	Will Not Occur			

Section VII - Spill or Leak Procedures

Steps to be Taken in Case Material is Released or Spilled MATERIAL FOAMS PROFUSELY, SHOVEL AND RECOVER AS MUCH AS POSSIBLE. RINSE REMAINDER TO SEWER. MATERIAL IS COMPLETELY BIODEGRADABLE.

Waste Disposal Method SMALL QUANTITIES MAY BE DISPOSED OF IN SEWER. LARGE QUANTITIES SHOULD BE DISPOSED OF ACCORDING TO LOCAL REQUIREMENTS FOR NON-HAZARDOUS DETERGENT

Section VIII - Special Protection Information

Respiratory Protection (Specify Type) DUST MASK

Ventilation	Local Exhaust	NORMAL	Special	N.A.
	Mechanical (General)	N.A.	Other	N.A.

Protective Gloves	USEFUL - NOT REQUIRED	Eye Protection	USEFUL - NOT REQUIRED

Other Protective Equipment NOT REQUIRED

Section IX - Special Precautions

Precautions to be Taken in Handling and Storing SHOULD BE STORED IN A DRY AREA TO PREVENT CAKING

Other Precautions NO SPECIAL REQUIREMENTS OTHER THAN THE GOOD INDUSTRIAL HYGIENE AND SAFETY PRACTICES EMPLOYED WITH ANY INDUSTRIAL CHEMICAL.

HEAT STRESS PREVENTION: See attached

Control Measures:

Recognition of heat illnesses:

General First Aid:

UNUSUAL OR SPECIAL HAZARDS: (For ex., Tsunamis, etiologic hazards & indigenous pathogens etc.)

CHEMICAL HAZARDS:

The attached table summarizes the physical, chemical and toxicological data of key hazardous materials identified at the study site.

Notes: _____

ARNESON TIMBER SITE

Task Safety and Health Risk Analysis

All activities onsite have certain hazards associated with them that are common to any site work. Listed below are the common hazards with appropriate precautions and avoidance measures:

	PHYSICAL HAZARDS	PRECAUTIONS
_____	Heat Stress	Be aware of heat stress symptoms and treatment; drink plenty of fluids; take sufficient breaks. Follow attached heat stress SOP(s).
_____	Sunburn	Apply sunscreen; wear hat, cap, long sleeves, etc., as needed.
__X__	Cold Stress	Be aware of cold stress symptoms and treatment; wear warm, dry clothing; take sufficient breaks. Follow attached cold stress procedures(s)
__X__	Uneven or slippery terrain	Be aware of trip/fall hazard; walk carefully; wear boot covers if needed.
_____	Debris onsite	Be aware of debris; walk carefully.
__X__	Overhead obstructions	Be aware of overhead piping and other objects; wear hard hat.
__X__	Proximity to heavy equipment	Be aware of heavy equipment operations and back-up alarm; keep safe distance from moving equipment; non-essential workers out of area.
__X__	Noise	Wear appropriate ear protection; use hand signals.
__X__	Flammable fuels for equipment and generators	Keep away from ignition sources and/or flames; store properly.
_____	Deteriorated drums and/or tanks	Avoid contact unless activity involves contact; walk carefully near them; keep safe distance from them during handling, pumping, etc; don't move or stand on them.
_____	Compressed gas cylinders on site	Avoid contact; don't move; be aware of projectile hazard if cylinder knocked over and neck broken.
_____	Ponds, lagoons,	Be aware of drowning danger

	pit on site	especially in PPE; walk on edges carefully; avoid going out on catwalks over liquids; be aware of hardened "crusts" covering liquids.
X	Brushy, wooded areas	Wear safety glasses; walk carefully.
	Confined space	Do not enter without special safety meeting, procedures, safety equipment, emergency planning. Complete confined space entry form; follow SOP attached. Permission of OSC and SSO.
	Flammable chemicals present at site	Keep away from ignition sources or flames; use intrinsically safe and/or sparkproof equipment or tools as much as possible. Fire extinguisher on hand during handling.
	Explosive, shock-sensitive or unstable materials at site	Be very careful when working near these; avoid contact; don't move, jar, shake, or open.
	Bulging drums or containers	Be very careful when working near them; avoid contact. Don't move, open, jar, or touch.
	Radioactive materials onsite	Monitor with proper equipment; wear TLD badge; avoid contact; wear proper PPE (See V.B.). Special entry procedures, decon, etc.
	Snakes, spiders, scorpions, ticks, bees, dogs, etc.	Be aware of potential hazard of bite and treatment. Walk carefully; wear safety boots. Use insect repellent as needed. Be aware of warning signs. Watch for fire ant mounds, bee hives, hornet nests, high brush, piles of debris, overhead obstructions, etc.
X	Sharp edges, debris, nails, etc.	Be aware of cut/puncture hazard; wear appropriate protective gloves and boots.
	Dark rooms, areas	Use flashlights; walk carefully; stay near buddies; use radios if needed.

CHEMICALS	PRECAUTIONS

X	Contaminated air	Wear proper PPE for the task to avoid inhalation; [redacted] be aware of nature of chemical hazard.
X	Contaminated soil/ sludges/liquids	Wear proper PPE for the task to to avoid direct contact; [redacted] PPE depends upon activity, chemical, etc; be aware of nature of chemical hazard and possible slippery walking surface.
X	Chemicals known or suspected to be present at site	Be aware of nature and types of chemicals present, effects, symptoms of exposure, treatment; see chemical info in [redacted] attachments; be aware of physical hazards of the chemicals (ex. flammable, unstable, explosive). Avoid contact; wear proper PPE for task to prevent exposure. [redacted]
	Splash by liquids present at site	Wear Level C protective clothing with Saranex coveralls and/or splash apron; work carefully.
X	Decontamination solvents (Alconox, hexane, etc.)	Be aware of chemical and physical properties and contents of attached MSDS sheet. Wear proper PPE during use. Keep hexane away from ignition sources.

BIOLOGICAL MATERIALS	PRECAUTIONS
Medical wastes	Be aware of potentially infectious waste; avoid contact. Wear proper PPE for the task (See V.B.), especially gloves and respiratory protection. Watch for needles, syringes, etc. Be aware of possible presence of chemical and radioactive waste also; wear TLD; air monitor for radiation and organic vapors.

TRAINING SSIGNMENTS

All site staff will h e completed the OSHA 40-hour Hazardous Waste Operations Training, 24-hour onsite supervised t ming and appropriate annual updated. In addition, site supervisors will have completed OSHA r Supervisory Training and First Aid/CPR Training. Occasional site workers that will not receive ex sures exceeding permissible exposure limits (geophysical and land surveyors) require only 24 ho of OSHA Hazardous Waste Operations Training and two days onsite training and supervision.

Documentation th raining assignments have been met will be required prior to site entry.

MEDICAL URVEILLANCE REQUIREMENTS

All site staff will h e medical clearance to perform work on hazardous waste site, following protocols at least as stringent a hose defined in the Ogden Medical Surveillance Program.

Documentation of edical clearance will be required prior to site entry.

TABLE 2

INITIAL ASS NMENTS OF PROTECTION LEVELS, TRAINING, AND MEDICAL SU VEILLANCE ASSIGNMENTS FOR SITE WORK TASKS

Task Name	Level of Protection	Haz Waste Train. 40-Hr	24-Hr	Med Surv.
SAMPLING	D	X	X	X
EXCAVATIO	D	X	X	X
MANAGEME T	D	X	X	X

AIR S RVEILLANCE

EXPOS E MONITORING:

Type	Frequency
Backgro d	
Perimete	Twice per day using direct-reading instruments during intrusive activities.
Personne	At least twice per day in the breathing zone of those with the highest anticipated exposure during intrusive activities.
Area	

EQUIPM NT:

Eq ment	Contaminant
High V ume Personnel Samplers	PCP

ACTION EVELS: (Action levels should be established for upgrading/downgrading PPE, work stoppages, and evacuation)

Equipme	Action Level	Action
High Vol e Perso l Sampler	0.5 mg/m^3	Stop work, employ respirator add water to soil

SITE CONTROL

SITE SECURITY: (Summarize below)

The site and a buffer area are controled by a barbed wire fence with a locked gate.

Signs are posted to properly warn the Public.

VISITOR ACCESS: (Summarize below)

Access to the site is through the gate. Immediately inside the gate, to the north, is the clean area, or staging area. Visitors should park on the east side of Shady Lane on Mr. Leezy's property.

WORK ZONES: (Summarize below)

Support/Clean Zone (S/CZ): The clean zone is in the extreme north east corner of the site. It will be designated by white plastic tape.

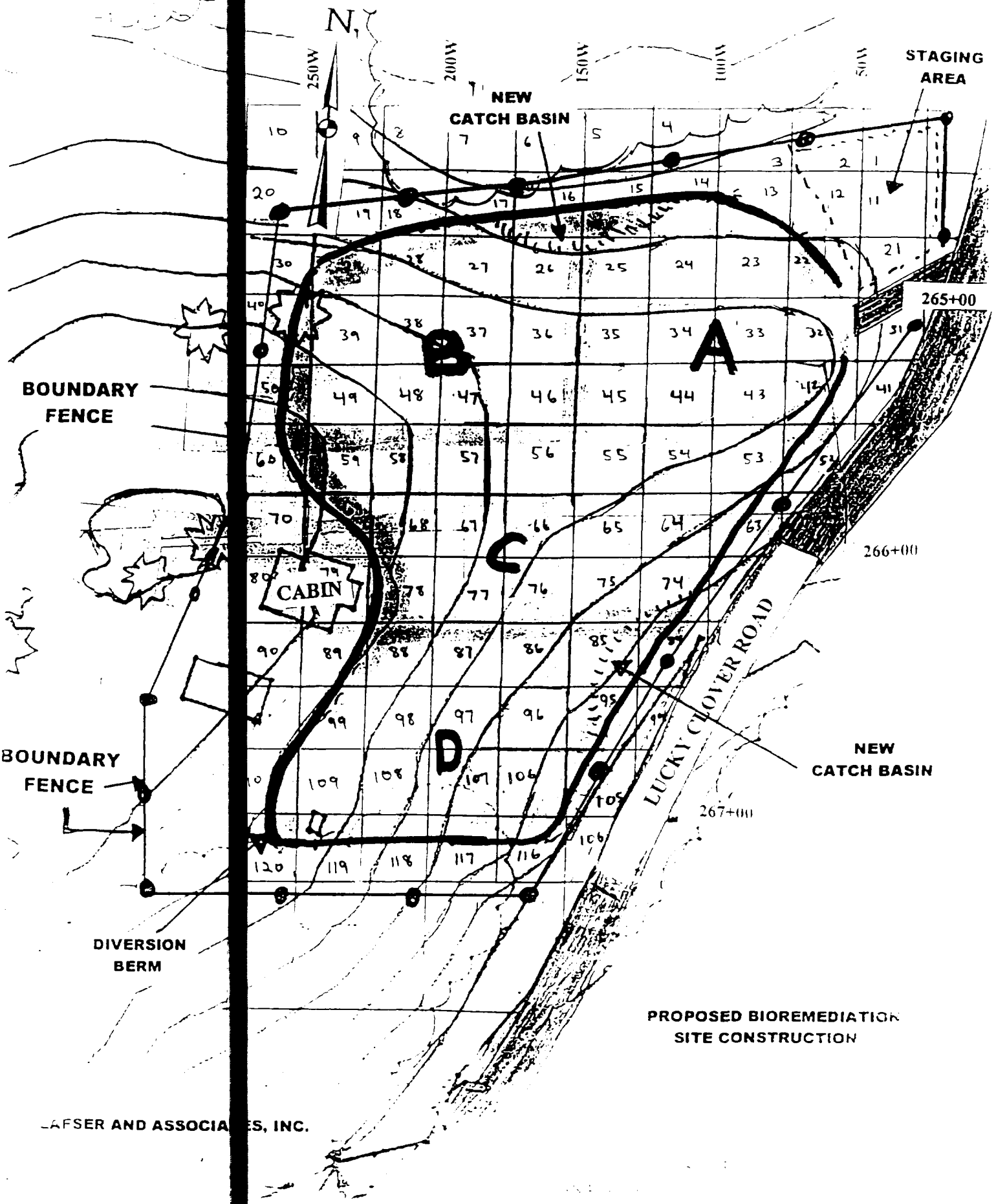
Contamination Reduction Zone (CRZ): The contamination zone is designated by the perimeter barbed wire fence.

Transition Zone (TZ): This zone will be designated by blue plastic tape.

Exclusion Zone (EZ):

OTHER: (Summarize below)

ARNESON TIMBER SITE
STEELVILLE, MISSOURI



LAFSER AND ASSOCIATES, INC.

SAFE WOR PRACTICES:

1) Unauthorized rsonnel are not allowed onsite, particularly in the Exclusion Zone.

2) Work groups l always consists of at least two (2) team members.

3) Wind-flags w e positioned onsite so that work can be preformed upwind as much as possible.

4) Smoking, eati drinking, chewing gum or tobacco, taking medication, and applying cosmetics will not be permitt within any restricted or exclusion zone.

5) Wearing of co ct lenses is prohibited.

6) Open flames a not allowed anywhere onsite without an operating permit from the facility.

7) Personnel und he obvious influence of alcohol or controlled substances are not allowed onsite.

8) Personnel will oid skin contact with contaminated or potentially-contaminated media. If such contact occurs e contaminated clothing will be removed and the affected areas washed thoroughly with soap and ter.

9) Personnel will scard and replace any damaged, or heavily soiled protective clothing.

10) Personnel sho notify the onsite health and safety coordinator of any defective monitoring , emergency, or er safety equipment.

11) A supply of po ble water, electrolyte replacement solutions, a shaded area and sufficient lighting will be mainta d onsite; sanitary facilities will be accessible to personnel.

12) All site person will familiarize themselves with these and the emergency procedures by use of daily tailgate s ty meetings.

DOCUMEN ATION AND RECORDKEEPING

The onsite health a safety coordinator must carefully document the implementation of this health and safety plan and wil erefore establish and maintain project-specific Health and Safety Files.
The file may conta he following records:

- Certif tion letter(s) of medical and training requirements
- Signe ertification page of this health and safety plan
- Signe Visitor and Contractor Orientation Forms
- Signe Daily Tailgate Safety Meeting Forms
- Air su eillance records of environmental and exposure monitoring
- Heat s ss monitoring records
- Super or's report of personnel accidents or environmental incidents
- Safety udit records including violations and remedial action plans
- Daily alth and safety notations in the Site Log, held by the Site Manager
- Docu ntation of changes made to this health and safety plan

DECONTA INATION PROCEDURES: (Procedures for the decontamination of sampling tools a other related equipment are specified in the work plan and/or QA plan. Note that separate areas sh d be established for personnel, sampling and/or heavy equipment decontamination.)

PERSONNEL CONTAMINATION: (Summarize equipment and procedures below)

Equipment: S MPLING AND OTHER SMALL ITEMS WILL BE WASHED WITH ALCONOX A D WATER FOLLOWED BY A FINAL RINSE

Decon Solution: ALCONOX AND WATER

Procedures: R USABLE TOOLS AND PPE WILL BE CLEANED OF VISIBLE S L AND SCRUBBED WITH A BRUSH WITH WATER, A D RINSED.

HEAVY EQUI ENT DECONTAMINATION: (Summarize equipment and procedures below)

USE HAND QUIPMENT TO REMOVE AS MUCH SOIL AS POSSIBLE, FOLLOWED Y A WATER PRESSURE WASH

DISPOSAL PR EDURES: (Summarize equipment and procedures below)

DISPOSAB E SAMPLING EQ AND PPE WITH VISIBLE SOIL CONTAMINATION WILL BE C TAINERIZED AND PROPERLY DISPOSED

SPECIAL INST UCTIONS: (Summarize equipment and procedures below)

Emergency Dec

EMERGEN Y RESPONSE

General: CONT CT SITE MANAGER OR IF NOT AVAILABLE, CONSTRUCTION MANAGER. HUT DOWN ALL EQUIPMENT, NOTIFY APPROPRIATE EMERGENC SERVICE.

Emergency Coor ator (EC):

FRED A. LA SER

Safe Refuge Area

OFFICE AR A OR VEHICLES.

BLOODBO NE PATHOGEN EXPOSURE CONTROL PLAN

1. Exposure De mination:

   Any field pe nnel trained in first aid response has the potential to be exposed to blood borne pathogens. T ks where exposures could occur include a bleeding injury from working around rotating or he y equipment.

2. Exposure Co ol

   A. Personal otection equipment: While rendering first aid where exposure to blood may occur, [Enter Co pany Name] employees will don protective gloves (N-Dex undergloves or Nitrile overglov). These gloves should be readily available at any hazardous waste site.

   B. Hepatiti Vaccination: First aid providers whose primary job assignment is not first aid admini ion do not need to receive a pre-exposure hepatitis B vaccine. However, all first aid- oviders assisting in any situation involving the presence of blood – regardless of wheth or not a specific exposure incident occurred – must be offered the full Hepatiti immunization series no later than 24 hours after an incident.

   C. Exposur ncident Evaluation: All first aid incidents involving exposures must be reported to [Enter c pany name] Human Resources and Health and Safety before the end of the work shift in w ch the incident occurs. A first aid incident report must be completed describing the circu stances of the accident and response. Following a report of an exposure incident, [Enter C pany Name] shall make immediately available to the exposed employee a confide l medical evaluation and follow-up.

SITE – SPE IFIC RESPONSE SCENARIOS:

Natural Disaste

REPORT TO SITE MANAGER, SHUT DOWN EQUIPMENT, SEEK SHELTER

Injury Accident Project Personnel or Visitors

REPORT TO SITE MANAGER, APPLY FIRST AID, CONTACT APPROPRIATE EMERGENCY SERVICE

Spill of Hazardo Materials

REPORT TO SITE MANAGER, TAKE APPROPRIATE ACTIONS TO CONTAIN SPILL AND LEAN UP CONTAMINATION.

Fire or Explosio

REPORT TO SITE MANAGER; NOTIFY APPROPRIATE EMERGENCY SERVICE

EMERGENCY REFERENCE LIST (Keep posted in vehicles and near communication system)

MEDICAL EMERGENCIES:

Hospital Name: MO. BAPTIST HOSPITAL Hospital Address: SULLIVAN MO.

Hospital Telephone: 573-468-221

Distance: See attached map
APPROX. 30 MILES

EMERGENCY SERVICES:

Service:	Name:	Telephone Number:
Ambulance:	911 OR 573-775-221	
Fire Department:	911 OR 573-775-2708	STEELVILLE
~~Security~~		
Poison Control Center:	PHELPS CO. HOSP., ROLLA, MO.	573-364-1322
~~OTHER~~		

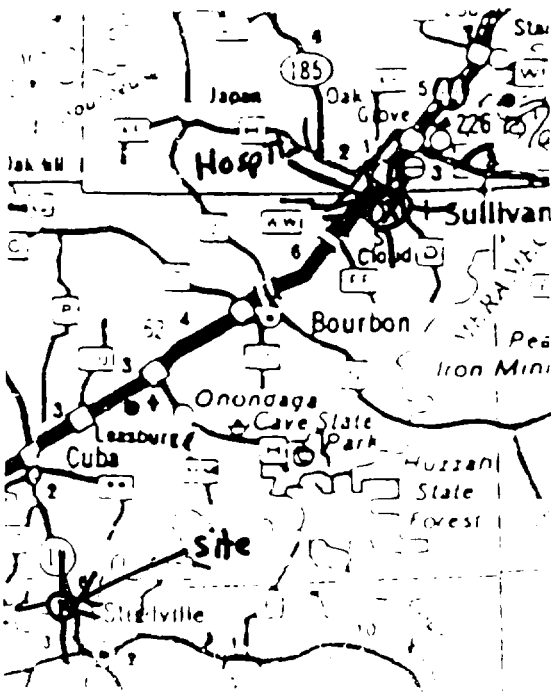
CALL LIST:

Title:	Name:	Telephone Number:
H & S Manager	FRED LAFSER	314-570-6969 (MOBILE)
Project Manager	(SAME)	
Field Manager	(SAME)	
H & S Coordinator	(SAME)	
Client Contact	ARNE ARNESON	314-692-9999
~~OSHA~~ EPA	TIM CURRY	913-551-7636

EMERGENCY EQUIPMENT: (Check all that apply)

(X) First Aid Kit	(X) Fire Extinguishers	(X) Water
( ) SCBA	( ) Escape Packs _____	( ) Alarms
( ) Spill Equipment	(X) Mobile Phone	( ) Fire Blanket
( ) Other _____		

MAP TO HOSPITAL



DIRECTIONS TO HOSPITAL

FROM THE SITE TAKE LUCKY CLOVER ROAD NORTH TO ROUTE 19, THEN PROCEED NORTH TO I-44 AND GO EAST TO SULLIVAN, MO. AND EXIT AT THE 225 EXIT. TURN RIGHT OFF EXIT RAMP AND THEN TURN RIGHT AGAIN ON THE SOUTH SERVICE RD. CONTINUE TO HWY AND TURN LEFT. PROCEED 1 1/2 MILES AND TURN RIGHT ON S PPINGTON BRIDGE RD. GO 1/2 BLOCK TO MISSOURI BAPT T HOSPITAL.

HEALTH A ND SAFETY PLAN CERTIFICATION

I have had the op tunity to read and ask questions about this health and safety plan. My signature indicates that I un rstand the procedures and restrictions of this plan and agree to abide by them.

Signatur	Company	Date

FORMS

SITE SAFETY PLAN

FIRST AID INCIDENT REPORT

Date for Report:

Date of Accident:

Person Completing This Report:

Description of the Accident: (time, location, event, description of injuries)

Accident Victim Name:

Name(s) of First Aid Provider(s):

Bloodborne Pathogen Exposure Incident Evaluation:

The First Aid Responder was exposed to blood or other potentially infectious materials by contact with the:

( ) No Exposure

( ) Eye
( ) Mouth
( ) Other Mucous Membrane
( ) Non Intact Skin (cuts, abrasions)
( ) Needlestick
( ) Human bite

Exposure Control Precautions Taken: describe any protection measures taken by the responder (i.e., gloves, face mask, etc.).

Please forward this completed form to Human Resources and your Divisional Health and Safety Manager.

APPENDIX A

TAILGATE SAFETY MEETING REPORT

TAILGATE SAFETY MEETING REPORT

Date _____ Site ARNESON TIMBER SITE--STEELVILLE MO.

Attendees _____

_____

_____

ORDER OF BUSINESS

Topic(s) discussed _____

_____

_____

Safety Suggestions _____

_____

_____

Action taken on previous meeting suggestions _____

_____

_____

Emergency Information _____

_____

_____

Inquiries and accidents since previous meeting _____

_____

_____

Additional comments _____

_____

_____

Meeting conducted by _____ Title _____

Signature _____ Date _____

TAILGATE SAFETY MEETING REPORT

Date _____ Site ARNESON TIMBER SITE

Attendees _____

_____

_____

ORDER OF BUSINESS

Topic(s) discussed _____

_____

_____

Safety Suggestions _____

_____

_____

Action taken on previous meeting suggestions _____

_____

_____

Emergency Information _____

_____

_____

Inquiries and accidents since previous meeting _____

_____

_____

Additional comments _____

_____

_____

Meeting conducted by _____ Title _____

Signature _____ Date _____

APPENDIX B

VISITOR AND SUBCONTRACTOR

HEALTH AND SAFETY SITE ORIENTATION

SITE S[redacted]FETY PLAN

VISITOR AND

SUBC[redacted]NTRACTOR VISITOR HEALTH AND SAFETY ORIENTATION

SITE Arnes[redacted]n Timber Site

ONSITE HEA[redacted]TH AND SAFETY COORDINATOR Fred A. Lafser

SITE DESCR[redacted]TION Former wood treating site with soils contaminated with Pentachlorophenol (PCP). 1.07 [redacted]cres of vacant land surface. Heavy equipment may be operating.

POSSIBLE S[redacted]E CONTAMINANTS AND HAZARDS PCP is toxic. Dust inhalation and skin contact should be a[redacted]ded. Heavy equipment or trucks may be operating.

The informat[redacted] summarized below is important for you to read and fully understand. This information has been extracted from the site [redacted]ecific Health and Safety Plan, and has been compiled to help insure your health and safety onsite. If you have any question[redacted]garding the information presented below, please ask your escort for clarification.

HEALT[redacted] SAFETY AND SECURITY INFORMATION

1. You mu[redacted]sign in and out of the Visitor Log Book maintained at the site. This assists in identifying all the visitors at the site [redacted]the event of an emergency.
2. If your [redacted]siness takes you beyond the designated VISITOR'S AREAS, you must be escorted. If you are observed unesco[redacted]d in an unauthorized area, you will be asked to leave immediately.
3. Areas [redacted]rked with yellow and black tape stating "CAUTION – DO NOT ENTER" demarcate where the Exclusion (contar[redacted]ated) areas begin. You are not allowed to enter these areas.
4. Access [redacted] the contaminated area is strictly forbidden to all visitors unless they have approval of the client and can produc[redacted]dequate written proof of adequate training and medical certification prior to arrival onsite.
5. Hard h[redacted]s and visitor safety glasses must be worn at all times onsite. Your escort will provide you with this safety equipm[redacted]nt.
6. Please [redacted]ad and follow all safety signs onsite. The signs are there to alert you to possible physical and chemical hazards.
7. Eatin[redacted]nd smoking is not allowed onsite. You may eat or smoke in designated clean areas or in your vehicle.

8. Observe the proper lookout, *tag-out* procedure before working on electrical and/or rotating equipment.

9. Normal subcontractor shift hours coincide with the regular [Site Name] work schedule.

10. Report any accident or injury (even if minor to you) to the Site Health and Safety Coordinator.

11. No one under the age of 18 is permitted onsite without prior approval of the client.

12. No domestic animals are permitted onsite.

13. Complete cooperation with the Health and Safety Plan must be maintained. Any violation may result in expulsion from the site.

14. In the event of a site emergency, please walk immediately to the designated meeting area for the site. You will receive further instructions from this location. Please stay in this meeting area until the all clear signal is given from the site Health and Safety Coordinator or offsite emergency support personnel.

15. Please cooperate fully with those in authority in the event of an emergency.

ACKNOWLEDGEMENT OF INFORMATION

ARNESON TIMBER CO.

I have read and understand the above information provided by [Company Name] XXXXXXXXX and have had an opportunity to direct questions of a health and safety nature, and have received adequate answer or explanations from my escort or other site staff member.

Visitor Signature	Print Name	Affiliation	Date

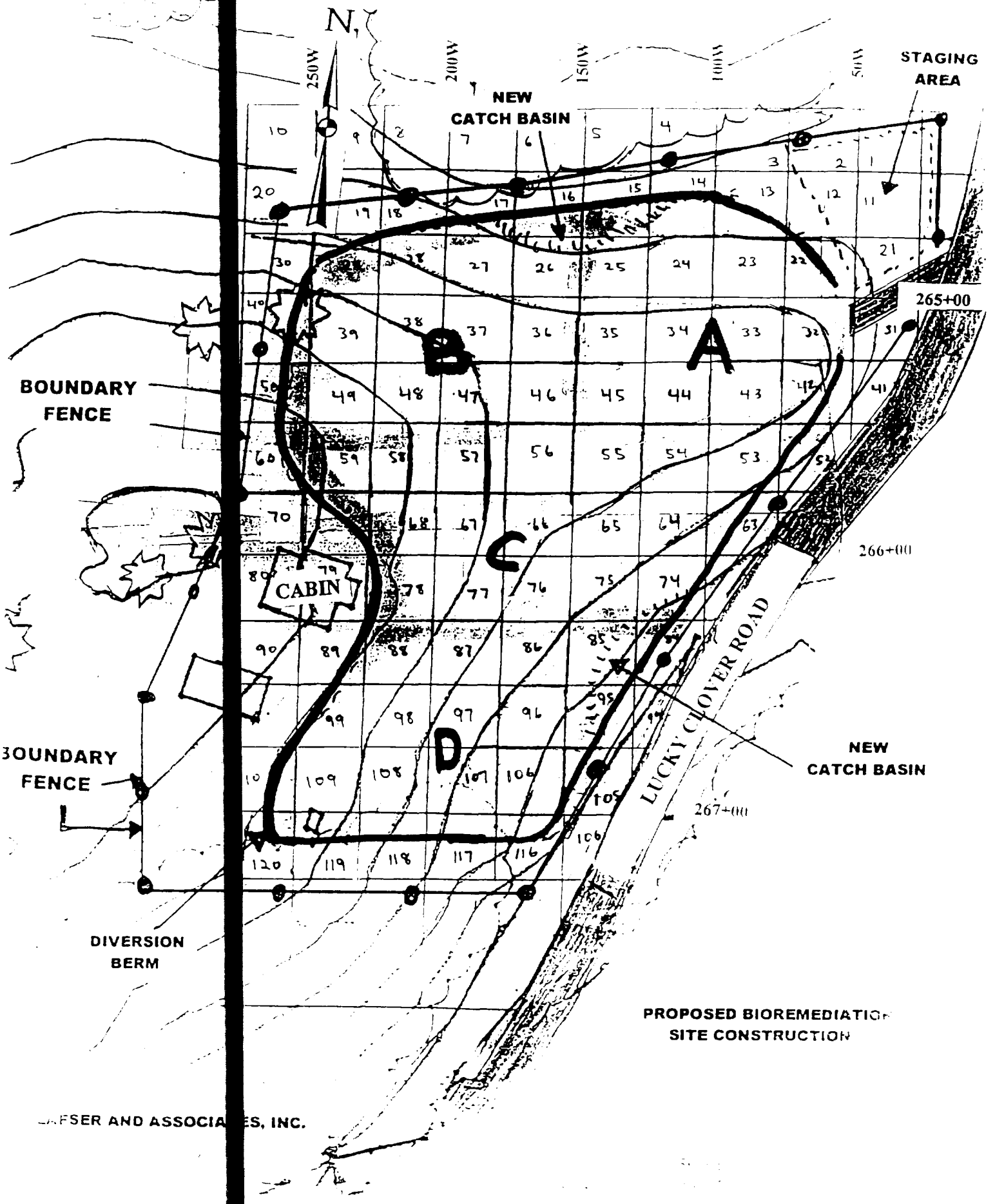
ARNESON TIMBER SITE
PCP BIOREMEDIATION PLAN

A study with recommendations from the Washington University Environmental Engineering Program follows. These recommendations further define the bioremediation program for the site.

The site set-up is described in earlier sections of the RAWP - Removal Strategy. It is expected that the levels of PCP in soil will be reduced to below 9.1 mg/kg after 2 warm seasons.

Final clearance sampling is expected to occur in October, 2001. If results indicate levels above 37.7 mg/kg PCP, management and monitoring of the site will continue for up to an additional 3 years or until levels below 37.7 mg/kg are obtained. The site will be left on the State Registry and the fence will remain. The site will have met the goals of the order. If and when the soil levels fall below 9.1 mg/kg, application will be made to MDNR to remove the site from the registry. The landfarm will them be seeded with a mixture approved by ATC, the landowner and EPA.

ARNESON TIMBER SITE
STEELVILLE, MISSOURI



ARSER AND ASSOCIATES, INC.

Feasibili Investigation for Bioremediation of PCP-Contaminated Soil at the Arneson Timber Company, Crawford County, MO

Brian A. Wrenn, Ph.D.
Environmental Engineering Program
Civil Engineering Department
Washington University
Campus Box 1180
One Brookings Dr.
St. Louis, MO 63130
(314) 935-8144

Submitted to:
Fred Lafser
Lafser & Associates, Inc.
409 Greenfield Dr.
St. Louis, MO 63132-4208
(314) 994-7001

July 29, 1999

Introducti

Site Descr tion:

The A neson Timber Company site is an approximately 1 acre site located near the top of a ridge in ar nincorporated, wooded area in Crawford County, MO. This site was used for timber cutting an wood-preserving operations between 1978 and January 1983. Pentachlorophenol (PCP) dis ved in diesel fuel was the only wood-preserving chemical used at this site.

The s face soil is a stony loam 8 to 15 inches deep underlain by a well-developed red clay. The prese e of a large number of stones and a high percentage of clay are important factors for bioremedi on feasibility at this site. PCP concentrations ranging from below the detection limit to 1,400 m /kg soil were observed during site investigations. (The median PCP concentration for all sam es that were analyzed is 1.1 mg/kg, and 75% of all analyzed samples contained less than 10 m PCP/kg soil.) Most contaminated soil samples were collected from within12 inches of the gro d surface, but deeper contamination is suspected in the vicinity of a filled former sludge co inment basin and the former drip pad.

Tech al grade PCP is about 85-90% PCP, with the remainder consisting of lower chlorinate phenols (esp. tri- and tetrachlorophenols). Polychlorinated dibenzodioxins and dibenzofu ns (PCDD/Fs) are also frequent contaminants of technical-grade PCP formulations. The most undant dioxin in technical-grade PCP is octachloro-*p*-dibenzodioxin (OCDD), which is relative nontoxic (Crosby, 1981). Dioxins have been observed at the Arneson Timber site at concentra ns up to 10 µg/kg as 2,3,7,8-TCDD equivalents, which is equal to the EPA's action level for P DD/Fs in industrial-use soils at depths of 0 to 48 inches. Tri- and tetrachlorophenols are biodeg dable, but dioxins are not.

Proposed reatment:

The p posed remedial action for PCP-contaminated soil at this site is landfarming, which relies on a obic biodegradation of the target contaminants. Contaminated soil will be excavated and place n a treatment cell that will be surrounded by soil berms to control run-on and runoff. The soil i he treatment cell will be monitored and managed to optimize biodegradation of PCP and its fue oil carrier.

PCP d diesel fuel are both known to be biodegradable by a wide variety of common aerobic m roorganisms (McAllister *et al.*, 1996; Song *et al.*, 1990). In many cases, the organisms hat catalyze biodegradation reactions use the contaminants as growth substrates (Saber an Crawford, 1985; Song *et al.*, 1990). Some microorganisms can transform PCP and diesel-fue ydrocarbons during growth on other organic substrates (Banerji and Bajpai, 1994; Sato and e, 1996; Lamar *et al.*, 1990). This type of "cometabolic" process is the basis for a well-studi technology for treating PCP-contaminated soil: inoculation with white-rot fungi (Lamar *et* ., 1990; Lamar and Dietrich, 1990; EPA, 1995). Unfortunately, white-rot fungi mineraliz i.e., convert to harmless inorganic products like CO_2 and H_2O) only a small percentag f the PCP that is removed (Lamar *et al.*, 1990). The remainder is converted to volatile p ducts, such as pentachloroanisole, or incorporated into soil organic matter. Incorpora n of PCP into soil humus is not accompanied by a significant amount of dechlorin on, and the ultimate fate and environmental effects of the chlorinated aromatic residues i soil organic matter are unknown. Therefore, processes that result in contaminant mineraliz on, such as occurs when indigenous soil bacteria grow on PCP, are preferable.

Factors Affecting the Biodegradation of PCP

As with any aerobic biological process in which the target contaminant supports microbial growth, the success of PCP bioremediation at the Arneson Timber site will depend on:

1) the presence of a competent microbial population (i.e., PCP degraders);

2) an adequate rate of oxygen transfer to the soil;

3) the presence of sufficient available nutrients;

4) neutral pH (between about 6 and 8.5); and

5) soil moisture content maintained within the optimal range (60 to 80% of the field capacity)

In addition to the factors listed above, PCP bioremediation frequently benefits from addition of wood chips, sawdust, or compost. These materials serve several functions, including provision of additional carbon sources to support growth of a diverse microbial population (which often results in more complete biodegradation), adsorption of PCP (which reduces the toxicity to soil microbes), and as a bulking agent to improve the water-holding and oxygen-transfer characteristics of the soil.

A study was conducted to determine whether PCP degraders are present in contaminated soil at the Arneson Timber site. Data collected during this study conclusively demonstrated that microorganisms with the ability to grow on and degrade PCP exist at this site (see Appendix). Also, the size of the total heterotrophic microbial population is within the range expected for surface soil (Alexander, 1977; Konopka and Turco, 1991), which indicates that unrecognized toxicity is probably not a problem.

In landfarming, aeration is provided by frequent tilling. The soil moisture content can be managed by irrigation and provision of an underdrain or leachate collection system. The nutrient concentrations and pH of the contaminated soil can be adjusted by addition of appropriate amendments (e.g., commercial or organic fertilizer for nutrients, lime, crushed limestone, or sulfur for pH control).

Recommendations for Bioremediation of PCP-Contaminated Soil at the Arneson Timber Site

(1) The soil at the Arneson Timber site is a mixture of clay, gravel, and a loamy sand. The clay and gravel, in particular, will make it difficult to mix the soil and amendments during bed preparation. The clay will reduce the rate of oxygen transfer into the soil and will make moisture management difficult. Therefore, addition of wood chips or sawdust as a bulking agent will be very beneficial at this site.

   a) The quantity of bulking agent added to PCP-contaminated soils varies from zero to 100% of the volume of soil treated (Laine and Jorgensen, 1997; Laine *et al.*, 1997; Truel *et al.*, 1994; Johnston *et al.*, 1997; McGinnis *et al.*, 1991). The poor quality of this soil suggests that a relatively large proportion of sawdust or wood chips should be used. A volume of sawdust or wood chips equal to 50-100% of the volume of soil that will be treated should be mixed with the contaminated soil during construction of the landfarm treatment cell.

      - Since a relatively large volume of bulking agent is recommended, and the high clay and gravel content of the contaminated soil will reduce the efficiency of mixing, the

awdust or wood chips should be added gradually over the first 2-3 months of peration. The amendments can be mixed into the soil bed during regularly heduled tilling operations.

b) Sin this soil, especially areas dominated by clay, appears to contain relatively small am nts of natural organic matter, addition of compost or composted manure may also be ben icial. If compost is added, reduce the volume of sawdust so the total amount of am dment does not exceed 100% of the volume of the contaminated soil.

- lthough composted yard waste and composted manure will both provide beneficial ganic matter, composted manure will also function as a slow-release source of trients (see Recommendation 3). Composted chicken manure and livestock dding material are particularly useful as nutrient amendments.

(2) The pH nd moisture content of soil samples collected for microbial enumeration are given in Table 1 The averages reported are based on analysis of five independent replicate samples from ea location.

Ta e 1: Characteristics of soil samples collected from the Arneson Timber site

Loc on	soil type	contamination status	average pH	soil moisture (%)*
drip ad	gravel and loam	stained, oily	6.47 ± 0.20	13.5 ± 3.6
catch asin	clay and gravel	stained, oily	5.60 ± 0.52	16.8 ± 4.3
backg und	silty loam	clean	6.75 ± 0.44	10.6 ± 1.4

*soil m sture = mass water per mass dry soil x 100%

a) Alth gh the pH of the soil collected near the former drip pad is within an acceptable rang the soil collected from the former catch basin area is too acidic. If a sufficient quan y of the soil in the landfarm treatment cell is from the catch basin or an area with simil soil characteristics, pH adjustment will be necessary. The pH should be main ined between about 6.5 and 8. The pH can be adjusted by adding crushed lime ne or lime, but the exact quantity required must be determined empirically.

- T landfarm treatment cell, with all other amendments added, should be constructed b ore the need for pH adjustment is determined. If pH adjustment is necessary, the a unt of crushed limestone that is needed can be estimated and the appropriate a unt can be added during one of the regularly scheduled tilling operations.

b) The s l moisture content listed above is for the native soil. Assuming the porosity is abou 0% and the solids density is 2.65 g/cm^3, these values represent 65% (clean back und) to 104% (catch basin) saturation. The optimum soil moisture content for biore diation is between 60 and 80% of saturation.

- If e soil becomes water logged following a heavy rain, anaerobic conditions could d elop. Although temporary anaerobiosis will not affect the long-term prospects for b ogical treatment of these soils, aerobic biodegradation will not occur as long as ox gen is absent. Therefore, a drainage system should be provided if maximum re diation rates are desired.

c) T soil pH and moisture content of the landfarm treatment cell can be monitored in the fi d using a Kelway Soil Acidity and Moisture Tester (Model HB-2). The field in rument should be calibrated periodically according to the manufacturer's re mmendations.

(3) The n rient content of the soil should be adjusted by addition of commercial fertilizer (e.g., amm um nitrate plus soluble phosphate). Sufficient fertilizer should be added to raise the nitrog concentration to about 250 to 300 mg N/kg soil as ammonium and/or nitrate and the phosp te concentration to about 25 to 50 mg P/kg soil as P_2O_5 or as phosphate (Laine and Jorge en, 1997; Laine *et al.*, 1997; Trudell *et al.*, 1994).

- A rnatively, composted manure or livestock bedding material can be added to supply nu ents (see Recommendation 1). If manure or bedding material is used, the readily av lable nutrient fraction (i.e., ammonia, urea, and soluble phosphorus) should be de rmined in addition to total N and P.

(4) The co taminated soil in the landfarm should be tilled frequently enough to maintain a soil-gas O_2 oncentration of at least 5% (Sims, 1996). To insure that this aeration requirement is met, t soil should be treated in maximum lifts of 12 to 15 inches, and it should be tilled freque y. Tilling every other week for the first few months followed by monthly tilling therea r should provide adequate aeration.

- Si e biological activity will be minimal during the winter, tilling should not be co ucted during cold weather. Tilling should be stopped when the average daytime te erature falls below 40° F and should not begin again until it rises above 40° F in the sp g. In general, this will probably be between about October 15 and April 15.

References

Alexander M. 1977. *Introduction to Soil Microbiology*, 2nd Ed., John Wiley and Sons, Inc., New ork.

Banerji, S. and R.K. Bajpai. 1994. Cometabolism of PCP by microbial species. J. Hazardous Mate ls 39: 19-31.

Crosby, D. 1981. Environmental chemistry of pentachlorophenol. Pure Appl. Chem. 53: 1051 080.

EPA. 199 Bioremediation Field Initiative Site Profile: Escambia Wood Preserving Site. U.S. EPA/ RD/OSWER. EPA/540/F-95/506G.

Fredrickso J.K., T.R. Garland, R.J. Hicks, J.M. Thomas, S.W. Li, and K.M. McFadden. 1989. Litho phic and heterotrophic bacteria in deep subsurface sediments and their relation to sedim t properties. Geomicrobiol. J. 7: 53-66.

Konopka. A and R. Turco. 1991. Biodegradation of organic compounds in vadose zone and aquife sediments. Appl. Environ. Microbiol. 57: 2260-2268.

Laine, M.M and K.S. Jorgensen. 1997. Effective and safe composting of chlorphenol-conta nated soil in pilot scale. Environ. Sci. Technol. 31: 371-378.

Laine, M.M H. Haario, and K.S. Jorgensen. 1997. Microbial functional activity during comp ing of chlorophenol-contaminated sawmill soil. J. Microbiol. Methods 30: 21-32.

Lamar and Dietrich. 1990. In situ depletion of PCP from contaminated soils by *Phanerochaete* sp. AEM 56: 3093-3100.

Lamar, R. T., J.A. Glaser, and T.K. Kirk. 1990. Fate of pentachlorophenol (PCP) in sterile soils inoculated with the white-rot basidiomycete *P. chrysosporium*: mineralization, volatilization, and depletion of PCP. Soil Biology and Biochemistry 22: 433-440.

Johnston, P.G., M.A. Becerra, R.L. Lutz, M.A. Staton, C.A. Axtel, and B.D. Bass. 1997. Fungal remediation of PCP and TNT contaminated soil in the field. In: *Proceedings, Fourth International In Situ and On-Site Bioremediation Symposium: Volume 2*, pp. 537-544. Battelle Press, Columbus, OH

McAllister, K.A., H. Lee, and J.T. Trevors. 1996. Microbial degradation of pentachlorophenol. Biodegradation 7: 1-40.

McGinnis, J.D., H. Borazjani, M. Hannigan, F. Hendrix, L. McFarland, D. Pope, D. Strobel, and J. Wagner. 1991. Bioremediation studies at a northern California Superfund site. J. Hazard. Materials 28: 145-158.

Saber, D.L. and R.L. Crawford. 1985. Isolation and characterization of *Flavobacterium* strains that degrade pentachlorophenol. Appl. Environ. Microbiol. 50: 1512-1518.

Sims, R.C. 1996. In: *Tech Trends*. September, 1996. http://www.clu-in.org/PRODUCTS/NEWSLTRS/TTREND/ttwstwtr.htm.

Song, H.G., K. Wang, and R. Bartha. 1990. Bioremediation potential of terrestrial fuel spills. Appl. Environ. Microbiol. 56: 652-656.

Trudell, M., J.M. Markowitch, D.G. Thomson, C.W. Fulton, and R.E. Hofmann. 1994. *In situ* bioremediation at a wood-preserving site in a cold, semi-arid climate: feasibility and field pilot design. In: *Bioremediation of Chlorinated and Polycyclic Aromatic Hydrocarbon Compounds*, R.E. Hinchee, A. Leeson, L. Semprini, and S.K. Ong (eds.), pp. 99-116. Lewis Publishers, Boca Raton, FL.

Winter, B. and W. Zimmermann. 1992. Degradation of halogenated aromatics by actinomycetes. In: *Metal Ions in Biological Systems, Vol. 28: Degradation of Environmental Pollutants by Microorganisms and Their Metalloenzymes*, pp. 157-203. H. Sigel and A. Sigel (Eds.), Marcel Dekker, Inc., NY.

Appendix: Microbial Enumeration in Soil Samples from the Arneson Timber Site

Sample Locations and Collection Procedures:

Soil samples were collected from the Arneson Timber Site and an adjacent uncontaminated area on June 2, 1999. Sample locations were chosen based on the known distribution of PCP-contaminated soil at the site. One set of samples was collected from a largely unvegetated area immediately north of the former drip pad, and another set was collected from a similarly unvegetated area in the vicinity of the former catch basin. The background samples were collected from a heavily vegetated area about 100 ft south of the property line on the same ridge as the former wood-treatment facility. Five samples were collected from each site. In both sets of samples collected from areas with known contamination, one sample was collected from a position that was within the unvegetated perimeter but which had substantial plant growth. In both of these cases, the vegetated soil appeared to be free of oily contamination, whereas the samples collected from the unvegetated areas were dark stained and had an oily odor and texture.

Samples were collected from within 6 to 12 inches of the ground surface by digging a small hole, then using sterile stainless steel spoons to remove soil samples from its wall. Aseptic technique was used to collect the samples: the spoons were soaked in 70% ethanol in between uses and flamed using a propane torch immediately before each use. Two spoons were used for each sample. One sterile spoon was used to remove soil from the wall that may have come into contact with the shovel used to dig the hole, and the second sterile spoon was used to collect samples from the freshly exposed sections of the walls. These precautions ensured that soil and/or bacteria were not transferred between sample locations by any of the sampling equipment.

Methods for Bacterial Enumeration:

Four groups of bacteria were enumerated in the soil samples:

(1) heterotrophic bacteria,

(2) PCP-tolerant heterotrophic bacteria,

(3) PCP-cometabolizing bacteria, and

(4) bacteria capable of using PCP as the sole growth substrate.

Heterotrophic bacteria, PCP-tolerant heterotrophs, and PCP cometabolizers were enumerated by plate counts on solid media. A dilute, nutritionally complex medium (1:20 strength PYG peptone-yeast extract-glucose, medium) was solidified with agar (1.5%) for the plate counts. PCP-tolerant bacteria were enumerated on agar plates containing 1:20 PYG plus PCP (200 mg/L). PCP cometabolizers were enumerated on the PYG + PCP plates by counting colonies that were surrounded by a clearing zone, which indicates that PCP biodegradation had occurred. (The PCP precipitated in the agar forming a hazy suspension of solid PCP. Metabolism of PCP in the vicinity of active colonies reduced the concentration to below its solubility limit resulting in the appearance of a clear "halo" around the colonies.) Bacteria able to grow on PCP were enumerated using a most-probable-number (MPN) procedure with an aqueous mineral salts medium containing PCP (100 mg /L) as the sole source of carbon and energy.

Soil samples were prepared for these enumeration procedures by shaking 10 g soil in 100 mL of a 0.1% tetrasodium pyrophosphate solution (pH = 7.1) for 1 hour at 400 rpm on a gyratory shaker to release the bacteria from the soil surfaces. The supernatant liquids from each sample were diluted by a serial 10-fold dilution procedure using 0.1% tetrasodium pyrophosphate as the dilution medium. For plate counts, 0.1 mL of selected dilutions were transferred to the surface of an agar plate, and the liquid was spread over the surface using a bent glass rod, which were soaked in 70% ethanol between uses and flamed immediately before each use to sterilize the surface. Colonies were counted after incubation in the dark for 1 week at room temperature. For the MPN procedures, 1.0 mL of selected dilutions (from 10^0 to 10^{-6} times the initial concentration) were added to 9.0 mL of sterile PCP-containing mineral salts medium. The MPN tubes were incubated in the dark at room temperature for 5 weeks prior to scoring them for growth. Wells were considered to be positive for growth if the PCP concentration (measured by absorbance at 320 nm) was reduced by about 20% ($P < 0.05$ for comparison of A_{320} in inoculated tubes to the average A_{320} of 10 uninoculated tubes).

Results:

The plate count data are reported in Table 2 and shown in Figure 1. Total heterotrophic bacteria are relatively abundant at all three sample locations, indicating that generally inhibitory conditions do not exist at this site. The number of heterotrophic bacteria present in the catch basin samples is significantly lower ($P < 0.05$) than at the other two sites, but the size of the microbial population is still within the range expected for surface soils. The smaller size of the microbial population in the catch basin samples might be due to the low pH of this soil or its very high clay and low natural organic matter content (Fredrickson *et al.*, 1989; Konopka and Turco, 1991). At all three locations, PCP-tolerant heterotrophic bacteria and PCP-cometabolizing bacteria constitute a very small fraction of the total microbial population. Both groups of bacteria represent similar proportions of the total microbial populations (2% and 1% for PCP-tolerant and PCP-cometabolizing bacteria, respectively) at the two PCP-contaminated locations, but they are a much smaller fraction of the microbial population (0.3% and <0.2%, respectively) at the clean background location. PCP cometabolizers were not observed in samples from the background location. The approximately order-of-magnitude lower abundance of PCP-tolerant and PCP-cometabolizing microorganisms in the background samples is expected, because there is no selective pressure that would give such organisms a competitive advantage in the clean soil.

Table 2: Plate count data from Arneson Timber site soil samples

	log(CFU/g soil)		
Sample Location	total heterotrophs	PCP-tolerant heterotrophs	PCP cometabolizers
Background	6.61 ± 0.18	4.03 ± 0.74	ND*
Drip Pad	6.78 ± 0.43	5.13 ± 1.51	4.79 ± 1.04
Catch Basin	6.02 ± 0.49	4.32 ± 0.93	3.77 ± 0.15

*ND = not detected; the detection limit for these assays was 10^3 CFU/g soil

Bac ia able to grow on PCP as the sole source of carbon and energy were enumerated by MPN. T results are presented in Table 3. Nine of ten samples collected from contaminated locations t the Arneson Timber site were positive for bacteria capable of growing on PCP, but none of t five samples collected from the uncontaminated background location contained detectab numbers of bacteria with this ability. These data are consistent with the plate count data. Th elative numbers of PCP degraders at the Drip Pad and Catch Basin locations are also consisten with the plate count data: higher numbers of bacteria capable of using PCP as the sole growth-s porting substrate were observed in samples collected near the former drip pad than from the rmer catch basin.

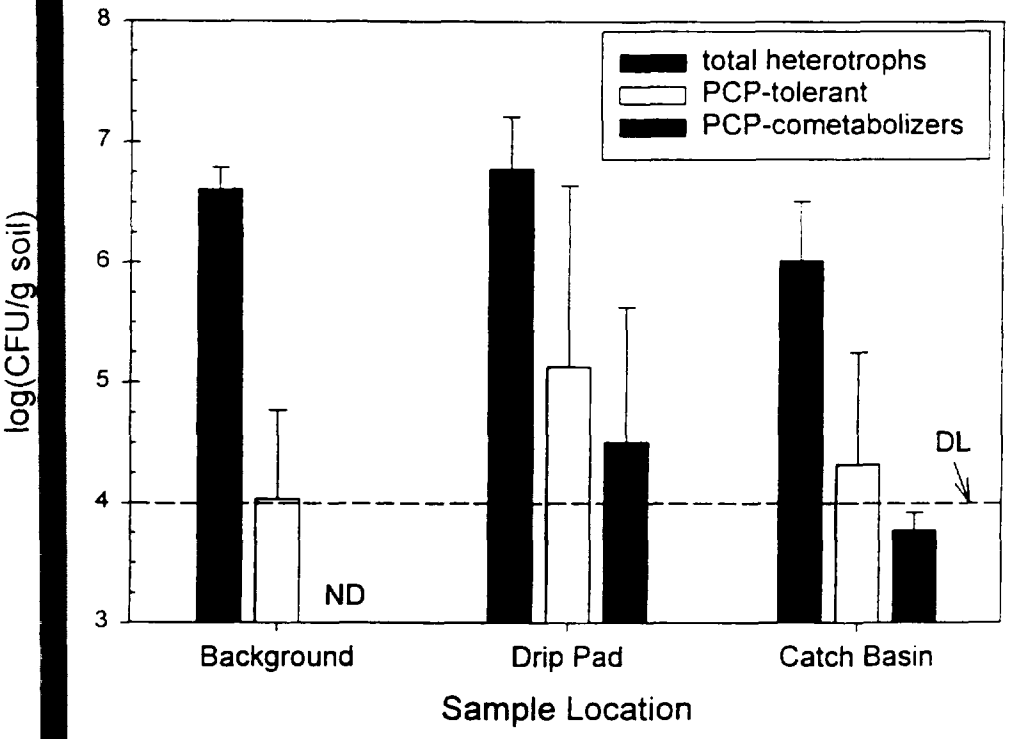


Figure 1: Microbial abundance at the Arneson Timber site. Total heterotrophs represent an estimate of the total size of the microbial population at this site, whereas the PCP-tolerant and PCP-cometabolizing bacteria provide estimates of the proportion of the population that has adapted to the presence of PCP in the soil. PCP cometabolizers were not detected (ND) in soil samples from the clean background site. The detection limit (DL) of these procedures was 10^4 CFU/g soil. PCP degraders were detected in only one of five samples collected in the vicinity of the former catch basin, but they were detected in three of five samples collected near the former drip pad.

Note at the numbers reported in Table 3 are probably conservative estimates, because in addition t being required to use PCP as the sole source of carbon and energy, the bacteria enumerate in the MPN procedure were also forced to make all their own vitamins and other growth fa rs. Microbial communities frequently involve cross-feeding interactions in which

the organism will supply one or more cofactors required for the growth of other organisms. The dilution-extinction method upon which the MPN procedure is based precludes these types of interactions, and no alternative source of vitamins (e.g., yeast extract) was provided in this experimental design.

Table: Enumeration of bacteria with the ability to use PCP as the sole growth-supporting substrate by a most-probable-number (MPN) procedure

Sample Location	log MPN ± SD (PCP degraders/g soil)
Background	ND*
Drip Pad	2.16 ± 1.05
Catch Basin	2.80 ± 1.17

*ND = not detected; the detection limit for this procedure was 10 organisms/g soil

Conclusions:

A healthy heterotrophic microbial population is present in contaminated soil at the Arneson Timber site. The density of heterotrophic bacteria (10^6 to 10^7 CFU/g soil) is within the normal range for surface soils (Alexander, 1977; Konopka and Turco, 1991), and the population present in contaminated soil from the vicinity of the former drip pad is comparable to the population present in uncontaminated soil from outside the site boundary. This suggests that unrecognized toxicity that could interfere with bioremediation does not exist at this site. The slightly lower heterotrophic microbial population present in the soil from the former catch basin probably reflects the soil composition (a dense, low-pH clay) rather than the effects of pollution from wood-treating wastes (Fredrickson *et al.*, 1989; Konopka and Turco, 1991).

Although PCP-degrading bacteria constitute a small fraction of the total heterotrophic microbial population in contaminated soils at the Arneson Timber site, their presence suggests that bioremediation can be an effective remedy at this site. The presence of bacteria that are able to use PCP as the sole source of carbon and energy in nine of ten samples collected from contaminated locations provides conclusive evidence that the appropriate metabolic potential exists at this site. The population of bacteria that can metabolize PCP while growing on other substrates is approximately 2 orders of magnitude larger than the population that can grow on PCP as the sole substrate. Amendment of the contaminated soil with degradable organic matter is likely to result in an increase in the size of this population. The absence of organisms adapted to growth on, or in the presence of PCP, in samples collected from the uncontaminated background location strongly suggests that the microbial population within the site boundaries has adapted to the presence of contaminants in the soil.

Although this study did not attempt to determine which factors limit the growth of PCP degraders at the Arneson Timber site, the proposed treatment, which is based on an extensive survey of PCP-bioremediation literature, should stimulate the growth of these organisms and increase their relative abundance in the microbial population. The PCP biodegradation rate will increase directly in proportion to the increase in the size of the PCP degrading population. The ultimate result of this process will be bioremediation of the contaminated soil.

Feasibility Study for Bioremediation of PCP-Contaminated Soil

Brian A. Wrenn, Ph.D.
Environmental Engineering Program
Civil Engineering Department
Washington University
Campus Box 1180
One Brookings Dr.
St. Louis, MO 63130
(314) 935-8144
bawrenn@seas.wustl.edu

Introduction

Pentachlorophenol (PCP) is a widely used wood-preserving chemical, and its use has resulted in soil contamination at many sites. PCP is a biocide that is toxic to many living organisms, including soil bacteria, at sufficiently high concentrations. Also, it is extensively chlorinated, rendering it resistant to biotransformation reactions that are catalyzed by many enzymes that act on less highly chlorinated aromatic substrates. For years it was considered to be recalcitrant, but it is now clear that it is biodegradable by a wide variety of microorganisms, including bacteria and fungi. Many of the bacteria that catalyze PCP biodegradation are common soil bacteria, including *Pseudomonas* sp. and actinomycetes, such as *Arthrobacter* sp., *Corynebacterium* sp., and *Rhodococcus* sp. (Liedehaus and Schmidt, 1992; Winter and Zimmermann, 1992).

The biodegradability of PCP suggests that bioremediation might be a viable alternative for cleanup of PCP-contaminated soil. A large number of laboratory and field studies of PCP bioremediation have been conducted. Aerobic bioremediation technologies, such as landfarming, composting, and biopiles, have been successful in many of these studies (McGinnis *et al.*, 1991; McGinnis *et al.*, 1994; Laine and Jorgensen, 1996). Although many studies have shown that indigenous microorganisms at PCP-contaminated sites are capable of effecting the bioremediation, this is not always the case (Barbeau *et al.*, 1997). As a result, bioaugmentation – the addition of exogenous microorganisms selected for specific metabolic capabilities – has been another popular approach in PCP bioremediation studies (Colores *et al.*, 1995; Barbeau *et al.*, 1997; Kastanev *et al.*, 1998; Tuomela *et al.*, 1999). One such application, bioaugmentation with white-rot fungi, has been demonstrated as part of the EPA's SITE Program (EPA, 19[illegible]). In the EPA study, a 64% reduction in PCP concentration was achieved during a 20-week field study in plots that were inoculated with fungal spores. This is despite the fact that the initial concentration of PCP in the soil was about 1,000 mg/kg, a concentration that is normally toxic to bacteria.

Feasibility Study

The feasibility study should begin with a thorough review of the PCP-bioremediation literature to identify conditions and amendments that are likely to result in rapid biodegradation of PCP at this small former wood-preserving site. In particular, the literature study will attempt to determine the concentrations of nutrients and soil moisture

t have been used at other sites where PCP bioremediation was successful. The use of king agents and organic soil amendments will also be investigated. Finally, we will empt to determine the levels of PCP-degrading bacteria that are typical at successful s. The information gathered during this literature survey will be used to design any sibility experiments that are conducted and to evaluate the results. This information also be used to design an effective full-scale treatment process and performance nitoring program. The literature survey can be conducted for **$750 to $1,000** and will **1 to 2 weeks.**

Experimental work to determine the feasibility of PCP bioremediation at this site can conducted in two ways. The simplest approach is to enumerate PCP-degrading teria in representative soil samples from the site. In one study involving full-scale tment of PCP-contaminated soil by composting (Laine *et al.*, 1997), this was found to he best indicator of chlorophenol degradation potential. A more comprehensive roach is to conduct a detailed microcosm study, in which microbial numbers and abolic activity are monitored along with PCP concentration over a period of 2-3 nths in bench-scale laboratory reactors. The microcosm study can be designed in a that allows evaluation of various amendments (e.g., nutrient addition, pH or moisture trol, chemical preoxidation). A brief description of the scope of each of these roaches is provided below.

meration of PCP-Degrading Bacteria:

One of the most important factors in determining the success of bioremediation ects is the presence of competent microorganisms. Competent microorganisms are e that are capable of catalyzing the reaction of interest, usually biodegradation of the t contaminant(s). In cases where the target contaminant is degraded by bacteria that biquitous (e.g., gasoline or other light petroleum hydrocarbons), this is not as rtant as when the target contaminants are biodegradable by a more restricted group icroorganisms. PCP falls into the later category.

When PCP degraders are enumerated, it is important to distinguish between bacteria can grow using PCP as the sole source of carbon and energy and those that tabolize the compound. Cometabolism is a fortuitous biotransformation reaction provides no benefit to the microorganism catalyzing the reaction (with the possible ption of toxicity reduction). The strategies used to optimize bioremediation will be different for sites where bacteria that can use PCP as a growth-supporting substrate resent than for sites dominated by bacteria that degrade PCP by cometabolism. efore, we propose to separately enumerate bacteria that can grow on PCP and eria that can cometabolize PCP during growth on other substrates. We will also erate total heterotrophic bacteria to assess the overall metabolic status of the minated soil. Low numbers of total heterotrophs suggest the presence of toxic ounds that could interfere with bioremediation.

Microorganisms that can grow on PCP will be enumerated on selective media, in PCP is provided as the only source of carbon and energy. We may use either solid plate counts) or liquid (i.e., most-probable-number) media to accomplish this task.

Microorganisms that cometabolize PCP will be enumerated following growth on lective medium (similar to the medium used to enumerate the total heterotrophic

pulation). If solid medium is used, the PCP will be provided in an overlayer and metabolizers will be identified by formation of a clearing zone or colored products. If uid medium is used, dilutions that contain PCP degraders will be identified by rmation of colored products from dimerization of quinone intermediates.

Total heterotrophic microorganisms will be enumerated by plate counts on diluted TG (yeast extract-peptone-trypticase-glucose) agar.

These three groups of microorganisms should be enumerated in about 10 samples t are representative of different soil conditions on the site. For example, the samples uld accurately reflect the range of PCP concentrations that exist on site and any ferences in soil texture, composition, or organic carbon content should also be resented. Ideally, each soil sample should be split for analysis of PCP concentration d any other contaminants of concern) and microbial numbers. Approximately 100 g oil is required for each sample. Triplicate subsamples from each of the 10 soil nples will be analyzed for each group of microorganisms. (A total of 3 replicates x 3 robial groups x 10 representative soil samples = 90 independent enumerations will be ducted.)

This level of feasibility testing can be performed for approximately **$2,000 to $2,500** will require **3 to 4 weeks**.

rocosm Studies:

The goals of microcosm studies would be to: (1) demonstrate that biodegradation of P can occur under optimal conditions using real contaminated soil from the site; (2) to e uate the effects of amendments on the biodegradation kinetics; and (3) to determine w ther the site's cleanup goals can be achieved in a reasonable period of time through b emediation. The first goal requires that PCP disappearance from the microcosms be d mented and strong evidence that the disappearance occurs through a biological m hanism. The second goal requires collection of data that allows comparison of b egradation kinetics between and among treatments. Finally, the third goal requires e polation from laboratory data to field conditions to estimate the rate and extent of c up that can be expected if bioremediation is chosen as the full-scale cleanup a native. This is the most difficult goal to achieve, because extrapolation from the lab to e field always involves a high degree of uncertainty. The better the microcosms s late field conditions – especially with respect to temperature, contaminant c entrations and availability, oxygen transfer rates, and moisture content – the better th rediction will be.

Proper experimental design is critical for the success of microcosm experiments. Th includes use of proper controls to estimate abiotic removal rates, adequate rep cation of each treatment, and repeated measures to provide kinetic data. The most im rtant response variables include PCP concentration, size of the microbial population, an e products of biodegradation. It is also useful to semi-continuously measure sur gate parameters of biodegradation progress and microcosm conditions. In par ular, the soil respiration rate (oxygen consumption and carbon dioxide production) sh d be measured at frequent intervals. A detailed work plan can be provided if there is s ous interest in pursuing this alternative.

The cost of a laboratory microcosm study will be $20,000 to $25,000 and will take 3 4 months.

commendations

Because this site is relatively small, we recommend that the feasibility study be ited to the literature survey and enumeration of PCP degraders in the site soil. The sence or absence of PCP degraders in the contaminated soils is the single most portant factor governing whether bioremediation will be a feasible cleanup alternative , if it is, how to best optimize the process. The literature survey will enable us to ermine which amendments should be added and how to best monitor the site to ensure optimal conditions are maintained.

Since the site is relatively small, the cost of the laboratory microcosm study will be a nificant fraction of the cost of the cleanup, and the added value will be slight due to uncertainties associated with extrapolating from the lab to the field. In addition, the e required to complete a microcosm study would delay the start of the cleanup until at t August, leaving only a few months of good weather in the field season. A better on is to use some of the money that would be spent on the microcosm study for eased monitoring of PCP biodegradation activity under actual field conditions. This tional monitoring will allow us to determine whether performance is adequate or if er optimization is required. In the worst case scenario, in which performance is tantially below expectations, we can conduct a targeted microcosm study beginning e fall so we will be ready to complete the cleanup during the next field season.

rences

eau, C., L. Deschenes, D. Karamenev, Y. Comeau, and R. Samson. 1997. Bioremediation of pentachlorophenol-contaminated soil by bioaugmentation using activated soil. Appl. Microbiol. Biotechnol 48: 745-752.

res, G.M., P.M. Radehaus, and S.K. Schmidt. 1995. Use of a pentachlorophenol degrading bacterium to bioremediate highly contaminated soil. Appl. Biochem. Biotechnol. 54: 271-275.

1995. Bioremediation Field Initiative Site Profile: Escambia Wood Preserving Site. U.S. EPA/ORD/OSWER. EPA/540/F-95/506G.

nanev, D.G. C. Chavarie, and R. Sampson. 1998. Soil immobilization: New oncept for biotreatment of soil contaminants. Biotech. Bioeng. 57: 471-476.

, M.M. and K.S. Jorgensen. 1996. Straw compost and bioremediated soil as nocula for the bioremediation of chlorophenol-contaminated soil. Appl. Environ. licrobiol. 62: 1507-1513.

M.M., H. Haario, and K.S. Jorgensen. 1997. Microbial functional activity during mposting of chlorophenol-contaminated sawmill soil. J. Microbiol. Meth. 30: 21-

nis, G.D., H. Borazjani, M. Hannigan, F. Hendrix. L. McFarland, D. Pope, D. robel, and J. Wagner. 1991. Bioremediation studies at a northern California perfund site. J. Hazard. Mater. 28: 145-158.

M innis, D., R.R. DuPont, K. Everhart, and G. St. Laurent. 1994. Evaluation and management of field soil pile bioventing systems for the remedi tion of PCP contaminated surface soils. Environ. Technol. 15: 729-739.

Ra haus, P.M and S.K. Schmidt. 1992. Characterization of a novel *Pseudomonas* sp. that mineralizes high concentrations of pentachlorophenol. Appl. Environ. Microbiol. 58: 2879-2885.

Tu nela, M., M. Lyytikainen, P. Oivanen, and A. Hatakka. 1999. Mineralization and conversion of pentachlorophenol (PCP) in soil inoculated with the white-rot fungus *Trametes versicolor*. Soil Biol. Biochem. 31: 65-74.

Wi er, B. and W. Zimmermann. 1992. Degradation of halogenated aromatics by actinomycetes. In: *Metal Ions in Biological Systems, Vol 28: Biodegradation of Environmental Pollutants by Microorganisms and their Metalloenzymes*., pp. 157-203. H. Sigel and A. Sigel (eds.). Marcel Dekker, Inc. New York.

METHOD 8270C

SEMIVOLATILE ORGANIC COMPOUNDS
BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. The following compounds can be determined by this method:

Compounds	CAS No[a]	Appropriate Preparation Techniques[b]				
		3510	3520	3540/ 3541	3550	3580
Acenaphthene	83-32-9	X	X	X	X	X
Acenaphthene-d_{10} (IS)		X	X	X	X	X
Acenaphthylene	208-96-8	X	X	X	X	X
Acetophenone	98-86-2	X	ND	ND	ND	X
2-Acetylaminofluorene	53-96-3	X	ND	ND	ND	X
1-Acetyl-2-thiourea	591-08-2	LR	ND	ND	ND	LR
Aldrin	309-00-2	X	X	X	X	X
2-Aminoanthraquinone	117-79-3	X	ND	ND	ND	X
Aminoazobenzene	60-09-3	X	ND	ND	ND	X
4-Aminobiphenyl	92-67-1	X	ND	ND	ND	X
3-Amino-9-ethylcarbazole	132-32-1	X	X	ND	ND	ND
Anilazine	101-05-3	X	ND	ND	ND	X
Aniline	62-53-3	X	X	ND	X	X
o-Anisidine	90-04-0	X	ND	ND	ND	X
Anthracene	120-12-7	X	X	X	X	X
Aramite	140-57-8	HS(43)	ND	ND	ND	X
Aroclor 1016	12674-11-2	X	X	X	X	X
Aroclor 1221	11104-28-2	X	X	X	X	X
Aroclor 1232	11141-16-5	X	X	X	X	X
Aroclor 1242	53469-21-9	X	X	X	X	X
Aroclor 1248	12672-29-6	X	X	X	X	X
Aroclor 1254	11097-69-1	X	X	X	X	X
Aroclor 1260	11096-82-5	X	X	X	X	X
Azinphos-methyl	86-50-0	HS(62)	ND	ND	ND	X
Barban	101-27-9	LR	ND	ND	ND	LR
Benzidine	92-87-5	CP	CP	CP	CP	CP
Benzoic acid	65-85-0	X	X	ND	X	X
Benz(a)anthracene	56-55-3	X	X	X	X	X
Benzo(b)fluoranthene	205-99-2	X	X	X	X	X
Benzo(k)fluoranthene	207-08-9	X	X	X	X	X
Benzo(g,h,i)perylene	191-24-2	X	X	X	X	X
Benzo(a)pyrene	50-32-8	X	X	X	X	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
p-Benzoquinone	106-51-4	OE	ND	ND	ND	X
Benzyl alcohol	100-51-6	X	X	ND	X	X
α-BHC	319-84-6	X	X	X	X	X
β-BHC	319-85-7	X	X	X	X	X
δ-BHC	319-86-8	X	X	X	X	X
γ-BHC (Lindane)	58-89-9	X	X	X	X	X
Bis(2-chloroethoxy)methane	111-91-1	X	X	X	X	X
Bis(2-chloroethyl) ether	111-44-4	X	X	X	X	X
Bis(2-chloroisopropyl) ether	108-60-1	X	X	X	X	X
Bis(2-ethylhexyl) phthalate	117-81-7	X	X	X	X	X
4-Bromophenyl phenyl ether	101-55-3	X	X	X	X	X
Bromoxynil	1689-84-5	X	ND	ND	ND	X
Butyl benzyl phthalate	85-68-7	X	X	X	X	X
Captafol	2425-06-1	HS(55)	ND	ND	ND	X
Captan	133-06-2	HS(40)	ND	ND	ND	X
Carbaryl	63-25-2	X	ND	ND	ND	X
Carbofuran	1563-66-2	X	ND	ND	ND	X
Carbophenothion	786-19-6	X	ND	ND	ND	X
Chlordane (NOS)	57-74-9	X	X	X	X	X
Chlorfenvinphos	470-90-6	X	ND	ND	ND	X
4-Chloroaniline	106-47-8	X	ND	ND	ND	X
Chlorobenzilate	510-15-6	X	ND	ND	ND	X
5-Chloro-2-methylaniline	95-79-4	X	ND	ND	ND	X
4-Chloro-3-methylphenol	59-50-7	X	X	X	X	X
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	X	ND	ND	ND	X
1-Chloronaphthalene	90-13-1	X	X	X	X	X
2-Chloronaphthalene	91-58-7	X	X	X	X	X
2-Chlorophenol	95-57-8	X	X	X	X	X
4-Chloro-1,2-phenylenediamine	95-83-0	X	X	ND	ND	ND
4-Chloro-1,3-phenylenediamine	5131-60-2	X	X	ND	ND	ND
4-Chlorophenyl phenyl ether	7005-72-3	X	X	X	X	X
Chrysene	218-01-9	X	X	X	X	X
Chrysene-d12 (IS)		X	X	X	X	X
Coumaphos	56-72-4	X	ND	ND	ND	X
p-Cresidine	120-71-8	X	ND	ND	ND	X
Crotoxyphos	7700-17-6	X	ND	ND	ND	X
2-Cyclohexyl-4,6-dinitro-phenol	131-89-5	X	ND	ND	ND	LR
4,4'-DDD	72-54-8	X	X	X	X	X
4,4'-DDE	72-55-9	X	X	X	X	X
4,4'-DDT	50-29-3	X	X	X	X	X
Demeton-O	298-03-3	HS(68)	ND	ND	ND	X
Demeton-S	126-75-0	X	ND	ND	ND	X
Diallate (cis or trans)	2303-16-4	X	ND	ND	ND	X

Compound	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
2,4-Diamitoluene	95-80-7	DC,0E(42)	ND	ND	ND	X
Dibenz(a,cridine	224-42-0	X	ND	ND	ND	X
Dibenz(a,anthracene	53-70-3	X	X	X	X	X
Dibenzofun	132-64-9	X	X	ND	X	X
Dibenzo(apyrene	192-65-4	ND	ND	ND	ND	X
1,2-Dibron-3-chloropropane	96-12-8	X	X	ND	ND	ND
Di-n-butyl thalate	84-74-2	X	X	X	X	X
Dichlone	117-80-6	OE	ND	ND	ND	X
1,2-Dichlobenzene	95-50-1	X	X	X	X	X
1,3-Dichlobenzene	541-73-1	X	X	X	X	X
1,4-Dichlobenzene	106-46-7	X	X	X	X	X
1,4-Dichlobenzene-d_4 (IS)		X	X	X	X	X
3,3'-Dichlobenzidine	91-94-1	X	X	X	X	X
2,4-Dichlorphenol	120-83-2	X	X	X	X	X
2,6-Dichlorphenol	87-65-0	X	ND	ND	ND	X
Dichlorovo	62-73-7	X	ND	ND	ND	X
Dicrotopho	141-66-2	X	ND	ND	ND	X
Dieldrin	60-57-1	X	X	X	X	X
Diethyl phtlate	84-66-2	X	X	X	X	X
Diethylstilbtrol	56-53-1	AW,0S(67)	ND	ND	ND	X
Diethyl sulfe	64-67-5	LR	ND	ND	ND	LR
Dihydrosafle	56312-13-1	ND	ND	ND	ND	ND
Dimethoate	60-51-5	HE,HS(31)	ND	ND	ND	X
3,3'-Dimethybenzidine	119-90-4	X	ND	ND	ND	LR
Dimethylamoazobenzene	60-11-7	X	ND	ND	ND	X
7,12-Dimetbenz(a)-anthracen	57-97-6	CP(45)	ND	ND	ND	CP
3,3'-Dimethenzidine	119-93-7	X	ND	ND	ND	X
α,α-Dimethhenethylamine	122-09-8	ND	ND	ND	ND	X
2,4-Dimethyhenol	105-67-9	X	X	X	X	X
Dimethyl phalate	131-11-3	X	X	X	X	X
1,2-Dinitrobzene	528-29-0	X	ND	ND	ND	X
1,3-Dinitrobzene	99-65-0	X	ND	ND	ND	X
1,4-Dinitrobzene	100-25-4	HE(14)	ND	ND	ND	X
4,6-Dinitro-2methylphenol	534-52-1	X	X	X	X	X
2,4-Dinitropnol	51-28-5	X	X	X	X	X
2,4-Dinitrotene	121-14-2	X	X	X	X	X
2,6-Dinitrotene	606-20-2	X	X	X	X	X
Dinocap	39300-45-3	CP,HS(28)	ND	ND	ND	CP
Dinoseb	88-85-7	X	ND	ND	ND	X
Dioxathion	78-34-2	ND	ND	ND	ND	ND
Diphenylami	122-39-4	X	X	X	X	X
5,5-Diphenydantoin	57-41-0	X	ND	ND	ND	X
1,2-Diphenydrazine	122-66-7	X	X	X	X	X

Compou[redacted]s	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
Di-n-octy[redacted]hthalate	117-84-0	X	X	X	X	X
Disulfoto[redacted]	298-04-4	X	ND	ND	ND	X
Endosulf[redacted] I	959-98-8	X	X	X	X	X
Endosulf[redacted] II	33213-65-9	X	X	X	X	X
Endosulf[redacted] sulfate	1031-07-8	X	X	X	X	X
Endrin	72-20-8	X	X	X	X	X
Endrin al[redacted]hyde	7421-93-4	X	X	X	X	X
Endrin ke[redacted]ne	53494-70-5	X	X	ND	X	X
EPN	2104-64-5	X	ND	ND	ND	X
Ethion	563-12-2	X	ND	ND	ND	X
Ethyl car[redacted]mate	51-79-6	DC(28)	ND	ND	ND	X
Ethyl met[redacted]nesulfonate	62-50-0	X	ND	ND	ND	X
Famphur	52-85-7	X	ND	ND	ND	X
Fensulfot[redacted]on	115-90-2	X	ND	ND	ND	X
Fenthion	55-38-9	X	ND	ND	ND	X
Fluchloral	33245-39-5	X	ND	ND	ND	X
Fluoranth[redacted]e	206-44-0	X	X	X	X	X
Fluorene	86-73-7	X	X	X	X	X
2-Fluorob[redacted]henyl (surr)	321-60-8	X	X	X	X	X
2-Fluorop[redacted]nol (surr)	367-12-4	X	X	X	X	X
Heptachlo[redacted]	76-44-8	X	X	X	X	X
Heptachlo[redacted]epoxide	1024-57-3	X	X	X	X	X
Hexachlor[redacted]benzene	118-74-1	X	X	X	X	X
Hexachlor[redacted]utadiene	87-68-3	X	X	X	X	X
Hexachlor[redacted]yclopentadiene	77-47-4	X	X	X	X	X
Hexachlor[redacted]thane	67-72-1	X	X	X	X	X
Hexachlor[redacted]hene	70-30-4	AW,CP(62)	ND	ND	ND	CP
Hexachlor[redacted]ropene	1888-71-7	X	ND	ND	ND	X
Hexameth[redacted]hosphoramide	680-31-9	X	ND	ND	ND	X
Hydroquin[redacted]e	123-31-9	ND	ND	ND	ND	X
Indeno(1,2[redacted]-cd)pyrene	193-39-5	X	X	X	X	X
Isodrin	465-73-6	X	ND	ND	ND	X
Isophorone	78-59-1	X	X	X	X	X
[redacted]osafrole	120-58-1	DC(46)	ND	ND	ND	X
[redacted]pone	143-50-0	X	ND	ND	ND	X
Leptophos	21609-90-5	X	ND	ND	ND	X
Malathion	121-75-5	HS(5)	ND	ND	ND	X
Maleic anh[redacted]ride	108-31-6	HE	ND	ND	ND	X
Mestranol	72-33-3	X	ND	ND	ND	X
Methapyrile[redacted]e	91-80-5	X	ND	ND	ND	X
Methoxych[redacted]	72-43-5	X	ND	ND	ND	X
3-Methylch[redacted]anthrene	56-49-5	X	ND	ND	ND	X
4,4'-Methyl[redacted]ebis (2-chloro[redacted]niline)	101-14-4	OE,OS(0)	ND	ND	ND	LR

Compound	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
4,4'-Methylenebis (N,N-dimethylaniline)	101-61-1	X	X	ND	ND	ND
Methyl methanesulfonate	66-27-3	X	ND	ND	ND	X
2-Methylnaphthalene	91-57-6	X	X	ND	X	X
Methyl parathion	298-00-0	X	ND	ND	ND	X
2-Methylphenol	95-48-7	X	ND	ND	ND	X
3-Methylphenol	108-39-4	X	ND	ND	ND	X
4-Methylphenol	106-44-5	X	ND	ND	ND	X
Mevinphos	7786-34-7	X	ND	ND	ND	X
Mexacarbate	315-18-4	HE,HS(68)	ND	ND	ND	X
Mirex	2385-85-5	X	ND	ND	ND	X
Monocrotophos	6923-22-4	HE	ND	ND	ND	X
Naled	300-76-5	X	ND	ND	ND	X
Naphthalene	91-20-3	X	X	X	X	X
Naphthalene-d_8 (IS)		X	X	X	X	X
1,4-Naphthoquinone	130-15-4	X	ND	ND	ND	X
1-Naphthylamine	134-32-7	OS(44)	ND	ND	ND	X
2-Naphthylamine	91-59-8	X	ND	ND	ND	X
Nicotine	54-11-5	DE(67)	ND	ND	ND	X
5-Nitroacenaphthene	602-87-9	X	ND	ND	ND	X
2-Nitroaniline	88-74-4	X	X	ND	X	X
3-Nitroaniline	99-09-2	X	X	ND	X	X
4-Nitroaniline	100-01-6	X	X	ND	X	X
5-Nitro-o-anisidine	99-59-2	X	ND	ND	ND	X
Nitrobenzene	98-95-3	X	X	X	X	X
Nitrobenzene-d_5 (surr)		X	X	X	X	X
4-Nitrobiphenyl	92-93-3	X	ND	ND	ND	X
Nitrofen	1836-75-5	X	ND	ND	ND	X
2-Nitrophenol	88-75-5	X	X	X	X	X
4-Nitrophenol	100-02-7	X	X	X	X	X
5-Nitro-o-toluidine	99-55-8	X	X	ND	ND	X
Nitroquinoline-1-oxide	56-57-5	X	ND	ND	ND	X
N-Nitrosodi-n-butylamine	924-16-3	X	ND	ND	ND	X
N-Nitrosodiethylamine	55-18-5	X	ND	ND	ND	X
N-Nitrosodimethylamine	62-75-9	X	X	X	X	X
N-Nitrosomethylethylamine	10595-95-6	X	ND	ND	ND	X
N-Nitrosodiphenylamine	86-30-6	X	X	X	X	X
N-Nitrosodi-n-propylamine	621-64-7	X	X	X	X	X
N-Nitrosomorpholine	59-89-2	ND	ND	ND	ND	X
N-Nitrosopiperidine	100-75-4	X	ND	ND	ND	X
N-Nitrosopyrrolidine	930-55-2	X	ND	ND	ND	X
Octamethyl pyrophosphoramide	152-16-9	LR	ND	ND	ND	LR
4,4'-Oxydianiline	101-80-4	X	ND	ND	ND	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
Parathion	56-38-2	X	X	ND	ND	X
Pentachlorobenzene	608-93-5	X	ND	ND	ND	X
Pentachloronitrobenzene	82-68-8	X	ND	ND	ND	X
Pentachlorophenol	87-86-5	X	X	X	X	X
Perylene-d_{12} (IS)		X	X	X	X	X
Phenacetin	62-44-2	X	ND	ND	ND	X
Phenanthrene	85-01-8	X	X	X	X	X
Phenanthrene-d_{10} (IS)		X	X	X	X	X
Phenobarbital	50-06-6	X	ND	ND	ND	X
Phenol	108-95-2	DC(28)	X	X	X	X
Phenol-d$_{6}$ (surr)		DC(28)	X	X	X	X
1,4-Phenylenediamine	106-50-3	X	ND	ND	ND	X
Phorate	298-02-2	X	ND	ND	ND	X
Phosalone	2310-17-0	HS(65)	ND	ND	ND	X
Phosmet	732-11-6	HS(15)	ND	ND	ND	X
Phosphamidon	13171-21-6	HE(63)	ND	ND	ND	X
Phthalic anhydride	85-44-9	CP,HE(1)	ND	ND	ND	CP
2-Picoline (2-Methylpyridine)	109-06-8	X	X	ND	ND	ND
Piperonyl sulfoxide	120-62-7	X	ND	ND	ND	X
Pronamide	23950-58-5	X	ND	ND	ND	X
Propylthiouracil	51-52-5	LR	ND	ND	ND	LR
Pyrene	129-00-0	X	X	X	X	X
Pyridine	110-86-1	ND	ND	ND	ND	ND
Resorcinol	108-46-3	DC,OE(10)	ND	ND	ND	X
Safrole	94-59-7	X	ND	ND	ND	X
Strychnine	57-24-9	AW,0S(55)	ND	ND	ND	X
Sulfallate	95-06-7	X	ND	ND	ND	X
Terbufos	13071-79-9	X	ND	ND	ND	X
Terphenyl-d_{14}(surr)	1718-51-0	X	X	ND	X	X
1,2,4,5-Tetrachlorobenzene	95-94-3	X	ND	ND	ND	X
2,3,4,6-Tetrachlorophenol	58-90-2	X	ND	ND	ND	X
Tetrachlorvinphos	961-11-5	X	ND	ND	ND	X
Tetraethyldithiopyrophosphate	3689-24-5	X	X	ND	ND	ND
Tetraethylpyrophosphate	107-49-3	X	ND	ND	ND	X
Thionazin	297-97-2	X	ND	ND	ND	X
Thiophenol (Benzenethiol)	108-98-5	X	ND	ND	ND	X
Toluene diisocyanate	584-84-9	HE(6)	ND	ND	ND	X
o-Toluidine	95-53-4	X	ND	ND	ND	X
Toxaphene	8001-35-2	X	X	X	X	X
2,4,6-Tribromophenol (surr)	118-79-6	X	X	X	X	X
1,2,4-Trichlorobenzene	120-82-1	X	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	ND	X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X	X
Trifluralin	1582-09-8	X	ND	ND	ND	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
2,4,5-Trimet aniline	137-17-7	X	ND	ND	ND	X
Trimethyl ph phate	512-56-1	HE(60)	ND	ND	ND	X
1,3,5-Trinitrc enzene	99-35-4	X	ND	ND	ND	X
Tris(2,3-dibr opropyl) phosphate	126-72-7	X	ND	ND	ND	LR
Tri-p-tolyl ph phate	78-32-0	X	ND	ND	ND	X
O,O,O-Trieth phosphorothioate	126-68-1	X	ND	ND	ND	X

[a] Chemical A stract Service Registry Number
[b] See Sec. 1 for other acceptable preparation methods.

KEY TO AN YTE LIST

IS = Th compound may be used as an internal standard.
surr = Th compound may be used as a surrogate.
AW = Ac rption to walls of glassware during extraction and storage.
CP = N reproducible chromatographic performance.
DC = U vorable distribution coefficient (number in parenthesis is percent recovery).
HE = Hy rolysis during extraction accelerated by acidic or basic conditions (number in pa nthesis is percent recovery).
HS = Hy rolysis during storage (number in parenthesis is percent stability).
LR = Lo response.
ND = N determined.
OE = O dation during extraction accelerated by basic conditions (number in parenthesis is pe cent recovery).
OS = O dation during storage (number in parenthesis is percent stability).
X = G ater than 70 percent recovery by this technique.

1.2 In addition to the sample preparation methods listed in the above analyte list, Method 3542 descri s sample preparation for semivolatile organic compounds in air sampled by Method 0010 (Table 1 contains surrogate performance data), Method 3545 describes an automated solvent extraction d ce for semivolatiles in solids (Table 12 contains performance data), and Method 3561 describes a supercritical fluid extraction of solids for PAHs (see Tables 13, 14, and 15 for performanc data).

1.3 Method 8270 can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatizatio as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly po silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbon and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including ni phenols. See Table 1 for a list of compounds and their characteristic ions that have been evalu ed.

In most cases, Method 8270 is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analytes. When these analytes have been identified by another technique, Method 8270 is appropriate for confirmation of the presence of these analytes when concentration in the extract permits. Refer to Sec. 7.0 of Methods 8081 and 8082 for guidance on calibration and quantitation of multicomponent analytes such as the Aroclors, Toxaphene, and Chlordane.

1.4 The following compounds may require special treatment when being determined by this method:

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

1.4.2 Under the alkaline conditions of the extraction step from aqueous matrices, α-BHC, γ-BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected.

1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.4.4 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

1.4.5 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

1.4.6 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4.7 Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. The analyst needs to use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4.8 Toluene diisocyanate rapidly hydrolyses in water (half-life of less then 30 min.). Therefore, recoveries of this compound from aqueous matrices should not be expected. In addition, in solid matrices, toluene diisocyanate often reacts with alcohols and amines to produce urethane and ureas and consequently cannot usually coexist in a solution containing these materials.

1.4.9 In addition, analytes in the list provided above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.5 The estimated quantitation limit (EQL) of Method 8270 for determining an individual compound is approximately 660 µg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples (see Table 1). EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (refer to Method 3500) and, if necessary, sample cleanup procedures (refer to Method 3600).

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.4 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interference. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system

4.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

4.1.2 Column - 30 m x 0.25 mm ID (or 0.32 mm ID) 1 µm film thickness silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

4.1.3 Mass spectrometer

4.1.3.1 Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 3 when 1 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.3.2 An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. The mass spectrometer must be capable of producing a mass spectrum for DFTPP which meets the criteria in Table 3 when 5 or 50 ng are introduced.

4.1.4 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

4.1.5 Data system - A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.1.6 Guard column (optional) - (J&W Deactivated Fused Silica, 0.25 mm ID x 6 m, or equivalent) between the injection port and the analytical column joined with column joiners (Hewlett-Packard Catalog No. 5062-3556, or equivalent).

4.2 Syringe - 10-µL.

4.3 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

4.4 Balance - Analytical, capable of weighing 0.0001 g.

4.5 Bottles - glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

5.0 REAGENTS

5.1 Reagent grade inorganic 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 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

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

5.3.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure mate[illegible]. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to vol[illegible]e in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analy[illegible] When compound purity is assayed to be 96% or greater, the weight may be used witho[illegible] correction to calculate the concentration of the stock standard. Commercially-prepared stock [illegible]andards may be used at any concentration if they are certified by the manufacturer or by an[illegible] dependent source.

5.3.2 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, [illegible]rotected from light, at -10°C or less or as recommended by the standard manufacturer. Stock [illegible]andard solutions should be checked frequently for signs of degradation or evaporation, espec[illegible]ly just prior to preparing calibration standards from them.

5.3.3 Stock standard solutions must be replaced after 1 year or sooner if comparison with q[illegible]lity control check samples indicates a problem.

5.3.4 It is recommended that nitrosamine compounds be placed together in a separate calibra[illegible]n mix and not combined with other calibration mixes. When using a premixed certified standa[illegible], consult the manufacturer's instructions for additional guidance.

5.3.5 Mixes with hydrochloride salts may contain hydrochloric acid, which can cause analyti[illegible] difficulties. When using a premixed certified standard, consult the manufacturer's instruc[illegible]ns for additional guidance.

5.4 In[illegible]rnal standard solutions - The internal standards recommended are 1,4-dichlorob[illegible]zene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} [illegible]ee Table 5). Other compounds may be used as internal standards as long as the requirements [illegible]ven in Sec. 7.3.2 are met.

5.4.1 Dissolve 0.200 g of each compound with a small volume of carbon disulfide. Transfe[illegible]r to a 50 mL volumetric flask and dilute to volume with methylene chloride so that the final sol[illegible]nt is approximately 20% carbon disulfide. Most of the compounds are also soluble in smal[illegible]l volumes of methanol, acetone, or toluene, except for perylene-d_{12}. The resulting solution [illegible]will contain each standard at a concentration of 4,000 ng/µL. Each 1 mL sample extract [illegible]dergoing analysis should be spiked with 10 µL of the internal standard solution, resulting[illegible] a concentration of 40 ng/µL of each internal standard. Store at -10°C or less when not in u[illegible]. When using premixed certified solutions, store according to the manufacturer's docume[illegible]ed holding time and storage temperature recommendations.

5.4.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a [illegible]more dilute internal standard solution may be required. Area counts of the internal standard [illegible]eaks should be between 50-200% of the area of the target analytes in the mid-point calibrati[illegible] analysis.

5.5 GC[illegible]MS tuning standard - A methylene chloride solution containing 50 ng/µL of decafluorotrip[illegible]henylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/µL each of 4[illegible]'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column perfo[illegible]rmance. Store at -10°C or less when not in use. If a more sensitive mass spectrometer i[illegible]s employed to achieve lower detection levels, a more dilute tuning solution may be necessary. W[illegible]hen using premixed certified solutions, store according to the manufacturer's documented h[illegible]olding time and storage temperature recommendations.

5.6 Calibration standards - A minimum of five calibration standards should be prepared at five different concentrations. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in actual samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method.

5.6.1 It is the intent of EPA that all target analytes for a particular analysis be included in the calibration standard(s). These target analytes may not include the entire list of analytes (Sect. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.6.2 Each 1-mL aliquot of calibration standard should be spiked with 10 µL of the internal standard solution prior to analysis. All standards should be stored at -10°C or less, and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification standard should be prepared weekly and stored at 4°C. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.7 Surrogate standards - The recommended surrogates are phenol-d_6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}. See Method 3500 for instructions on preparing the surrogate solutions.

5.7.1 Surrogate Standard Check: Determine what the appropriate concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards. It is recommended that this check be done whenever a new surrogate spiking solution is prepared.

NOTE: Method 3561 (SFE Extraction of PAHs) recommends the use of bromobenzene and p-quaterphenyl to better cover the range of PAHs listed in the method.

5.7.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute surrogate solution may be necessary.

5.8 Matrix spike and laboratory control standards - See Method 3500 for instructions on preparing the matrix spike standard. The same standard may be used as the laboratory control standard (LCS).

5.8.1 Matrix Spike Check: Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery. It is recommended that this check be done whenever a new matrix spiking solution is prepared.

5.8.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute matrix and LCS spiking solution may be necessary.

5.8.3 Some projects may require the spiking of the specific compounds of interest, since the spiking compounds listed in Method 3500 would not be representative of the compounds of interest required for the project. When this occurs, the matrix and LCS spiking

standards should be prepared in methanol, with each compound present at a concentration appropriate for the project.

5.9 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents - All solvents should be pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Store the sample extracts at -10°C, protected from light, in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa.

7.0 PROCEDURE

7.1 Sample preparation

7.1.1 Samples are normally prepared by one of the following methods prior to GC/MS analysis.

Matrix	Methods
Air	3542
Water	3510, 3520, 3535
Soil/sediment	3540, 3541, 3545, 3550, 3560, 3561
Waste	3540, 3541, 3545, 3550, 3560, 3561, 3580

7.1.2 In very limited applications, direct injection of the sample into the GC/MS system with a 10-µL syringe may be appropriate. The detection limit is very high (approximately 10,000 µg/L). Therefore, it is only permitted where concentrations in excess of 10,000 µg/L are expected.

7.2 Extract cleanup - Extracts may be cleaned up by any of the following methods prior to GC/MS analysis.

Analytes of interest	Methods
Anilines, aniline derivatives	3620
Phenols	3630, 3640, 8041[a]
Phthalate esters	3610, 3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine pesticides & PCBs	3610, 3620, 3630, 3660, 3665
Nitroaromatics and cyclic ketones	3620, 3640
Polynuclear aromatic hydrocarbons	3611, 3630, 3640
Haloethers	3620, 3640
Chlorinated hydrocarbons	3620, 3640
Organophosphorus pesticides	3620

Analytes of interest	Methods
Petroleum waste	3611, 3650
Acid, base, neutral, and acid priority pollutants	3640

[a] Method 8041 includes a derivatization technique followed by GC/ECD analysis, if interferences are encountered on GC/FID.

7.2 Initial calibration

Establish the GC/MS operating conditions, using the following recommendations as guidance.

Mass range:	35-500 amu
Scan time:	1 sec/scan
Initial temperature:	40°C, hold for 4 minutes
Temperature program:	40-270°C at 10°C/min
Final temperature:	270°C, hold until benzo[g,h,i]perylene elutes
Injector temperature:	250-300°C
Transfer line temperature:	250-300°C
Source temperature:	According to manufacturer's specifications
Injector:	Grob-type, splitless
Injection volume:	1-2 µL
Carrier gas:	Hydrogen at 50 cm/sec or helium at 30 cm/sec
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

Split injection is allowed if the sensitivity of the mass spectrometer is sufficient.

7.3.1 The GC/MS system must be hardware-tuned using a 50 ng injection of DFTPP. Analyses must not begin until the tuning criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of DFTPP from the instrument manufacturer, the following approach has been shown to be useful: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak.

7.3.1.2 Use the DFTPP mass intensity criteria in Table 3 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g. CLP, Method 525, or manufacturer's instructions), provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

7.3.1.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD

ould not exceed 20%. (See Sec. 8.0 of Method 8081 for the percent breakdown lculation). Benzidine and pentachlorophenol should be present at their normal sponses, and no peak tailing should be visible.

7.3.1.4 If degradation is excessive and/or poor chromatography is noted, the ection port may require cleaning. It may also be necessary to break off the first 6-12 of the capillary column. The use of a guard column (Sec. 4.1.6) between the injection t and the analytical column may help prolong analytical column performance.

7 .2 The internal standards selected in Sec. 5.4 should permit most of the components of inter t in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standa s. Use the base peak ion from the specific internal standard as the primary ion for quanti ion (see Table 1). If interferences are noted, use the next most intense ion as the quanti on ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

7 .3 Analyze 1-2 µL of each calibration standard (containing internal standards) and tabulat e area of the primary characteristic ion against concentration for each target analyte (as indi ted in Table 1). A set of at least five calibration standards is necessary (see Sec. 5.6 and M od 8000). The injection volume must be the same for all standards and sample extract Figure 1 shows a chromatogram of a calibration standard containing base/neutral and ac analytes.

C culate response factors (RFs) for each target analyte relative to one of the internal standar as follows:

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

w re:

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

7.3 System performance check compounds (SPCCs)

7.3.4.1 A system performance check must be performed to ensure that mi num average RFs are met before the calibration curve is used. For semivolatiles, the ystem Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hex chlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol.

7.3.4.2 The minimum acceptable average RF for these compounds is 0.050. The e SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the hromatographic system begins to deteriorate or the standard material begins to det orate. They are usually the first to show poor performance. Therefore, they must me the minimum requirement when the system is calibrated.

7.3.4.3 If the minimum response factors are not met, the system must be eva ated, and corrective action must be taken before sample analysis begins. Possible pro ems include standard mixture degradation, injection port inlet contamination, con mination at the front end of the analytical column, and active sites in the column or chr atographic system. This check must be met before sample analysis begins.

3.5 Calibration check compounds (CCCs)

7.3.5.1 The purpose of the CCCs are to evaluate the calibration from the andpoint of the integrity of the system. High variability for these compounds may be dicative of system leaks or reactive sites on the column. Meeting the CCC criteria is t a substitute for successful calibration of the target analytes using one of the pproaches described in Section 7.0 of Method 8000.

7.3.5.2 Calculate the mean response factor and the relative standard deviation SD) of the response factors for each target analyte. The RSD should be less than or ual to 15% for each target analyte. However, the RSD for each individual CCC (see able 4) must be less than or equal to 30%.

$$\text{ean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

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

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

7.3.5.3 If the RSD of any CCC is greater than 30%, then the chromatographic stem is too reactive for analysis to begin. Clean or replace the injector liner and/or pillary column, then repeat the calibration procedure beginning with Sec. 7.3.

7.3.5.4 If the CCCs are not included in the list of analytes for a project, and erefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000.

.6 Evaluation of retention times - The relative retention time (RRT) of each target analyt n each calibration standard should agree within 0.06 RRT units. Late-eluting target analyt usually have much better agreement.

.7 Linearity of target analytes - If the RSD of any target analytes is 15% or less, then the re ive response factor is assumed to be constant over the calibration range, and the averag relative response factor may be used for quantitation (Sec. 7.6.2).

7.3.7.1 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 i Method 8000 for additional calibration options. One of the options must be applied to C/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 designates a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8270 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.7.2 When the RSD exceeds 15%, the plotting and visual inspection of a bration curve can be a useful diagnostic tool. The inspection may indicate analytical blems, including errors in standard preparation, the presence of active sites in the omatographic system, analytes that exhibit poor chromatographic behavior, etc.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000B for method blank performance criteria.

7.4.4 System performance check compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC in the calibration verification standard must meet a minimum response factor of 0.050. This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.4.5 Calibration check compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Table 4 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference for each CCC is less than or equal to 20%, then the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift) for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCCs are not included in the list of analytes for a project,

and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that sample extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

7.5.2 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 µL of the internal standard solution to the 1-mL concentrated sample extract obtained from sample preparation.

7.5.3 Inject a 1-2 µL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 7.3). The volume to be injected should contain 100 ng of base/neutral and 200 ng of acid surrogates (assuming 100% recovery), unless a more sensitive GC/MS system is being used and the surrogate solution is less concentrated then that listed in Sec. 5.7. The injection volume must be the same volume used for the calibration standards.

7.5.4 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (40 ng/µL, unless a more sensitive GC/MS system is being used).

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.5 The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. However, SIM

may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6 Qualitative analysis

7.6.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with a 50% abundance in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be identified, quantitated and reported as the sum of both compounds by the GC.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the

analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

7.7.2 If the RSD of a compound's response factor is 15% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (Sec. 7.3.5). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_s should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.7.5 Quantitation of multicomponent compounds (e.g., Toxaphene, Arochlors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Method 8081 or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10 ng/µL in the concentrated sample extract.

7.6 Structural isomers that produce very similar mass spectra should be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are quantitated as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be summed and reported as the sum of both compounds.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Sec. 7.0 of Method 8000 and include calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8270:

8.2.1 The GC/MS system must be tuned to meet the DFTPP criteria listed in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the calibration verification acceptance criteria in Sec. 7.4 each 12 hours.

8.2.4 The RRT of the sample component must fall within the RRT window of the standard component provided in Sec. 7.6.1.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 3000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed, a septum is changed), see the guidance in Sec 8.2 of Method 8000 regarding whether recalibration of the system must take place.

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. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 Method 8250 (the packed column version of Method 8270) was tested by 15 laboratories using organic-free reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations ranging from 5 to 1,300 µg/L. Single operator accuracy and precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7. These values are presented as guidance only and are not intended as absolute acceptance criteria. Laboratories should generate their own acceptance criteria for capillary column method performance. (See Method 8000.)

9.2 Chromatograms from calibration standards analyzed with Day 0 and Day 7 samples were compared to detect possible deterioration of GC performance. These recoveries (using Method 3510 extraction) are presented in Table 8.

9.3 Method performance data (using Method 3541 Automated Soxhlet extraction) are presented in Table 9. Single laboratory accuracy and precision data were obtained for semivolatile organics in a clay soil by spiking at a concentration of 6 mg/kg for each compound. The spiking solution was mixed into the soil during addition and then allowed to equilibrate for approximately 1 hour prior to extraction. The spiked samples were then extracted by Method 3541 (Automated Soxhlet). Three determinations were performed and each extract was analyzed by gas chromatography/ mass spectrometry following Method 8270. The low recovery of the more volatile compounds is probably due to volatilization losses during equilibration. These data are listed in Table 10 and were taken from Reference 7.

9.4 Surrogate precision and accuracy data are presented in Table 11 from a field dynamic spiking study based on air sampling by Method 0010. The trapping media were prepared for analysis by Method 3542 and subsequently analyzed by Method 8270.

9.5 Single laboratory precision and bias data (using Method 3545 Accelerated Solvent Extraction) for semivolatile organic compounds are presented in Table 12. The samples were conditioned spiked samples prepared and certified by a commercial supplier that contained 57 semivolatile organics at three concentrations (250, 2500, and 12,500 µg/kg) on three types of soil (clay, loam and sand). Spiked samples were extracted both by the Dionex Accelerated Solvent Extraction system and by Perstorp Environmental Soxtec™ (automated Soxhlet). The data presented in Table 12 represents seven replicate extractions and analyses for each individual sample and were taken from reference 9. The average recoveries from the three matrices for all analytes and all replicates relative to the automated Soxhlet data are as follows: clay 96.8%, loam 98.7% and sand 102.1%. The average recoveries from the three concentrations also relative to the automated Soxhlet data are as follows: low-101.2%, mid-97.2% and high-99.2%.

9.6 Single laboratory precision and bias data (using Method 3561 SFE Extraction of PAHs with a variable restrictor and solid trapping material) were obtained for the method analytes by the extraction of two certified reference materials (one, EC-1, a lake sediment from Environment Canada and the other, HS-3, a marine sediment from the National Science and Engineering Research Council of Canada, both naturally-contaminated with PAHs). The SFE instrument used for these extractions was a Hewlett-Packard Model 7680. Analysis was by GC/MS. Average recoveries from six replicate extractions range from 85 to 148% (overall average of 100%) based on the certified value (or Soxhlet value if a certified value was unavailable for a specific analyte) for the lake sediment. Average recoveries from three replicate extractions range from 73 to 133% (overall average 92%) based on the certified value for the marine sediment. The data are found in Tables 13 and 14 and were taken from Reference 10.

9.7 Single laboratory precision and accuracy data (using Method 3561 SFE Extraction of PAHs with a fixed restrictor and liquid trapping) were obtained for twelve of the method analytes by the extraction of a certified reference material (a soil naturally contaminated with PAHs). The SFE instrument used for these extractions was a Dionex Model 703-M. Analysis was by GC/MS. Average recovenes from four replicate extractions range from 60 to 122% (overall average of 89%) based on the certified value. Following are the instrument conditions that were utilized to extract a 3.4 g sample: Pressure - 300 atm; Time - 60 min.; Extraction fluid - CO_2; Modifier - 10% 1:1 (v/v) methanol/methylene chloride; Oven temperature - 80°C; Restrictor temperature - 120°C; and, Trapping fluid - chloroform (methylene chloride has also been used). The data are found in Table 15 and were taken from Reference 11.

10.0 REFERENCES

1. Eichelberger, J.W., Harris, L.E., and Budde, W.L., "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry Systems", Analytical Chemistry, 47, 995-1000, 1975.

2. "Method Detection Limit for Methods 624 and 625", Olynyk, P., Budde, W.L., and Eichelberger, J.W., unpublished report, October 1980.

3. "Interlaboratory Method Study for EPA Method 625-Base/Neutrals, Acids, and Pesticides", Final Report for EPA Contract 68-03-3102.

4. Burke, J.A., "Gas Chromatography for Pesticide Residue Analysis: Some Practical Aspects", Journal of the Association of Official Analytical Chemists (AOAC), 48, 1037, 1965.

5. Lucas, S.V., Komfeld, R.A., "GC-MS Suitability Testing of RCRA Appendix VIII and Michigan List Analytes ", U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268, February 20, 1987, Contract No. 68-03-3224.

6. Engel, T.M., Komfeld, R.A., Warner, J.S., and Andrews, K.D., "Screening of Semivolatile Organic Compounds for Extractability and Aqueous Stability by SW-846, Method 3510", U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268, June 5, 1987, Contract 68-03-3224.

7. Lopez-Avila, V. (W. Beckert, Project Officer); "Development of a Soxtec Extraction Procedure for Extraction of Organic Compounds from Soils and Sediments"; U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Las Vegas, NV, October 1991; EPA 600/X-91/140.

8. Bursey, J., Merrill, R., McAllister, R., and McGaughey, J., "Laboratory Validation of VOST and SemiVOST for Halogenated Hydrocarbons from the Clean Air Act Amendments List", Vol. 1 and 2, U.S. Environmental Protection Agency, EPA 600/R-93/123a and b, (NTIS PB 93-227163 and 93-227171), Research Triangle Park, NC, July 1993.

9. Richter, B., Ezzell, J., and Felix, D., "Single Laboratory Method Validation Report: Extraction of Target Compound List/Priority Pollutant List BNAs and Pesticides using Accelerated Solvent Extraction (ASE) with Analytical Validation by GC/MS and GC/ECD", Document 101124, Dionex Corporation, Salt Lake City, UT, June 16, 1994.

10. Lee, H., Peart, T.E., Hong-You, R.L., and Gere, D.R., "Supercritical Carbon Dioxide Extraction of Polycyclic Aromatic Hydrocarbons from Sediments", J. Chromatography, A 653 83-91 (1993).

11. Personal communication from Sue Warner, EPA Region 3, Central Regional Laboratory, 839 Bestgate Road, Annapolis, MD 21401.

TABLE 1

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
2-Picoline	3.75[a]	93	66,92
Aniline	5.68	93	66,65
Phenol	5.77	94	65,66
Bis(2-chloroethyl) ether	5.82	93	63,95
2-Chlorophenol	5.97	128	64,130
1,3-Dichlorobenzene	6.27	146	148,111
1,4-Dichlorobenzene-d_4 (IS)	6.35	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	6.78	108	79,77
1,2-Dichlorobenzene	6.85	146	148,111
N-Nitroso-ethylethylamine	6.97	88	42,43,56
Bis(2-chloroisopropyl) ether	7.22	45	77,121
Ethyl carbamate	7.27	62	44,45,74
Thiophenol (Benzenethiol)	7.42	110	66,109,84
Methyl methanesulfonate	7.48	80	79,65,95
N-Nitroso-n-propylamine	7.55	70	42,101,130
Hexachloroethane	7.65	117	201,199
Maleic anhydride	7.65	54	98,53,44
Nitrobenzene	7.87	77	123,65
Isophorone	8.53	82	95,138
N-Nitrosodiethylamine	8.70	102	42,57,44,56
2-Nitrophenol	8.75	139	109,65
2,4-Dimethylphenol	9.03	122	107,121
p-Benzoquinone	9.13	108	54,82,80
Bis(2-chloroethoxy)methane	9.23	93	95,123
Benzoic acid	9.38	122	105,77
2,4-Dichlorophenol	9.48	162	164,98
Trimethyl phosphate	9.53	110	79,95,109,140
Ethyl methanesulfonate	9.62	79	109,97,45,65
1,2,4-Trichlorobenzene	9.67	180	182,145
Naphthalene-d_8 (IS)	9.75	136	68
Naphthalene	9.82	128	129,127
Hexachlorobutadiene	10.43	225	223,227
Tetraethyl pyrophosphate	11.07	99	155,127,81,109
Diethyl sulfate	11.37	139	45,59,99,111,125
4-Chloro-3-methylphenol	11.68	107	144,142
2-Methylnaphthalene	11.87	142	141
2-Methylphenol	12.40	107	108,77,79,90
Hexachloropropene	12.45	213	211,215,117,106,141
Hexachlorocyclopentadiene	12.60	237	235,272

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
N-Nitroso[illegible]rrolidine	12.65	100	41,42,68,69
Acetophe[illegible]ne	12.67	105	71,51,120
4-Methylp[illegible]nol	12.82	107	108,77,79,90
2,4,6-Tric[illegible]orophenol	12.85	196	198,200
o-Toluidin[illegible]	12.87	106	107,77,51,79
3-Methylp[illegible]nol	12.93	107	108,77,79,90
2-Chloron[illegible]hthalene	13.30	162	127,164
N-Nitroso[illegible]eridine	13.55	114	42,55,56,41
1,4-Pheny[illegible]nediamine	13.62	108	80,53,54,52
1-Chloron[illegible]hthalene	13.65[a]	162	127,164
2-Nitroani[illegible]e	13.75	65	92,138
5-Chloro-2[illegible]methylaniline	14.28	106	141,140,77,89
Dimethyl p[illegible]halate	14.48	163	194,164
Acenaphth[illegible]ene	14.57	152	151,153
2,6-Dinitro[illegible]uene	14.62	165	63,89
Phthalic a[illegible]ydride	14.62	104	76,50,148
o-Anisidin[illegible]	15.00	108	80,123,52
3-Nitroanil[illegible]e	15.02	138	108,92
Acenaphth[illegible]ne-d_{10} (IS)	15.05	164	162,160
Acenaphth[illegible]ne	15.13	154	153,152
2,4-Dinitro[illegible]enol	15.35	184	63,154
2,6-Dinitro[illegible]enol	15.47	162	164,126,98,63
4-Chloroan[illegible]ne	15.50	127	129,65,92
Isosafrole	15.60	162	131,104,77,51
Dibenzofur[illegible]	15.63	168	139
2,4-Diamin[illegible]oluene	15.78	121	122,94,77,104
2,4-Dinitrot[illegible]uene	15.80	165	63,89
4-Nitrophe[illegible]	15.80	139	109,65
2-Naphthyl[illegible]mine	16.00[a]	143	115,116
1,4-Naphth[illegible]quinone	16.23	158	104,102,76,50,130
p-Cresidine[illegible]	16.45	122	94,137,77,93
Dichlorovo[illegible]	16.48	109	185,79,145
Diethyl phth[illegible]ate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trime[illegible]ylaniline	16.70	120	135,134,91,77
N-Nitrosodi[illegible]-butylamine	16.73	84	57,41,116,158
4-Chloroph[illegible]yl phenyl ether	16.78	204	206,141
Hydroquino[illegible]	16.93	110	81,53,55
4,6-Dinitro-[illegible]methylphenol	17.05	198	51,105
Resorcinol	17.13	110	81,82,53,69
N-Nitrosodi[illegible]enylamine	17.17	169	168,167
Safrole	17.23	162	104,77,103,135

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Hexamethylphosphoramide	17.33	135	44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	127,129,65,39
Diphenylamine	17.54[a]	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	214,179,108,143,218
1-Naphthylamine	18.20	143	115,89,63
1-Acetyl-2-thiourea	18.22	118	43,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	145,173,146,132,91
2,4,5-Trichlorophenol	18.47	196	198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	133,161,162
Pentachlorophenol	19.25	266	264,268
5-Nitro-o-toluidine	19.27	152	77,79,106,94
Thionazine	19.35	107	96,97,143,79,68
4-Nitroaniline	19.37	138	65,108,92,80,39
Phenanthrene-d_{10} (IS)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	75,50,76,92,122
Mevinphos	19.90	127	192,109,67,164
Naled	20.03	109	145,147,301,79,189
1,3-Dinitrobenzene	20.18	168	76,50,75,92,122
Diallate (cis or trans)	20.57	86	234,43,70
1,2-Dinitrobenzene	20.58	168	50,63,74
Diallate (trans or cis)	20.78	86	234,43,70
Pentachlorobenzene	21.35	250	252,108,248,215,254
5-Nitro-o-anisidine	21.50	168	79,52,138,153,77
Pentachloronitrobenzene	21.72	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	21.73	174	101,128,75,116
Di-n-butyl phthalate	21.78	149	150,104
2,3,4,6-Tetrachlorophenol	21.88	232	131,230,166,234,168
Dihydrosafrole	22.42	135	64,77
Demeton-O	22.72	88	89,60,61,115,171
Fluoranthene	23.33	202	101,203
1,3,5-Trinitrobenzene	23.68	75	74,213,120,91,63
Dicrotophos	23.82	127	67,72,109,193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	43,264,41,290
Bromoxynil	23.90	277	279,88,275,168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	192,67,97,109
Phorate	24.10	75	121,97,93,260

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Sulfallate	24.23	188	88,72,60,44
Demeton-S	24.30	88	60,81,89,114,115
Phenacetin	24.33	108	180,179,109,137,80
Dimethoate	24.70	87	93,125,143,229
Phenobarbital	24.70	204	117,232,146,161
Carbofuran	24.90	164	149,131,122
Octamethyl pyrophosphoramide	24.95	135	44,199,286,153,243
4-Aminobiphenyl	25.08	169	168,170,115
Dioxathion	25.25	97	125,270,153
Terbufos	25.35	231	57,97,153,103
α,α-Dimethylphenylamine	25.43	58	91,65,134,42
Pronamide	25.48	173	175,145,109,147
Aminoazobenzene	25.72	197	92,120,65,77
Dichlone	25.77	191	163,226,228,135,193
Dinoseb	25.83	211	163,147,117,240
Disulfoton	25.83	88	97,89,142,186
Fluchloralin	25.88	306	63,326,328,264,65
Mexacarbate	26.02	165	150,134,164,222
4,4'-Oxydianiline	26.08	200	108,171,80,65
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	152,141,169,151
Phosphamidon	26.85	127	264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	185,41,193,266
Methyl parathion	27.03	109	125,263,79,93
Carbaryl	27.17	144	115,116,201
Dimethylaminoazobenzene	27.50	225	120,77,105,148,42
Propylthiouracil	27.68	170	142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d12 (IS)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	125,127,93,158
Kepone	28.18	272	274,237,178,143,270
Fenthion	28.37	278	125,109,169,153
Parathion	28.40	109	97,291,139,155
Anilazine	28.47	239	241,143,178,89
Bis(2-ethylhexyl) phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	106,196,180
Carbophenothion	28.58	157	97,121,342,159,199
5-Nitroacenaphthene	28.73	199	152,169,141,115
Methapyrilene	28.77	97	50,191,71
Isodrin	28.95	193	66,195,263,265,147

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Captan	29.47	79	149,77,119,117
Chlorfenvinphos	29.53	267	269,323,325,295
Crotoxyphos	29.73	127	105,193,166
Phosmet	30.03	160	77,93,317,76
EPN	30.11	157	169,185,141,323
Tetrachlorvinphos	30.27	329	109,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	167,195
Barban	30.83	222	51,87,224,257,153
Aramite	30.92	185	191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	139,253,111,141
Fensulfothion	31.87	293	97,308,125,292
Ethion	32.08	231	97,153,125,121
Diethylstilbestrol	32.15	268	145,107,239,121,159
Famphur	32.67	218	125,93,109,217
Tri-p-tolyl phosphate[b]	32.75	368	367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d_{12} (IS)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	241,239,120
5,5-Diphenylhydantoin	33.40	180	104,252,223,209
Captafol	33.47	79	77,80,107
Dinocap	33.47	69	41,39
Methoxychlor	33.55	227	228,152,114,274,212
2-Acetylaminofluorene	33.58	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	201,229
3-Methylcholanthrene	35.07	268	252,253,126,134,113
Phosalone	35.23	182	184,367,121,379
Azinphos-methyl	35.25	160	132,93,104,105
Leptophos	35.28	171	377,375,77,155,379
Mirex	35.43	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,119,217,219,199
Dibenz(a,j)acridine	36.40	279	280,277,250
Mestranol	36.48	277	310,174,147,242
Coumaphos	37.08	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	151,150,300

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	135,105,77
Hexachlorophene	47.98	196	198,209,211,406,408
Aldrin	--	66	263,220
Aroclor 1016	--	222	260,292
Aroclor 1221	--	190	224,260
Aroclor 1232	--	190	224,260
Aroclor 1242	--	222	256,292
Aroclor 1248	--	292	362,326
Aroclor 1254	--	292	362,326
Aroclor 1260	--	360	362,394
α-BHC	--	183	181,109
β-BHC	--	181	183,109
δ-BHC	--	183	181,109
γ-BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin	--	263	82,81
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr)	--	172	171
2-Fluorophenol (surr)	--	112	64
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d_5 (surr)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d_6 (surr)	--	99	42,71
Terphenyl-d_{14} (surr)	--	244	122,212
2,4,6-Tribromophenol (surr)	--	330	332,141
Toxaphene	--	159	231,233

IS = internal standard
surr = surrogate
[a]Estimated retention times
[b]Substitute for the non-specific mixture, tricresyl phosphate

TABLE 2

ESTIMATED QUANTITATION LIMITS (EQLs) FOR SEMIVOLATILE ORGANICS

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
Acenaphthene	10	660
Acenaphthylene	10	660
Acetophenone	10	ND
2-Acetylaminofluorene	20	ND
1-Acetyl-2-thiourea	1000	ND
2-Aminoanthraquinone	20	ND
Aminoazobenzene	10	ND
4-Aminobiphenyl	20	ND
Anilazine	100	ND
o-Anisidine	10	ND
Anthracene	10	660
Aramite	20	ND
Azinphos-methyl	100	ND
Barban	200	ND
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
p-Benzoquinone	10	ND
Benzyl alcohol	20	1300
Bis(2-chloroethoxy)methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
4-Bromophenyl phenyl ether	10	660
Bromoxynil	10	ND
Butyl benzyl phthalate	10	660
Captafol	20	ND
Captan	50	ND
Carbaryl	10	ND
Carbofuran	10	ND
Carbophenothion	10	ND
Chlorfenvinphos	20	ND
4-Chloroaniline	20	1300
Chlorobenzilate	10	ND
5-Chloro-2-methylaniline	10	ND
4-Chloro-3-methylphenol	20	1300
3-(Chloromethyl)pyridine hydrochloride	100	ND
2-Chloronaphthalene	10	660

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
2-Chlorophol	10	660
4-Chlorophyl phenyl ether	10	660
Chrysene	10	660
Coumaphos	40	ND
p-Cresidine	10	ND
Crotoxypho	20	ND
2-Cyclohex 4,6-dinitrophenol	100	ND
Demeton-O	10	ND
Demeton-S	10	ND
Diallate (cis r trans)	10	ND
Diallate (tra or cis)	10	ND
2,4-Diamino luene	20	ND
Dibenz(a,j) idine	10	ND
Dibenz(a,h) thracene	10	660
Dibenzofura	10	660
Dibenzo(a, yrene	10	ND
Di-n-butyl p halate	10	ND
Dichlone	NA	ND
1,2-Dichloro enzene	10	660
1,3-Dichloro enzene	10	660
1,4-Dichloro enzene	10	660
3,3'-Dichlor enzidine	20	1300
2,4-Dichloro henol	10	660
2,6-Dichloro henol	10	ND
Dichlorovos	10	ND
Dicrotophos	10	ND
Diethyl phth ate	10	660
Diethylstilbe rol	20	ND
Diethyl sulfa	100	ND
Dimethoate	20	ND
3,3'-Dimeth ybenzidine	100	ND
Dimethylam bazobenzene	10	ND
7,12-Dimeth benz(a)anthracene	10	ND
3,3'-Dimeth enzidine	10	ND
a,a-Dimethy henethylamine	ND	ND
2,4-Dimethy henol	10	660
Dimethyl ph alate	10	660
1,2-Dinitrob zene	40	ND
1,3-Dinitrob zene	20	ND
1,4-Dinitrob zene	40	ND
4,6-Dinitro-2 methylphenol	50	3300
2,4-Dinitrop nol	50	3300

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
2,4-Dinitrotol ne	10	660
2,6-Dinitrotol ne	10	660
Dinocap	100	ND
Dinoseb	20	ND
5,5-Diphenylh dantoin	20	ND
Di-n-octyl pht late	10	660
Disulfoton	10	ND
EPN	10	ND
Ethion	10	ND
Ethyl carbam e	50	ND
Bis(2-ethylhe) phthalate	10	660
Ethyl methan ulfonate	20	ND
Famphur	20	ND
Fensulfothion	40	ND
Fenthion	10	ND
Fluchloralin	20	ND
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobe zene	10	660
Hexachlorobu diene	10	660
Hexachlorocy pentadiene	10	660
Hexachloroeth ne	10	660
Hexachloroph ne	50	ND
Hexachloropr ene	10	ND
Hexamethylph sphoramide	20	ND
Hydroquinone	ND	ND
Indeno(1,2,3-)pyrene	10	660
Isodrin	20	ND
Isophorone	10	660
Isosafrole	10	ND
Kepone	20	ND
Leptophos	10	ND
Malathion	50	ND
Maleic anhydr e	NA	ND
Mestranol	20	ND
Methapyrilene	100	ND
Methoxychlor	10	ND
3-Methylchola hrene	10	ND
4,4'-Methylen s(2-chloroaniline)	NA	ND
Methyl methar sulfonate	10	ND
2-Methylnaph alene	10	660
Methyl parath n	10	ND
2-Methylphen	10	660
3-Methylphen	10	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
4-Methylph ol	10	660
Mevinphos	10	ND
Mexacarba	20	ND
Mirex	10	ND
Monocroto os	40	ND
Naled	20	ND
Naphthalen	10	660
1,4-Naphth uinone	10	ND
1-Naphthyla ine	10	ND
2-Naphthyla ine	10	ND
Nicotine	20	ND
5-Nitroacen hthene	10	ND
2-Nitroanilin	50	3300
3-Nitroanilin	50	3300
4-Nitroanilin	20	ND
5-Nitro-o-an dine	10	ND
Nitrobenzen	10	660
4-Nitrobiphe l	10	ND
Nitrofen	20	ND
2-Nitrophenc	10	660
4-Nitrophenc	50	3300
5-Nitro-o-tolu ine	10	ND
4-Nitroquinol e-1-oxide	40	ND
N-Nitrosodi-n utylamine	10	ND
N-Nitrosodie lamine	20	ND
N-Nitrosodiph nylamine	10	660
N-Nitroso-di- opropylamine	10	660
N-Nitrosopipe dine	20	ND
N-Nitrosopyr dine	40	ND
Octamethyl p ophosphoramide	200	ND
4,4'-Oxydianil e	20	ND
Parathion	10	ND
Pentachlorob zene	10	ND
Pentachloront benzene	20	ND
Pentachlorop nol	50	3300
Phenacetin	20	ND
Phenanthrene	10	660
Phenobarbital	10	ND
Phenol	10	660
1,4-Phenylene amine	10	ND
Phorate	10	ND
Phosalone	100	ND
Phosmet	40	ND
Phosphamidon	100	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
Phthalic anhy ide	100	ND
2-Picoline	ND	ND
Piperonyl sulf ide	100	ND
Pronamide	10	ND
Propylthioura	100	ND
Pyrene	10	660
Pyridine	ND	ND
Resorcinol	100	ND
Safrole	10	ND
Strychnine	40	ND
Sulfallate	10	ND
Terbufos	20	ND
1,2,4,5-Tetra orobenzene	10	ND
2,3,4,6-Tetra orophenol	10	ND
Tetrachlorvin os	20	ND
Tetraethyl pyr hosphate	40	ND
Thionazine	20	ND
Thiophenol (B nzenethiol)	20	ND
o-Toluidine	10	ND
1,2,4-Trichlor enzene	10	660
2,4,5-Trichlor henol	10	660
2,4,6-Trichlor henol	10	660
Trifluralin	10	ND
2,4,5-Trimeth niline	10	ND
Trimethyl pho hate	10	ND
1,3,5-Trinitrob nzene	10	ND
Tris(2,3-dibro opropyl) phosphate	200	ND
Tri-p-tolyl pho hate(h)	10	ND
O,O,O-Triethy hosphorothioate	NT	ND

[a] Sample E Ls are highly matrix-dependent. The EQLs listed here are provided for guidance and may not a ays be achievable.

[b] EQLs list for soil/sediment are based on wet weight. Normally, data are reported on a dry weight ba , therefore, EQLs will be higher based on the % dry weight of each sample. These EQLs are ased on a 30-g sample and gel permeation chromatography cleanup.

ND = Not D ermined
NA = Not A licable
NT = Not T ed

Other Matrice	Factor[c]
High-concent ion soil and sludges by ultrasonic extractor	7.5
Non-water mi ble waste	75

[c] EQL = (EQL r Low Soil/Sediment given above in Table 2) x (Factor)

TABLE 3

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a,b]

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

[a] Data taken from Reference 3.
[b] Alternate tuning criteria may be used, (e.g., CLP, Method 525, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 4

CALIBRATION CHECK COMPOUNDS (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 5

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethyl-phenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	

TABLE 5
(continued)

Phenanth[illegible]ne-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminob[illegible]henyl	Benzidine	Benzo(b)fluor-anthene
Anthracer[illegible]	Benzo(a)anthracene	Benzo(k)fluor-anthene
4-Bromop[illegible]enyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)-perylene
Di-n-butyl[illegible]hthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro[illegible]-methyl-phenol	Chrysene	Dibenz(a,j)acridine
Diphenyla[illegible]ne	3,3'-Dichlorobenzidine	Dibenz(a,h)-anthracene
Fluoranth[illegible]e	p-Dimethylaminoazobenzene	
Hexachlor[illegible]enzene	Pyrene	
N-Nitrosod[illegible]henylamine	Terphenyl-d_{14} (surr)	
Pentachlo[illegible]phenol	7,12-Dimethylbenz-(a)anthracene	
Pentachlo[illegible]nitrobenzene	Di-n-octyl phthalate	
Phenaceti[illegible]	Indeno(1,2,3-cd) pyrene	
Phenanthr[illegible]e	3-Methylchol-anthrene	
Pronamide		

(surr) = su[illegible]gate

TABLE 6

MULTILABORATORY PERFORMANCE DATA[a]

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L)	Range p, p_s (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192

TABLE 6
(continued)

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L)	Range p, p_s (%)
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in µg/L

$\bar{x}$ = Average recovery for four recovery measurements, in µg/L

p, p_s = Measured percent recovery

D = Detected; result must be greater than zero

[1] Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 7

METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Compound	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Acenaphthene	0.96C+0.19	0.15$\bar{x}$-0.12	0.21$\bar{x}$-0.67
Acenaphthylene	0.89C+0.74	0.24$\bar{x}$-1.06	0.26$\bar{x}$-0.54
Aldrin	0.78C+1.66	0.27$\bar{x}$-1.28	0.43$\bar{x}$+1.13
Anthracene	0.80C+0.68	0.21$\bar{x}$-0.32	0.27$\bar{x}$-0.64
Benz(a)anthracene	0.88C-0.60	0.15$\bar{x}$+0.93	0.26$\bar{x}$-0.21
Benzo(b)fluoranthene	0.93C-1.80	0.22$\bar{x}$+0.43	0.29$\bar{x}$+0.96
Benzo(k)fluoranthene	0.87C-1.56	0.19$\bar{x}$+1.03	0.35$\bar{x}$+0.40
Benzo(a)pyrene	0.90C-0.13	0.22$\bar{x}$+0.48	0.32$\bar{x}$+1.35
Benzo(g,h,i)perylene	0.98C-0.86	0.29$\bar{x}$+2.40	0.51$\bar{x}$-0.44
Benzyl butyl phthalate	0.66C-1.68	0.18$\bar{x}$+0.94	0.53$\bar{x}$+0.92
β-BHC	0.87C-0.94	0.20$\bar{x}$-0.58	0.30$\bar{x}$+1.94
δ-BHC	0.29C-1.09	0.34$\bar{x}$+0.86	0.93$\bar{x}$-0.17
Bis(2-chloroethyl) ether	0.86C-1.54	0.35$\bar{x}$-0.99	0.35$\bar{x}$+0.10
Bis(2-chloroethoxy)methane	1.12C-5.04	0.16$\bar{x}$+1.34	0.26$\bar{x}$+2.01
Bis(2-chloroisopropyl) ether	1.03C-2.31	0.24$\bar{x}$+0.28	0.25$\bar{x}$+1.04
Bis(2-ethylhexyl) phthalate	0.84C-1.18	0.26$\bar{x}$+0.73	0.36$\bar{x}$+0.67
4-Bromophenyl phenyl ether	0.91C-1.34	0.13$\bar{x}$+0.66	0.16$\bar{x}$+0.66
2-Chloronaphthalene	0.89C+0.01	0.07$\bar{x}$+0.52	0.13$\bar{x}$+0.34
4-Chlorophenyl phenyl ether	0.91C+0.53	0.20$\bar{x}$-0.94	0.30$\bar{x}$-0.46
Chrysene	0.93C-1.00	0.28$\bar{x}$+0.13	0.33$\bar{x}$-0.09
4,4'-DDD	0.56C-0.40	0.29$\bar{x}$-0.32	0.66$\bar{x}$-0.96
4,4'-DDE	0.70C-0.54	0.26$\bar{x}$-1.17	0.39$\bar{x}$-1.04
4,4'-DDT	0.79C-3.28	0.42$\bar{x}$+0.19	0.65$\bar{x}$-0.58
Dibenzo(a,h)anthracene	0.88C+4.72	0.30$\bar{x}$+8.51	0.59$\bar{x}$+0.25
Di-n-butyl phthalate	0.59C+0.71	0.13$\bar{x}$+1.16	0.39$\bar{x}$+0.60
1,2-Dichlorobenzene	0.80C+0.28	0.20$\bar{x}$+0.47	0.24$\bar{x}$+0.39
1,3-Dichlorobenzene	0.86C-0.70	0.25$\bar{x}$+0.68	0.41$\bar{x}$+0.11
1,4-Dichlorobenzene	0.73C-1.47	0.24$\bar{x}$+0.23	0.29$\bar{x}$+0.36
3,3'-Dichlorobenzidine	1.23C-12.65	0.28$\bar{x}$+7.33	0.47$\bar{x}$+3.45
Dieldrin	0.82C-0.16	0.20$\bar{x}$-0.16	0.26$\bar{x}$-0.07
Diethyl phthalate	0.43C+1.00	0.28$\bar{x}$+1.44	0.52$\bar{x}$+0.22
Dimethyl phthalate	0.20C+1.03	0.54$\bar{x}$+0.19	1.05$\bar{x}$-0.92
2,4-Dinitrotoluene	0.92C-4.81	0.12$\bar{x}$+1.06	0.21$\bar{x}$+1.50
2,6-Dinitrotoluene	1.06C-3.60	0.14$\bar{x}$+1.26	0.19$\bar{x}$+0.35
Di-n-octyl phthalate	0.76C-0.79	0.21$\bar{x}$+1.19	0.37$\bar{x}$+1.19
Endosulfan sulfate	0.39C+0.41	0.12$\bar{x}$+2.47	0.63$\bar{x}$-1.03
Endrin aldehyde	0.76C-3.86	0.18$\bar{x}$+3.91	0.73$\bar{x}$-0.62
Fluoranthene	0.81C+1.10	0.22$\bar{x}$-0.73	0.28$\bar{x}$-0.60
Fluorene	0.90C-0.00	0.12$\bar{x}$+0.26	0.13$\bar{x}$+0.61
Heptachlor	0.87C-2.97	0.24$\bar{x}$-0.56	0.50$\bar{x}$-0.23
Heptachlor epoxide	0.92C-1.87	0.33$\bar{x}$-0.46	0.28$\bar{x}$+0.64

TABLE 7
(continued)

Compound	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Hexachlor enzene	0.74C+0.66	0.18$\bar{x}$-0.10	0.43$\bar{x}$-0.52
Hexachlor utadiene	0.71C-1.01	0.19$\bar{x}$+0.92	0.26$\bar{x}$+0.49
Hexachlor thane	0.73C-0.83	0.17$\bar{x}$+0.67	0.17$\bar{x}$+0.80
Indeno(1,2 -cd)pyrene	0.78C-3.10	0.29$\bar{x}$+1.46	0.50$\bar{x}$-0.44
Isophoron	1.12C+1.41	0.27$\bar{x}$+0.77	0.33$\bar{x}$+0.26
Naphthale	0.76C+1.58	0.21$\bar{x}$-0.41	0.30$\bar{x}$-0.68
Nitrobenze	1.09C-3.05	0.19$\bar{x}$+0.92	0.27$\bar{x}$+0.21
N-Nitrosod n-propylamine	1.12C-6.22	0.27$\bar{x}$+0.68	0.44$\bar{x}$+0.47
Aroclor 12	0.81C-10.86	0.35$\bar{x}$+3.61	0.43$\bar{x}$+1.82
Phenanthr e	0.87C+0.06	0.12$\bar{x}$+0.57	0.15$\bar{x}$+0.25
Pyrene	0.84C-0.16	0.16$\bar{x}$+0.06	0.15$\bar{x}$+0.31
1,2,4-Trich robenzene	0.94C-0.79	0.15$\bar{x}$+0.85	0.21$\bar{x}$+0.39
4-Chloro-3- ethylphenol	0.84C+0.35	0.23$\bar{x}$+0.75	0.29$\bar{x}$+1.31
2-Chloroph nol	0.78C+0.29	0.18$\bar{x}$+1.46	0.28$\bar{x}$+0.97
2,4-Dichlor henol	0.87C-0.13	0.15$\bar{x}$+1.25	0.21$\bar{x}$+1.28
2,4-Dimeth henol	0.71C+4.41	0.16$\bar{x}$+1.21	0.22$\bar{x}$+1.31
2,4-Dinitrop enol	0.81C-18.04	0.38$\bar{x}$+2.36	0.42$\bar{x}$+26.29
2-Methyl-4, dinitrophenol	1.04C-28.04	0.10$\bar{x}$+42.29	0.26$\bar{x}$+23.10
2-Nitropher	0.07C-1.15	0.16$\bar{x}$+1.94	0.27$\bar{x}$+2.60
4-Nitropher	0.61C-1.22	0.38$\bar{x}$+2.57	0.44$\bar{x}$+3.24
Pentachlor henol	0.93C+1.99	0.24$\bar{x}$+3.03	0.30$\bar{x}$+4.33
Phenol	0.43C+1.26	0.26$\bar{x}$+0.73	0.35$\bar{x}$+0.58
2,4,6-Trichl ophenol	0.91C-0.18	0.16$\bar{x}$+2.22	0.22$\bar{x}$+1.81

x' = Ex cted recovery for one or more measurements of a sample containing a concentration of in µg/L.

s_r' = Ex cted single analyst standard deviation of measurements at an average concentration of $\bar{x}$ n µg/L.

S' = Ex cted interlaboratory standard deviation of measurements at an average concentration fou of $\bar{x}$, in µg/L.

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

$\bar{x}$ = Ave ge recovery found for measurements of samples containing a concentration of C, in µg/

[a] Criteria om 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based rectly on the method performance data in Table 7. Where necessary, the limits for recover have been broadened to assure applicability of the limits to concentrations below those u d to develop Table 7. These values are for guidance only. Appropriate derivation of accepta e criteria for capillary columns should result in much narrower ranges. See Method 8000 fo nformation on developing and updating acceptance criteria for method performance.

TABLE 8

EXTRACTION EFFICIENCY AND AQUEOUS STABILITY RESULTS

Compound	Percent Recovery on Day 0		Percent Recovery on Day 7	
	Mean	RSD	Mean	RSD
3-Amino-9-[illegible]ylcarbazole	80	8	73	3
4-Chloro-1,2-[illegible]henylenediamine	91	1	108	4
4-Chloro-1,3-[illegible]henylenediamine	84	3	70	3
1,2-Dibromo[illegible]-chloropropane	97	2	98	5
Dinoseb	99	3	97	6
Parathion	100	2	103	4
4,4'-Methyle[illegible]bis(N,N-dimethylaniline)	108	4	90	4
5-Nitro-o-tol[illegible]dine	99	10	93	4
2-Picoline	80	4	83	4
Tetraethyl di[illegible]iopyrophosphate	92	7	70	1

Data taken f[illegible]m Reference 6.

TABLE 9

MEAN PERCENT RECOVERIES AND PERCENT RSD VALUES FOR SEMIVOLATILE ORGANICS FROM SPIKED CLAY SOIL AND TOPSOIL BY AUTOMATED SOXHLET EXTRACTION (METHOD 3541) WITH HEXANE-ACETONE (1:1)[a]

Compound	Clay Soil		Topsoil	
	Mean Recovery	RSD	Mean Recovery	RSD
1,3-Dichlorobenzene	0	--	0	--
1,2-Dichlorobenzene	0	--	0	--
Nitrobenzene	0	--	0	--
Benzal chloride	0	--	0	--
Benzotrichloride	0	--	0	--
4-Chloro-2-nitrotoluene	0	--	0	--
Hexachlorocyclopentadiene	4.1	15	7.8	23
2,4-Dichloronitrobenzene	35.2	7.6	21.2	15
3,4-Dichloronitrobenzene	34.9	15	20.4	11
Pentachlorobenzene	13.7	7.3	14.8	13
2,3,4,5-Tetrachloronitrobenzene	55.9	6.7	50.4	6.0
Benefin	62.6	4.8	62.7	2.9
alpha-BHC	58.2	7.3	54.8	4.8
Hexachlorobenzene	26.9	13	25.1	5.7
delta-BHC	95.8	4.6	99.2	1.3
Heptachlor	46.9	9.2	49.1	6.3
Aldrin	97.7	12	102	7.4
Isopropalin	102	4.3	105	2.3
Heptachlor epoxide	90.4	4.4	93.6	2.4
trans-Chlordane	90.1	4.5	95.0	2.3
Endosulfan I	96.3	4.4	101	2.2
Dieldrin	129	4.7	104	1.9
2,5-Dichlorophenyl-4-nitrophenyl ether	110	4.1	112	2.1
Endrin	102	4.5	106	3.7
Endosulfan II	104	4.1	105	0.4
p,p'-DDT	134	2.1	111	2.0
2,3,6-Trichlorophenyl-4'-nitrophenyl ether	110	4.8	110	2.8
2,3,4-Trichlorophenyl-4'-nitrophenyl ether	112	4.4	112	3.3
Mirex	104	5.3	108	2.2

[a] The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g; the spiking concentration was 500 ng/g, except for the surrogate compounds at 1000 ng/g, 2,5-Dichlorophenyl-4-nitrophenyl ether, 2,3,6-Trichlorophenyl-4-nitrophenyl ether, and 2,3,4-Trichlorophenyl-4-nitrophenyl ether at 1500 ng/g, Nitrobenzene at 2000 ng/g, and 1,3-Dichlorobenzene and 1,2-Dichlorobenzene at 5000 ng/g.

TABLE 10

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR THE EXTRACTION OF SEMIVOLATILE ORGANICS FROM SPIKED CLAY BY AUTOMATED SOXHLET (METHOD 3541)[a]

Compound	Mean Recovery	RSD
Phenol	47.8	5.6
Bis(2-chloroe yl)ether	25.4	13
2-Chlorophen	42.7	4.3
Benzyl alcoh	55.9	7.2
2-Methylphen	17.6	6.6
Bis(2-chloroi propyl)ether	15.0	15
4-Methylphen	23.4	6.7
N-Nitroso-di- propylamine	41.4	6.2
Nitrobenzene	28.2	7.7
Isophorone	56.1	4.2
2-Nitrophenol	36.0	6.5
2,4-Dimethylp enol	50.1	5.7
Benzoic acid	40.6	7.7
Bis(2-chloroe oxy)methane	44.1	3.0
2,4-Dichlorop nol	55.6	4.6
1,2,4-Trichlor enzene	18.1	31
Naphthalene	26.2	15
4-Chloroanilin	55.7	12
4-Chloro-3-m ylphenol	65.1	5.1
2-Methylnaph alene	47.0	8.6
Hexachlorocy pentadiene	19.3	19
2,4,6-Trichlor henol	70.2	6.3
2,4,5-Trichlor henol	26.8	2.9
2-Chloronaph alene	61.2	6.0
2-Nitroaniline	73.8	6.0
Dimethyl phth ate	74.6	5.2
Acenaphthyle	71.6	5.7
3-Nitroaniline	77.6	5.3
Acenaphthen	79.2	4.0
2,4-Dinitrophe ol	91.9	8.9
4-Nitrophenol	62.9	16
Dibenzofuran	82.1	5.9
2,4-Dinitrotolu ne	84.2	5.4
2,6-Dinitrotolu ne	68.3	5.8
Diethyl phthal e	74.9	5.4
4-Chlorophen phenyl ether	67.2	3.2
Fluorene	82.1	3.4
4-Nitroaniline	79.0	7.9

TABLE 10
(continued)

Compound	Mean Recovery	RSD
4,6-Dinitro-2 ethylphenol	63.4	6.8
N-Nitrosodip enylamine	77.0	3.4
4-Bromophe l-phenyl ether	62.4	3.0
Hexachlorob zene	72.6	3.7
Pentachloro enol	62.7	6.1
Phenanthren	83.9	5.4
Anthracene	96.3	3.9
Di-n-butyl ph alate	78.3	40
Fluoranthene	87.7	6.9
Pyrene	102	0.8
Butyl benzyl thalate	66.3	5.2
3,3'-Dichloro nzidine	25.2	11
Benzo(a)anth cene	73.4	3.8
Bis(2-ethylhe l) phthalate	77.2	4.8
Chrysene	76.2	4.4
Di-n-octyl ph alate	83.1	4.8
Benzo(b)fluo nthene	82.7	5.0
Benzo(k)fluo nthene	71.7	4.1
Benzo(a)pyre e	71.7	4.1
Indeno(1,2,3-)pyrene	72.2	4.3
Dibenzo(a,h) thracene	66.7	6.3
Benzo(g,h,i)p ylene	63.9	8.0
1,2-Dichlorob zene	0	--
1,3-Dichlorob zene	0	--
1,4-Dichlorob zene	0	--
Hexachloroet ne	0	--
Hexachlorobu diene	0	--

[a] Number of eterminations was three. The operating conditions for the Soxtec apparatus were as follows: mersion time 45 min; extraction time 45 min; the sample size was 10 g clay soil; the spike conc ration was 6 mg/kg per compound. The sample was allowed to equilibrate 1 hour after spikin

Data taken fr Reference 7.

TABLE 11

PRECISION AND BIAS VALUES FOR METHOD 3542[1]

Compound	Mean Recovery	Standard Deviation	Relative Standard Deviation Percent
2-Fluorophenol	74.6	28.6	38.3
Phenol-d_5	77.8	27.7	35.6
Nitrobenzene-d_5	65.6	32.5	49.6
2-Fluorobiphenyl	75.9	30.3	39.9
2,4,6-Tribromophenol	67.0	34.0	50.7
Terphenyl-d_{14}	78.6	32.4	41.3

[1] The surrogate values shown in Table 11 represent mean recoveries for surrogates in all Method 0010 matrices in a field dynamic spiking study.

TABLE 12

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES
AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Phenol	83.3	78.7	135.9	73.9	82.8	124.6	108.8	130.6	89.7	102.0
Bis(2-chloroethyl)ether	102.1	85.1	109.1	96.0	88.0	103.6	122.3	119.9	90.8	**101.9**
2-Chlorophenol	100.8	82.6	115.0	93.8	88.9	111.1	115.0	115.3	91.9	**101.6**
1,3-Dichlorobenzene	127.7	129.7	110.0	*364.2	129.9	119.0	*241.3	*163.7	107.1	**120.6**
1,4-Dichlorobenzene	127.9	127.0	110.5	*365.9	127.8	116.4	*309.6	*164.1	105.8	**119.2**
1,2-Dichlorobenzene	116.8	115.8	101.3	*159.2	113.4	105.5	*189.3	134.0	100.4	**112.5**
2-Methylphenol	98.9	82.1	119.7	87.6	89.4	111.0	133.2	128.0	92.1	**104.7**
Bis(2-chloroisopropyl)ether	109.4	71.5	108.0	81.8	81.0	88.6	118.1	148.3	94.8	**100.2**
o-Toluidine	100.0	89.7	117.2	100.0	*152.5	120.3	100.0	*199.5	102.7	**110.3**
N-Nitroso-di-n-propylamine	103.0	79.1	107.7	83.9	88.1	96.2	109.9	123.3	91.4	**98.1**
Hexachloroethane	97.1	125.1	111.0	*245.4	117.1	128.1	*566.7	147.9	103.7	**118.6**
Nitrobenzene	104.8	82.4	106.6	86.8	84.6	101.7	119.7	122.1	93.3	**100.2**
Isophorone	100.0	86.4	98.2	87.1	87.5	109.7	135.5	118.4	92.7	**101.7**
2,4-Dimethylphenol	100.0	104.5	140.0	100.0	114.4	123.1	100.0	*180.6	96.3	**109.8**
2-Nitrophenol	80.7	80.5	107.9	91.4	86.7	103.2	122.1	107.1	87.0	**96.3**
Bis(chloroethoxy)methane	94.4	80.6	94.7	86.5	84.4	99.6	130.6	110.7	93.2	**97.2**
2,4-Dichlorophenol	88.9	87.8	111.4	85.9	87.6	103.5	123.3	107.0	92.1	**98.6**
1,2,4-Trichlorobenzene	98.0	97.8	98.8	123.0	93.7	94.5	137.0	99.4	95.3	**104.2**
Naphthalene	101.7	97.2	123.6	113.2	102.9	129.5	*174.5	114.0	89.8	**106.1**
4-Chloroaniline	100.0	*150.2	*162.4	100.0	125.5	*263.6	100.0	*250.8	114.9	**108.1**
Hexachlorobutadiene	101.1	98.7	102.2	124.1	90.3	98.0	134.9	96.1	96.8	**104.7**
4-Chloro-3-methylphenol	90.4	80.2	114.7	79.0	85.2	109.8	131.6	116.2	90.1	**99.7**
2-Methylnaphthalene	93.2	89.9	94.6	104.1	92.2	105.9	146.2	99.1	93.3	**102.1**
Hexachlorocyclopentadiene	100.0	100.0	0.0	100.0	100.0	6.8	100.0	100.0	*238.3	**75.8**
2,4,6-Trichlorophenol	94.6	90.0	112.0	84.2	91.2	103.6	101.6	95.9	89.8	**95.9**
2,4,5-Trichlorophenol	84.4	91.9	109.6	96.1	80.7	103.6	108.9	83.9	87.9	**94.1**
2-Chloronaphthalene	100.0	91.3	93.6	97.6	93.4	98.3	106.8	93.0	92.0	**96.2**
2-Nitroaniline	90.0	83.4	97.4	71.3	88.4	89.9	112.1	113.3	87.7	**92.6**
2,6-Dinitrotoluene	83.1	90.6	91.6	86.4	90.6	90.3	104.3	84.7	90.9	**90.3**
Acenaphthylene	104.9	95.9	100.5	99.0	97.9	108.8	118.5	97.8	92.0	**101.7**
3-Nitroaniline	*224.0	115.6	97.6	100.0	111.8	107.8	0.0	111.7	89.0	**92.9**
Acenaphthene	102.1	92.6	97.6	97.2	96.9	104.4	114.2	92.0	89.0	**98.4**
4-Nitrophenol	0.0	93.2	121.5	18.1	87.1	116.6	69.1	90.5	84.5	**75.6**
2,4-Dinitrotoluene	73.9	91.9	100.2	84.7	93.8	98.9	100.9	84.9	87.3	**90.7**

TABLE 12 (cont.)

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES
AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Dibenzofuran	89.5	91.7	109.3	98.5	92.2	111.4	113.8	92.7	90.4	**98.8**
4-Chlorophenyl phe ether	83.0	94.5	98.7	95.7	94.3	94.2	111.4	87.7	90.3	**94.4**
Fluorene	85.2	94.9	89.2	102.0	95.5	93.8	121.3	85.7	90.9	**95.4**
4-Nitroaniline	77.8	114.8	94.5	129.6	103.6	95.4	*154.1	89.3	87.5	**99.1**
N-Nitrosodiphenylan e	82.6	96.7	93.8	92.9	93.4	116.4	97.5	110.9	86.7	**96.8**
4-Bromophenyl phe ether	85.6	92.9	92.8	91.1	107.6	89.4	118.0	97.5	87.1	**95.8**
Hexachlorobenzene	95.4	91.7	92.3	95.4	93.6	83.7	106.8	94.3	90.0	**93.7**
Pentachlorophenol	68.2	85.9	107.7	53.2	89.8	88.1	96.6	59.8	81.3	**81.2**
Phenanthrene	92.1	93.7	93.3	100.0	97.8	113.3	124.4	101.0	89.9	**100.6**
Anthracene	101.6	95.0	93.5	92.5	101.8	118.4	123.0	94.5	90.6	**101.2**
Carbazole	94.4	99.3	96.6	105.5	96.7	111.4	115.7	83.2	88.9	**99.1**
Fluoranthene	109.9	101.4	94.3	111.6	96.6	109.6	123.2	85.4	92.7	**102.7**
Pyrene	106.5	105.8	107.6	116.7	90.7	127.5	103.4	95.5	93.2	**105.2**
3,3'-Dichlorobenzidir	100.0	*492.3	131.4	100.0	*217.6	*167.6	100.0	*748.8	100.0	**116.5**
Benzo(a)anthracene	98.1	107.0	98.4	119.3	98.6	104.0	105.0	93.4	89.3	**101.5**
Chrysene	100.0	108.5	100.2	116.8	93.0	117.0	106.7	93.6	90.2	**102.9**
Benzo(b)fluoranthen	106.6	109.9	75.6	121.7	100.7	93.9	106.9	81.9	93.6	**99.0**
Benzo(k)fluoranthen	102.4	105.2	88.4	125.5	99.4	95.1	144.7	89.2	78.1	**103.1**
Benzo(a)pyrene	107.9	105.5	80.8	122.3	97.7	104.6	101.7	86.2	92.0	**99.9**
Indeno(1,2,3-cd)pyr	95.1	105.7	93.8	126.0	105.2	90.4	*133.6	82.6	91.9	**102.7**
Dibenz(a,h)anthrace	85.0	102.6	82.0	118.8	100.7	91.9	*142.3	*71.0	93.1	**98.6**
Benzo(g,h,i)perylene	98.0	0.0	81.2	0.0	83.6	78.6	*128.7	83.0	94.2	**66.4**
Average	95.1	94.3	**101.0**	95.5	96.5	**104.1**	113.0	*100.9	92.5	

Values greater tha 150% were not used to determine the averages, but the 0% values were used

TABLE 13

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT EC-1, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	(27.9)[b]	41.3 ± 3.6	(148)	8.7
Acenaphthylene	(0.8)	0.9 ± 0.1	(112)	11.1
Acenaphthene	(0.2)	0.2 ± 0.01	(100)	0.05
Fluorene	(15.3)	15.6 ± 1.8	(102)	11.5
Phenanthrene	15.8 ± 1.2	16.1 ± 1.8	102	11.2
Anthracene	(1.3)	1.1 ± 0.2	(88)	18.2
Fluoranthene	23.2 ± 2.0	24.1 ± 2.1	104	8.7
Pyrene	16.7 ± 2.0	17.2 ± 1.9	103	11.0
Benz(a)anthracene	8.7 ± 0.8	8.8 ± 1.0	101	11.4
Chrysene	(9.2)	7.9 ± 0.9	(86)	11.4
Benzo(b)fluoranthene	7.9 ± 0.9	8.5 ± 1.1	108	12.9
Benzo(k)fluoranthene	4.4 ± 0.5	4.1 ± 0.5	91	12.2
Benzo(a)pyrene	5.3 ± 0.7	5.1 ± 0.6	96	11.8
Indeno(1,2,3-cd)pyrene	5.7 ± 0.6	5.2 ± 0.6	91	11.5
Benzo(g,h,i)perylene	4.9 ± 0.7	4.3 ± 0.5	88	11.6
Dibenz(a,h)anthracene	(1.3)	1.1 ± 0.2	(85)	18.2

[a] Relative standard deviations for the SFE values are based on six replicate extractions.

[b] Values in parentheses were obtained from, or compared to, Soxhlet extraction results which were not certified.

Data are taken from Reference 10.

TABLE 14

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT HS-3, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	9.0 ± 0.7	7.4 ± 0.6	82	8.1
Acenaphthylene	0.3 ± 0.1	0.4 ± 0.1	133	25.0
Acenaphthene	4.5 ± 1.5	3.3 ± 0.3	73	9.0
Fluorene	13.6 ± 3.1	10.4 ± 1.3	77	12.5
Phenanthrene	85.0 ± 20.0	86.2 ± 9.5	101	11.0
Anthracene	13.4 ± 0.5	12.1 ± 1.5	90	12.4
Fluoranthene	60.0 ± 9.0	54.0 ± 6.1	90	11.3
Pyrene	39.0 ± 9.0	32.7 ± 3.7	84	11.3
Benz(a)anthracene	14.6 ± 2.0	12.1 ± 1.3	83	10.7
Chrysene	14.1 ± 2.0	12.0 ± 1.3	85	10.8
Benzo(b)fluoranthene	7.7 ± 1.2	8.4 ± 0.9	109	10.7
Benzo(k)fluoranthene	2.8 ± 2.0	3.2 ± 0.5	114	15.6
Benzo(a)pyrene	7.4 ± 3.6	6.6 ± 0.8	89	12.1
Indeno(1,2,3-cd)pyrene	5.0 ± 2.0	4.5 ± 0.6	90	13.3
Benzo(g,h,i)perylene	5.4 ± 1.3	4.4 ± 0.6	82	13.6
Dibenz(a,h)anthracene	1.3 ± 0.5	1.1 ± 0.3	85	27.3

[a] Relative standard deviations for the SFE values are based on three replicate extractions.

Data are taken from Reference 10.

TABLE 15

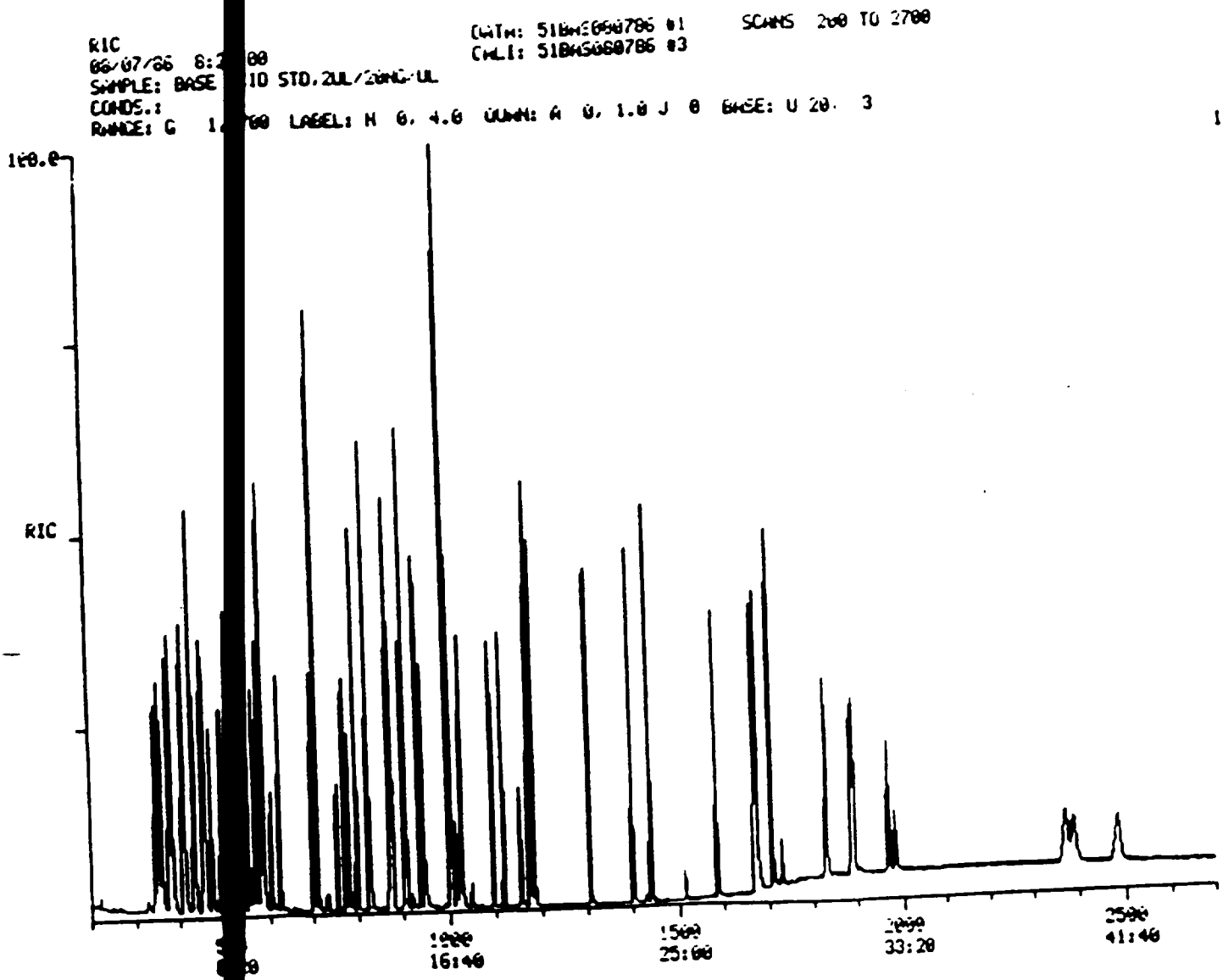
SINGL LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs ROM A CERTIFIED REFERENCE SOIL SRS103-100, USING METHOD 3561 (SFE - LIQUID TRAP)

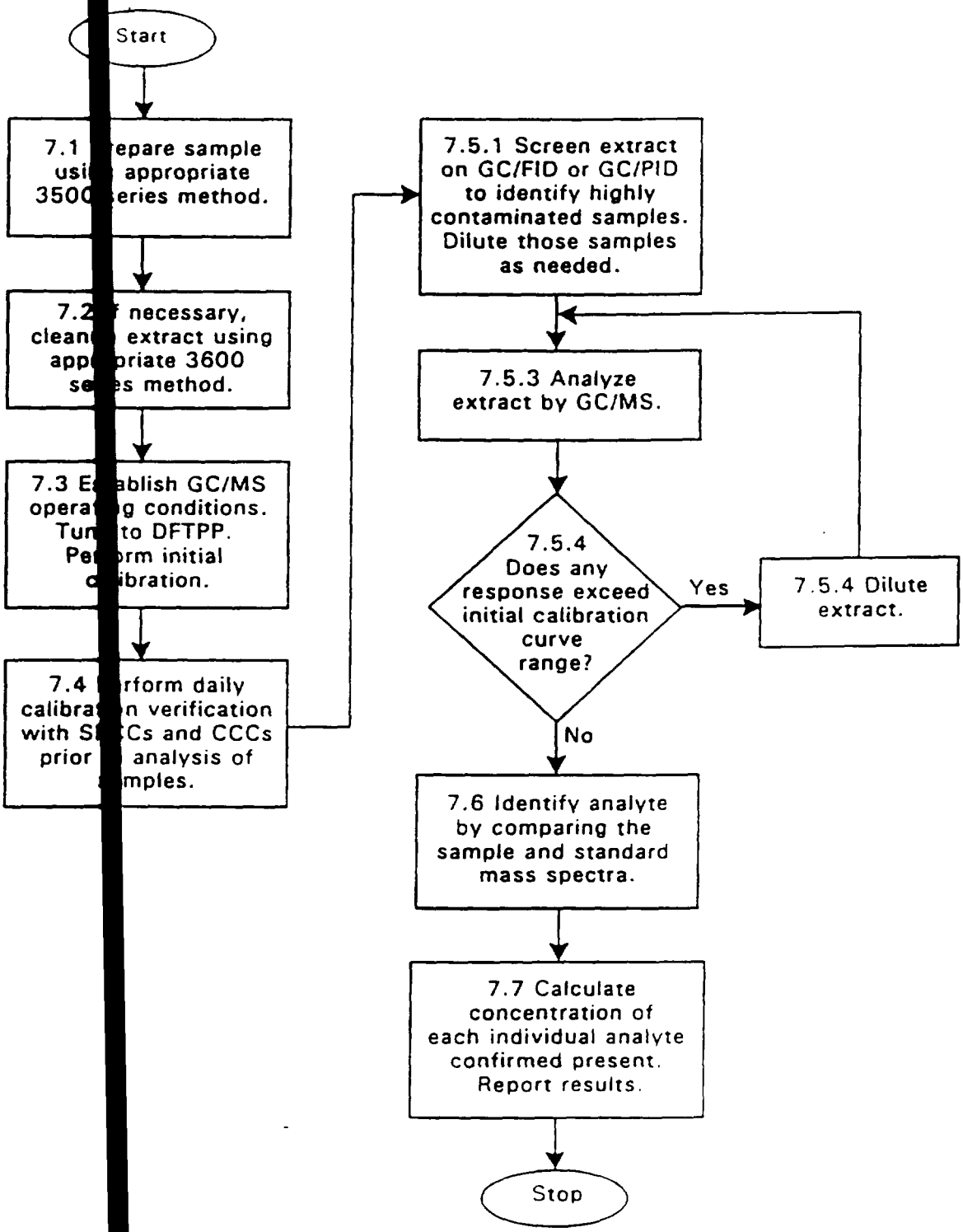
Compoun	Certified Value (mg/kg)			SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthal e	32.4	±	8.2	29.55	91	10.5
2-Methyln hthalene	62.1	±	11.5	76.13	122	2.0
Acenapht ne	632	±	105	577.28	91	2.9
Dibenzofu n	307	±	49	302.25	98	4.1
Fluorene	492	±	78	427.15	87	3.0
Phenanth ne	1618	±	340	1278.03	79	3.4
Anthracen	422	±	49	400.80	95	2.6
Fluoranth e	1280	±	220	1019.13	80	4.5
Pyrene	1033	±	285	911.82	88	3.1
Benz(a)an racene	252	±	38	225.50	89	4.8
Chrysene	297	±	26	283.00	95	3.8
Benzo(b)f oranthene + Benzo(k) oranthene	153	±	22	130.88	86	10.7
Benzo(a)p ene	97.2	±	17.1	58.28	60	6.5

[a] Relative tandard deviations for the SFE values are based on four replicate extractions.

Data ar aken from Reference 11.

FIGURE 1
GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD




Start
7.1 Prepare sample using appropriate 3500 series method.
7.2 If necessary, clean up extract using appropriate 3600 series method.
7.3 Establish GC/MS operating conditions. Tune to DFTPP. Perform initial calibration.
7.4 Perform daily calibration verification with SPCCs and CCCs prior to analysis of samples.
7.5.1 Screen extract on GC/FID or GC/PID to identify highly contaminated samples. Dilute those samples as needed.
7.5.3 Analyze extract by GC/MS.
7.5.4 Does any response exceed initial calibration curve range?
Yes
7.5.4 Dilute extract.
No
7.6 Identify analyte by comparing the sample and standard mass spectra.
7.7 Calculate concentration of each individual analyte confirmed present. Report results.
Stop

PENTACHLOROPHENOL

Method no.	39
Matrix:	Air
Target concentration:	0.5 mg/m³ (OSHA PEL/TWA)
Procedure:	Samples are collected by drawing a known volume of air through two specially prepared XAD-7 adsorbent tubes which are connected in series. Following desorption with methanol the samples are analyzed by high performance liquid chromatography with ultraviolet (UV) detection.
Recommended air volume and sampling rate:	48 L at 0.2 L/min
Reliable quantitation limit:	0.007 mg/m³
Standard error of estimate: (Figure 4.1.)	6.9%
Special requirements:	Sampling tubes must be obtained from the laboratory. Prior to sampling, remove the XAD-7 resin-filled front section of the sampling device and save it. This resin packed tube serves as a cap to trap any PCP which might volatilize from the filter after sampling. Immediately after the completion of sampling, re-attach this section onto the front section of the sampling device for shipment to the laboratory. (Section 2.)
Status of method:	A sampling and analytical method which has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

Date: October 1982 Chemist: Kevin Cummins

Organic Methods Evaluation Branch
OSHA Analytical Laboratory
Salt Lake City, Utah

1. Gener[illegible] Discussion

1.1. [illegible]ackground

.1.1. History

This air sampling procedure for pentachlorophenol (PCP) uses two laboratory prepared XAD-7 sampling tubes connected in series. A small glass fiber disc is placed ahead of the resin bed of the front tube to trap any aerosols present in the air. The second tube serves as a backup section in the unlikely event of breakthrough.

Unlike a previous NIOSH sampling method which employed ethylene glycol (Ref. 5.1.), or the frequently used aqueous sodium hydroxide solution, this method does not require the use of a cumbersome bubbler for sampling.

Previous evaluations of air sampling methods for phenol and cresol, and a preliminary study performed with a mixture of all of the chlorinated phenols indicate that XAD-7 resin will probably serve as a versatile sampling medium for a wide variety of phenolic compounds (Ref. 5.2.).

The majority of the air sampling evaluations performed with PCP in this study used an aerosol generation system which is described in Section 4.5. The effectiveness of XAD-7 as a sampling media was established by comparing the collection efficiency of both commercial (SKC Inc., Eighty-Four, PA.) and laboratory prepared XAD-7 sampling tubes with a variety of other samplers. Similar efficiencies were observed for XAD-7, silica gel, 0.1 N sodium hydroxide bubblers, isopropanol bubblers, and a glass fiber filter in a cassette followed by a XAD-7 sampling tube. The volatile nature of PCP was clearly demonstrated in aerosol studies. Approximately two thirds of the total PCP collected was observed to have passed through the glass fiber filter and was trapped on the backup XAD-7 tube. However, efficient collection of a sodium pentachlorophenate (NaPCP) aerosol was observed for the glass fiber filter alone. Since the recommended sampling device is a glass fiber filter placed ahead of the XAD-7 resin bed, this sampling device should effectively trap NaPCP. The HPLC analytical method, however, does not distinguish between PCP and NaPCP.

The high capacity of XAD-7 resin for PCP was clearly established in breakthrough studies performed with laboratory prepared XAD-7 sampling tubes. No breakthrough was observed after sampling a 0.82 mg/m^3 PCP aerosol atmosphere for over 4.5 h at 1 L/min, or after sampling a 10 mg/m^3 PCP vapor atmosphere for over 6 h at 1 L/min (Section 4.5.).

Laboratory prepared XAD-7 sampling tubes using 8-mm o.d., 6-mm i.d. glass tubing were selected for use in preference to the smaller commercial XAD-7 sampling tubes for a number of reasons: the large diameter opening of the laboratory-packed tube may improve aerosol sampling efficiency (Ref. 5.3.); the 8-mm diameter opening permits easy insertion of a glass fiber disc for trapping an aerosol in a one-unit system; and finally the Soxhlet extracted XAD-7 resin used in the laboratory prepared tubes, unlike the commercial XAD-7 resin, is free of a contaminant which will interfere with the HPLC analysis of the lower-molecular-weight chlorinated phenols.

In the course of this evaluation it was observed that low recoveries of PCP were obtained for samples which were spiked with a stock solution of PCP directly onto the glass fiber filter disc mounted ahead of the resin bed. No losses were observed, however, if the PCP was spiked into the resin bed of the sampling device. (Section 1.2.8.) The losses from the filter can most likely be attributed to volatilization since no adsorption on glass fiber filters has been observed. Since no differences in recovery have been observed for this sampling method versus other collection methods in sampling an aerosol generated atmosphere of PCP, the loss of PCP from the aerosol generated samples must be much less than for the glass fiber filter spiked samples. In actual work environments it is anticipated that the collection of PCP on the sampling device will more closely resemble the pattern observed for the laboratory generated aerosol samples. The analysis of individual components of the sampling tube of aerosol generated samples indicates that approximately two-thirds of the sample is collected on the resin bed while approximately one-third is found on the filter. Thus, only a portion of the sample is retained on the filter for possible loss. If this is the case, only minimal losses of PCP from the glass fiber filter disc can be expected. In order to ensure that no sample loss is experienced, an XAD-7 packed glass tube will be used to cap the sampling device and capture any PCP which might escape following sampling.

A number of reverse and normal phase high performance liquid chromatographic methods have been published for the analysis of PCP in a variety of matrices (Refs. 5.1 and 5.4 - 5.9.). Gas chromatographic analysis of PCP both by derivatization and by direct analysis is also reported (Refs. 5.8. - 5.9.). The GC methods, although generally considered to be more difficult to perform than HPLC, are more sensitive, and can be used to complement HPLC. Thin layer chromatography and colorimetry have also been used to analyze PCP (Ref. 5.6.)

In this method, PCP is analyzed by reverse-phase HPLC

using a UV detector set at 220 nm. A cyano-alkyl bonded column is used with a water-acetonitrile mobile phase containing phosphoric acid to suppress the ionization of the acidic analyte. A Zorbax ODS column exhibited a higher capacity than the CN column for the chlorinated phenols, however, it offered little or no improvement in selectivity. Using the recommended conditions, PCP can be separated from all of the other chlorinated phenols in approximately 8 min.

As with many industrial chemicals which can be absorbed through the skin, the determination of PCP or NaPCP air concentration is not a total measure of occupational exposure. For example, significant exposure to NaPCP can occur through skin contact with lumber which has been sprayed with a dilute aqueous solution of NaPCP to prevent wood darkening. Elevated urinary PCP levels among lumber workers who handle NaPCP-treated lumber have been observed in the absence of significant air exposures. Urinary PCP analysis may represent a reliable means of measuring total exposure to PCP or NaPCP. In a preliminary study conducted at this laboratory, PCP recoveries of over 90% were obtained for urine spiked at the 1 ppm level. Following acidification, the urine was extracted using commercial octadecasilane extraction columns, and analyzed by HPLC/UV. Although UV detection is less sensitive than electrochemical or electron capture detection, this method can provide a means for rapidly screening for significant exposures to PCP or NaPCP.

1 .2. Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.)

The toxic properties of PCP and NaPCP have been well studied due to its toxic nature and wide industrial and commercial use. Dusts in excess of 1 mg/m^3 produce eye, nose, and throat irritation among the unacclimated, although acclimation has been reported to occur at air concentrations as high as 2.4 mg/m^3 (Ref. 5.10.). Prolonged skin exposure can result in an acne-like dermatitis. In addition to their contact irritant properties, both PCP and NaPCP can be absorbed through the skin to produce a systemic effect. The toxic action of PCP is believed to be the result of its ability to uncouple oxidative phosphorylation enzyme systems. (Ref. 5.11.) The resultant failure to generate sufficient stores of energy by normal metabolic pathways is manifested by elevated body temperatures (hyperpyrexia), and an increase in respiration and heart rate. Intoxication is characterized by weakness, loss of appetite (anorexia), weight loss, and profuse sweating (Ref. 5.10.). Headaches, dizziness, nausea, vomiting, shortness of breath (dyspnea), and chest pain may also be experienced. The risk of a toxic effect due to PCP exposure is more pronounced at high ambient

temperatures or among persons with impaired liver or kidney function. The world literature reports 31 deaths among 50 reported poisonings resulting from PCP use (Ref. 5.12.). Included in this total is a death resulting from a 14 h exposure in a spraying operation. Fatalities from the skin absorption of PCP due to the uncontrolled use of a 1.5-3% PCP/NaPCP solution in fuel oil have also been reported. Acute gastric LD_{50}s for mice and rats of 130 and 184 mg/kg respectively have been reported for PCP. A dermal LD_{50} of 96 mg/kg in rats has been reported (Ref. 5.12.).

Concern has been expressed about the presence of highly toxic dibenzo-p-dioxins and dibenzofurans which are contaminants in PCP formed from the condensation of chlorophenols in the production process (Refs. 5.13 and 5.14.). Although less than 0.05 ppm of the highly toxic 2,3,7,8-tetrachlorodibenzo-para-dioxin is reported in one analysis of PCP, parts-per-million levels of the toxic hexachlorodibenzo-para-dioxin, and parts-per-thousand levels of the less toxic octachlorodibenzo-para-dioxin were reported in the same sample (Ref. 5.14.).

Based on available information, the International Agency for Research on Cancer (IARC) does not make a judgment on the carcinogenicity of PCP (Ref. 5.14.). In one animal study an excess of hepatomas was observed in one of two strains of male mice upon subcutaneous injection of a single dose of PCP. Two other separate studies using mice and rats were negative. No human data was reported to be available for evaluation by IARC.

.1.3. Potential workplace exposure.

In 1974 there were 23.4 million kg of PCP used in the United States (Ref. 5.14.). Over 80% of this total was used in the wood industry. Pentachlorophenol and its sodium salt are primarily used as antimicrobial and antifungal agents. PCP in a fuel oil mixture is used to treat and preserve posts, logs, and lumber in the lumber industry. PCP is also used in the construction industry to prevent mold formation on tiles, building surfaces and concrete blocks; in the leather industry to protect upper leather in shoes; and in the paint industry for the protection of protein-based latex paints (Ref. 5.15.).

The sodium salt of pentachlorophenol is used as an antimicrobial agent in starch or protein-based adhesives, and in the protection of leather and paints. The sodium salt is also used in the paper industry to protect stored pulp and fiberboard from mildew and rot; in the oil industry to prevent the growth of bacteria in drilling muds; in the textile industry to prevent mold formation on finished yarns and cloth in storage; and in water treatment to

prevent the growth of algae, fungi and bacteria. The pentachlorophenol salt is primarily used in aqueous solutions. A dilute aqueous solution of the sodium salt is used to spray freshly milled lumber to prevent darkening of the wood by fungi.

.1.4. Physical properties (Ref. 5.16. unless otherwise indicated)

	Pentachlorophenol	Sodium salt
CAS no.:	87-86-5	--
molecular weight:	266.35	288.34
melting point:	191°C (anh)	--
	184°C ($1 \cdot H_2O$)	--
boiling point:	293.08°C	--
specific gravity:	1.978 at 22°C	--
solubility:	water - 35 ppm at 50°C. soluble in ether, alcohol, and benzene	water - 15% at 4°C, soluble in alcohol & acetone
vapor pressure at 15°C:	0.00019 mm Hg	--
dissociation constant at 25°C, (KA):	1.2×10^{-5} (Ref. 5.15.)	--
synonyms and trade names:		
pentachlorophenol:	Chem-Tol, Permacide, Penta, Santophen 20, Dowicide 7, Eurisan, Cuprinol, Penchloral, PCP, Pentachlorofenol	
sodium salt:	Santobrite, Dowicide G	
physical appear.:	PCP and NaPCP are white or tan solids with a distinct phenolic odor.	

1.2. imit defining parameters (The analyte air concentrations listed hrough this method are based on an air volume of 48 L and a solent desorption volume of 2 mL)

.2.1. Detection limit of the analytical procedure

The detection limit of the analytical procedure is 4.1 ng per injection. This is the amount of the analyte which

2.3. Reliable quantitation limit

The reliable quantitation limit is 0.33 µg per sample (0.007 mg/m³) for pentachlorophenol. This is the smallest amount of analyte which can be quantitated within the requirements of a recovery of at least 75% and a precision (1.96 SD) of ±25% or better. (Section 4.2.)

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e reliable quantitation limit and detection limits reported in e method are based upon optimization of the instrument for the allest possible amount of analyte. When the target concentration of an analyte is exceptionally higher than these limits, they y not be attainable at the routine operating parameters.

2.4. Sensitivity

The sensitivity of the analytical procedure over a concentration range representing 0.5 to 2 times the target concentration based on the recommended air volume is 23,184 area units per µg/mL for pentachlorophenol. The sensitivity is determined from the slope of the calibration curve. The sensitivity may vary with different instruments or instrumental conditions. (Section 4.4.)

2.5. Recovery

The recovery of pentachlorophenol from samples used in a 19-day storage remained above 99% when the samples were stored at ambient conditions in the dark. (Section 4.8.) The recovery of the analyte from the collection medium during storage must be 75% or greater.

2.6. Precision (Analytical procedure only)

The pooled coefficient of variation obtained from replicate determinations of analytical standards at 0.5, 1 and 2 times the target concentration is 0.010 (Section 4.3.).

2.7. Precision (overall procedure)

The precision at the 95% confidence level for the 19 day storage test is ±13.5% for pentachlorophenol. (Section 4.8.) The overall procedure must provide results at the target concentration that are ±25% or better at the 95% confidence level.

2.8. Reproducibility

Three spiked samples were prepared by injection of a stock standard of PCP in methanol into the resin bed of the sample tube and were given along with a draft copy of this

method to a chemist unassociated with this evaluation for analysis. The samples were analyzed after 22 days storage at room temperature. The average recovery for the three samples loaded with 12.1, 24.3 and 40.5 µg of PCP respectively was 101%. The recovery ranged from 98 to 105%.

Three other samples were also prepared at the same time by spiking PCP directly onto the glass fiber filter disc which is placed ahead of the resin bed and these samples were analyzed in conjunction with the three resin-bed spiked samples. The average recovery for these three samples with the same loadings as the resin spiked samples was 80%. The range was from 67 to 89%. Similar results were also obtained by the chemist who developed this method upon analysis of an identical set of six samples which were prepared at the time of the above samples and analyzed at approximately the same time. The low recoveries obtained from samples spiked onto the glass fiber filter most probably can be attributed to volatilization losses since no adsorption of PCP onto glass fiber filters has been observed. The excellent recoveries obtained upon analysis by an independent chemist of samples spiked into the resin bed, although based on limited data, indicate good reproducibility. (Section 4.8.)

1.3. dvantages

3.1. The two solid sorbent sampling tubes in series represent a more convenient method than the previous methods using bubbler solutions.

3.2. The analysis for pentachlorophenol is rapid, sensitive, and precise.

1.4. sadvantages

4.1. The method has not been field tested.

4.2. The sampling tubes are not commercially available.

2. Sampli Procedure

2.1. pparatus

1.1. A constant flow personal sampling pump is used which can be calibrated to within ±5% of the recommended 0.2 L/min flow rate while the sampling train is in line.

1.2. The sampling tubes consist of two 50-mm long by 8-mm o.d., 6-mm i.d. glass tubes which are packed with approximately 175 mg (15-mm bed length) of XAD-7 resin held in place with two silanized glass wool plugs. The tubes are butted together using a connector made from a 9/32-in. diameter plastic cap from which the closed end has been removed.

The first sampling tube in the series also contains an 8-mm glass fiber filter disc as a precautionary measure to trap any aerosols of the analyte. A number 4 cork borer is used to cut out the discs from Gelman (Ann Arbor, Michigan, USA) Type A 35-mm glass fiber filters. The over-sized glass fiber discs are fitted snugly into the sample tube using a stirring rod to tamp the filter in place over the glass wool plugs. Placed ahead of the front section sampling tube both before and after sampling is a third glass tube packed with XAD-7 resin which is identical to the backup section tube. This extra tube serves as a cap to trap any PCP which may volatilize off the filter following sampling. Commercially purchased XAD-7 20/50 mesh (Rohm and Haas, Philadelphia, PA USA) is prepared for use by a 24-h Soxhlet extraction with HPLC grade methanol followed by evaporation and resieving to remove fines.

2.2. eagents

one required

2.3. echnique

3.1. Properly label all three sections of the sampling device prior to sampling.

3.2. Before sampling, remove and save the front glass tube section containing XAD-7 resin which will serve as a cap following completion of sampling.

3.3. Attach the sampling tubes to the pump using a section of flexible, plastic tubing so that the adsorbent tube containing the glass fiber filter serves as the front sampling section. Do not place any tubing ahead of the sampling device. Attach the sampling device in the workers breathing zone in such a manner that it does not impede work performance.

.3.4. After sampling for the appropriate time, remove the sampling device from the pump, cap the front end of the device with the resin-filled glass tube and cap the back end of the device with a plastic cap. Insure that the caps are well fitted and label the sampling tubes with OSHA seals (Form 21).

.3.5. Include at least one blank for each sampling set. The blank should be handled in the same manner as the samples with the exception that air is not drawn through it.

.3.6. Any bulk samples submitted for analysis must be shipped in separate containers to avoid contamination of the air samples.

.3.7. List any potential interferences on the sample data sheet.

2.4. reakthrough

ince XAD-7 resin has a very high capacity for pentachlorophenol, he determination of the amount of analyte which can be collected rom an atmosphere before breakthrough occurs was found to be xperimentally difficult to determine and of little practical alue. Two studies were performed, however, to demonstrate the igh capacity of the resin for pentachlorophenol.

n the first study, an XAD-7 sampling tube without a glass fiber ilter insert was used to sample an independently measured 0.82 g/m³ PCP aerosol of 2.2 µm in diameter which was generated by an erosol generation system. No breakthrough to the backup sampling ube was observed after 275 L of dry air was sampled at 1 L/min or 4.6 h. A more complete description of the experiment and the erosol generation system is discussed in Section 4.5.

n the second study the ability of a XAD-7 sampling tube to collect PCP vapors was investigated under high humidity conditions. o breakthrough was observed after sampling 378 L of a 10 mg/m³ tmosphere of PCP at 1 L/min with an XAD-7 sampling tube containng a glass fiber filter. Details of this study are discussed in ection 4.5.

2.5. esorption efficiency (solvent adsorption effect)

he values obtained for desorption efficiency of PCP over the ange of 0.5 to 2 times the PEL averaged 118% for XAD-7 sampling ubes. This high value is probably the result of a change in the oncentration of PCP in solution due to the preferential adsorption of methanol onto XAD-7 resin. This effect is proportional to he amount of XAD-7 used in the sampling tube and can best be llustrated by the linear graph obtained from plotting the observed PCP concentration versus the amount of XAD-7 added to 2 mL f methanol containing a known amount of PCP. (Figure 4.6.) The igh correlation coefficient of 0.956 observed for the line trongly suggests that the observed response is a function of the mount of XAD-7 used. Since this effect is apparently not concentration dependent, a reliable correction for sample results can e made if a uniform weight of XAD-7 resin is used in all sampling ubes. (Section 4.6.)

2.6. ecommended air volume and sampling rate

48-L air sample obtained by sampling at 0.2 L/min for 4 h is ecommended for pentachlorophenol. If necessary, the sensitivty of the analytical method will permit a sampling period as hort as 15 min at 0.2 L/min for determination of the analyte at he target concentration.

2.7. Interferences

There are no known interferences to the sampling procedure.

2.8. Safety precautions

2.8.1. Attach the sampling equipment to the worker in such a manner that it will not interfere with work performance or safety.

2.8.2. Follow all safety practices that apply to the work area being sampled.

3. Analytical Procedure

3.1. Apparatus

3.1.1. A high performance liquid chromatograph equipped with sample injector, a cyano bonded phase HPLC column, a variable wavelength UV detector, and a chart recorder are needed for the analysis. A Waters 6000A pump, a Waters WISP 710 auto sampler, a Perkin-Elmer LC-55 UV-Visible detector and a Zorbax 25-cm × 4.6-mm i.d. CN bonded phase column were used in this study.

3.1.2. An electronic integrator or other suitable means of measuring detector response is required. The Hewlett-Packard 3354 data system was used in this study.

3.1.3. Various sizes of volumetric glassware and pipettes are needed for sample and standard preparations.

3.1.4. Three-milliliter (or larger) screw-cap or crimp-type vials are needed for desorbing the XAD-7 sampling adsorbent. Four-milliliter Waters WISP vials were used in this study.

3.1.5. Small brown glass bottles fitted with inert cap liners are needed to store standard solutions.

3.1.6. A repetitive dispenser capable of accurately delivering the desorption solution is needed.

3.2. Reagents

3.2.1. HPLC grade methanol and acetonitrile.

3.2.2. Reagent grade phosphoric acid.

3.2.3. HPLC grade water. Water obtained from a Milli-Q reagent grade water system (Millipore, Inc., Bedford, Mass.) was used in this study.

3.2.4. A reagent grade standard of pentachlorophenol is required. Pentachlorophenol, Chem Service, Lot #PS-7, West Chester,

PA was used in this study.

3.3. andard preparation

3.1. Prepare a stock solution of pentachlorophenol by accurately weighing approximately 200 mg of the standard and transferring it to a 25-mL volumetric flask. Dilute to volume with methanol. Dilute this stock solution 1/25 to yield an approximate 320 µg/mL solution in methanol. Prepare 1/50, 1/25 and 2/25 dilutions of the 320 µg/mL PCP solution to yield standards which correspond approximately to 0.5, 1, and 2 times the PEL for the recommended sampling conditions.

3.2. As an alternative method of preparing standards, spike appropriate quantities of the stock PCP standard into vials containing 2 mL of methanol and the same approximate weight of XAD-7 resin as was used in the sampling tube (175 mg). These spiked samples can be used as standards, or they can be used to determine desorption efficiencies with standard solutions as outlined above. No correction for the solvent adsorption effect will need to be made if the standards are prepared by spiking PCP into solutions containing 175 mg of XAD-7 resin.

3.4. mple preparation

epare samples for analysis by transferring the entire contents the sampling tube including both glass wool plugs, the resin, d the glass fiber disc into a 4-mL vial. Considerable care must exercised in transferring the samples to the vials to avoid mple loss from static build-up on the XAD-7 beads. The transfer n best be accomplished if the glass fiber disc is first transrred to the sample vial and the front glass wool plug partially moved. Then with the sampling tube inverted into the vial, use small spatula or glass stirring rod to force the entire contents the tube including both glass wool plugs into the vial. Rinse e inside of the sampling tube into the sampling vial with two mL portions of methanol using a 1-mL repetitive dispenser.

3.5. alysis

5.1. Prepare a high performance liquid chromatograph for sample analysis using the HPLC conditions listed below.

column:	Zorbax 25-cm × 4.6-mm i.d. CN bonded phase
mobile phase:	40/60 (v/v) acetonitrile/water containing approximately 0.1% by volume of phosphoric acid.
flow rate:	1.3 mL/min
UV detector:	220 nm
injection volume:	25 µL
retention time:	8.1 min

5.2. Analyze the front and back sampling tubes and the sample cap tube separately. Verify that the sample responses lie within the range of the responses observed for the standards. (See chromatogram, Figure 4.9.)

5.3. Since column-to-column variations do occur, it is important to ensure that PCP is being separated from the other chlorinated phenols. The injection of a tetra/pentachlorophenol standard mixture will produce baseline separation between pentachlorophenol and the tetrachlorophenol isomers if the analytical conditions are properly selected. Under these conditions the other chlorinated phenols are not interferences.

3.6. Interferences

6.1. Any compound which has the same retention time as pentachlorophenol is a potential interference. Under the recommended analytical conditions none of the mono-, di-, tri-, or tetrachlorophenols interfered with the analysis. (See chromatogram, Figure 4.10.)

6.2. Comparison of peak height ratios of analyte response at two wavelengths for both samples and standards is a valuable confirmatory technique in HPLC. (See UV scan, Figure 4.11.) Normal phase HPLC methods and gas chromatographic methods may also be used for sample confirmation. GC/mass spectrometry is an additional confirmatory technique which may be used.

3.7. Calculations

7.1. Prepare a standard calibration curve of area response versus concentration for PCP by determining the least squares fit equation for the curve. Calculate the analyte concentration in the samples by entering their response values into the equation and solving for the sample concentration. A laboratory data system, or one of many small hand-held calculators, can be used to perform these calculations.

7.2. Combine the amount of analyte found on all sections including the sample tube cap. Express results in mg/m^3 using the following equation:

$$mg/m^3 = \frac{(\mu g/mL \text{ analyte})\ (2\text{-mL desorption vol.})\ (1\ mg)}{(\text{air volume in } m^3)\ (1000\ \mu g)\ (\text{solvent adsorption effect})}$$

Note: No solvent adsorption effect correction will need to be applied if standards have been prepared in solutions containing XAD-7 resin.

To convert to ppm at 760 mm and 25°C:

$$ppm = (mg/m^3)(24.46)/(MW \text{ of analyte})$$

24.46 = the molar volume at 760 mm and 25°C.
MW = 266.35 for pentachlorophenol

3.8. Safety precautions

3.8.1. Minimize exposure to pentachlorophenol by performing standard preparations in a well ventilated hood.

3.8.2. Avoid all skin contact with pentachlorophenol.

3.8.3. Restrict the use of solvents to hoods which provide adequate ventilation.

3.8.4. Wear safety glasses in laboratory areas at all times.

4. Backup data

4.1. Detection limit for analytical procedure

The detection limit for the analytical procedure is 4.1 ng for PCP. This is based on a 25-µL injection of a 0.163-ng/µL standard, and represents approximately five times the baseline noise. (Figure 4.1.)

4.2. Detection limit of the overall procedure and reliable quantitation limit

The detection limit of both the overall procedure and the reliable quantitation limit is 0.33 µg per sample (0.007 mg/m³) for PCP.

Six XAD-7 sampling tubes were spiked with 8 µL of 40.4 µg/mL PCP in methanol, then capped and stored overnight in a laboratory drawer. Assuming complete recovery, this amount of analyte is equivalent to the detection limit of the analytical procedure. The following day the samples were desorbed in 2 mL of methanol and analyzed. The percent recoveries (corrected for the solvent desorption effect) are reported below.

Table 4.2.
Detection Limit Data

% recovery	statistics
104	
104	$\bar{X}$ = 104
104	SD = 3.8
98	1.96 SD = 7.4
110	
104	

4.3. ecision

e pooled coefficient of variation over a range of 0.5 to 2 times e target concentration for pentachlorophenol was obtained from ltiple 25-µL injections of three standard solutions. The results are listed in Table 4.3.

Table 4.3.
Sensitivity and Precision Data

× target conc. µg/mL	0.5× 5.99	1× 12.0	2× 24.0
area counts	137934	283403	560373
	138276	287608	561265
	137543	278956	554505
	136555	278511	548815
	137254	276153	550071
$\bar{X}$	137470	280856	555574
SD	600	4085	5122
CV	0.0044	0.015	0.0092

$\overline{CV}$ = 0.010

4.4. nsitivity

e slope of the calibration curve over the range of 0.5 to 2 mes the target concentration for the analytes represents the nsitivity of the method. The sensitivity determined in this nner is 23,184 area units per µg/mL for PCP (Figure 4.4.).

4.5. eakthrough

eakthrough studies for PCP were performed using both aerosol neration and vapor generation systems. The aerosol generation stem used in this study consists of a syringe drive pump which s used to meter a 0.313 mg/mL standard of PCP in isopropanol to a model 3050 Bergland-Liu Vibrating Orifice Monodisperse rosol Generator (Thermo-System Inc., St. Paul, Minn.) at 0.16 /min. The frequency of the orifice was set at 7.5 KHz with a ecision Dynascan model 3010 function generator (Chicago, IL.) to oduce a 2.2 µm monodisperse aerosol of PCP. The aerosol was en diluted with 2 m^3/h. of dry laboratory air as it passed vertically up through the electrostatic neutralizer. After exiting e neutralizer at a right angle, the aerosol then passed through proximately 3 ft of 1.5-in. flexible plastic tubing before entering the sample chamber. The sampling chamber consists of a -in. by 20-in. cylinder of clear acrylic plastic in which a diffuser plate is mounted near the top. Samples were collected via y of seven sampling ports mounted in the bottom of the chamber.

Flow rates were controlled by means of critical orifices attached to a vacuum pump.

Attached to one of the sampling ports in the chamber was a section of tubing used to sample the total mass of the aerosol in the chamber with a Thermo System Model 3203 Particle Mass Monitor. An air flow of approximately 20 L/min was maintained in the sampling chamber by means of a calibrated pressure-vacuum pump connected via 3/4-in. PVC tubing to three outlets in the bottom of the sampling chamber. Excess air flow from the aerosol generator and all sampled air was first drawn through a HEPA filter before it was vented to the atmosphere.

Ideally this aerosol generation system, when used in conjunction with the particle mass monitor to measure the actual concentration of the aerosol generated, should permit the evaluation of a test method independent of other sampling methods. In practice, however, some discrepancies were obtained between the amount of PCP present in the generated atmosphere as measured by the analysis of various sampling tubes or bubblers and with the levels measured by the particle mass monitor.

Several preliminary evaluations of mass monitor results versus the results of various sampling methods for PCP were in good agreement. Subsequent studies, however, indicated that although the total PCP concentration had increased in the system, the particle mass monitor was underestimating the total PCP concentration. This difference in results could possibly be attributed to an increase in the amount of vapor present in the generated atmosphere which would not be detected by the particle mass monitor.

Although the particle mass monitor could not be used as an independent measure of the total PCP present in the system, the good agreement obtained using various sampling methods to measure the total PCP concentration establishes confidence that the actual PCP concentration is known with some degree of certainty.

The average concentration of the atmosphere generated in the chamber was determined to be 0.82 mg/m^3 for the breakthrough study. This was determined by analysis of a series of XAD-7 tubes and one silica gel packed sampling tube which sampled adjacent to the XAD-7 tube used for the breakthrough study. This concentration was considerably less than the calculated concentration of 1.5 mg/m^3. The observed deposition of the aerosol in the neutralizer may have accounted for the difference.

In this study, no breakthrough was observed for XAD-7 after sampling 275 L of the dry laboratory air at 1 L/min. It might also

nown to be much less effective than XAD-7 in collecting phenol nd cresol, it is anticipated that silica gel will lack the ver- atility offered by XAD-7.

second attempt at evaluating breakthrough for PCP was performed sing a crude vapor generation system. This system consisted of a ringe drive pump which delivered 0.01 mL/min of 4 mg/mL PCP in thanol into one end of a glass tee which was wrapped with heat pe and packed with silanized glass wool. A rheostat set at 65% full scale was used to heat the sampling tee. Air, with a lative humidity of 80% at 23°C, was drawn through the tee at 1 min and onto an XAD-7 sampling tube mounted at the other end of e tee. No breakthrough was observed during a 6.3 h sampling riod. The total amount of PCP collected on the sampling media s 3912 µg. This corresponds to an average concentration of 10 g/m^3 over the entire sampling period. Of this total only 105 µg s found on the glass fiber disc which preceded the resin bed. e actual amount of PCP deposited on the sample tube is consider- ly less than expected. However, considerable condensation of P ahead of the sampling media was observed.

is vapor study performed under humid conditions, and the aerosol udy performed with dry laboratory air indicate the recommended mpling device is very effective for sampling PCP vapors or aero- ls.

4.6. esorption efficiency (solvent adsorption effect)

e average desorption efficiency from spiked samples over the nge of 0.5 to 2 times the target concentration is 118%. This gh desorption efficiency is probably due to a solvent adsorption fect as discussed in Section 2.5. Six XAD-7 sampling tubes were ch spiked with either 1.5, 3, or 6 µL of a 7492 µg/mL PCP stan- rd in methanol. All of the 18 samples were prepared by spiking P standard directly on the glass fiber disc contained within the D-7 sampling tube. The samples were then capped and stored over ght under ambient conditions and analyzed the following day. The sults are presented in Table 4.6. (Similar high desorption fficiencies are obtained for samples prepared by spiking PCP tandard directly onto the XAD-7 resin.)

Table 4.6.
Desorption Efficiency

x target conc.	0.5x	1x	2x
desorption	121	118	117
efficiency,	121	120	119
%	126	123	117
	121	122	113
	121	114	116
	113	119	109
$\bar{X}$	120	119	115

$\bar{\bar{X}}$ = 118

4.7. orage test

stability problems were observed for PCP upon storage at either bient or refrigerated conditions. Storage samples were gen- ated using approximately the same aerosol concentration as was ed in the breakthrough study. Mass monitor determinations of e total aerosol concentration generated for storage samples were wer than the results observed from the analysis of sampling bes. This may be due to the presence of a volatile fraction of P in the atmosphere which was not detected by the particle mass nitor. Since there exists no reliable means of independently termining the total PCP concentration generated in this study, e percent recovery of each sample was determined relative to the erall average PCP concentration. The overall average PCP concen- ation for the ambient and the refrigerated stored samples were 77 and 0.79 mg/m³ respectively. All of the storage samples were nerated in groups of six by sampling the aerosol atmosphere for proximately 48 min at 1 L/min. Seven sets of samples were gen- ated over a one-day period. One sample of each set was analyzed mediately to ensure that no major fluctuations in the PCP con- ntration had occurred during the generation process. Of the maining 35 samples, two were randomly discarded and three were alyzed immediately for the zero day storage. The remaining 30 mples were then randomly split into equal ambient and refriger- ed groups for storage. All of the stored samples were capped d stored in the dark either at ambient conditions on a labora-

Table 4.7.
Storage Tests

storage time (days)	% recovery (refrigerated)			% recovery (ambient)		
0	94.1	97.1	97.2	96.9	100	100
5	99.7	95.2	106	99.0	101	104
8	99.6	107	102	94.3	102	104
12	93.5	107	107	92.1	98.5	104
15	101	101	lost	94.2	111	102
19	97.1	101	101	96	95	102

4.8. eproducibility

AD-7 packed glass tubes containing a glass fiber filter disc were piked either in the resin bed or on the surface of the glass iber filter with either 1.5, 3.0, or 5.0 µL of a 8094 µg/mL stan- ard of PCP in methanol. The samples were capped with plastic aps and then stored in the dark at room temperature until sub- itted for analysis. The samples were analyzed after 22 days by a hemist unassociated with the method. The recoveries are shown in ables 4.8.1. and 4.8.2.

Table 4.8.1.
Resin Spiked Samples

µg spiked	% recovery
12.1	98
24.3	105
40.5	100
	$\bar{X}$ = 101

Table 4.8.2.
Filter Spiked Samples

µg spiked	% recovery
12.1	89
24.3	85
40.5	65
	$\bar{X}$ = 80

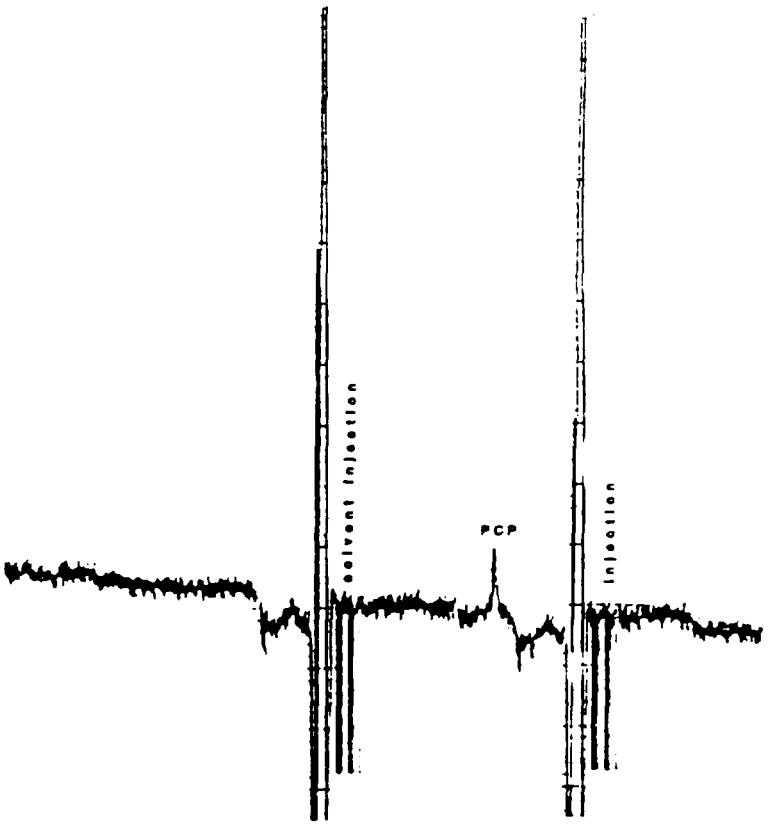


Figure 4.1 Detection limit for PCP.

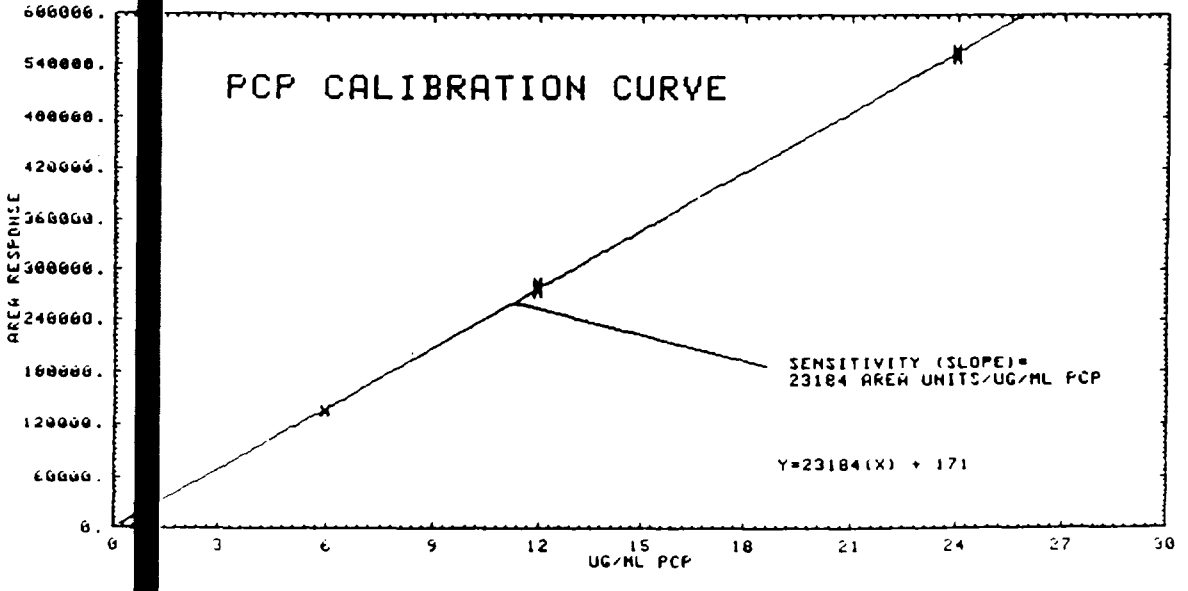


Figure 4.4 Calibration curve for PCP.

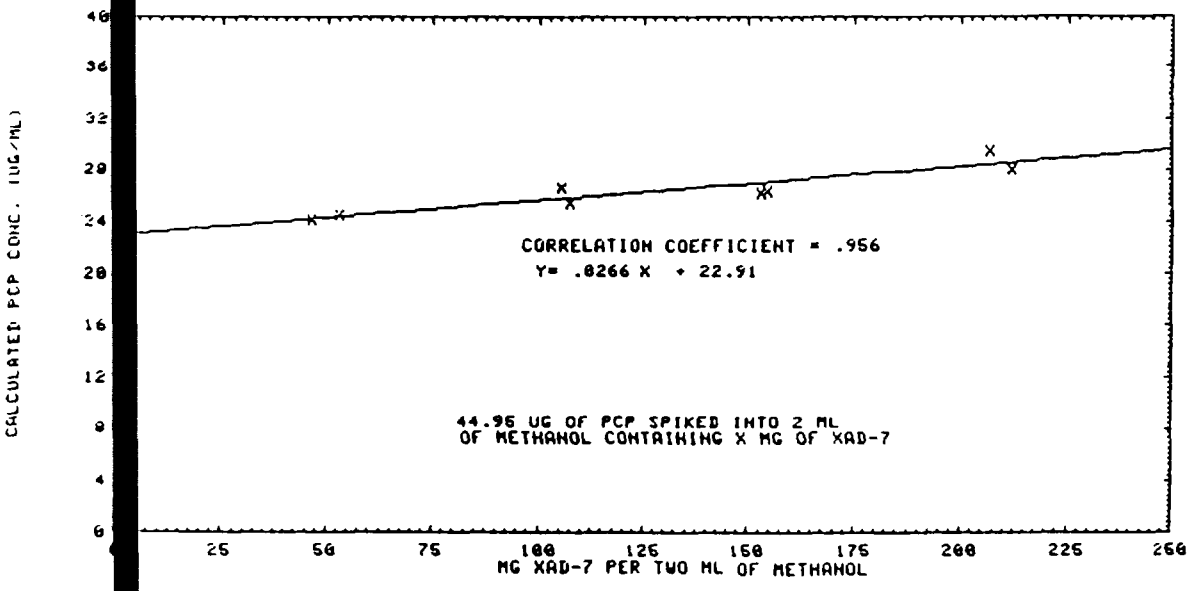


Figure 4 . Solvent adsorption effect of XAD-7.

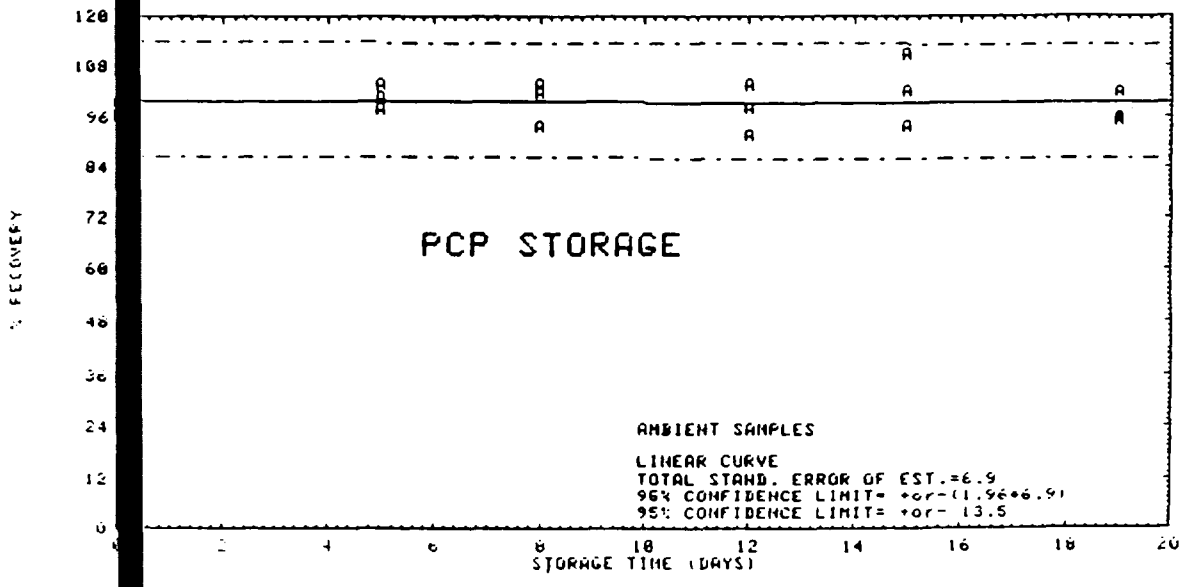


Figure 4 .1. Ambient storage for PCP.

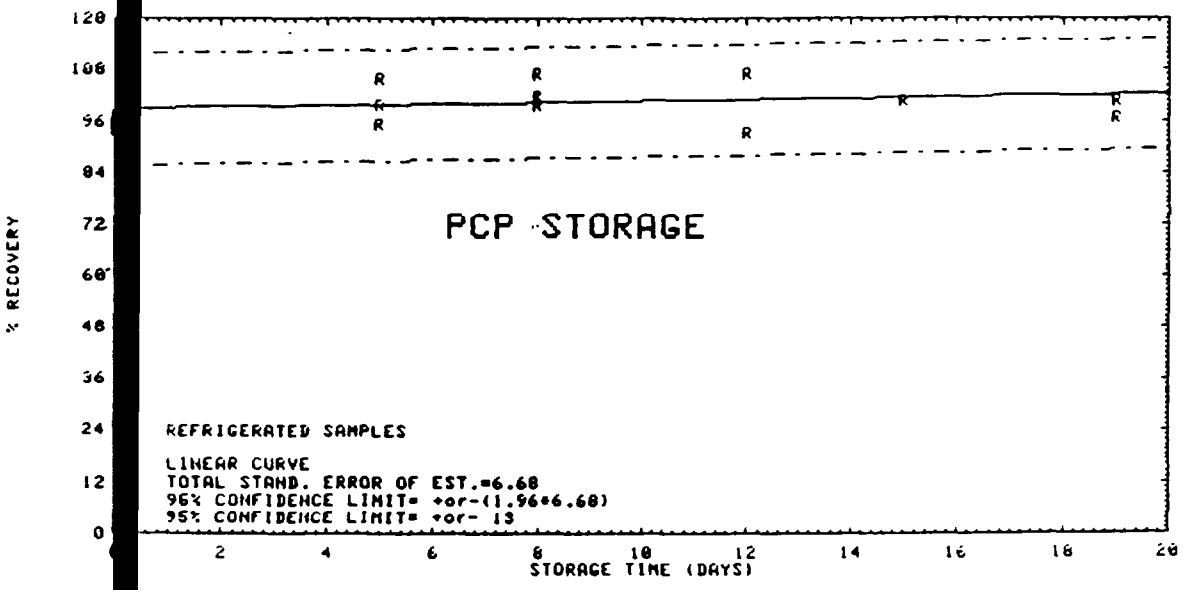


Figure 4.2. Refrigerated storage for PCP.

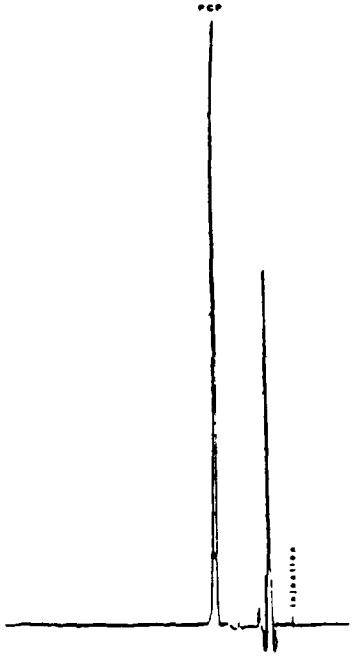


Figure 4.9. Chromatogram of pentachlorophenol.

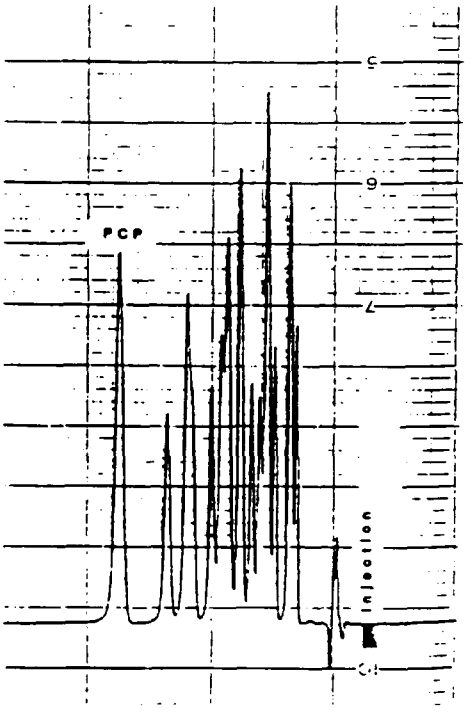


Figure 4.10. HPLC chromatogram of a mixture of mono-, di-, tri-, tetra-, and penta-chlorophenols.

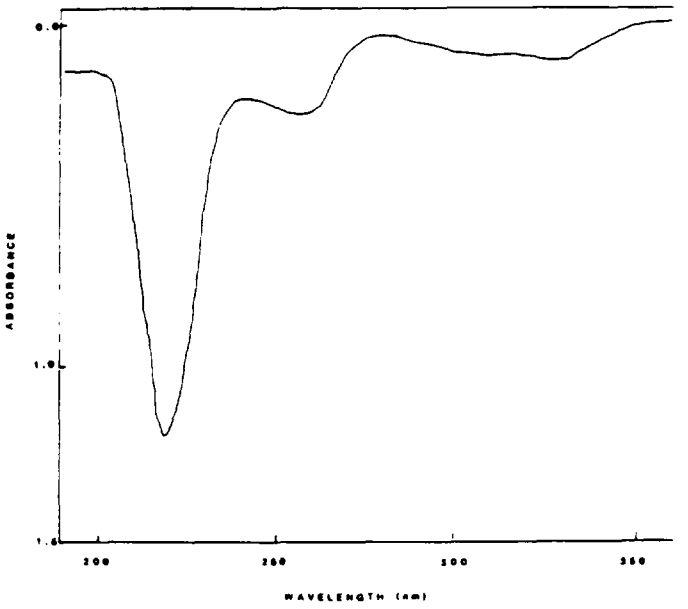


Figure 4.11. UV scan of PCP in methanol.

5. References

5.1. "NIOSH Manual of Analytical Methods", 2nd ed.; USDHEW/PHS/CDC/NIOSH, USDHEW (NIOSH) Publication No. 78 - 175, Vol. 4, Method S297, 1978.

5.2. Cummins, Kevin, "Phenol and Cresols," Method No. 36, Organic Methods Evaluation, OSHA Laboratory, Salt Lake City, Utah 1982 (unpublished).

5.3. Beaulieu, Harry J.; Eidino, A.V.; Arlington, Kim L.B.; Bachan, Roy M. American Ind. Hygiene Assoc. J. (1980), 41, (10), 758-65.

5.4. Ivanov, Zlata; Magee, R.J. Microchemical Journal (1980), 25, 543-47.

5.5. Ugland, Karin; Ludanes, Elsa; Greibrokk, Tyge J. Chromatogr. (1981), 213, 83-90.

5.6. Ervin, H.E.; McGinnis, G.D. J. Chromatogr. (1980), 190, 203-207.

5.7. Daniels, C.R.; Swan, E.P. J. Chrom. Sci. (1979), 17, 628-30.

5.8. Mundy, D.E.; Machin, A.F. J. Chromatogr. (1981), 216, 229-38.

5.9. Edgerton, Thomas R.; Moseman, Robert F. J. Chrom. Sci. (1980), 18, 25-29.

5.10. Mackinson, Frank W.; Stricoff, R. Scott; Partridge, Lawrence J., editors, "Occupational Health Guideline for Pentachlorophenol", USDHHS/PHS/CDC/NIOSH, (NIOSH) Publication No. 81-123, Vol. II, 1981.

5.11. Weinbach, E.C. Proc. Nat. Acad. Sci. (1956), 43, (6), 393-97.

5.12. "Documentation of the Threshold Limit Values", 4th ed., American Conference of Government Industrial Hygienists, Cincinnati, OH., (1980), p. 323.

5.13. Choudhary, Gangadhar, "Chemical Hazards in the Workplace" ACS Symposium Series 149, American Chemical Society, Washington, D.C., (1981), 319-342.

5.14. International Agency for Research on Cancer, "IARC Monographs on the Evaluation of the Carcinogenic Risk of Chemicals to Humans", IARC, Lyon, Switzerland, (1979), Vol. 20, 303-325.

5.15. E.R. Freiter, "Chlorophenols", "Kirk-Othmer Encyclopedia of Chemical Technology", 3rd ed., John Wiley and Sons, N.Y., (1980), Vol 5, 864-872.

5.16. "Pentachlorophenol and Sodium Pentachlorophenate". Hygienic Guide Series, American Industrial Hygiene Association, Vol. II, Akron, Ohio, (1970).

1. General discussion

1.1. Background

1.1.1. History

The recommended air sampling procedure listed in the OSHA Field Operations Manual for methylamine is collection in midget impingers containing sulfuric acid (Ref. 5.1.). The analysis is done by gas chromatography. Impingers are cumbersome to use in the field and the analysis of free low molecular weight amines is difficult by gas chromatography (Ref. 5.2.). Thus better sampling and analytical procedures were needed for methylamine.

In NIOSH methods 221, 277, and S148 (Refs. 5.3. - 5.5.) silica gel is recommended for collection of methylamine in air. It was later found by NIOSH that low molecular weight amines are not stable after being collected on silica gel (Ref. 5.6.).

In this evaluation, it was found that methylamine could be collected on a sampling tube containing XAD-7 resin coated with 10% NBD chloride (7-chloro-4-nitrobenzo-2-oxa-1,3-diazole) by weight. A stable derivative is formed on the coated resin. The derivative is extracted with 5% (w/v) NBD chloride in tetrahydrofuran (THF) and analyzed by high-performance liquid chromatography. Similar procedures have been successfully evaluated for dimethylamine (OSHA Method 34) (Ref. 5.7.) and ethylamine (OSHA Method 36) (Ref. 5.8.) and may be applicable for other volatile aliphtic amines.

1.1.2. Toxic effects (This section is quoted directly from "Occupational Health Guidelines for Chemical Hazards" (Ref. 5.9.) and is for information only and should not be taken as the basis of OSHA policy.)

> "Methylamine gas is a severe eye and respiratory irritant. The LD_{50} was 0.1 to 0.2 g/kg in rats exposed orally to a 40% aqueous solution of methylamine. One case of bronchitis in a chemical worker has been reported; concentrations measured in the workroom ranged from 2 to 60 ppm; the duration of the exposure was not given. Brief exposures to 20 to 100 ppm are said to produce transient irritation of the eyes, nose, and throat. No symptoms of irritation are produced from longer exposures at less than 10 ppm. One drop of 5% aqueous solution caused conjunctival hemorrhage, superficial corneal opacities, and edema in experimental animals; a 40% solution caused corneal damage in rabbits. A 40% solution caused necrosis when applied to the skin of a rabbit. Dermatitis

and conjunctivitis are occasionally observed in workers after prolonged exposure to the vapor."

1.1.3. Potential workplace exposure

Following are some common operations in which exposure to methylamine may occur as reported in "Occupational Health Guidelines for Chemical Hazards." (Ref. 5.9.)

Methylamine is used:

in production of insecticides, herbicides, fungicides, surfactants, rocket fuels, explosives, pharmaceuticals, photographic chemicals, dyes, textiles, dye assists, rubber and anticorrosive chemicals.

as a polymerization inhibitor of hydrocarbons during distillation.

to prevent coagulation and webbing in natural and synthetic latex.

to prevent polymerization in paint removers.

1.1.4. Physical properties (Ref. 5.9.)

molecular weight:	31.1
boiling point:	-6.32°C (760 mm Hg)
color:	colorless gas
specific gravity:	0.656 (water = 1)
formula:	CH_3NH_2
vapor pressure at 20°C:	not pertinent
flash point:	not applicable (gas)
odor:	ammonia-like
flammable limits in air, % by volume:	lower: 5; upper: 21
autoignition temperature:	430°C
synonyms:	anhydrous methylamine, monomethylamine

1.2 Limit defining parameters (The methylamine air concentrations listed throughout this method are based on an air volume of 10 L and a solvent desorption volume of 2 mL. Air concentrations given in ppm are referenced to 25°C and 760 mm Hg.)

1.2.1. Detection limit of the analytical procedure

The detection limit of the analytical procedure is 1.9 ng per injection. This is the amount of methylamine which will give a peak whose height is approximately five times baseline noise. (Section 4.1.)

QUALITY ASSURANCE/QUALITY CONTROL

MANUAL

ENVIRONMETRICS, INC.

11401 MOOG DRIVE
ST. LOUIS, MO 63146

(314) 432-05 (VOICE)
(800) 333-FA T (VOICE)
(314) 432-49 (FAX)

Revision 991014

Table of Contents

List of Tables and Forms

The following representatives of **Environmetrics, Inc.** Laboratory have read and approved this Quality Assurance Manual.

Eugene Scheide — 10/28/99

Dr. Eugene Scheide
CEO/Laboratory Director — Date

Wayne Cooper — 10/28/99

Wayne Cooper
Director of Technical & Quality Services — Date

Keith Earhart — 10/28/99

Keith Earhart
Laboratory Manager — Date

Sue Ferguson — 10-28-99

Sue Ferguson
Manager — Date

Section 1

QUALITY ASSURANCE OBJECTIVES

Introduction

The overall Q lity Assurance Objective at Environmetrics is to develop and implement procedures fo chain-of-custody, laboratory analysis, and reporting that will provide legally defensible res ts and insure the generation of reliable and valid analytical data. Specific procedures to e used for chain-of-custody, calibration, laboratory analysis, reporting, internal quality contro audits, preventative maintenance, and corrective actions are described in other sections of th document. The purpose of this section is to define QA goals for accuracy, precision, and ompleteness. Establishment of these goals allows the clients of Environmetrics to judge the a equacy of the results being obtained. These items are addressed in the EPA Manual on A lytical Quality Control in Water and Wastewater Laboratories (EPA-600/4- -019, March 1979). This manual and Section 1 of the SW-846 will always be followed in a lysis of water and soil/sediment for organic and inorganic parameters.

The overall g ls and objectives of the Environmetrics laboratories are to provide sufficient information t allow the technicians, chemists, section managers, and the laboratory director to initiate or rei orce programs of analytical QC that emphasize early recognition and correction of factors leadi to breakthrough in the validity of data. Therefore, the purpose of this Quality Assurance M ual is to insure that the objectives related to sample analysis are met.

Quality Assu nce

Quality Assu nce (QA) is defined as procedures that are necessary in order to produce reliable results in sam ling and analysis. In general, this refers to uniform field techniques, good laboratory pr tices and good standard operating procedures that are used to maintain consistency a l uniformity of the above. If a deviation from analysis, sampling, etc. is necessary, go l documentation is needed to assure that there is a valid reason for the deviation from protoco or standard operating procedure. This is also needed to maintain integrity of the data.

Quality Con l

Quality Con l (QC) is defined as the set of operational techniques and activities that are used for obtaining rescribed standards of performance in the monitoring and measurement process.

Accuracy

Accuracy is th closeness of agreement between an observed value and an acceptable reference value. The Q accuracy objectives for quantitative analysis is expressed in terms of recovery of surrogate com ounds or spiked analytes. The equations used to calculate percent recoveries of surrogate com ounds and spiked analytes added to a sample are given in Section 10.

The QA accu y objective for quantitative analysis is generally 80% for the lower limit and 120% for the per limit. Environmetrics realizes that there are some compounds for which this recovery is no possible. In all cases, the laboratory will strive to achieve the stated goal for accuracy.

The specific c mpounds used for spiking must be verified by the use of EPA acceptable reference stan rds. These accuracy objectives shall apply to results obtained for reference standards, spi d samples, and performance evaluation samples.

Precision

The objective r precision where duplicates or replicate analyses have been performed are as follows:

- Analy s of Duplicate or Replicate Samples

  One o he QA objectives is that the results of quantitative analysis for duplicate or replic samples be within the limits specified in methods and protocols described in SW-8 and 40CFR, part 136.

- Analy s of Surrogates or Analyte Spikes

  One o he QA objectives is that the standard analysis of surrogate compounds or analyte spikes n Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples and duplicate sampl from a given site be within the limits specified in methods or generated by Envir metrics laboratory.

The equation sed to calculate precision are given in Section 10.

Completenes

Environmetri defines the degree of completeness as the percent of samples for which acceptable an ytical data are generated. The equation used to calculate completeness is given in Section 10. The QA objective for most programs is 95%.

Representativeness

Representativeness expresses the degree to which sample data accurately represents the site, a specific matrix or parameter variations at a sampling point. Representativeness is a qualitative parameter which is dependent on both the proper design of the sampling program and proper laboratory protocol. The representative criterion is best satisfied by making certain that sampling locations, procedures, and quantity are selected based on the project objectives, and that proper analytical procedures are utilized, preservation requirements are met and holding times are not exceeded in the lab.

Comparability

Comparability expresses the confidence with which one data set can be compared with another. Sample data will be comparable with other measurement data if consistent documented analytical procedures are used for similar samples and sampling methods and conditions.

Section 2

SAMPLING PROCEDURES
CONTAINERS, PRESERVATIVES, HOLDING TIMES

Introduction

Before sampli of any site is performed, the client's Project Manager for the site should meet with Environm rics' QA Officer, or his designee. The purpose of the meeting will be to establish the sampling m thodology to be employed, and the tests that will be performed on the samples. A Sample Col tion Planning Request Form will be completed. When possible, the QA Officer and field samp ng team will establish the sensitivity required for the analytical tests. This will be possible in cas where the approximate levels of suspected regulated substances that are present in the samples e known.

After the plan ng meeting, the client will notify the laboratory and provide specifics of scope of the required se ices. Sample containers, preservatives and shipping containers, "blue ice", and chain of custo forms will be provided by Environmetrics. The QA Officer will ensure that, as part of the sam ling plan, sufficient sample is available for analysis.

Environmetric Inc. laboratory will supply bottles, coolers, containers, and chain-of-custody forms to clien for collecting water, hazardous waste and soil/sediment samples. After samples have been tak , they should be sent to Environmetrics for analysis within 24 hours after collection. Ty cally, the holding time begins from the date of collection in the field. For some programs the lding time begins the day the samples are delivered to the laboratory.

Tables 2.1, 2. 2.3, and 2.4 present the holding times and type of containers and preservatives to be used. Typ lly, the requirements of 40CFR, part 136 will be followed. However, SW-846, CLP, and drin ng water sampling procedures are also presented.

For details on ample Receipt, refer to SOP # 500-001.

For details on ample Storage, refer to SOP # 500-002.

Preparation of Sample Containers

Environmetrics, Inc. supplies new and pre-cleaned sample containers. These containers are purchased from nationally known vendors, who in many cases supply containers to the USEPA and state agencies, and are cleaned in conjunction with the procedures set by the EPA for quality-controlled sample containers.

All pre-cleaned containers are supplied as either Level I or Level II certified. All Level I and Level II containers are cleaned in accordance with the attached cleaning procedures. Level I containers are supplied with Certificates of Analysis outlined as follows:

Cleaning Procedures	***Type of Container***	***Analysis***
"A"	All clear glass All amber glass	Metals Pesticides Semivolatiles
"B"	Vials with septa Bottles with septa	Volatiles
"C"	HDPE plastic bottles	Metals Cyanide Fluoride
"D"	Cubitainers	Conductivity

The Certificate of Analysis includes the bottle type and QA Level, description, lot number, date analyzed , and the names of the compounds analyzed with their detection limits. A certification for Level II containers is also supplied. This Level II certification verifies that the containers are washed in conjunction with the procedures set by the EPA for quality-controlled sample containers.

The selection, storage, distribution, and control of sample containers is under the supervision of the director of operations and the shipping/receiving supervisor.

Depending on the container requirements of a project, Level I or Level II containers can be provided. Level I containers should only be used for critical/trace analyses.

The cleaning procedures are established as follows:

Cleaning Procedure A: (all clear and amber glass containers)

1. Wash bottles, liners, and caps in laboratory grade non-phosphate detergent.
2. Rinse three times.
3. Rinse with 1:1 nitric acid.
4. Rinse three times with ASTM Type 1 organic free water.
5. Oven dry for one hour.
6. Rinse with hexane.
7. Oven dry for one hour.

Analyzed for metals, pesticides, and semivolatiles.

Cleaning Procedure B: (all vials and bottles with septa lid)

1. Wash bottles, liners, and caps in laboratory grade non-phosphate detergent.
2. Rinse three times with tap water.
3. Rinse three times with ASTM Type 1 water.
4. Oven dry for one hour.

Analyzed for volatiles.

Cleaning Procedure C: (HDPE plastic bottles)

1. Wash bottles, liners, and caps in laboratory grade non-phosphate detergent.
2. Rinse three times.
3. Rinse with 1:1 nitric acid.
4. Rinse three times with ASTM Type 1 organic-free water.
5. Air dry.

Analyzed for metals, cyanide, and fluoride.

Cleaning Procedure D: (Cubitainers)

1. Rinse three times with deionized water.
2. Let stand forty-eight hours with DI water.
3. Air dry.

Table 2.1 Preservatives and Holding Times for SW-846, 3rd ed.

Parameter	Container	Preservative	Holding Time Soil	Water
Volatiles by GC/MS and GC	Water - 40ml glass vial with Teflon lined septa. Soil - Glass with Teflon-lined septa.	Cool, 4°C. pH ≤ 2 with HCl	14 days	14 days
Pesticides/PCB	Glass - Teflon-lined lid.	Cool, 4°C	• Extract within 14 days • Analyze within 40 days	• Extract within 7 days • Analyze within 40 days
Extractable Oraganics	Glass, Teflon-lined lid.	Cool, 4°C	• Extract within 14 days • Analyze within 40 days	• Extract within 7 days • Analyze within 40 days
Metals	Plastic or Glass	HNO3 to pH ≤ 2	6 months	6 months
Mercury	Plastic or Glass	HNO3 to pH ≤ 2	28 days	28 days
Cyanide	Plastic or Glass	NaOH to pH ≥ 12. Cool to 4 C. Add 0.6 grams ascorbic acid if residual chlorine present.	14 days	14 days
Chromium	Plastic or Glass	HNO3 to pH ≤ 2	24 hours	24 hours

Table 2.2 Preservatives and Holding Times for Contract Laboratory Program

Parameter	Container	Preservative	Holding Time	
			Water	Soil
Volatiles by GC/MS and GC	Water - 40ml glass vial with Teflon lined septa. Soil - Glass with Teflon-lined septa.	Cool, 4°C pH ≤ 2 with HCl	10 days	10 days
Pesticides/PC	Glass - Teflon-lined lid.	Cool, 4°C	• Extract within 10 days • Analyze within 40 days	• Extract within 5 days • Analyze within 40 days
Extractable Oraganics	Glass, Teflon-lined lid.	Cool, 4°C	• Extract within 10 days • Analyze within 40 days	• Extract within 5 days • Analyze within 40 days
Metals	Plastic or Glass	HNO_3 to pH ≤ 2	6 months	6 months
Mercury	Plastic or Glass	HNO_3 to pH ≤ 2	26 days	26 days
Cyanide	Plastic or Glass	NaOH to pH ≥ 12. Cool to 4 C. Add 0.6 grams ascorbic acid if residual chlorine present.	14 days	14 days
Chromium V	Plastic or Glass	HNO3 to pH ≤ 2	24 hours	24 hours

Table 2.3 Preservatives and Holding Times

40 CFR 136

Parameter	Container	Preservative	Holding Time Water
Volatiles by GC/MS and GC	Water - 40ml glass vial with Teflon lined septa.	Cool, 4° C. pH ≤ 2 with HCl. Add sodium thiosulfate if residual chlorine present.	14days
Pesticides/PCB	Glass - Teflon-lined lid.	Cool, 4°C Adjust pH to 5 - 9 if extraction is not within 72 hrs Add sodium thiosulfate if residual chloine & aldrin is to be determined.	• Extract within 7 days • Analyze within 40 days
Extractable Oragantics	Glass, Teflon-lined lid.	Cool, 4°C. Add sodium thiosulfate if residual chlorine present.	• Extract within 7days • Analyze within 40 days
Metals	Plastic or Glass	HNO_3 to pH ≤ 2	6 months
Mercury	Plastic or Glass	HNO_3 to pH ≤ 2	26 days
Cyanide	Plastic or Glass	NaOH to pH ≥ 12. Cool to 4 C. Add 0.6 grams ascorbic acid if residual chlorine present.	14 days
Chromium VI	Plastic or Glass	HNO3 to	24 hours

Table 2.4 Preservatives and Holding Times
EPA 600/R-92/129
EPA 600/4-79-20

Parameter	Container	Preservative	Holding Time - Water
Volatiles by GC/MS and GC	Water - 40ml glass vial with Teflon lined septa.	Cool, 4° C. pH ≤ 2 with HCl. Add ascorbic acid if residual chlorine present.	14days
Pesticides/PCB	Glass - Teflon-lined lid.	Cool, 4°C (Add mercuric chloride if biological degradation is noted.) Add sodium thiosulfate if residual chlorine & aldrin is to be determined.	• Extract within 7 days • Analyze within 14 days
Herbicides	Glass - Teflon-lined lid.	Cool, 4°C Add mercuric chloride. Add sodium thiosulfate if residual chlorine & aldrin is to be determined.	• Extract within 7 days • Analyze within 28 days
Extractable Organics by GC/MS	Glass, Teflon-lined lid.	Cool, 4°C. Add sodium sulfite if residual chlorine present. Adjust pH ≤ 2 with HCL for unchlorinated water.	• Extract within 7days • Analyze within 30 days
Metals	Plastic or Glass	HNO_3 to pH ≤ 2	6 months
Mercury	Plastic or Glass	HNO_3 to pH ≤ 2	28 days
Cyanide, Total & Amenable	Plastic or Glass	NaOH to pH ≥ 12. Cool to 4 C. Add 0.6 grams ascorbic acid if residual chlorine present.	14 days (24 hours when sulfide is present)

Section 3

SAMPLE CUSTODY PROCEDURES

Introduction

The procedur and definitions described below are necessary to maintain data validity and control. This cludes the sample numbering system, custody of samples in the field, transportation the laboratory via a certified carrier, and at the laboratory and all associated transfers of cu ody.

Definition

A sample is i someone's custody if:

a) It in his/her actual possession, or
b) It in his/her view, after being in his/her physical possession, or
c) It in his/her physical possession and locked up so that no one can tamper with i or
d) It kept in a secured area restricted to authorized personnel only.

Field Sampli Operations

The field sam ler will keep a bound field notebook, in which is recorded conditions and activities rela d to each sample collection. The sample will be placed in the correct container appropriately arked with a sample label. If the sample remains in the custody of the sampler, as described ove, no sample seals will be required. (All Illinois drinking water samples must specify the n e of the person who collected the sample on the Chain-of-Custody.)

Transfer of C stody

Samples coll ted by client's personnel will remain in the custody of the designated field custodian un delivery to the laboratory sample custodian or the certified carrier. The form shown in Fig e 3.1 is a chain of custody form that must be completed by the sample carrier and the laborator custodian.

Laboratory C stody Procedures

The followin procedures will be followed by the laboratory:

All s ples shall be handled by a minimum number of people.

The laboratory shall set aside "secured sample storage areas". These areas will be clean, dry, refrigerated, stored separately.

A specific person shall be designated custodian and an alternate designated to act in the custodian's absence. All incoming samples shall be received by the custodian, (or his/her designee) who shall indicate receipt by signing the accompanying chain of custody sheets.

The sample custodian shall maintain a log to record, for each sample, the person delivering the sample, the person receiving the sample, the date and time received, the source of sample, the sample identification or log number, how the sample was transported to the laboratory and the condition in which the sample was received (sealed, unsealed, broken container, or other pertinent remarks). A standardized format will be established for log book entries.

The custodian shall ensure that samples with heat- sensitive, light-sensitive and/or other unusual physical characteristics, or requiring special handling, are properly stored and maintained prior to analysis.

The laboratory area will be maintained as a secured area, restricted to authorized personnel only.

Laboratory personnel are responsible for the care and custody of the sample once it is handed over to them, and should be prepared to testify that the sample was in their possession and view, or secured in the laboratory at all times, from the moment it was received from the custodian until the time the analyses are completed. The laboratory personnel will return samples to the secure storage area after aliquoting the samples so that holding times for other parameters will not be invalidated by leaving samples at room temperature too long.

The samples and their associated extracts will be held for thirty days after the final report has been delivered to the client. The time and date of discard will be recorded in the log. Data sheets will be kept secured by the lab custodian.

11401 Moog Drive
St. Louis, MO 63146-3560
(314) 432-0550

CUSTODY TRANSFER RECORD / LABORATORY WORK REQUEST

COC # 10004

COMPANY _____ CONTACT _____

ADDRESS _____ DATE _____

CITY STATE ZIP _____ FAX ( ) _____

PHONE ( ) _____ E-MAIL _____

PROJECT # _____

PO # _____

PAGE _____ OF _____

Turnaround Time: ☐ Normal (8-10 business days) ☐ Rush (5 business days) ☐ Fastrak (3 business days)

☐ 1-2 Business Days ☐ Same Day Due Date _____

Delivery Mode: ☐ Fax ☐ E-Mail ☐ Phone Call

INSTRUCTIONS _____

SAMPLE IDENTIFICATION

ANALYSES REQUEST

ITEM	SITE CODE / SAMPLE DESCRIPTION	DATE COLLECTED	TIME COLLECTED	PRESERV.	CONTAINER
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
14					
15					

ITEMS TRANSFERRED	RELINQUISHED BY	Date	Time	RECEIVED BY	Date	Time	REASON for TRANSFER

SPECIAL NOTES / INSTRUCTIONS

Section 4

CALIBRATION PROCEDURES AND FREQUENCY

Introduction

Calibration pr edures and frequency of calibration are specified in the following tables for the most frequent performed analytical tests. Environmetrics' chemists and technicians follow the latest EPA an RCRA protocols and generate hard copy reports to show that these criteria have been met prio o and during sample analysis.

Metals Anal s

The followin criteria found in Table 4.4 will be used in assessing calibration data for ICP and AAS method Samples will not be analyzed unless these criteria are met.

Initial calibra n curves must show a correlation coefficient between 0.995 and 1.000 when method of sta dard addition (MSA) is used.

Organic An ses

The followin criteria found in Tables 4.1 through 4.3 will be used in assessing calibration data for GC/MS m thods. Samples will not be analyzed unless these criteria are met:

Other Orga c Methods

All organics alysis methods other than those listed above will follow procedures dictated in the individual m hods for all calibrations, surrogates and internal standards, QC check samples, matrix spike mples, etc.

Inorganic M thods

All inorgani analysis methods will follow procedures dictated in the individual methods for all calibrations. rrogates and internal standards, QC check samples, matrix spike samples, etc.

General Cali ration

If the approve test method specifies the generation of an initial calibration curve but does not establish crite a for the number of standards, initial or continuing verification of the calibration, the following uidelines will be used.

Number of S ndards:

1. Deter ne a percent relative standard deviation (%RSD) of:
   - th nalyses of a minimum of seven replicate measurements of a standard with a co centration at one to three times the MDL; or
   - th respone or calibration factors of at least three standards having concentrations that co r the expected calibration range.

2. From e following table, determine the minium number of calibration standards to be used the initial calibration curve by correlating the %RSD with the number of required calib ion standards.

%RSD	Number of Calibration Standards
0 - <2	1**
2 - <10	3
10 -<25	5
>25	7

** Assumes linearity through the origin (0,0). For analytes for which there is no origin (such as pH), a two point calibration curve shall be used.

3. The mber of calibration standards as dtermined form the table and a blank shall be used to ge rate the initial curve.

4. If the alibration curve is not linear (see section - Linearity Test) and the approved test meth t allows for the use of non-linear calibration curves, additional calibration stand ds shall be used to define the calibration.

Reporting R ge - if the approved method specifies the generation and use of a calibration curve, all sample re lts shall be reported from sample analyes with the range of the curve, except when the approve method specifically allow otherwise (i.e. ICP analyes above the highest calibration concentratio but within the linear dynamic range).

Linearity Tes

1. The i ial calibration curve is consider linear when:
   - th correlation coefficient from the linear regression analyses is 0.995 or greater;
   - th %RSD of the response factors is 15% or less;
   - th %RSD of the calibration factors is 30% or less.

2. The l oratory shall utilize the same method for determining analytical results as the meth used to determine the curve linearity.

Initial Calibr on Verification:

1. ICV s ndards shall be prepared from a second source, where available. Otherwise the ICV ll be prepared by an analyst other then the one preparing the calibration standards.
2. If the CV standard concentration is not specified, it shall be a 10% to 50% of the maxi um of the calibration range.
3. If the CV acceptance criteria is not specified, the results of the check standard shall be withi 15% of the true value.
4. If the CV fails to meet the acceptance criteria - either:
   - s pend sample analyses and take corrective action to be followed immediately by a re alysis of the ICV; or
   - i nediately reanalyze the ICV, and either
     - continue analyses if the reanalysis of the ICV meets acceptance criteria, or
     - terminate sample analysis, reject samples for which the ICV failed to meet criteria, and establish and reverify a new initial calibration.

Continuing C libration Verification:

1. CCV andards shall be prepared from the initial calibration curve standards or from a secon source.
2. If the CV standard concentration is not specified, it shall be a 25% to 50% of the maxi um of the calibration range.
3. The C V shall be analyzed once per 20 samples or every 12 hours, whichever is more frequ t.
4. If the CV acceptance criteria is not specified, the results of the check standard shall be withi 15% of the true value.
5. If the CV fails to meet the acceptance criteria - either:
   - s pend sample analyses and take corrective action to be followed immediately by a re alysis of the CCV; or
   - i nediately reanalyze the CCV, and either
     - continue analyses if the reanalysis of the CCV meets acceptance criteria, or
     - terminate sample analysis, reject samples for which the CCV failed to meet criteria, and establish and reverify a new initial calibration.

6. Whenever the generation of a new initial calibration curve and verification of the new initial calibration curve are required, the laboratory shall reanalyze all samples analyzed since the last CCV check standard which met the CCV acceptance criteria, except for those instances where the CCV acceptance criteria was exceed high and there are non-detect results for the corresponding analyte in the samples associated with the CCV check standard. In those instances, the non-detect results may be reported.

Table 4.1

TUNING AND CALIBRATION REQUIREMENTS

	SW-846	Method	Method	Method	SW-846	Method	Method
	8260						
TUNING CRITERIA							
FREQUENCY	12 hours	12 hours	24 hours	8 hours	12 hours	12 hours	24 hours
TUNING COMPOUND ION ABUNDANCE FOR BROMOFLUOROBENZENE							
MASS							
50 as Percent of 95	15 to 40	8 to 40	15 to 40	15 to 40	NA	NA	NA
75 as Percent of 95	30 to 60	30 to 66	30 to 60	30 to 80	NA	NA	NA
95 as Percent of 95	Base = 100	Base = 100	Base = 100	Base = 100	NA	NA	NA
96 as Percent of 95	5 to 9	5 to 9	5 to 9	5 to 9	NA	NA	NA
173 as Percent of 174	< 2	< 2	< 2	< 2	NA	NA	NA
174 as Percent of 95	> 50	50 to 120	> 50	> 50	NA	NA	NA
175 as Percent of 174	5 to 9	4 to 9	5 to 9	5 to 9	NA	NA	NA
176 as Percent of 174	> 95 but < 101	93 to 101	> 95 but < 101	> 95 but < 101	NA	NA	NA
177 as Percent of 176	5 to 9	5 to 9	5 to 9	5 to 9	NA	NA	NA
FOR DFTPP							
MASS							
51 as Percent of 198	NA	NA	NA	NA	30 to 60	30 to 80	30 to 60
68 as Percent of 69	NA	NA	NA	NA	< 2	< 2	< 2
69 Present	NA	NA	NA	NA	YES	YES	YES
70 as Percent of 69	NA	NA	NA	NA	< 2	< 2	< 2
127 as Percent of 198	NA	NA	NA	NA	40 to 60	25 to 75	40 to 60
197 as Percent of 198	NA	NA	NA	NA	< 1	< 1	< 1
198 as Percent of 198	NA	NA	NA	NA	Base = 100	Base = 100	Base = 100
199 as Percent of 198	NA	NA	NA	NA	5 to 9	5 to 9	5 to 9
275 as Percent of 198	NA	NA	NA	NA	10 to 30	10 to 30	10 to 30
365 as Percent of 198	NA	NA	NA	NA	> 1	> 0.75	> 1
441 as Percent of 443	NA	NA	NA	NA	< mass 443	< mass 443	< mass 443
442 as Percent of 198	NA	NA	NA	NA	> 40	40 to 110	> 40
443 as Percent of 442	NA	NA	NA	NA	17 to 23	15 to 20	17 to 23

Table 4.1 (continued)

TUNING AND CALIBRATION REQUIREMENTS

	SW-846 8240 & 8260	Method	Method	Method	SW-846	Method	Method
CALIBRATION CRITERIA							
INITIAL CALIBRATION							
CALIBRATION TYPE	Average Response	Average Response	Average Response	Average Response 1st and 2nd Order	Average Response	Average Response	Average Response
CALIBRATION POINTS	5	5	MINIMUM 3	MINIMUM 3	5	5	MINIMUM 3
AMOUNTS	Working Range Low standard Near and above MDL	10,20,50,100, 200 PPB	Working Range Low standard Near and above MDL	Working Range Low standard Near and above MDL	Working Range Low standard Near and above MDL	20,50,80,120, 160 PPB	Working Range Low standard Near and above MDL
CONTINUING CALIBRATION CHECK							
FREQUENCY	PER TUNE	PER TUNE	PER TUNE	PER TUNE	PER TUNE	PER TUNE	PER TUNE
AMOUNT	MID POINT	MID POINT	20 PPB	MID POINT	MID POINT	MID POINT	MID POINT
INTERNAL STANDARD AREAS CALIBRATION TO PREVIOUS CALIBRATION	-50 to +100% LAST IC or CC	Calibration to Sample -50 to +100%	NS	± 30% LAST IC ± 50% LAST CC	-50 to + 100% LAST IC or CC	Calibration to Samples. -50 to + 100	NS

MDL = METHOD DETECTION LIMIT
IC = INITIAL CALIBRATION
CC = CONTINUING CALIBRATION
DFTPP = DECAFLUOROTRIPHENYLPHOSPHINE
NA = NOT APPLICABLE TO THIS METHOD
NS = NOT SPECIFIED IN THIS METHOD

SPCC & CCC ACCEPTANCE CRITERIA
VOLATILE METHODS

	SW-846-8240			SW-846-8260			CLP 3/90			Method 624			Method 524.2		
COMPOUND	SPCC RF	CCC RSD	% DRIFT	SPCC RF	CCC RSD	% DRIFT	MIN RF	MAX RSD	% DIFF	SPCC RF	CCC RSD	% DIFF	MIN RF	MAX RSD	% DIFF
Acetone	NS	15*	NS	NA	15*	NA	0.010	NS	NS	NA	NA	NA	NA	NA	NA
2-Butanone	NS	15*	NS	NA	15*	NA	0.010	NS	NS	NA	NA	NA	NA	NA	NA
Carbon disulfide	NS	15*	NS	NA	15*	NA	0.010	NS	NS	NA	NA	NA	NA	NA	NA
Hexanone	NS	15*	NS	NA	15*	NA	0.010	NS	NS	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	NS	15*	NS	NA	15*	NA	0.010	NS	NS	NA	NA	NA	NA	NA	NA
Toluene-d8	NS	NS	NS	NS	NS	NS	0.010	NS	NS	NA	NA	NA	NA	NA	NA
1,2-Dichloroethane-d4	NS	NS	NS	NA	NA	NA	0.010	NS	NS	NS	NS	NS	NS	NS	NS
Benzene	NS	15*	NS	NS	15*	NS	0.500	20.5	25.0	NS	35	37-151	NS	20	30
Bromobenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Bromochloromethane	NA	NA	NA	NS	15*	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Bromodichloromethane	NS	15*	NS	NS	15*	NS	0.200	20.5	25.0	NS	35	35-155	NS	20	30
Bromofluorobenzene	NS	NS	NS	NS	NS	NS	0.200	20.5	25.0	NS	NS	NS	NS	NS	NS
Bromoform	0.10	NS	NS	0.10	15*	NS	0.100	20.5	25.0	NS	35	45-169	NS	20	30
Bromomethane	NS	15*	NS	NS	15*	NS	0.100	20.5	25.0	NS	35	D-242	NS	20	30
n-Butylbenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
sec-Butylbenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
tert-Butylbenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Carbon Tetrachloride	NS	15*	NS	NS	15*	NS	0.100	20.5	25.0	NS	35	70-140	NS	20	30
Chlorobenzene	0.300	NS	NS	0.30	15*	NS	0.500	20.5	25.0	NS	35	37-160	NS	20	30
Chloroethane	NS	15*	NS	NS	15*	NS	0.010	NS	NS	NS	35	14-230	NS	20	30
Chloroform	NS	30	20	NS	30	20	0.200	20.5	25.0	NS	35	51-138	NS	20	30
Chloromethane	0.300	NS	NS	0.10	15*	NS	0.010	NS	NS	NS	35	D-273	NS	20	30
2-Chlorotoluene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
4-Chlorotoluene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Dibromochloromethane	NS	15*	NS	NS	15*	NS	0.100	20.5	25.0	NS	35	53-149	NS	20	30
1,2-Dibromo-3-Chloropropane	NS	NS	NS	NS	15*	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,2-Dibromoethane	NS	NS	NS	NS	15*	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Dibromomethane	NS	NS	NS	NS	15*	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,2-Dichlorobenzene	NS	NS	NS	NS	15*	NS	NA	NA	NA	NS	35	18-190	NS	20	30
1,3-Dichlorobenzene	NS	NS	NS	NS	15*	NS	NA	NA	NA	NS	35	59-156	NS	20	30
1,4-Dichlorobenzene	NS	NS	NS	NS	15*	NS	NA	NA	NA	NS	35	18-190	NS	20	30
Dichlorodifluoromethane	NS	NS	NS	NS	15*	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,1-Dichloroethane	0.300	NS	NS	0.10	15*	NS	0.200	20.5	25.0	NS	35	59-155	NS	20	30
1,2-Dichloroethane	NS	15*	NS	NS	15*	NS	0.100	20.5	25.0	NS	35	49-155	NS	20	30
1,1-Dichloroethene	NS	30	20	NS	30	20	0.100	20.5	25.0	NS	35	D-234	NS	20	30
cis-1,2-Dichloroethene	NS	15*	NS	NS	NS	NS	0.010	NS	NS	NA	NA	NA	NS	20	30
trans-1,2-Dichloroethene	NS	15*	NS	NS	NS	NS	0.010	NS	NS	NS	35	54-156	NS	20	30
1,2-Dichloropropane	NS	30	20	NS	30	20	0.010	NS	NS	NS	35	D-210	NS	20	30
1,3-Dichloropropane	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30

Table 4.2 (continued)

SPCC & CCC ACCEPTANCE CRITERIA
VOLATILE METHODS

	SW-846-8240			SW-846-8260			CLP 3/90			Method 624			Method 524.2		
COMPOUND	SPCC RF (I/C)	CCC RSD (I)	% DIFF (C)	SPCC RF (I/C)	CCC RSD (I)	% DIFF (C)	MIN (I/C)	MAX (I)	% (C)	SPCC (I/C)	CCC (I)	% (C)	MIN	MAX (I)	% (C)
2,2-Dichloropropane	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,1-Dichloropropene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
cis-1,3-Dichloropropene	NS	15*	NS	NS	15*	NS	0.200	20.5	25.0	NS	35	D-227	NS	20	30
trans-1,3-Dichloropropene	NS	15*	NS	NS	15*	NS	0.100	20.5	25.0	NS	35	17-183	NS	20	30
Ethylbenzene	NS	15*	20	NS	30	20	0.100	20.5	25.0	NS	35	37-162	NS	20	30
Hexachlorobutadiene	NS	NS	NS	NS	15*	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Isopropylbenzene	NS	NS	NS	NS	15*	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Methylene Chloride	NS	15*	NS	NS	15*	NS	0.010	NS	NS	NS	35	D-221	NS	20	30
Naphthalene	NS	NS	NS	NS	15*	NS	NA	NA	NA	NA	NA	NA	NS	20	30
n-Propylbenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Styrene	NS	15*	NS	NS	NS	NS	0.300	20.5	25.0	NA	NA	NA	NS	20	30
1,1,1,2-Tetrachloroethane	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,1,2,2-Tetrachloroethane	0.300	NS	NS	0.30	NS	NS	0.500	20.5	25.0	NS	35	46-157	NS	20	30
Tetrachloroethene	NS	15*	NS	NS	NS	NS	0.200	20.5	25.0	NS	35	64-148	NS	20	30
Toluene	NS	30	20	NS	30	20	0.400	20.5	25.0	NS	35	47-150	NS	20	30
1,2,3-Trichlorobenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,2,4-Trichlorobenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,1,1-Trichloroethane	NS	15*	NS	NS	NS	NS	0.100	20.5	25.0	NS	35	52-162	NS	20	30
1,1,2-Trichloroethane	NS	15*	NS	NS	NS	NS	0.100	20.5	25.0	NS	35	52-150	NS	20	30
Trichloroethene	NS	15*	NS	NS	NS	NS	0.300	20.5	25.0	NS	35	71-157	NS	20	30
Trichlorofluoromethane	NS	15*	NS	NS	NS	NS	NA	NA	NA	NS	35	17-181	NS	20	30
1,2,3-Trichloropropane	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,2,4-Trimethylbenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
1,3,5-Trimethylbenzene	NS	NS	NS	NS	NS	NS	NA	NA	NA	NA	NA	NA	NS	20	30
Vinyl Chloride	NS	30	20	NS	30	20	0.100	20.5	25.0	NS	35	D-251	NS	20	30
o-Xylene	NS	15*	NS	NS	15*	NS	0.300	20.5	25.0	NA	NA	NA	NS	20	30
m-Xylene	NS	15*	NS	NS	15*	NS	0.300	20.5	25.0	NA	NA	NA	NS	20	30
p-Xylene	NS	15*	NS	NS	15*	NS	0.300	20.5	25.0	NA	NA	NA	NS	20	30

SPCC = SYSTEM PERFORMANCE CHECK COMPOUNDS
CCC = CALIBRATION CHECK COMPOUNDS
RSD = RELATIVE STANDARD DEVIATION
RF = RESPONSE FACTOR
NA = NOT APPLICABLE
NS = NOT SPECIFIED

I = INITIAL CALIBRATION CRITERIA
C = DAILY (TUNE PERIOD) CALIBRATION CRITERIA
D = DETECTED
* = CALIBRATION CURVES ($r \geq 0.99$) USED IF RSD EXCEEDS 15%

Acrolein, Acrylonitrile, 2-chloro-ethylvinyl-ether, and cyanogen chloride exhibit poor purging efficiency and or are unstable and therefore do not have SPCC and CCC criteria. Any compound not found in this table but is contained in appendix 9, CLP, 8240, 8260, 624, and 524.2 can be considered not specified.

Table 4.3

SPCC & CCC ACCEPTANCE CRITERIA
SEMIVOLATILE METHODS

		SW-846-8270			CLP 3/90			Method 625		
	COMPOUND	SPCC RF	CCC RSD	% DIFF	SPCC RF	CCC RSD	% DIFF	SPCC RF	CCC RSD	% DIFF
2-F	rophenol	NS	NS	NS	0.600	20.5	25.0	NS	35	20
Ph	l-d5 (d6)	NS	NS	NS	0.800	20.5	25.0	NS	35	20
Ph	l	NS	30	30	0.800	20.5	25.0	NS	35	20
bis	-chloroethyl)Ether	NS	NS	NS	0.700	20.5	25.0	NS	35	20
2-C	rophenol-d4	NS	NS	NS	0.800	20.5	25.0	NA	NA	NA
2-C	rophenol	NS	NS	NS	0.800	20.5	25.0	NS	35	20
1,3	chlorobenzene	NS	NS	NS	0.600	20.5	25.0	NS	35	20
1,4	chlorobenzene	NS	30	30	0.500	20.5	25.0	NS	35	20
1,2	chlorobenzene-d4	NS	NS	NS	0.400	20.5	25.0	NA	NA	NA
1,2	chlorobenzene	NS	NS	NS	0.400	20.5	25.0	NS	35	20
2-I	hylphenol	NS	NS	NS	0.700	20.5	25.0	NA	NA	NA
2,2	vbis(1-Chloropropane)	NS	NS	NS	0.010	NS	NS	NS	35	20
4-I	hylphenol	NS	NS	NS	0.600	20.5	25.0	NA	NA	NA
N-	roso-Di-n-Propylamine	0.050	NS	NS	0.500	20.5	25.0	NS	35	20
He	hloroethane	NS	NS	NS	0.300	20.5	25.0	NS	35	20
Ni	enzene-d5	NS	NS	NS	0.200	20.5	25.0	NS	35	20
Ni	enzene	NS	NS	NS	0.200	20.5	25.0	NS	35	20
Iso	rone	NS	NS	NS	0.400	20.5	25.0	NS	35	20
2-I	opnenol	NS	30	30	0.100	20.5	25.0	NS	35	20
2,4	imethylphenol	NS	NS	NS	0.200	20.5	25.0	NS	35	20
bis	-Chloroethoxy) Methane	NS	NS	NS	0.300	20.5	25.0	NS	35	20
2,4	ichlorophenol	NS	30	30	0.200	20.5	25.0	NS	35	20
1,2	Trichlorobenzene	NS	NS	NS	0.200	20.5	25.0	NS	35	20
Na	thalene	NS	NS	NS	0.700	20.5	25.0	NS	35	20
He	hlorobutadiene	NS	30	30	0.010	NS	NS	NS	35	20
4-C	oro-3-Methylphenol	NS	30	30	0.200	20.5	25.0	NS	35	20
2-I	thylnaphthalene	NS	NS	NS	0.400	20.5	25.0	NS	35	20
2,4	Trichlorophenol	NS	30	30	0.200	20.5	25.0	NS	35	20
2,4	Trichlorophenol	NS	NS	NS	0.200	20.5	25.0	NA	NA	NA
2-C	oronaphthalene	NS	NS	NS	0.800	20.5	25.0	NS	35	20
2-C	oropiphenyl	NS	NS	NS	0.700	20.5	25.0	NA	NA	NA
Di	thyl Phthalate	NS	NS	NS	0.010	NS	NS	NS	35	20
Ac	aphthylene	NS	NS	NS	1.300	20.5	25.0	NS	35	20
2,6	initrotoluene	NS	NS	NS	0.200	20.5	25.0	NS	35	20
Ac	aphthene	NS	30	30	0.800	20.5	25.0	NS	35	20
2,4	initrophenol	0.050	NS	NS	0.010	NS	NS	NS	35	20
4-N	ropnenol	0.050	NS	NS	0.010	NS	NS	NS	35	20
Di	nzofuran	NS	NS	NS	0.800	20.5	25.0	NA	NA	NA
Di	ylphthalate	NS	NS	NS	0.010	NS	NS	NS	35	20
2,4	initrotoluene	NS	NS	NS	0.200	20.5	25.0	NS	35	20
4-C	orophenyl-phenyl-ether	NS	NS	NS	0.400	20.5	25.0	NS	35	20
Fl	ene	NS	NS	NS	0.900	20.5	25.0	NS	35	20
4,6	initro-2-Methylphenol	NS	NS	NS	0.010	20.5	25.0	NS	35	20
2,4	-Tribromophenol	NS	NS	NS	0.100	NS	NS	NA	NA	NA
4-B	omophenyl-phenyl-ether	NS	NS	NS	0.100	20.5	25.0	NS	35	20
He	chlorobenzene	NS	NS	NS	0.800	20.5	25.0	NS	35	20
Pe	achlorophenol	NS	30	30	0.050	20.5	25.0	NS	35	20
Ph	anthrene	NS	NS	NS	0.700	20.5	25.0	NS	35	20
An	racene	NS	NS	NS	0.700	20.5	25.0	NS	35	20
Ca	rbazole	NS	NS	NS	0.800	NS	NS	NA	NA	NA
Di	utylphthalate	NS	NS	NS	0.010	NS	NS	NS	35	20
Fl	anthene	NS	NS	NS	0.600	20.5	25.0	NS	35	20
Py	ne	NS	NS	NS	0.700	20.5	25.0	NS	35	20
Be	nzyl Butyl	NS	NS	NS	0.500	20.5	25.0	NS	35	20

Table 4.3 (continued)

SPCC & CCC ACCEPTANCE CRITERIA
SEMIVOLATILE METHODS

		SW-846-8270			CLP 3/90			Method 625		
	COMPOUND	SPCC RF	CCC RSD	% DIFF	SPCC RF	CCC RSD	% DIFF	SPCC RF	CCC RSD	% DIFF
Ber	butylphthalate	NS	NS	NS	0.010	NS	NS	NS	35	20
3,3'	chlorobenzidine	NS	NS	NS	0.010	NS	NS	NS	35	20
Ber	a)Anthracene	NS	NS	NS	0.800	20.5	25.0	NS	35	20
Chr	ne	NS	NS	NS	0.700	20.5	25.0	NS	35	20
bis-	Ethylhexyl)-phthalate	NS	NS	NS	0.010	NS	NS	NS	35	20
Di-	)ctyl Phthalate	NS	30	30	0.010	NS	NS	NS	35	20
Ber	(b)Fluoranthene	NS	NS	NS	0.700	20.5	25.0	NS	35	20
Ber	(a)Fluoranthene	NS	NS	NS	0.700	20.5	25.0	NS	35	20
Ber	(a)Pyrene	NS	30	30	0.700	20.5	25.0	NS	35	20
Ind	)(1,2,3-cd)Pyrene	NS	NS	NS	0.500	20.5	25.0	NS	35	20
Dib	zo(a,h)Anthracene	NS	NS	NS	0.400	20.5	25.0	NS	35	20
Ber	(g,h,i)Perylene	NS	NS	NS	0.500	20.5	25.0	NS	35	20
3-N	oaniline	NS	NS	NS	0.010	NS	NS	NA	NA	NA
N-N	rosodiphenylamine	NS	30	30	0.010	NS	NS	NA	NA	NA
4-B	oaniline	NS	NS	NS	0.010	NS	NS	NA	NA	NA
4-C	)roaniline	NS	NS	NS	0.010	NS	NS	NA	NA	NA
Hex	hlorocyclopentadiene	0.050	NS	NS	0.010	NS	NS	NA	NA	NA
2-N	oaniline	NS	NS	NS	0.010	NS	NS	NA	NA	NA

SPCC = SYSTEM PERFORMANCE CHECK COMPOUNDS.
CCC = CALIBRATION CHECK COMPOUNDS.
RSD = RELATIVE STANDARD DEVIATION.
RF = RESPONSE FACTOR.
NA = NOT APPLICABLE.
NS = NOT SPECIFIED.
NOTE: Any compound not found in this table, but is contained in appendix 9, CLP, 8270, and 625 can be considered not specified.

Table 4.4

CALIBRATION REQUIREMENTS for METALS ANALYSIS

	SW-846 6010	Method 200.7	Method CLP	Method 245.1	SW-846 7470A	SW-846	Method
CALIBRATION CRITERIA							
INITIAL CALIBRATION							
CALIBRATION POINTS	Per instrument manufacturer specs. Minium of one standard and a blank	Per instrument manufacturer specs. Minium of one standard and a blank	Per instrument manufacturer specs. Minium of one standard and a blank	5 levels + blank	5 levels + blank	Mininum of 3 levels + blank	Minimum of 4 levels + blank
AMOUNTS				Range: 0.5 - 10 µg/L	Range: 0.5 - 10 µg/L		
CONTINUING CALIBRATION CHECK							
FREQUENCY	Beginning, end, and every 10 samples	After Calibration, every 10 samples & end of run	Beginning, end, and every 10 samples	After calibration, every 10 samples, & end of run	After every 10 samples	Every 10 samples or every 10 injections for GFAA	Daily or every 20 samples
AMOUNT	Mid Range (CCV)	Mid Point (IPC)	Mid Range (CCV)	Mid Level	Mid Level	Mid Level	Low Level
CRITERION	± 10% of stated value	ICV = ±5%R CCV = ±10%R	± 10% of stated value	ICV = ±5%R CCV = ±10%R	± 20% of stated value	± 20% of stated value	%R = ±10%

MDL = METHOD DETECTION LIMIT
IC = INITIAL CALIBRATION
CC = CONTINUING CALIBRATION

Table 4.5

CALIBRATION PROCEDURES AND FREQUENCY

Parameter	Initial Calibration	Continuing Calibration	Comments
Polynuclear Aromatics / P A	3-5 point (checked with every sample set).	One midpoint standard and blank on daily basis.	QC check standard when new stock standards are used.
Pesticides / P B	3-5 point (checked with every sample set).	One midpoint standard and blank on daily basis.	QC check standard when new stock standards are used.
PCB (Oil & V pe)	3-5 point (checked with every sample set).	One midpoint standard and blank on daily basis.	QC check standard when new stock standards are used.
VOA by GC	3-5 point (checked with every sample set).	One midpoint standard and blank on daily basis.	QC check standard when new stock standards are used.
TPH	3-5 point (checked with every sample set).	One midpoint standard and blank on daily basis.	QC check standard when new stock standards are used.
Fuels Finger nt (GC/FID)	3 point (checked with every sample set) using appropriate petroleum product (e.g. gasoline, diesel, jet fuel, etc.).	One midpoint standard and blank, daily or every 12 hours.	External standard technique. When possible, standards are to be site specific. QC check standard when new stock standards are used.

Section 5

ANALYTICAL PROCEDURES

Introducti

Selection d implementation of analytical procedures must be done such that chemists and techni ans can produce accurate and reproducible data. All sources of bias and error must be m imized and well-known to trained laboratory staff. The sampling process must be s nd and related to the intended use of analytical procedures. Therefore, at Environm ics, all aspects of the project are discussed with the client before analytical procedure re selected. In most cases, the procedures recommended in federal regulation CWA, RCRA, CERCLA/SARA, TSCA, and SDWA) are followed. For non target com unds and unusual sample matrices, the laboratory staff will document all deviations om the accepted analytical procedures.

In general he analytical procedures will take the following points under consideration:

1. ample matrix
2. reparation of samples
3. etection limits
4. hoice of instrumentation/Analytical Methods
5. reparation of Calibration standards
6. olding times
7. uality Assurance/Quality Control
8. raining of chemists/technicians
9. ata Handling/Validation
10. nstrument maintenance
11. orrective action for out of control events
12. afety

When a p posal becomes a task order, the client service group will discuss the specifics of the pro t with the lab personnel. A quality assurance project plan (QAPP) may be prepared f long term contracts. The analytical methods that will be used are in most cases fron pproved EPA, ASTM, or Standard Methods. Methods are translated into Standard perating Procedures (SOP). SOP's are updated and copies are maintained in each labo ory section.

Table 5.1

Example
MDL & PQL for VOLATILES ANALYSIS
by GC/MS

PARAMETERS	MDL (actual)	8240 PQL	8260 PQL	CLP CRDL	624 MDL	ApplX PQL	524.2 MDL (actual)
Dichlorodifluoromethane	0.28	---	5	---	---	5	0.05
Chloromethane	0.65	10	10	10	---	10	0.12
Vinyl Chloride	0.34	10	10	10	---	10	0.03
Bromomethane	0.58	10	10	10	---	100	0.20
Chloroethane	0.13	10	10	10	---	10	0.01
Trichlorofluoromethane	0.41	---	5	---	---	5	0.05
Acrolein	1.3	---	---	---	---	5	---
1,1-Dichloroethene	0.37	5	5	10	2.8	5	0.11
Methyl Iodide (Iodomethane)	0.24	---	5	---	---	5	0.16
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.40	---	---	---	---	---	0.11
Acetone	0.64	100	100	10	---	100	0.54
Carbon Disulfide	0.55	100	100	10	---	5	0.10
Allyl Chloride	0.82	---	---	---	---	100	0.13
Acetonitrile	1.5	---	---	---	---	100	0.11
Methylene Chloride	0.99	5	5	10	2.8	5	0.25
Acrylonitrile	1.7	---	100	---	---	5	0.09
trans-1,2-Dichloroethene	0.24	5	5	---	1.6	5	0.10
cis-1,2-Dichloroethene	0.35	5	5	---	---	---	0.12
Bromochloromethane	0.44				---		
1,1-Dichloroethane	0.42	5	5	10	4.7	5	0.10
Vinyl Acetate	0.41	50	---	---	---	5	0.17
Chloroprene	Na	---	---	---	---	5	---
Methyl-tert-butyl ether	0.25				---		
2-Butanone (MEK)	0.30	100	---	10	---	100	0.15
2,2-Dichloropropane	0.50	---	5	---	---	---	0.14
Propionitrile*	0.44	---	---	---	---	5	0.15
Methacrylonitrile	0.39	---	---	---	---	5	0.10
Chloroform	0.28	5	5	10	1.6	5	0.10
1,1,1-Trichloroethane	0.48	5	5	10	3.8	5	0.10
Carbon tetrachloride	0.51	5	5	10	2.8	5	0.20
1,1-Dichloropropene	0.21	---	5	---	---	---	0.10
Isobutyl alcohol	0.88	---	---	---	---	50*	0.11
1,2-Dichloroethane	0.20	5	5	10	2.8	5	0.12
Benzene	0.41	5	5	10	4.4	5	0.10
Trichloroethene	0.20	5	5	10	1.9	5	0.09
1,2-Dichloropropane	0.20	5	5	10	---	5	0.11
Methyl Methacrylate	0.43	---	---	---	---	5	0.12
1,4-Dioxane	0.45	---	---	---	---	150	0.11

Concentration (µg/L/Kg)

Table 5.1 (continued)

Example
MDL & PQL for VOLATILES ANALYSIS
by GC/MS

PARAMETERS	MDL (actual)	8240 PQL	8260 PQL	CLP CRDL	624 MDL	AppIX PQL	524.2 MDL (actual)
Dibromome[illegible]ne	0.20	---	5	---	---	5	0.14
Bromodichl[illegible]omethane	0.14	5	5	10	2.2	5	.013
2-Chloroeth[illegible]vinyl ether	0.61				---		
2-Nitroprop[illegible]e	0.48	---	---	---	---	---	0.17
trans-1,3-D[illegible]loropropene	0.22	5	---	10	---	5	0.14
4-Methyl-2[illegible]ntanone	0.43	50	50	10	---	50	0.12
Toluene	0.27	5	5	10	6.0	5	0.10
cis-1,3-Dic[illegible]ropropene	0.24	5	5	10	5.0	5	0.12
Ethyl Meth[illegible]ylate	0.28	---	---	---	---	5	0.10
1,1,2-Trich[illegible]oethane	0.21	5	5	10	5.0	5	0.10
Tetrachlorc[illegible]hene	0.20	5	5	10	4.1	5	0.10
1,3-Dichlo[illegible]ropane	0.14	---	5	---	---	---	0.13
2-Hexanon[illegible]	0.44	50	50	10	---	50	0.12
Dibromoch[illegible]romethane	0.20	5	5	10	3.1	5	0.12
1,2-Dibron[illegible]thane	0.20	---	5	---	---	5	0.14
Chloroben[illegible]ne	0.21	5	5	10	6.0	5	0.10
1,1,1,2-Te[illegible]chloroethane	0.21	---	5	---	---	5	0.12
Ethyl benz[illegible]e	0.21	5	5	10	7.2	5	0.10
m & p-Xy[illegible]e	0.29	5	5	10	---	5	0.16
o-Xylene	0.42	5	5	10	---	5	0.10
Styrene	0.15	5	5	10	---	5	0.10
Bromofor[illegible]	0.27	5	5	10	4.7	5	0.14
Isopropylb[illegible]zene (Cumene)	0.20	---	5	---	---	---	0.12
1,1,2,2-Te[illegible]chloroethane	0.32	5	5	10	6.9	5	0.11
Bromober[illegible]ne	0.31	---	5	---	---	---	0.13
trans-1,4-[illegible]hloro-2-Butene	0.32	---	5	---	---	5	0.12
1,2,3-Tric[illegible]ropropane	0.34	---	5	---	---	5	0.12
n-Propylb[illegible]zene	0.14	---	5	---	---	---	0.12
2-Chlorot[illegible]ene	0.23	---	5	---	---	---	0.11
1,3,5-Trim[illegible]thylbenzene	0.20	---	5	---	---	---	0.11
4-Chlorot[illegible]ene	0.25	---	5	---	---	---	0.10
tert-Butyl[illegible]nzene	0.24	---	5	---	---	---	0.10
1,2,4-Trim[illegible]thylbenzene	0.36	---	5	---	---	10*	0.23
sec-Butyl[illegible]nzene	0.28	---	5	---	---	---	0.11
1,3-Dichl[illegible]obenzene	0.21	---	10	---	---	10*	0.12
p-Isoprop[illegible]oluene	0.24	---	5	---	---	---	0.11
1,4-Dichl[illegible]obenzene	0.26	---	10	---	---	10*	0.12

Concentrat[illegible]n ug/L(Kg)

Table 5.1 (continued)

Example
MDL & PQL for VOLATILES ANALYSIS
by GC/MS

PARAMET	RS	MDL (actual)	8240 PQL	8260 PQL	CLP CRDL	624 MDL	AppIX PQL	524.2 MDL (actual)
1,2-Dichlo	enzene	0.34	---	10	---	---	10*	0.12
n-Butylben	ne	0.35	---	5	---	---	---	0.11
1,2-Dibron Chloroprop	3- e	0.34	---	5	---	---	5	0.18
1,2,3-Trich	obenzene	0.15	---	5	---	---	---	0.18
Hexachlor	itadiene	0.41	---	10	---	---	10*	0.16
Naphthaler		0.43	---	10	---	---	10*	0.10
1,2,4-Trich	obenzene	0.23	---	5	---	---	10*	0.16

Concentration µg/ L(Kg)

Table 5.2

Example MDL & PQL
for SEMI-VOLATILES ANALYSIS
by GC/MS

PARAMETERS	MDL (Water)	MDL (Soil)	8270 PQL (Water)	8270 PQL (Soil)	CLP CRDL (Water)	CLP CRDL (Soil)	625 MDL (Water)	AppIX PQL (Water)
Pyridine	1.72	71.6	---	---	---	---	---	10
N-Nitrosodiethylamine	3.14	52.1	---	---	---	---	---	10
Ethyl Methanesulfonate	2.07	38.6	---	---	---	---	---	10
2-Picoline	1.97	26.2	---	---	---	---	---	10
N-Nitrosomethylethylamine	4.03	162	---	---	---	---	---	10
Methyl methanesulfonate	1.79	36.3	---	---	---	---	---	10
N-Nitrosodimethylamine	2.04	37.4	---	---	---	---	---	10
1,4-Benzoquinone	2.88	53.3	---	---	---	---	---	---
Ethyl methanesulfonate	1.92	40.3	---	---	---	---	---	10
Pentachloroethane	3.48	27.8	---	---	---	---	---	10
Aniline	2.53	75.2	---	---	---	---	---	10
Phenol	1.79	27.9	10	330	10	330	5.7	10
Bis(2-chloroethyl)ether	2.42	30.3	10	330	10	330	3.3	10
2-Chlorophenol	2.84	37.1	10	330	10	330	---	10
1,3-Dichlorobenzene	2.36	38.5	10	330	10	330	1.9	10
1,4-Dichlorobenzene	2.46	37.2	10	330	10	330	4.4	10
Benzyl Alcohol	2.72	25.8	10	330	---	---	---	20
1,2-Dichlorobenzene	2.59	35.3	10	330	10	330	1.9	10
Bis(2-Chloroisopropyl)ether	2.52							
2-Methylphenol	2.38	41.6	10	330	10	330	---	10
2,2-Oxybis(1-chloropropane)	1.87	46.2	---	---	10	330	---	---
Acetophenone	1.99	29.9	---	---	---	---	---	10
1-Nitrosopiperidine	1.73	31.5	---	---	---	---	---	10
4-Nitroso-n-Morpholine	1.56	36.6	---	---	---	---	---	10
o-Toluidine	2.07	83.4	---	---	---	---	---	10
1,2-Dibromo-3-chloropropane	2.19	33.9	---	---	---	---	---	10
4-Methylphenol	2.52	43.5	10	330	10	330	---	10
N-Nitroso-n-propylamine	2.15	35.3	10	330	10	330	---	10
Hexachloroethane	2.29	36.8	10	330	10	330	1.6	10
Nitrobenzene	2.15	28.1	10	330	10	330	1.9	10
1-Nitrosopyrrolidine	1.65	34.2	---	---	---	---	---	10
Isophorone	1.84	29.7	10	330	10	330	2.2	10
2-Nitrophenol	2.37	34.0	10	330	10	330	3.6	10
2,4-Dimethylphenol	2.10	85.3	10	330	10	330	2.7	10
α,α-Dimethylphenethylamine	Nd	nd	---	--	---	--	---	10

Concentration (ug/L or Kg)

Table 5.2 (continued)

Example MDL & PQL
for SEMI-VOLATILES ANALYSIS
by GC/MS

PARAMETERS	MDL (Water)	MDL (Soil)	8270 PQL (Water)	8270 PQL (Soil)	CLP CRDL (Water)	CLP CRDL (Soil)	625 MDL (Water)	AppIX PQL (Water)
Benzoic ac	0.64	61.8	50	1700	---	---	---	---
Bis(2-Chlo thoxy)methane	2.00	27.6	10	330	10	330	5.31	10
O,O,O-Triethylpho horothioate	2.39	44.8	---	---	---	---	---	10
2,4-Dichlor henol	2.35	27.1	10	330	10	330	2.7	10
1,2,4-Trich obenzene	2.14	33.6	10	330	10	330	1.9	10
Naphthalen	2.69	32.5	10	330	10	330	1.6	10
Hexachlorc opene	3.81	40.0	---	---	---	---	---	10
2,6-Dichlo henol	2.10	40.1	---	---	---	---	---	10
4-Chloroan ne	2.76	22.0	10	330	10	330	---	---
Hexachlorc tadiene	2.22	38.2	10	330	10	330	0.9	10
p-Phenyler amine	Nd	nd	---	---	---	---	---	10
N-Nitroso- n-butylamine	1.88	26.7	---	---	---	---	---	10
Safrole	2.07	34.3	---	---	---	---	---	10
4-Chloro-3 ethylphenol	1.47	33.3	10	330	10	330	3.0	20
2-Methyln thalene	1.76	28.2	10		10	330	---	10
1,2,4,5-Te chlorobenzene	2.84	39.8	---	---	---	---	---	10
Hexachlorc clopentadiene	2.55	46.7	10	330	10	330	---	10
2,4,6-Trich rophenol	1.83	49.9	10	330	10	330	2.7	10
2,4,5-Trich rophenol	3.27	48.6	10	330	50	1700	---	10
2-Chloron thalene	1.62	31.3	10	330	10	330	1.9	10
Isosafrole	2.13	25.0	---	---	---	---	---	10
1,4-Naphth uinone	1.37	38.1	---	---	---	---	---	10
2-Nitroani e	1.32	23.7	50	1700	50	1700	---	50
Dimethylp alate	1.51	28.4	10	330	10	330	1.6	10
Acenaphth ne	1.34	20.3	10	330	10	330	3.5	10
3-Dinitrob zene	1.42	33.3	---	---	---	---	---	10
2,6-Dinitro luene	0.94	32.4	10	330	10	330	1.9	10
3-Nitroani e	1.14	35.6	50	1700	50	1700	---	50
Acenaphth e	0.98	30.4	10	330	10	330	1.9	10
2,4-Dinitro henol	2.89	32.4	50	1700	50	1700	42	50
4-Nitrophe l	4.06	67.6	50	1700	50	1700	2.4	50
Dibenzofu n	1.01	34.0	10	330	10	330	---	10
Pentachlor enzene	1.96	28.4	---	---	---	---	---	10
2,4-Dinitro luene	1.21	27.2	10	330	10	330	5.7	10
1-Naphthyl ine	nd	nd	---	---	---	---	---	10
2-Naphthy ine	nd	nd	---	---	---	---	---	10
2,3,4,6-Te chlorophenol	nd	nd	---	---	---	---	---	10

Concentration: ug/L or ug/Kg

Table 5.2 (continued)

Example MDL & PQL
for SEMI-VOLATILES ANALYSIS
by GC/MS

PARAMETERS	MDL (Water)	MDL (Soil)	8270 PQL (Water)	8270 PQL (Soil)	CLP CRDL (Water)	CLP CRDL (Soil)	625 MDL (Water)	AppIX PQL (Water)
Diethyl phthalate	3.21	27.0	10	330	10	330	1.9	10
5-Nitro-o-toluidine	1.62	37.1	---	---	---	---	---	10
Thionazin	1.31	29.4	---	---	---	---	---	
4-Chlorophenyl phenyl ether	1.69	22.6	10	330	10	330	4.2	10
Fluorene	1.27	24.4	10	330	10	330	1.9	10
4-Nitroaniline	2.06	27.9	50	1700	50	1700	---	50
Atrazine	1.07		---	---	---	---	---	---
4,6-Dinitro-2-methylphenol	1.51	54.7	50	1700	50	1700	24	50
N-Nitrosodiphenylamine (1)	0.79	32.0	10	330	10	330	1.9	10
Azobenzene	0.73	23.7	---	---	---	---	---	---
2,4,6-Tribromophenol	3.88	62.4	---	---	---	---	---	
sym-Trinitrobenzene	2.03	44.9	---	---	---	---	---	10
4-Bromophenyl phenyl ether	0.97	23.9	10	330	10	330	1.9	10
Sulfotep	1.20	23.3	---	---	---	---	---	10
Phorate	1.41	33.6	---	---	---	---	---	10
Diallate	1.02	30.7	---	---	---	---	---	10
Hexachlorobenzene	1.73	23.1	10	330	10	330	1.9	10
Phenacetin	1.15	36.5	---	---	---	---	---	10
Dimethoate	1.01	32.7	---	---	---	---	---	10
4-Aminobiphenyl	nd	nd	---	---	---	---	---	10
Pentachlorophenol	1.73	54.3	50	1700	50	1700	3.6	50
Pentachloronitrobenzene	2.04	31.4	---	---	---	---	---	10
Phenanthrene	1.29	19.3	10	330	10	330	5.4	10
Pronamide	1.46	38.7	---	---	---	---	---	10
Anthracene	0.83	25.4	10	330	10	330	1.9	10
Disulfoton	4.34	30.3	---	---	---	---	---	10
Dinoseb	3.34	34.9	---	---	---	---	---	10
Carbazole	0.96	29.8	---	---	---	---	---	
Methyl parathion	0.94	127	---	---	---	---	---	10
Di-n-Butylphthalate	1.95	42.6	10	330	10	330	2.5	10
4-Nitroquinoline-1-oxide	nd	nd	---	---	---	---	---	10
Parathion	1.30	41.3	---	---	---	---	---	10
Methapyrilene	5.07	70.9	---	---	---	---	---	10
Isodrin	1.66	33.3	---	---	---	---	---	10
Fluoranthene	1.07	21.2	10	330	10	330	2.2	10
Benzidine	Nd	nd	---	---	---	---	44	---
Pyrene	0.48	25.9	10	330	10	330	1.9	10

Concentration are in ug/Kg

Table 5.2 (continued)

Example MDL & PQL for SEMI-VOLATILES ANALYSIS by GC/MS

PARAMETERS	MDL (Water)	MDL (Soil)	8270 PQL (Water)	8270 PQL (Soil)	CLP CRDL (Water)	CLP CRDL (Soil)	625 MDL (Water)	ApplX PQL (Water)
Aramite	1.89	30.0	---	---	---	---	---	10
p-(Dimethyl[illegible]mo)-azobenzene	1.94	25.9	---	---	---	---	---	10
Chlorobenzi[illegible]e	1.88	29.8	---	---	---	---	---	10
Kepone	3.47	79.0	---	---	---	---	---	10
Famphur	22.7	277	---	---	---	---	---	10
Butyl benzy[illegible]hthalate	1.58	13.2	10	330	10	330	2.5	10
3,3'-Dichlor[illegible]enzidine	1.82	40.3	10	330	10	330	16.5	20
2-Acetylami[illegible]fluorene	1.45	31.6	---	---	---	---	---	10
Benzo(a)ant[illegible]acene	1.43	23.0	10	330	10	330	7.8	10
3,3'-Dimeth[illegible]enzidine	nd	nd	---	---	---	---	---	50
Chrysene	0.90	29.2	10	330	10	330	2.5	10
Bis(2-ethylh[illegible]yl)phthalate	1.21	42.7	10	330	10	330	2.5	10
Di-n-octyl p[illegible]halate	7.88	25.2	10	330	10	330	2.5	10
Benzo(b)flu[illegible]anthene	1.24	47.3	10	330	10	330	4.8	10
7,12-Dimet[illegible]benz(a)-anthracene	1.21	34.8	---	---	---	---	---	10
Benzo(k)flu[illegible]anthene	2.41	53.9	10	330	10	330	2.5	10
Benzo(a)py[illegible]e	1.51	21.6	10	330	10	330	2.5	10
3-Methylch[illegible]anthrene	2.19	26.9	---	---	---	---	---	10
Indeno(1,2,[illegible]3-cd)pyrene	1.28	27.1	10	330	10	330	3.7	10
Dibenzo(a,[illegible]h)anthracene	1.82	18.6	10	330	10	330	2.5	10
Benzo(g,h,i[illegible])perylene	1.61	28.3	10	330	10	330	4.1	10

Concentration [illegible]g/L/Kg)

Table 5.3

Example
MDL & PQL for PESTICIDES/PCB
by GC

PARAME	RS	8081 MDL (actual)	608 MDL (actual)	8081 PQL (Water)	8081 PQL (Soil)	CLP CRDL (Water)	CLP CRDL (Soil)	608 MDL (Water)	AppIX PQL (Water)
α-BHC		0.0005	0.0004	0.03	2.01	0.05	1.7	0.003	0.05
β-BHC		0.0010	0.0009	0.06	4.02	0.05	1.7	0.006	0.05
δ-BHC		0.0007	0.0009	0.09	6.03	0.05	1.7	0.009	0.10
γ-BHC (L	ane)	0.0007	0.0006	0.04	2.68	0.05	1.7	0.004	0.05
Heptachlo		0.0018	0.0005	0.03	2.01	0.05	1.7	0.003	0.05
Aldrin		0.0008	0.0005	0.04	2.68	0.05	1.7	0.004	0.05
Heptachlo	poxide	0.0008	0.0010	0.83	55.61	0.05	1.7	0.083	1.00
Endosulfa		0.0012	0.0014	0.14	9.38	0.05	1.7	0.014	0.10
Dieldrin		0.0008	0.0007	0.02	1.34	0.10	3.3	0.002	0.05
p,p'-DDE		0.0014	0.0009	0.04	2.68	0.10	3.3	0.004	0.05
Endrin		0.0013	0.0012	0.06	4.02	0.10	3.3	0.006	0.10
Endosulfa	l	0.0010	0.0012	0.04	2.68	0.10	3.3	0.004	0.05
p,p'-DDD		0.0010	0.0011	0.11	7.37	0.10	3.3	0.011	0.10
Endosulfa	ulfate	0.0011	0.0010	0.66	44.22	0.10	3.3	0.066	0.50
p,p'-DDT		0.0009	0.0012	0.12	8.04	0.10	3.3	0.012	0.10
Methoxyc	r	0.0063	0.0057	1.76	117.92	0.50	17.0	---	2.00
Endrin Ke	ne	0.0013	0.0010	---	---	0.10	3.3	---	---
Endrin Al	nyde	0.0052	0.0041	0.23	15.41	0.10	3.3	0.023	0.20
γ-Chlorda		0.0006	0.0005	---	---	0.05	1.7	---	
α-Chlorda		0.0010	0.0010	---	---	0.05	1.7	---	
Tech. Chl	dane	0.050	1.0 [1]	0.14	9.38	---	---	0.014	
Toxaphen		0.400	5.0 [1]	2.4	160.80	5.0	170.0	0.24	2.0
Aroclor 1		0.050	0.050	nd	nd	1.0	33.0	---	50 [2]
Aroclor 1	1 [2]		1.0	0.054	3.7.0	1.0	67.0	---	50 [2]
Aroclor 1	2 [2]		2.0	nd	nd	2.0	33.0	---	50 [2]
Aroclor 1	2 [2]		1.0	nd	nd	1.0	33.0	0.065	50 [2]
Aroclor 1	8 [2]		1.0	nd	nd	1.0	33.0	---	50 [2]
Aroclor 1	4 [2]		1.0	nd	nd	1.0	33.0	---	50 [2]
Aroclor 1	0	0.050	0.050	0.90	70.0	1.0	33.0	---	50 [2]

Concentrati n ug L(Kg)

[1] Based lowest calibration level.

[2] The PC show is an average value of PCB congeners.

Table 5.4

Example MDL & PQL
for POLYNUCLEAR AROMATICS
by HPLC

PARAME	ERS	MDL (Water)	MDL (Soil)	PQL (Soil)	8310 PQL (Water)	8310 PQL (Soil)	Illinois AQL (Water)	Illinois AQL (Soil)	
Naphthale		0.28	9.22	500	18.0	1206	10.0	660	
Acenaphth	ene	0.50	16.5	500	23.0	1541	10.0	660	
Acenaphth	e	0.50	1.67	500	18.0	1206	18.0	660	
Fluorene		0.03	0.93	112	2.1	141	2.1	140	
Phenanthr	e	0.02	0.80	500	6.4	429	6.4	660	
Anthracen		0.02	0.73	500	6.6	442	6.6	660	
Fluoranth	e	0.07	2.36	500	2.1	141	2.1	660	
Pyrene		0.08	2.50	144	2.7	181	2.7	180	
Benzo(a)a	racene	0.04	1.43	7	0.13	8.7	0.13	8.7	
Chrysene		0.03	0.83	80	1.5	101	1.5	100	
Benzo(b)f	oranthene	0.03	1.00	8.8	0.18	12.1	0.18	11.0	
Benzo(k)f	oranthene	0.07	2.36	8.8	0.17	11.4	0.17	11.0	
Benzo(a)p	ene	0.06	2.00	12.0	0.23	15.4	0.23	15.0	
Dibenzo(a	anthracene	0.12	4.03	16.0	0.30	20.1	0.30	20.0	
Benzo(ghi	erylene	0.10	3.33	41.0	0.76	50.9	0.76	51.0	
Indeno(1,2	-cd)pyrene	0.04	1.17	23.0	0.43	28.8	0.43	29.0	

Concentratio | µg/L(Kg)

Table 5.5

Example
MDL for HERBICIDES
by GC

PARAME	***RS***	*8151* ***MDL*** *(Actual)*		*515.1* ***MDL*** *(Water)*	*8151* ***MDL*** *(Water)*	*8151* ***MDL*** *(Soil)*	
Dalapon		0.004		1.3	1.3	0.12	
Dicamba		0.001		0.081	0.081	---	
Dichloropr		0.004		0.26	0.26	---	
2,4-D		0.002		0.2	0.2	0.11	
Pentachlor	henol	0.001		0.076	0.076	0.16	
2,4,5-TP (	vex)	0.001		0.075	0.075	0.28	
2,4,5-T		0.005		0.08	0.08	---	
Dinoseb		0.003		0.19	0.19	---	
2,4-DB		0.004		0.8	0.8	---	

Concentratio µg/ L(Kg)

Table 5.6

Example
MDL & IDL
for METALS by ICP
in Water

ELEME	*WAVE-LENGTH*	*TJA Env II IDL*	*200.7 MDL*	*6010A MDL*	*6010A PQL*
Aluminum	308.22	0.042	0.072	0.019	0.200
Antimony	206.83	0.033	0.013 *	0.038	0.300
Arsenic	193.70	0.030	0.005	0.016	0.500
Barium	493.41	0.002	0.001	0.001	0.020
Beryllium	313.11	0.002	0.0002	0.0004	0.003
Bismuth	223.06			0.086	
Boron	249.68	0.030	0.011	0.006	0.200
Cadmium	226.50	0.005	0.002	0.005	0.040
Calcium	317.93	0.015	0.012	0.025	0.400
Chromium	267.71	0.004	0.003	0.005	0.070
Cobalt	228.61	0.007	0.002	0.003	0.070
Copper	327.40	0.030	0.002	0.002	0.060
Iron	259.94	0.010	0.013	0.008	0.100
Lithium	670.78	0.030	0.011	0.088	0.050
Lead	220.35	0.043	0.002 *	0.034	0.400
Magnesium	285.21	0.020	0.013	0.030	0.200
Manganese	257.61	0.001	0.002	0.002	0.015
Molybdenu	202.03	0.014	0.005	0.024	0.150
Nickel	231.60	0.012	0.003	0.008	0.150
Phosphorus	214.91			0.146	
Potassium	766.49	0.121	0.055	0.145	0.500
Selenium	196.03	0.046	0.017 *	0.034	0.750
Silicon	288.s	0.033	0.071	0.047	0.500
Silver	328.07	0.004	0.005	0.003	0.070
Sodium	589.99	0.061	0.058	0.031	0.500
Strontium	407.77	0.004	0.001	0.001	0.050
Sulfur	180.73			0.058	
Thallium	190.86	0.063	0.028 *	0.026	0.400
Tin	189.99	0.034	0.013	0.024	0.200
Titanium	334.94	0.005	0.001	0.005	0.060
Vanadium	292.40	0.003	0.002	0.004	0.080
Zinc	213.86	0.005	0.010	0.004	0.020

Concentrati in mg/L(Kg)

* 200.7 is t an approved method for these elements per SWDA.

Table 5.7

Example
MDL for METALS
by ATOMIC ABSORPTION

ELEMEI		GFAAS METHOD	GFAAS MDL	AAS METHOD	AAS MDL	CVAAS METHOD	CVAAS MDL
Antimony		200.9	1.92				
Arsenic		200.9	0.85				
Beryllium		---	0.09	210.1			
Cadmium		213.2	0.13				
Calcium		---	---	215.1			
Chromium		218.2	0.31				
Copper		---	1.1	220.1			
Lead		200.9	2.20				
Magnesium		---	---	242.1			
Manganese		---	---	243.1			
Mercury		---	---	---	---	245.1	0.1
Nickel		---	---	249.1			
Potassium		---	---	258.1			
Selenium		200.9	0.84				
Silver		272.2	1.16				
Sodium		---	---	273.1			
Thallium		200.9	0.42				
Zinc		---	---	289.1			

Concentrati[illegible] in µg/L(Kg)0

GFAAS = G[illegible]phite Furnace Atomic Absorption
AAS = Dire[illegible]Aspiration Atomic Absorption
CVAAS = C[illegible]d Vapor Atomic Absorption

Table 5.8

MDL for METALS
by ATOMIC ABSORPTION

SW-846 METHODS

ELEMEI	GFAAS METHOD	GFAAS MDL	AAS METHOD	AAS MDL	CVAAS METHOD	CVAAS MDL
Antimony	7041	1.30	---	---		
Arsenic	7061	0.90	---	---		
Beryllium	7091	0.04	7090			
Cadmium	7131	0.08	---	---		
Calcium	---	----	7140			
Chromium	7191	0.40	---	---		
Copper	7211	155	7210			
Lead	7421	0.55	---	---		
Magnesium	---	---	7450			
Manganese	---	---	7460			
Mercury	---	---	---	---	7470	0.180
Nickel	---	---	7520			
Potassium	---	---	7610			
Selenium	7741	0.92	---	---		
Silver	7761	0.11	---	---		
Sodium	---	---	7770			
Thallium	7841		----	---		
Zinc	---	---	7950			

Concentrat s in µg/L(Kg)

GFAAS = G hite Furnace Atomic Absorption
AAS = Dire spiration Atomic Absorption
CVAAS = C l Vapor Atomic Absorption

Section 6

DA A RECORDING, REDUCTION, VALIDATION AND REPORTING

To produ data of high quality, the following steps are followed at Environmetrics:

- Data Recording and Data Maintenance
- Data Reduction
- Data Validation
- Data Review
- Data Reporting

Data Rec ding and Data Maintenance

The anal will record raw data produced by each section of the laboratory in notebooks or LIMS nerated worksheets. Notebooks are issued by the QA officer. The date of issuance d the laboratory section to which the notebook was issued is recorded in the Log of L book. Notebooks will be archived by the QA manager when they are filled or when the mployee will no longer use the notebook. Computer printouts (GC/MS, ICP, AAS) an hromatograms (GC, HPLC, and IC) will become a part of the project file.

Record k ping must allow historical reconstruction of all laboratory activities that produce resultant sample analytical data. This shall include interlaboratory transfers of sampl and sample extracts.

The labo ory shall maintain the following records:

1. I ntity of personnel involved in sampling (when possible), preparation, and te ing;
2. S nple container, preservation, and holding time compliance;
3. S nple identification, laboratory id, receipt and login information, and a eptance;
4. S nple storage and tracking: including shipping receipts (air bills), transmittal f ns, chain of custody and internal routing records;
5. S nple preparation: extraction and/or digestions procedures, cleanup and s aration procedures, volumes, weights, instrument printouts, meter readings, a reagents;
6. S nple analysis: all original raw data including worksheets and data output r ords including lab identification, date of analysis, instrument;
7. E ipment: use or injection log and maintenance logs;
8. C culations: written procedures (SOPs), all raw data and supporting information n ded to recreate calculation;

9. Q data: all associated tune, calibration, calibration verification, MS, MSD, LCS, m od blank data;
10. C es of final reports;
11. A ived SOPs;
12. A correspondence between the laboratory and the clients;
13. A corrective actions, audits, and audit responses;
14. P ample results and raw data
15. St dards and analytical reagents:
    - Traceable to national standards (if not possible, analysis of PE samples can be used to demonstrate that equipment is properly calibrated).
    - Certificate of the origin, purity, and traceability.
    - Date of receipt, date opened, and expiration date.
    - Log of working and intermediate standards: including date of preparation, preparer's initials, concentration, and traceability.
16. A inistrative records:
    - Personnel qualifications, education, experience, and training.
    - IDMP and any required repetitions of IDMP for each analyst.
    - Log of names, initials, and signatures.
17. R ord Retention:
    - Records pertaining to drinking water analyses that are associated with lab accreditation shall be retained for 10 years. Lead and copper analysis shall be retained for 12 years.
    - Records pertaining to environmental analyses that are associated with lab accreditation shall be retained for 5 years unless a long retention period is stipulated by another regulation or client.

Data Red tion

Environm rics analysts are responsible to reduce the raw data according to the requireme s indicated in the protocols. Sample matrix, sample size, % moisture, final volume, a ount injected, and other pertinent information will be used to calculate the concentra n of the desired analytes. In most protocols software is written to reduce the data auto tically. However, the analyst will review the data for correct identification and quant cation.

Data Vali tion

Data vali ion is performed by checking the results of an analytical batch or case of samples. reviewing a batch of samples, the QA officer, the laboratory director, and the section m agers will be checking the following items:

- Sample numbers on the chain of custody versus the submitted data for the batch.
- Deviations from the agreed protocol and the reasons for the deviations.
- Calibration data, method blanks, proper sample and QC sample frequency.
- Method blank, surrogate, lab control sample, (LCS), matrix spike recoveries and detection limits.
- Precision and accuracy and their control limits.
- Corrective action report for any of the above items if there was an out of control event.

Section managers and chemists can disallow the use of the data and will discuss the reasons with the QA officer or lab manager.

Data Review

After all data have been validated, the data management group will review the project/case file for completeness. The results will be entered into a summary report form. A full data package will be prepared if the client has requested copies of chromatograms, spectra, sample preparation forms, lab notebooks, etc. The final report will also be reviewed for data entry against the raw data in the file. A copy of the report and all the background information will be kept in the client's file.

Data Reporting

Environmetrics, Inc. provides three levels of reporting. The QA-5 level is the standard level provided for routine analysis reports. The QA-4 and QA-5 reporting levels are provided on request prior to the initiation of the project.

- QA-5 Reporting Level - (Standard Reporting)

  The deliverables for Reporting Level 5 includes the following standard information:

  1. sample results
  2. blank results
  3. chain of custody
  4. surrogate recovery results

  Level 5 is the normal reporting level with fees included on the Environmetrics Fee Schedule.

- QA-4 Reporting Level

  The QA-4 Reporting Level requires the analysis of matrix spikes, and matrix spike duplicates. The deliverables include the following items:

  1. QA-5 standard reporting
  2. cover letter
  3. laboratory control sample results
  4. matrix spike and duplicate results

  The matrix spikes and matrix spike duplicates will be charged as individual samples.

- QA-3 Reporting Level

  The QA-3 Reporting Level includes:

  1. items listed in the QA-5 and QA-4 reporting levels
  2. the results of the initial calibration
  3. the continuing calibration and blanks
  4. instrument printouts
  5. log book pages
  6. tentatively identified compounds

  The fees for QA-3 reporting will be based on the scope of work. All data on hard copy form and diskettes will be maintained by Environmetrics, Inc. for three (3) years or longer if requested by clients.

Section 7

INTERNAL QUALITY CONTROL

Introduction

Internal Quality Control procedures include blanks, replicates, spiked samples, control charts, internal standards, QC samples, surrogate spikes, calibration standards, reagent checks, and reference samples.

For every procedure listed in the scope of work, mandatory QC procedures will be performed. Environmetrics will use the specified QC except where additional QC will enhance data quality. Such changes will be documented appropriately in the laboratory's Quality Control Plan. The frequency of use required for QC procedures described below will be discussed with each client depending on the use of data and the existing regulations. However, SW-846 will be followed for most analytical tasks.

Blanks

Blanks may consist of field trip blanks, field equipment blanks and/or laboratory blanks.

Based on the client's request, a field trip blank will be used when the samples to be obtained will be analyzed for volatile organic compounds. The field trip blank will consist of organic-free distilled water placed in the same type of bottle in which the samples will be placed, preservatives added if necessary, and carried by the sampling team in the same container as the field samples.

Clients may submit a field equipment blank that will be used to determine sample adequacy of decontamination procedures in cases where samples come in contact with the sampling equipment.

Laboratory blanks are used to determine cleanliness, operation of equipment, etc. within the laboratory. The laboratory blank is carried through all stages of sample preparation and measurement. For organic analyses, surrogates and internal standards (if appropriate) are added to the blank. The laboratory blank should be analyzed prior to analyzing samples. In addition, a blank should be analyzed at a minimum of one is twenty samples or one for each analytical batch, whichever is more frequent. If contamination is found, it should be:

- Less then the laboratory's MDL for the analyte or less than the level of acceptable blank contamination specified in the approved quality assurance plan.
- Less than 5% of the regulatory level of the associated blank.

- Or less than 5% of the sample result of the same analyte.

The reason for the problem must be identified, corrected, and all subsequent standards, samples, etc. must be reanalyzed. Equipment should be clean enough so that the instrument blank shows no contamination above the instrument detection limit. The laboratory is responsible for making sure that laboratory blanks are run at the required frequency.

Laboratory Control Samples

Laboratory control samples shall be included with each analytical batch. The LCS consists of an aliquot of a clean matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis, indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the lab can perform the analysis in a clean matrix. The laboratory shall not use the analysis of matrix spike samples to override, ignore, or replace an LCS analysis that fails to meet criteria. LCS acceptance criteria are defined either in the individual methods or by control charting.

Field Duplicate Samples

Field duplicate samples are used to establish the representativeness of the field sampling. Environmetrics uses field duplicates to establish a background on sample homogeneity. A field duplicate is obtained by taking two separate samples at the same location in a presumably homogeneous area. Results from analysis of duplicate field samples may be used to calculate precision, but will be used to interpret the homogeneity of samples.

Laboratory Replicate Samples

Laboratory replicate samples are normally obtained by taking a presumably homogeneous field sample, splitting the sample, and carrying both samples through the entire analytical procedure including sample preparation. In some cases it is necessary to consume the entire sample during analysis (e.g., when the container walls must be washed). In this case, the field sampling team must obtain two samples. The analytical results from these samples are used for calculating the precision of the analyses.

Matrix Spike/Matrix Spike Duplicate

Laboratory matrix spike/matrix spike duplicates are obtained by taking a presumably homogeneous field sample, splitting the sample, spiking each split with predetermined quantities of stock solutions of certain analytes and carrying both samples through the entire analytical procedure, including sample preparation. The samples for matrix

spike/matrix spike duplicate are either chosen on a rotating basis amongst clients, locations, and waste streams or as directed by the client. In general, a matrix spike/matrix spike duplicate must be analyzed with every batch or every 20 samples when applicable. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated to assess the precision. (See Tables 7.1, 7.2, and 7.3 for spiking compounds and acceptance limits.)

If the approved test does not specify the spiking analytes, 10% of the analytes listed in the approved test method or a minimum of three analytes of interest must be spiked.

Method of Standard Additions

The method of Standard Additions (MSA) for metals analyzed by Furnace AA is required if recoveries of metals from the spiked sample is not between 85 and 115%. A standard addition is the addition of one or more required analytes to a sample immediately preceding the measurement procedure.

Surrogate Compounds

Surrogate compounds are added to all samples, standards, and blanks, whenever possible, when conducting analyses by approved test methods utilizing organic chromatograpy.

- Surrogate compounds and their acceptance limits for Volatile Organic Compounds by GC/MS are summarized in Table 7.1.
- Surrogate compounds and their acceptance limits for Semi-Volatile Organic Compounds by GC/MS are summarized in Table 7.2.
- Surrogate compounds and their acceptance limits for Volatile and Semi-Volatile Organic Compounds by GC are summarized in Table 7.3.

If surrogate acceptance limits are not met, perform the following:

- Check for errors in the calculations, surrogate solutions or internal standards. Examine the chromatogram for interfering peaks and integrated peak areas.
- Check instrument performance. If a problem is identified, correct the problem and re-analyze the extract.
- If sample dilution to the point that the surrogate recoveries can not be measured, the surrogate recoveries from a less diluted aliquot may be used to demonstrate that the surrogates were within acceptance limits.

- If on[redacted] one surrogate is found to exceed the high acceptance limits and sample is a non-[redacted]tect, then the sample need not be re-extracted and re-analyzed but reported as a [redacted]n-detect with comments in the narrative.

- If no[redacted]nstrument problem is found, the sample should be re-extracted and re-anal[redacted]ed.

- If, u[redacted]n re-analysis, the recovery is again not within limits, report the data as an "est[redacted]ated concentration". If the recovery is within limits in the re-analysis, repo[redacted] the re-analysis data. If the holding time for the method has expired prior to the [redacted] analysis, provide both the original and the re-analysis results and note the hold[redacted]g time problem.

- If su[redacted]ogate acceptance limits are not met in the blank, LCS, or check standard, all asso[redacted]ated samples must be re-extracted and re-analyzed.

Internal Sta[redacted]lards

Internal sta[redacted]lards will be used for quantitative analysis for all Volatile and Semi-Volatile Organics. [redacted]e appropriate internal standards are specified in appropriate protocols.

Initial Dem[redacted]nstration of Method Proficiency (IDMP)

Each analy[redacted] shall perform an IDMP study prior to initiation of sample analyses, unless the IDMP [redacted] not applicable to the approved test method, such as, TSS, TDS, TVS, TS, pH, color, [redacted]lor, temperature, dissolved O_2 or turbidity. The IDMP shall be repeated if there is a i[redacted]rument or method change.

IDMP sho[redacted]d be perfomed in accordance with the approved test method unless not available. [redacted] the IDMP procedure is not defined the following steps shall be performed:

A) A QC [redacted]eck sample shall be obtained from a certified source. If not available, the QC check [redacted]mple may be preapred by the lab using calibration standards that are prepared at a di[redacted]rent time than those used in instrument calibration.

B) Four a[redacted]quots of the QC check sample are prepared at a concentration of approx[redacted]nately 10 times the method-stated or lab calculated MDL.

C) Analy[redacted] the four aliquots according to the approved test method.

D) From t four results, calculate the average recovery and standard deviation for each analyte

E) For ea analyte, compare the standard deviation and average recovery to the corres nding acceptance criteria, for precision and accuracy in the approved test metho If the standard deviation and average recovery for all analytes meet the accept ce criteria, the analysis of actual samples may begin. If any one of the analyt exceed the acceptance range, the performance is unacceptable for that analyt

F) When e standard deviation or the average recovery of one or more of the tested analyt does not meeet the acceptance criteria, the analyst shall locate and correct the proble and repeat the IDMP.

Method D ection Limits

The labor ory shall determine MDLs using the procedures specified in 40 CFR Appendix , unless the approved test method specifies the procedure for MDL determina n or the determination of an MDL is not applicable to the approved test method, s h as, , TSS, TDS, TVS, TS, pH, color, odor, temperature, dissolved O_2 or turbidity.

A) Analy a minimum of seven replicates to determine the MDL.
- f seven replicates are analyzed, all analytical results shall be used to calculate he MDL.
- f more than seven replicates are analyzed, the laboratory shall only exclude nalytical results which the laboratory determines are outliers by utilizing a statistical outlier test.

B) The l oratory may define the MDL for multi-component analyses as the lowest conce ration for which pattern recognition is possible.

C) MDL for each approved test method shall be determined annually or when there is a chang in instrument type.

D) An M L is valid when:
- the calculated MDL is greater than 1/10 the MDL spiking concentration;
- the MDL spiking concentration is greater than the calculated MDL;
- the criteria for acceptable replicate percent recovery is met; and
- for drinking water, the MDLs are equal to or less than established regulatory limits.
-

E) The DL study shall be repeated if the criteria in subsection D are not met.

Table 7.1

Method Acceptance Criteria for Surrogates and Matrix Spiking Compounds in Water

VOLATILE METHODS

	SW-846 8240 IN % REC		SW-846 8260 IN % REC		METHOD CLP 3/90 IN % REC		METHOD 624 IN % REC		METHOD 524.2 IN % RPD	
BLANKS	1 in 20		1 in 20		1 in 12 hr		1 per day		1 per batch	
MS/MSD	1 in 20		1 in 20		1 in 20		1 MS in 20		Only w/Matrix	
SURROGATE	LOW/HIGH		LOW/HIGH		LOW/HIGH		LOW/HIGH		LOW/HIGH	
1,2-Dichloroeth -d4	76 / 114		NA		76 / 114		NS		NA	
Toluene-d8	88 / 110		88 / 110		88 / 110		NA		NA	
4-Bromofluorob zene	86 / 115		86 / 115		86 / 115		NS		80 / 120	
Dibromofluoror ane	NA		86 / 118		NA		NA		NA	
1,2-Dichlorobet e-d4	NA		NA		NA		NA		80 / 120	
SPIKING COMPOUNDS	LOW/HIGH	RPD	LOW/HIGH	RPD	LOW/HIGH	RPD	LOW/HIGH	RPD	LOW/HIGH	RPD
1,1-Dichloroeth	61 / 145	14	61 / 145	14	61 / 145	14	59 / 155	NS	80 / 120	NS
Benzene	76 / 127	11	76 / 127	11	76 / 127	11	37 / 151	NS	80 / 120	NS
Trichloroethene	71 / 120	14	71 / 120	14	71 / 120	14	71 / 157	NS	80 / 120	NS
Toluene	76 / 125	13	76 / 125	13	76 / 125	13	47 / 150	NS	80 / 120	NS
Chlorobenzene	75 / 130	13	75 / 130	13	75 / 130	13	37 / 160	NS	80 / 120	NS

SEMIVOLATILE METHODS

	SW-846 8270 IN % REC		METHOD CLP 3/90 IN % REC		METHOD 625 IN % REC	
BLANKS	1 in 20		1 in 20		1 per batch	
MS/MSD	1 in 20		1 in 20		1 LFM in 20	
SURROGATE	LOW/HIGH		LOW/HIGH		LOW/HIGH	
2-Fluoropheno	21 / 100		21 / 110		NS	
Phenol-d6 (d5)	10 / 94		10 / 110		NS	
2-Chlorophenol	NA		33 / 110		NA	
1,2-Dichlorobe ne-d4	NA		16 / 110		NA	
Nitrobenzene-d	35 / 114		35 / 114		NS	
2-Fluorobiphe	43 / 116		43 / 116		NA	
2,4,6-Tribrom enol	10 / 123		10 / 123		NA	
Terphenyl-d14	33 / 141		33 / 141		NA	
SPIKING COMPOUNDS	LOW/HIGH	RPD	LOW/HIGH	RPD	LOW/HIGH	RPD
Phenol	12 / 89	42	12 / 110	42	5 / 112	NS
2-Chlorophenol	27 / 123	40	27 / 123	40	23 / 134	NS
1,4-Dichlorobe ne	36 / 97	28	36 / 97	28	20 / 124	NS
N-Nitroso-di-n opylamine	41 / 116	38	41 / 116	38	D / 230	NS
1,2,4-Trichlor nzene	39 / 98	28	39 / 98	28	44 / 142	NS
4-Chloro-3-me phenol	23 / 97	42	23 / 97	42	22 / 147	NS
Acenaphthene	46 / 118	31	46 / 118	31	47 / 145	NS
4-Nitrophenol	10 / 80	50	10 / 80	50	D / 132	NS
2,4-Dinitrotol	24 / 96	38	24 / 96	38	39 / 139	NS
Pentachloropl	9 / 103	50	9 / 103	50	14 / 176	NS
Pyrene	26 / 127	31	26 / 127	31	52 / 115	NS

" REC = ercent Recovery
RPD = ve Percent Deviation
NA = Not Applicable to This Method
NS = Not Specified for This Method

Table 7.2

Method Acceptance Criteria for Surrogates and Matrix Spiking Compounds in Soil

VOLATILE METHODS

	SW-846 8240 IN % REC	SW-846 8260 IN % REC	METHOD CLP 3/90 IN % REC	METHOD 624 IN % REC	METHOD 524.2 IN % REC
BLANKS	1 in 20	1 in 20	1 in 12 hr	1 per day	1 per batch
MS/MSD	1 in 20	1 in 20	1 in 20	1 MS in 20	w/Matrix Effect
SURROGAT	LOW/HIGH	LOW/HIGH	LOW/HIGH	LOW/HIGH	LOW/HIGH
1,2-Dichloroeth -d4	70 / 121	NA	70 / 121	FWMO	FWMO
Toluene-d8	81 / 117	81 / 117	84 / 138	FWMO	FWMO
4-Bromofluorob zene	74 / 121	74 / 121	59 / 113	FWMO	FWMO
Dibromofluoror hane	NA	80 / 120	NA	FWMO	FWMO
1,2-Dichlorobe ne-d4	NA	NA	NA	FWMO	FWMO
SPIKING CO POUNDS	LOW/HIGH RPD	LOW/HIGH RPD	LOW/HIGH RPD	LOW/HIGH RPD	LOW/HIGH RPD
1,1-Dichloroeth	59 / 172 22	59 / 172 22	59 / 172 22	FWMO	FWMO
Benzene	66 / 142 21	66 / 142 21	66 / 142 21	FWMO	FWMO
Trichloroethene	62 / 137 24	62 / 137 24	62 / 137 24	FWMO	FWMO
Toluene	59 / 139 21	59 / 139 21	59 / 139 21	FWMO	FWMO
Chlorobenzene	60 / 133 21	60 / 133 21	60 / 133 21	FWMO	FWMO

SEMIVOLATILE METHODS

	SW-846 8270 IN % REC	METHOD CLP 3/90 IN % REC	METHOD 625 IN % REC
BLANKS	1 in 20	1 in 20	1 per batch
MS/MSD	1 in 20	1 in 20	1 LFM in 20
SURROGAT	LOW/HIGH	LOW/HIGH	LOW/HIGH
2-Fluoropheno	25 / 121	25 / 121	FWMO
Phenol-d6 (d5)	24 / 113	24 / 113	FWMO
2-Chlorophenc 4	NA	20 / 130	FWMO
1,2-Dichlorobe ne-d4	NA	20 / 130	FWMO
Nitrobenzene-	23 / 120	23 / 120	FWMO
2-Fluorobiphe	30 / 115	30 / 115	FWMO
2,4,6-Tribrom enol	19 / 122	19 / 122	FWMO
Terphenyl-d14	18 / 137	18 / 137	FWMO
SPIKING C MPOUNDS	LOW/HIGH RPD	LOW/HIGH RPD	LOW/HIGH
Phenol	26 / 90 35	26 / 90 35	FWMO
2-Chlorophen	25 / 102 50	25 / 102 50	FWMO
1,4-Dichlorob ene	28 / 104 27	28 / 104 27	FWMO
N-Nitroso-di-n opylamine	41 / 126 38	41 / 126 38	FWMO
1,2,4-Trichlor nzene	38 / 107 23	38 / 107 23	FWMO
4-Chloro-3-m phenol	26 / 103 33	26 / 103 33	FWMO
Acenaphthene	31 / 137 19	31 / 137 19	FWMO
4-Nitrophenol	11 / 114 50	11 / 114 50	FWMO
2,4-Dinitrotol e	28 / 89 47	28 / 89 47	FWMO
Pentachloroph l	17 / 109 47	17 / 109 47	FWMO
Pyrene	35 / 142 36	[illegible] / 142 [illegible]	FWMO

% REC = rcent Recovery NA = [illegible]

RPD = R ve Percent Deviation FWMO = [illegible]

Table 7.3

Method Acceptance Criteria for Blanks, Surrogates and Matrix Spiking Compounds

GC Volatile and Semivolatile Methods

	SW-846 8151A IN % REC	METHOD 8081A IN % REC	METHOD 8082 IN % REC	METHOD 1608 IN % REC	METHOD 8021 IN % REC
BLANKS	1 in 20	1 in 20	1 in 20	1 per batch	1 per batch
MS/MSD	1 in 20	1 in 20	1 in 20	NA	1 in 20
MS	NA	NA	NA	1 in 10	NA
SURROGATE	LOW/HIGH (Interim)	LOW/HIGH (Interim)	LOW/HIGH (Interim)	LOW/HIGH (Interim)	LOW/HIGH (Interim)
2,4-Dichlorophen cetic acid (DCAA)	LEL	NA	NA	NA	NA
Picloram	LEL	NA	NA	NA	NA
Decachlorobiphe (DCB)	NA	LEL	LEL	NS	NA
2,4,5,6-Tetrachlo m-xylene (TCMX)	NA	LEL	LEL	NS	NA
1,4-Difluorobenz	NA	NA	NA	NA	LEL
Fluorobenzene	NA	NA	NA	NA	LEL
4-Bromofluorob ne (BFB)	NA	NA	NA	NA	LEL
SPIKING CO OUNDS	LOW/HIGH (Interim)	LOW/HIGH (Interim)	LOW/HIGH (Interim)	LOW/HIGH (Interim)	LOW/HIGH (Interim)
2,4-D	LEL (±30%)	NA	NA	NA	NA
2,4,5-TP (Silvex)	LEL (±30%)	NA	NA	NA	NA
g-BHC (Lindane	NA	LEL (±30%)	NA	32/127	NA
Heptachlor	NA	LEL (±30%)	NA	34/111	NA
Aldrin	NA	LEL (±30%)	NA	42/122	NA
Dieldrin	NA	LEL (±30%)	NA	36/146	NA
Endrin	NA	LEL (±30%)	NA	30/147	NA
DDT	NA	LEL (±30%)	NA	25/160	NA
Alachlor 1660	NA	NA	LEL (±30%)	NA	NA
Benzene	NA	NA	NA	NA	LEL
Ethylbenzene	NA	NA	NA	NA	LEL
Tolune	NA	NA	NA	NA	LEL
m&p-Xylene	NA	NA	NA	NA	LEL
o-Xylene	NA	NA	NA	NA	LEL

% REC = cent Recovery.
RPD = Rel e Percent Deviation.
LEL = La ratory Established Limits.
NA = Not Applicable to This Method.
NS = Not Specified in This Method.

Section 8

PERFORMANCE AND SYSTEM AUDITS

Introductio

Performanc and systems audits are required to assure that all required QA/QC are being performed a d evaluation criteria followed.

Performanc Audit

A p formance audit is an independent check, by a person designated by labo atory management, or by an audit unit, to evaluate the data produced by a labo atory's analytical system, and is sometimes categorized as a quantitative app isal of quality. There are several ways that this can be done:

a) 'orksheet review.
b) ral worksheet review.
c) n-site analyst work review.
d) dependent or check sample examination.
e) ter- and intralaboratory check sample, or proficiency test sample analysis view.

Per rmance audits will consist of evaluations of all data to ensure that all req red QC checks are being made and evaluation criteria followed. The per rmance audits and data reviews will be conducted by the QA officer of the lab atory.

Internal la ratory audits will be performed on a quarterly basis by the quality assurance officer. U PA's audit form will be used to examine the following items:

- ersonnel background and their training program.
- tandard Operating Procedures (SOP) in each section.
- ample shipping/receiving area and chain of custody procedures.
- vailability of space and the neatness in each section.
- vailability of exhaust hoods, their operation, and testing program.
- he use of manual and electronic balances and their calibration program.
- rimary standards, solvents, chemicals use, storage and documentation.
- eagent water, deionized and HPLC for volatile analysis.
- laintenance of GC/MS, GC, AA and other instruments by laboratory ersonnel and service contract company.
- uality Assurance/Quality Control procedures and corrective actions.
- ata handling procedures, computer software and reports.
- aste disposal policies & procedures.

A report w l be generated by the QA officer and corrective actions cited in Section II will be taken t ring the method/analytes back in control. Reports will be submitted to the president the laboratory and also filed properly.

Systems A dit

A stems audit is an on-site inspection and review of a laboratory's QC system an s sometimes categorized as a qualitative appraisal of quality. It will cover an r all of the operational QC elements of the Quality Assurance program. Au ts performed by Environmetrics QA officer may include instrumentation and ba up, manpower, chain of custody, standard operating procedure, do mentation sample storage, QA/QC procedures, preventative maintenance, pr ciency testing, and personnel training.

Environm ics has been participating in USEPA PE studies such as the WP and WS Series.

Section 9

PRE[redacted]NTATIVE MAINTENANCE & CALIBRATION OF EQUIPMENT

Environm[redacted]rics has established a preventive maintenance program that is implemented by technician[redacted] and chemists in charge of analyzing samples by a specific instrument. Section managers [redacted] the organic and inorganic departments schedule and document all the activities [redacted]rformed. Table 9-1 shows the summary of Environmetrics equipment performa[redacted]e and maintenance schedules.

Table 9.1

EQ[redacted]IPMENT PERFORMANCE AND MAINTENANCE SCHEDULE

Atomic A[redacted]sorption Spectrophotometers

Each Us[redacted]	***As Needed***	***Quarterly or Annually***
1. If burne[redacted] to be used, clean slot and ins[redacted]l. After use, remove burner. Ri[redacted] spray chamber with distilled wa[redacted].	1. Dust and clean.	1. Disassemble nebulizer and clean.
2. Check a[redacted]nstrument parameters.	2. Request repair of any malfunctioning part.	2. Check gaskets and O-rings.
3. Align la[redacted] for maximum light throughput[redacted] the analytical wavelengt[redacted]	3. Replace D2 lamp.	
4. Align bu[redacted]er for best sensitivity.	4. Clean optics.	
5. Adjust g[redacted] flows and nebulizer for best se[redacted]tivity.	5. Replace fuels, oxidants, and drain tubing.	
6. Run star[redacted]rds.	6. Clean nebulizer.	
7. Run QC[redacted]mples.		

Table 9.1 (continued)

EQUIPMENT PERFORMANCE AND MAINTENANCE SCHEDULE

Inductively Coupled Plasma Spectrometers (ICP)

Each Use	*As Needed*	*Quarterly or Annually*
1. Check nebulizer aspiration tube. 2. Check aspiration rate. 3. Check standard calibration.	1. Clean ICP torch monthly.	1. Manufacturer's representative maintenance check.

Environmetrics maintains a service contract for ICP. This contract includes labors and parts for the ICP and data system.

Analytical Balances

Each Use	*As Needed*	*Quarterly or Annually*
1. Clean at each use.	1. Check accuracy with 2 NIST traceable Class S weights on each day of use. 2. Request repair if inaccurate or malfunctioning.	1. Service engineer visit. (Contract with Fisher Scientific). 2. Bi-annual calibration of balances service engineer. 3. Re-certify weights every 5 years.

Ovens

Each Use	*As Needed*	*Quarterly or Annually*
1. Check and log temperature. Recording the unique identification of the thermometer and oven, and initials of the responsible person.	1. NA	1. NA

Table 9.1 (continued)

EQUIPMENT PERFORMANCE AND MAINTENANCE SCHEDULE

Gas Chromatographs

Each Use	*As Needed*	*Quarterly or Annually*
1. Check cyl ers. 2. Run stand s, blanks, samples, and QC.	1. Change filters, septa, solvent and resin (Hall detector).	1. Performance instrument parameters. 2. Check ECD detector for radio-active leaks.

Environme ics maintains a service contract for all GC systems. This contract includes labor and parts f the instruments and data systems.

High Pres re Liquid Chromatography Systems (HPLC)

Each Use	*As Needed*	*Quarterly or Annually*
1. Check res oirs. 2. Run stand ls, blanks, samples, and QC.	1. Change guard columns. 2. Change pump seals. 3. Rebuild valves.	1. Performance instrument parameters.

Environm ics maintains a service contract for all HPLC systems. This contract includes labor and rts for the instruments and data systems.

Purge an Trap Systems

Each Use	*As Needed*	*Quarterly or Annually*
1. Check ins ment parameters. 2. Clean pu vessels.	1. Replace trap.	1. NA

Table 9.1 (continued)

EQUIPMENT PERFORMANCE AND MAINTENANCE SCHEDULE

Gas Chromatography/Mass Spectrometer

Each Use	*As Needed*	*Quarterly or Annually*
1. Daily maintenance of GC. a. Change septa. b. Replace injection port liners. 2. MS tune with DFTPP (BNAs) or BFB (VOAs). 3. Run standards, blanks, samples, and QC.	1. Change gas cylinders. 2. Trim column. 3. Replace ferrules. 4. Clean source (if tuning becomes difficult or curves are no longer linear). 5. Add oil to pump.	1. Performance Evaluation checks.

Environmentics maintains a service contract with Hewlett Packard Co. and Varian Instrument companies.

These contracts include labor and parts for GC/MS and the data system. However, the GC/MS operators are trained to clean the source and check the injection port liner, change septa and check columns for resolution/sensitivity/proper chromatography.

Refrigerators & Freezers

Each Use	*As Needed*	*Quarterly or Annually*
1. Refrigerator or freezer must be uniquely identified	1. Temperature check and logged daily. Recording the unique identification of the thermometer and oven, and initials of the responsible person. (Unless otherwise specified, samples requiring thermal preservation shall be stored at ±2°C of the specified temperature.)	1. NA

Table 9.1 (continued)

EQUIPMENT PERFORMANCE AND MAINTENANCE SCHEDULE

Deionized Organic-Free Water

Each Use	*As Needed*	*Quarterly or Annually*
1. NA	1. Conductivity check daily. See SOP 300-003. 2. Ion exchange bed changed. 3. Filters replaced.	1. NA

Vacuum Pumps & Air Compressors

Each Use	*As Needed*	*Quarterly or Annually*
1. NA	1. Check performance. 2. Lubricate 3. Check belts, etc.	1. NA

Conductivity Meter

Each Use	*As Needed*	*Quarterly or Annually*
1. Calibrate with standard KCl	1. Clean electrodes.	1. NA

Table 9.1 (continued)

EQUIPMENT PERFORMANCE AND MAINTENANCE SCHEDULE

pH Mete

Each Use	*As Needed*	*Quarterly or Annually*
1. Calibrate eter with a minimum a minimum two standard buffers in the appro iate pH range. (See SOP 3 092)	1. Check electrodes daily.	1. NA

Thermo ters (NIST-traceable) with 1°C or finer subdivisions.

Each Use	*As Needed*	*Quarterly or Annually*
1. Check fo racks or bubble in the merc	1.	1. Recalibrate every 5 years.

Thermo ters.

Each Use	*As Needed*	*Quarterly or Annually*
1. Check da age or for cracks or bubble in th liquid.	1.	1. Check working liquid-in-glass and digital thermometers annually against NIST-traceable thermometer 2. Check metal and continuously monitoring thermometers quarterly against NIST-traceable thermometer 3. Record the results in the appropriate log book.

Table 9.1 (continued)

EQUIPMENT PERFORMANCE AND MAINTENANCE SCHEDULE

Autopipet s and Dilutors.

Each Use	*As Needed*	*Quarterly or Annually*
1. If adjustab check volume setting.		1. Check delivery volumes gravi-metrically on an annual basis. Record results.

Glassware sed for purposes that may subject it to damage from heat or chemicals shall be of borosilic e glass. All volumetric glassware shall be ASTM class A.

Analytical tandards are traceaable to a national stanadard when available. Document the traceability o the national standard.

Analytical eagents are reagent grade (AR) or better and are documented with the date received, d e opened, and any appliable expiration date.

Section 10

SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

Assessment of Accuracy

Accuracy will be evaluated by comparing the mean recovery of surrogate compounds or spiked analytes against the goals identified in the client's scope of work. The recovery of a surrogate compound will be defined as:

$$\%\text{Recovery} = \frac{[\text{Grams of Surrogate Found in Sample}]}{[\text{Grams of Surrogate Added to Sample}]} \times 100\%$$

The recovery of a spiked analyte will be defined as:

$$\%\text{Recovery} = \frac{[\text{Total Analyte Found}] - [\text{Analyte Orginally Present}]}{[\text{Analyte Added}]}$$

Calculation of Mean Values and Estimates of Precision

The mean value, C, of a series of replicate measurements of concentration Ci, is calculated as:

$$C = \frac{1}{n} x \sum_{i=1}^{n} Ci$$

where n = number of replicate measurements. C and Ci, are both in mg/L or mg/kg.

The estimate of precision of duplicate measurements is expressed as the relative percent difference (RPD), where

$$RPD = \frac{C_2 - C_1}{\overline{C}} X100\%$$

The relative percent difference calculated will be compared with the respective goals identified in specific scope of work.

The estimate of precision of a series of replicate measurements (primarily used in GC/MS analysis) is expressed as the relative standard deviation (RSD), where

$$SD = \pm\sqrt{\frac{\sum_{i=1}^{n}(C_i - \overline{C})^2}{n-1}}$$

and

$$RSD = \frac{SD}{\overline{C}} x100\%$$

Completeness

Completeness will be evaluated by comparing the number of samples analyzed, as follows:

$$\text{Degree of Completeness} = \frac{[\text{Total \# of Samples with Acceptable Analytical Data}]}{[\text{Total \# of Samples Aquired for Analysis}]} x100\%$$

The goal for most programs will be at least 95%.

Environmetrics, Inc. will use the SW-846 or CLP criteria to assess precision, accuracy, and completeness of the analytical results. The following tables will be used for volatile, semivolatile, pesticide/PCB, metals, and PNA.

Section 11

CORRECTIVE ACTION

Introduction

This section identifies the data to be used in determining if a problem exists and the method of corrective action to be taken. The attached forms will be used to report the out-of-control incidents.

Data

For each analytical method employed, the laboratory will track, regularly, precision and accuracy by computing the Relative Percent Difference (RPD) for duplicate analysis along with periodic determinations of spiked sample recovery. The mean recovery and the RPD of the results will be computed. The data will be compiled for each type of sample matrix analyzed. These statistics will be updated as additional samples are performed and more data are collected. When either the precision from replicate analysis, the RPD, and/or the accuracy from recovery data exceeds the control limits, the procedure will be checked for calibration, quality of the standards and analytical techniques. Analysis will be stopped and corrective action will be taken.

Actions

Corrective action will include, but not necessarily be limited to:

1. Recalibration of instruments using freshly prepared calibration standards.
2. Replacement of solvent lots or other reagents that give unacceptable blank values.
3. Additional training of laboratory personnel, if necessary, to improve the overlap between operator skills and method requirements.
4. Reassignment of personnel.
5. Re-extraction and/or re-analysis as per method requirements.

After the corrective actions have been taken and satisfactory QC sample results are obtained, sample analyses performed while the procedure was "out of control" will be re-run.

Control Limits

Whenever the analytical procedure is " out of control ", the problem must be found, corrected, and the analysis repeated. Analytical results reported when the procedure is

operating "[redacted]ut of control" will not be used unless approved. The analytical procedure is to be consi[redacted]ered "out of control" when any of the following occurs:

- [redacted]Whenever the method blank result exceeds the detection limit required for the [redacted]arameter, with the exception of common laboratory solvents (e.g., methylene [redacted]hloride, acetone, etc.), which must be less than five times the required [redacted]etection limit for the parameter, or is greater than 5% of the regulatory level [redacted]f the associated blank, or is greater than 5% of the sample result of the same [redacted]nalyte.

- [redacted]Whenever one lab replicate varies by more than the limits set by specific [redacted]roject plan.

- [redacted]Whenever reference standards or laboratory fortified samples fall outside the [redacted]anges specified in respective methods or the limits specified by a specific [redacted]roject plan.

- [redacted]Whenever the surrogate recoveries fall outside the limits set by a specific [redacted]roject plan, screening of known or suspected highly contaminated samples is [redacted]trongly recommended to ensure adequate addition of surrogates and internal [redacted]tandards where applicable. Inclusion of such results will justify any inability [redacted]o comply with the prescribed limits.

- [redacted]Whenever the area of an internal standard, by GC/MS analysis, is less than [redacted]0% or greater than 200% of the area in the corresponding compound from [redacted]hat in the mid-point standard level of the most recent initial calibration. The [redacted]etention time for any internal standard may not vary by more than 30 seconds [redacted]rom that in the mid-point standard level of the most recent initial calibration.

Exception [redacted]o the above requirements will be in cases where they are not realistically achievable [redacted]y the USEPA method used (mainly applicable to organics analyses). Method specific co[redacted]trol limits may be substituted when appropriate with respect to sample matrix, amount, et[redacted]. Sample results outside the prescribed limits must be reextracted and/or reanalyzed [redacted]unless the laboratory is able to demonstrate that the problem is beyond the laboratory[redacted] control.

At Enviro[redacted]metrics, the technicians, chemists, and section managers are responsible for initiating c[redacted]rrective action. This report will be submitted to the laboratory director and the VP of [redacted]perations for review and comment. The laboratory directors will develop and approve th[redacted] corrective actions. The section managers are responsible for implementing the recom[redacted]endations and report the results. If the system is brought back to control, reruns or r[redacted]w sample analysis can begin. The corrective action forms and the backup data will be file[redacted] in the project file for client review and future use. Logbooks will also be used to do[redacted]ument all corrective action results. Information from corrective the action plan will be dis[redacted]ussed in the case narratives and submitted to the clients.

DATA REVIEW CHECKLIST (ORGANICS DEPT)

PROJECT/CLIENT #: _____ METHOD: _____

DATE SAMPLED: _____

ANALYSIS PERIOD: _____ to _____

ANALYST'S INITIALS: _____ DATE: _____

REVIEWER INITIALS: _____ DATE: _____

Do not place a checkmark in the provided space if the statement does not apply to the analysis or method

TUNING AND CALIBRATION INFORMATION

____1. Tuning and calibration meets method criteria (GC/MS only).
____2. Initial and continuing calibration meets with method criteria (all departments).
____3. Pesticide breakdown and resolution meets method criteria (GC only).
____4. Retention time criteria meets method specifications (all instrumentation).

SAMPLE INFORMATION

____1. Copy of Chain of Custody (internal, extract) signed, correct and present with package.
____2. ID's match between extract and internal chain of custody.
____3. Extraction and analysis hold times are met.
____4. Surrogate(s) recovery with method defined limits. If not, see narrative section.
____5. Spike(s) recovery and RPD(s) for the LCS, MSD and/or MS with method defined limits to assess accuracy and precision. If not, see narrative.
____6. Contamination within method defined limits for the method blank.
____7. Confirmation performed (column - Herb/Pest, GC/MS of BTEX & PCBs).
____8. All positive hits diluted in range of calibration.
____9. Sample receipt recorded in bound log book.
___10. Problems associated beyond the scope of the method and this checklist are noted in the narrative section.
___11. Corrective action, re-extraction in accordance with method and noted in the narrative.

REPORTING

____1. Tuning and calibration information present with package or on file.
____2. Sample reports (Form 1 report) present with data package and QC summary present if QA Level III or IV requested.
____3. All sample data generated present with this package.
____4. All valid hits initialed and dated. All line through(s) initialed and dated.
____5. Case Narrative present. This checklist for QA Level V or CLP type case narrative for QA Level II & IV.

FINAL REVIEW / SECOND REVIEW (SUPERVISOR OR PEER)

____1. This checklist filled out properly.
____2. All things check are actually done and present.
____3. Data is second reviewed and positive hits verified.
____4. QC summary forms if present check for correctness and completeness.
____5. Hold times met and correct method used.
____6. Narrative and billing portion filled out correctly.

SAMPLE SUMMARY AND NARRATIVE

METHOD: _____ _____ EXTRACTION METHOD: _____ (if applicable semivolatile only)

LAB ID	DILUTIONS	RERUNS OR RE-EXTRACTS	CONFIRM/PEST/HERB CLEAN-UP	CLP IS AREA	PROBLEMS WITH SAMPLES

This is the narrative section which will appear in the comments section of the labor report.

All sample(s) were ___ extracted ___ and in accordance with method(s) _____ and ___ (see above). All specified method ___ analyzed _____

criteria and limits for surrogate(s) and spiking compound(s) were ___ met for each sample ___, ___, ___ except for the following sample(s) and reason(s):

Sample(s) _____ _____ were ___ re-extracted ___, ___ and because of ___ low surrogate recoveries ___, ___ and ___
_____ _____ ___ re-analyzed ___, ___ and ___ contamination in blank ___, ___ and ___
_____ _____ ___ re-injected ___, ___ and ___ matrix interference(s)
_____ _____
_____ _____
_____ _____

Sample(s) _____ _____ were ___ diluted ___, ___ and because of ___ positive hits out of calibration range ___, ___ and ___
_____ _____ ___ confirmed ___, ___ and ___ matrix interferences ___, ___ and ___
_____ _____ ___ re-extracted ___, ___ and ___ positive hit(s) (Pest/Herb only)
_____ _____ ___ cleaned up ___ insufficient sample submitted
_____ _____ ___ sulfur contamination
_____ _____ ___ phthalates contamination

Dilution(s) and/or matrix interference(s) can and will result in elevated detection limits for positive hits ___, ___ and ___ and compounds if a matrix interference

Loss on drying (percent moisture) ___ was used in calculation for positive hit ___ and ___ results in elevated detection limit

Section 12

COMPLAINTS

Introduction

This section discusses procedures for dealing with complaints received from clients or other parties.

Procedures

The procedures for dealing with complaints are based on the type of complaint.

1. If the compliant is based on a typographical error and does not effect the result reported to the client, then the appropriate corrections will be made to all affected documents and marked with the date of correction and the initials of the individual making the correction,or

2. If the complaint is based on a reported result or compliance, the complaint will be recorded on the Quality Improvement Report (form 11.1) and an internal audit will be initiated. The results and action taken will be documented on the Quality Improvement Report. This report will become part of the analytical package and a copy will be added to the 'Complaints Binder', which will be maintained in the Client Services section. Any corrections made as a result of this audit must be clearly documented and marked with the date of correction and the initials of the individual making the correction.

Section 13

QUALITY ASSURANCE REPORTS TO MANAGEMENT

Review

On a period basis, Environmetrics' QA Officer will review all QC summaries, and other aspects of th project's QA program. A brief semi-annual assessment of the adequacy of the project's QA/QC program will be summarized in memorandum to the president of the laboratory, with a copy sent to the appropriate laboratory section managers. More frequent reports may be submitted if 1) a problem needing immediate attention is identified by the QA Officer or his/her designee, or 2) it is requested by the client. The memorandum will include:

a. Identification of any areas that appear to require remedial action.
b. Updates of new information pertaining to remedial actions.

Section 14

CONFIDENTIALITY and PROPRIETARY RIGHTS

To ensure confidentiality of test results, Environmetrics will only release and discuss test results with the company submitting the sample(s) for analysis. Exceptions and deviation are as follows:

- If the results are to be released and/or discussed with another company, this request may be documented by the client on the 'Chain of Custody', or
- If the results are to be released and/or discussed with another company, this request may be documented by a letter from the client, or
- If the results are to be released and/or discussed with another company, this request may be documented by the client filling out the 'Release of Results Form'.

Because of the public nature of telephones, fax machines, and computers, Environmetrics can not ensure confidentiality of data transmission to an individual within a company only to the company itself.

Form 14.1

Release of Results

As an employee of _____,
I hereby authorize Environmetrics to -

- ☐ Release of results
- ☐ Discussion of results

Sample identification _____

_____

To: _____

Of: _____

Signed: _____ date _____

Section 15

PHYSICAL FACILITIES

In July 199? Environmetrics moved into a new 13,000 square foot laboratory. This modern faci ty was built keeping in mind special design features that address the latest needs for pe forming trace organic and inorganic analyses of environmental samples. A 1,000 squar foot warehouse with a receiving dock provides ample room for shipping, receiving, a d storage.

Samples are brought to the shipping/receiving section and logged into a custom designed laboratory i formation management system (LIMS). A 150 square foot cold room is used to prop rly store samples (VOA samples are stored separately in the VOA laboratory).

The Inorgar c/Organic Prep Lab provides adequate facilities and services with over 700 square feet f bench space, six hoods, three refrigeration units (sample and standards storage), dis washer, sinks, tap and Type 1-SM water, power, and gas supplies.

The TCLP P ep Lab is equipped with 45 square feet of bench space, a hood, sink, and tap and Type 1- M water.

The Metals P rep Lab is equipped with 100 square feet of acid resistant slate bench space, a hood, sink and tap, Type 1-SM, and ASTM Type 1 water.

The Gas Ch omatography/Mass Spectrometry areas were given special design considerati s. The volatiles laboratory has features that keep the area completely sealed. Independen air conditioning and heating systems allow for complete separation of airflow fro the rest of the building. Continuous positive pressure minimizes sample contaminati n during analysis. These areas are also equipped with refrigerators and freezers for ample and standards storage, gas supplies, and over 250 square feet of bench, com uter, and workspace.

The Gas Ch omatograpy/HPLC/Ion Chromatography areas are equipped with refrigerator for sample and standards storage, gas supplies, and over 180 square feet of bench, com uter, and workspace.

The ICP/At mic Absorption Lab is equipped with gas supplies, sinks, tap and Type 1-SM water, pow assist exhaust hoods, and over 150 square feet of bench, computer, and workspace.

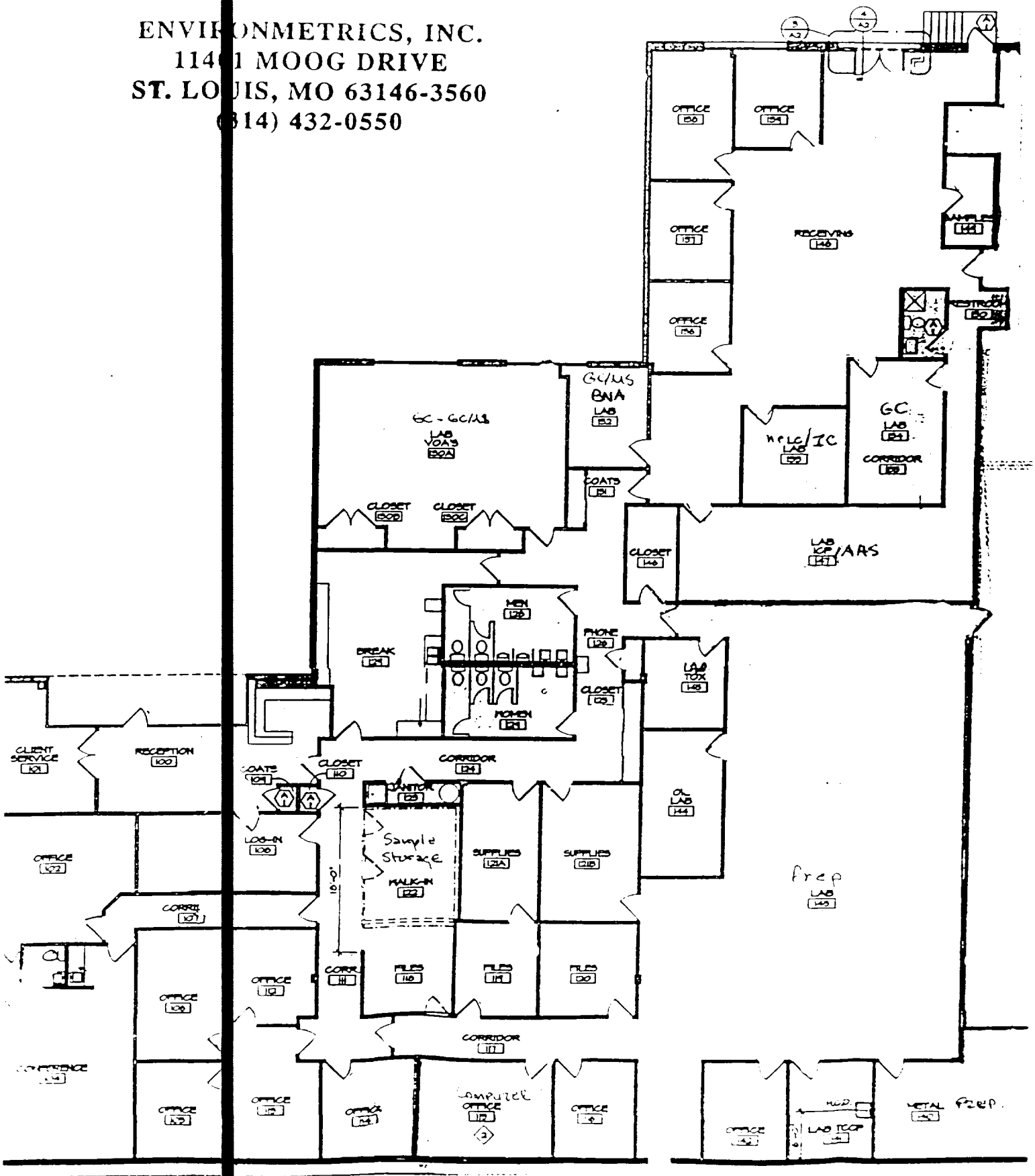
Environmetrics' Type 1-Standard Methods water is supplied by two systems. Each consisting of a pre-filter, carbon bed, mixed type II deionzation bed, mixed type II deionzation polisher bed, and monitored by Foxboro conductivity meters. The Type 1-SM water is further purified to Type 1-ASTM water by a Barnstead EASYPure RF Ultrapure water system.

For the safety of the analysts, this facility is equipped with a sprinkler system, emergency lighting, fire extinguishers, fire blankets, safety showers, eye wash stations, first aid stations, and spill kits. For a comprehensive review of safety practices, refer to Environmetrics Safety Manual.

Waste Containment area. Sample waste and waste produced from the analysis of samples is segregated by waste stream and stored in the waste containment area until it is removed by a waste disposal company. Drums containing liquid waste are stored atop liquid containment pallets.

This facility will support Environmetrics present and long term requirements for performing environmental analysis.

ENVIRONMETRICS, INC.
11401 MOOG DRIVE
ST. LOUIS, MO 63146-3560
(314) 432-0550



Section 16

INSTRUMENTATION

Organic Analysis Instrumentation

1. Hewlett Packard 5890/5970 Gas Chromatograph/Mass Spectrometer (GC/MS) with HP7673A Autosampler and ProLab Data System using HP EnviroQuant.

2. Hewlett Packard 5890/5970 Gas Chromatograph/Mass Spectrometer (GC/MS) with Tekmar LSC-2000 Purge and Trap Concentrator and ALS-2016 Autosampler and HP-1000 RTE-A Minicomputer Data System.

3. Varian Saturn Gas Chromatograph/Mass Spectrometer (GC/MS) with Tekmar LSC-2000 Purge and Trap Concentrator and ALS-2016 Autosampler and Saturn computer system for instrument control and data acquistion. Data processing is handled by ProLab Data System using HP EnviroQuant.

4. Varian Saturn Gas Chromatograph/Mass Spectrometer (GC/MS) with Tekmar LSC-2000 Purge and Trap Concentrator and ALS-2016 Autosampler and Saturn computer system for instrument control and data acquistion. Data processing is handled by ProLab Data System using HP EnviroQuant.

5. Varian 3400 Gas Chromatograph with PID and HECD Detectors, Tekmar LSC-2000 Purge and Trap Concentrator and ALS-2016 Autosampler and Justice Innovations ChromPerfect Data System.

6. Varian 3400 Gas Chromatograph with PID and FID Detectors, Tekmar LSC-2000 Purge and Trap Concentrator and ALS-2016 Autosampler and Justice Innovations ChromPerfect Data System.

7. Hewlett Packard 5890 Gas Chromatograph with dual Electron Capture Detectors (ECD), dual HP7673A Autosamplers, and Justice Innovations ChromPerfect Data System.

8. Hewlett Packard 5890 Gas Chromatograph with dual Electron Capture Detectors (ECD), dual HP7673A Autosamplers, and Justice Innovations ChromPerfect Data System.

9. Varian 3700 Gas Chromatograph with dual Electron Capture Detectors (ECD), 8000 Autosampler, and Justice Innovations ChromPerfect Data System.

10. Varian 400 Gas Chromatograph with Electron Capture Detectors (ECD), 8100 Autosampler, and Justice Innovations ChromPerfect Data System.

11. Varian 400 Gas Chromatograph with FID, 8030 Autosampler, and Justice Innovations ChromPerfect Data System.

12. Gilson Gradient 306 HPLC with 116 UV/VIS and 121 Fluorometer Detectors, 231 Autosampler, and GME-719 Data System/Controller.

13. Gilson Gel Permeation Chromatograph with 112 2UV/VIS detector, 231 Autosampler, FC203 Fraction Collector, and a Shimadzu C-R3A Integrator.

14. Zymark Turbo Vap Evaporator.

15. Summit Interests SIP-1000 Portable Gas Chromatograph with TCD and PID Detectors

16. Fisher A-160 Electronic Analytical Balance.

17. Fisher AD-4KD Electronic Balances.

18. Fisher AT-3000DR Electronic Balance.

19. Foxboro Miran 1A Infrared Spectrometer.

20. Heat Systems Ultrasonic Model W-385 Sonicator and two Tekmar Ultrasonic Model TM375 Sonicators.

21. Two Millipore TCLP ZHE Extractors.

22. Environmental Express TCLP Extractor.

Inorganic Analysis Instrumentation

1. Thermo Jarrell-Ash EnviroScan-II Inductively Coupled Plasma Spectrometer (ICP) with Data System.

2. Varian SpectrAA-400Z Zeeman Graphite Furnace Atomic Absorption Spectrometer (ZGFAAS) with Sample Dispenser and Data System.

3. Varian SpectrAA-800Z Zeeman Graphite Furnace Atomic Absorption Spectrometer (ZGFAAS) with Sample Dispenser and WIN95 Data System.

4. Varian SpectrAA-400A Flame Atomic Absorption Spectrometer (FAAS) with VGA-76 Hydride/Cold Vapor Mercury accessory, PSC-56 Autosampler and Data System.

5. Buck Scientific 400 Cold Vapor Mercury Analyzer.

6. Dohrmann DX-20A Total Organic Halogen Analyzer (TOX) with POX Sparger.

7. Two Shimadzu UV-1201 Spectrophotometers.

8. Hach COD Reactor.

9. Two Orion 920A Ion Specific Electrode (ISE) Meters.

10. Orion 8 Ionmeter.

11. Fisher Accumet 910 pH Meter.

12. Two Setaflash Flashpoint Tester.

13. Mettler AE-260 DeltaRange Electronic Balance.

14. Satorius LC 2200S Electronic Balance.

15. Floyd RMS-150 Microwave Digestion System.

16. IEC Centra-4B Centrifuge.

17. Brinkmann Rotavapor Model 461.

18. YSI Model 3560 Water Quality Monitor Portable pH, Temperature, Conductivity Meter.

19. Fisher Model 307C BOD Incubator.

20. Thermolyne Model 62700 Furnace.

21. Barnstead EASYPure RF Ultrapure Water System.

Laboratory Information Management System (LIMS)

Environmetrics laboratory information management system utilizes a company wide Ethernet computer network for sample tracking, data acquisition/reduction, data reporting, and accounting.

The hardware and software have been reviewed and are deemed Y2K compliant.

- Dell PowerEdge 2300 Server operating under Windows NT 4.0
- Gateway P5/100 Server operating under Novell Netware 4.1
- 35+ Dell, Gateway, other workstation computers
- REI's LabMage 2.1 LIMS system
- Sage's DACEasy Accouting 9.0 system

Section 17

VALIDATION PRACTICES

Performance Evaluation Programs

Environmetrics participates in the U.S. Environmental Protection Agency's Performance Evaluation Programs for both Water Supply (WS) and Water Pollutants. The results from these quarterly programs are submitted to the USEPA and final evaluations are submitted to each state in which Environmetrics is certified.

Environmetrics identification: MO00066.

Environmetrics also analyzes quarterly 'single-blind' quality control samples obtained from Environmental Resource Associates (ERA). These samples allow the laboratory to assess it's performance at the time of analysis.

Reference Materials

The laboratory shall utilize analytical standards that are traceable to a national standard when available. The laboratory shall document the traceability to a national standard.

The laboratory shall utilize analytical reagents of reagent grade (AR) or better. The laboratory shall document the date received, date opened and any applicable expiration date

CERTIFICATIONS

Environmetrics is certified in the following states:

State	Certification Number	Certification Type
Missouri	00910	Drinking Water
Kansas	E-10190	Drinking Water
Tennessee	02833	
Illinois	100253	Drinking Water
Kentucky	90092	Drinking Water
Nebraska		

For the most recent state by state listing of test methodologies for which Environmetrics is certified contact Environmetrics' Client Services.

Section 18

TEST METHODOLOGIES [1]

PARAMETER		Drinking Water	NRDES Water	RCRA Waste
Acidity		305.1	305.1	---
Alkalinity		2320B	310.1	---
Aluminum		200.7	200.7	6010
Ammonia			350.2	---
Antimony		200.9	200.9	6010/7041
Arsenic		200.9	200.9	6010/7060
Barium		200.7	200.7	6010
Beryllium		200.7	200.7	6010
BOD5		---	405.1	405.1
Boron		200.7	200.7	6010
Bromide		300.0	---	9056
Cadmium		200.7	200.7	6010
Calcium		200.7	200.7	6010
CBOD5		---	405.1	405.1
COD		---	410.4	410.4
Cation exchan	e capacity	---	---	9080
Chloride		300.0	325.3	9252/9056
Chlorine, total	esidual	---	330.2	---
Chromium		200.7	200.7	6010
Chromium, he		---	218.8/200.7	7195/6010
Cobalt		200.7	200.7	6010
Copper		200.7	200.7	6010
Cyanide, total		4500CN-c,e	335.2	9010
Cyanide, amen	ble to chlorination	4500CN-c,g	335.1	9010
Cyanide, weak	cid dissociable	---	4500CN i	---
Fluoride		300.0	340.1	9056
Hardness		130.2	130.2	130.2
PH		150.1	150.1	9040/9041/9045
Ignitability		---	---	1010/1020
Iron		200.7	200.7	6010
Lead		200.9	200.9	6010/7421
Magnesium		200.7	200.7	6010
Manganese		200.7	200.7	6010
Mercury		245.1	245.1	7470/7471
Molybdenum		200.7	200.7	6010
Nickel		200.7	200.7	6010
Nitrate		300.0	353.3	---
Nitrate Nitrite		300.0	353.3	---
Nitrite		300.0	353.3	---
Nitrogen, Kje	n	---	351.3	---
Oil & Grease		---	413.1	9070/9071

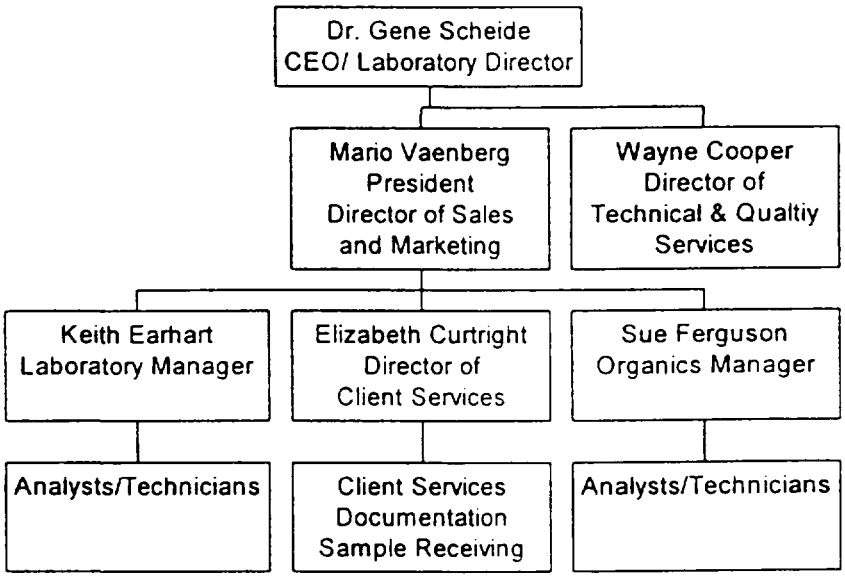
TEST METHODOLOGIES
(continued)

PARAMETER	Drinking Water	NPDES Water	RCRA Waste
TOC	---	415.1	9060
TOX	---	---	9020
Orthophospha	---	365.2	---
Oxygen, disso ed	---	360.1	---
Paint filter	---	---	9095
Phenol	---	420.1	9065
Phosphate	300.0		9056
Potassium	200.7	200.7	6010
Residue, total TS)	160.3	160.3	160.3
Residue, filter le (TDS)	2540C	160.1	160.1
Residue, nonf erable (TSS)	160.2	160.2	160.2
Residue, settle ble	160.5	160.5	160.5
Residue, volat e	160.4	160.4	160.4
Selenium	200.9	200.9	6010/7740
Silicon	---	200.7	6010
Silica	200.7	200.7	6010
Silver	200.7	200.7	6010/7761
Sodium	200.7	200.7	6010
Specific cond tance	2510B	120.1	9050
Strontium	---	200.7	6010
Sulfate	300.0	375.4	9038/9056
Sulfide	---	376.2	9030
Sulfite	---	377.1	
Surfactants	425.1	425.1	
Thallium	200.9	200.9	6010/7841
Tin	200.7	200.7	6010
Titanium	200.7	200.7	6010
Vanadium	200.7	200.7	6010
Zinc	200.7	200.7	6010
Volatile Orga c Cpds	524.2	624	8260
Volatile Arom tic Cpds (BTEX)	---	602	8020
Base/Neutrals & Acids	525	625	8270
Pesticides & PCBs	508	608	8080
Chlorophenox Herbicides	515.1		8150
Explosives	---	---	8330
TTHM	524.2	---	---
TCLP	---	---	1311
SPLP	---	---	1312
Total Petroleu Hydrocarbons	---	418.1	418.1 8015

1. For the most recent state by state listing of test methodologies and revisions for which Environmetrics is certified, contact Environmetrics' Client Services.

Section 19

LABORATORY ORGANIZATION



- CEO/Laboratory Director - Dr. Gene Scheide holds his Ph.D. in Analytical Chemistry from Louisiana State University in New Orleans.

  The laboratory director is responsible for the analytical and operational activities; supervision of personnel; assuring that sample acceptance criteria are met; that samples are logged into LIMS; that samples are properly labeled and that samples are properly stored; the production and quality of data reported; and designating laboratory supervisors, mangers, and quality assurances officers.

- President/Director of Sales and Marketing - Mario Vaenberg holds his MS in Environmental Sciences from Southern Illinois University in Edwardsville.

  The president/director of sales and marketing is responsible for general management; development and implementation of sales and marketing; and development of short and long term goal and objectives. The president has final authority in the absence of the CEO/Laboratory Director.

- Director of Technical and Quality Services - Wayne Cooper holds his BS in Chemistry from Washington University, St. Louis.

The quality assurance officer is responsible for coordinating QA/QC procedures and analytical data review procedures; verifying that QA/QC requirements are met; and conducting internal audits of the entire laboratory.

- Laboratory Manager - Keith Earhart - Core Studies in Chemistry/Biology with over 20 years experience in industrial and environmental applications.

  The laboratory manager is responsible for the analytical and operational activities; supervision of analysts and technicians; reviewing and verifying data produced by analysts-in-training or technicians; assuring that sample acceptance criteria are met; that samples are logged into LIMS; that samples are properly labeled and that samples are properly stored; the production and quality of data reported;

- Analysts - should hold a bachelor's degree in a natural or physical science; have completed a minimum of four hours of training (instrument manufacturer, professional organization, university, or qualified training facility); or served a two-week apprenticeship under an experienced analyst.

- Technicians - should hold a minimum of a high school diploma or equivalent; have completed a minimum of four hours of training (instrument manufacturer, professional organization, university, or qualified training facility); or served a two-week apprenticeship under an experienced analyst or technician.

Section 20

EMPLOYEE TRAINING

Each analyst and technician must receive appropriate training. Training is defined as:

1) Either:

   a) Satisfactorily completed a minimum of four hours training offered by the equipment manufacturer, a professional organization, a university or qualified training facility; or
   b) Served a two-week apprenticeship under an experienced analyst or technician;

2) Performed an IDMP (Initial Demonstration of Method Proficiency) study;

3) Performed an acceptable performance blind sample at least once per year;

4) Read, understood, and agreed to perform the most recent method, the approved method and/or standard operating procedure.

5) Documentation of training in each analyst's/technician's Training Summary persuant to SOP Number 100.005

Quality Assurance Plan
QAP

Data/Analysis Technologies, Inc.
7715 Corporate Blvd.
Plain City, Ohio 43064

August 1, 1999

Revision 5, August 1, 1999

Table of Contents

Appendix Listing

I. Introduction

The quality assurance program at Data/Analysis Technologies, Inc.(DAT) is designed to continuously improve the quality of the products through effective organizing, planning and execution of protocols. The involvement of employees in a total quality program, a commitment from management, and an ongoing quality improvement program insure the achievement of the organizations Quality Assurance Program Objectives.

II. Quality Assurance Policy

A. The laboratory will establish and maintain a Quality Assurance Program. A Quality Assurance Officer will be appointed, who will report directly to the President of the company.

B. Annual Quality Assurance Program reviews will be undertaken to assess the programs effectiveness.

C. Routine Audits of procedures and processes against standard operating procedures will be performed.

D. Training in the implementation of the Quality Assurance Program will be implemented and maintained.

The Quality Assurance Program at Data/Analysis Technologies, Inc. is designed to fulfill the needs of a rapidly expanding company to provide quality awareness to the staff, Quality Assured Products to our clients, and a commitment to total quality by the management of the Laboratory.

III. Organization

The Quality Assurance Officer reports directly to the office of the President. Each unit function is responsible for implementing the Quality Assurance Program into that specific part of the organization. Unit managers are responsible to the President. The laboratories Quality Assurance Objective will be met by assuring that:

A. Management is committed to a Total Quality Program

B. A direct reporting Quality Assurance Office is established

C. Training of staff

D. Routine QA audits and

E. Annual QA laboratory summary.

IV. Management

The management staff at the laboratory will implement the following program to assure the Quality Assurance Program will meet the objectives.

A. Establish a Quality Assurance Office.
This office will implement the Quality Assurance Program through training and audits. Reporting directly to the President of the laboratory.

B. Require QA project plans for each project undertaken by the laboratory.

C. Establish GLP (good laboratory practices) environment for the conduit of studies.

D. Provide information on QA goals to the organization.

E. Document QA Program on a annual basis.

V. Quality Assurance Officer

The Quality Assurance Officer will implement routine phase inspections, review final reports, review QAPP (Quality Assurance Program Plans) for the QA indicators, accuracy, precision, completeness, representativeness, comparability, and provide training on quality assurance procedures.

VI. Quality Assurance Project Plan (QAPP)

Quality Assurance Project Plans (QAPPs) are documents that precisely define the way a project, a group of allied projects, or a specific analytical program are to be conducted. It is within the QAPP that Data Quality Objectives (DQOs), and the means by which they are to be met are stated in detail. The general content and format required of a QAPP is specified in Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (USEPA, December 1980).

VII. Good Laboratory Practices

Good Laboratory Practices shall be followed by the staff in the conduction of all aspects of a TSCA, FIFRA or FDA study. Other non-regulated studies are to adhere to the intent of GLP in an effort to provide a level of continuity. This includes master schedule maintenance, final report review, phase inspections, protocol maintenance (SOPs), reports to management and certification statements. The Quality Assurance Unit must execute the GLP requirements described below:

Master schedule maintenance - The master schedule is a current record of all studies (projects). Documentation is indexed under test substance (the matrix in which TSCA,

FIFRA or FDA regulations for analyte content are applied) and contains the following information for each study:

- the test system (analytical method)
- the nature of the study
- the date the study was initiated
- the current status of the study
- the identity of the sponsor (client)
- the name of the study director

Final report review - Each final report must be reviewed by the QAO to assure that all protocols (methods and SOPs) were adequately described and followed, and raw data is accurately reflected in the reported data.

Phase inspections - Phase inspection records must be maintained within the QAU. Each record must show the date of inspection, the study (project) inspected, the phase of the study inspected, the inspector identity, findings and problems, corrective actions recommended and implemented, and a schedule for re-inspection of the study.

Protocol maintenance - The QAO must maintain copies of all protocols (SOPs, analytical methods, QAPPs, etc.) which are utilized in GLP studies.

Reports to management - The QAO must submit periodic written status reports to management and study directors, noting problems and corrective action taken on GLP studies.

Deviations from approved protocol - The QAO must determine that proper authorization and documentation exists when any deviations from approved protocol occurred in GLP studies.

QA Statement - The QAO must prepare and sign a statement of GLP compliance to be included with the final study report which presents dates and phases inspected and dates of reports to management. In case of noncompliance, the QA statement details any deviations/modifications to the protocols/SOPs with an evaluation of their impact on data quality.

VIII. Quality Assurance Goals

Measurable Quality Assurance goals are necessary to ensure the laboratory has a **Performance Based Program**. The QA indicators, vide supra, for each project are measurable quantities. Communication of the laboratory performance against the QA plan to the staff during the Annual QA Review, will assist to establish a strong program. It is the performance objective that each project meet the goals set forth in this document.

IX. Training

Training in the form of meetings, workshops, formal and on the job training will be documented and placed in the laboratory training file. Performance of new protocols (SOPs) will require documented training by the supervisor, prior to an analyst performing the protocol independently. The amount of training depends upon the task and is at the discretion of the supervisor.

X. QA Assessment

The Quality Assurance Officer will provide to management the annual assessment of the Quality Assurance Program at the laboratory. This report to the President will critique the program, its goals, its function and implementation at the laboratory level. Deficiencies are to be addressed by management.

The detailed job description for each position existing within the company is filed, updated as necessary, by the Personnel Department, and is available for inspection.

Brief resumes of the key DAT personnel are presented in Appendix D, the Qualification Summary.

XI. Recruitment Policy

The Personnel Department of Data/Analysis Technologies, Inc., uses several methods of recruitment.

> Data/Analysis Technologies, Inc. first announces open positions on the posting boards so that current employees have the first opportunity to apply for openings within the facility. These announcements are made one week before outside sources for candidates are sought. The outside sources used primarily are the local newspapers, placement agencies (temporary and permanent), local colleges and the State Employment Bureau.
>
> The recruitment process consists of collecting the applications and resumes, distributing them to the proper selecting official, setting up appointments as requested and interviewing with a representative from personnel, as well as, the appropriate staff. After the selection is made for the position, the candidates references are checked before making an offer. Data/Analysis Technologies, Inc. is an equal opportunity employer.

Training - On and Off Site

On Site training: Training goes on at different levels throughout the laboratory. On the job training relating directly to the position is done by the supervisor or other qualified staff. Cross training, supervisory and other related training takes place on a scheduled basis and is documented for training files.

Off Site training: This type of training takes place as needed. Recommendations and suggestions come from all levels of staff and are documented and updated regularly in the training files.

Record Maintenance

Resumes: Resumes are put in a uniform format upon hire. These resumes are updated on an annual basis or as needed.

Training: Training files are kept in the administrative office, and all new and ongoing training information is turned in on the 5th of each month and the records updated at that time.

Education and Experience: This information is updated with the resumes.

Safety and Health Policies

All personnel undertake orientation and on-the-job intensive training concerning safety issues. DAT policy in this respect is presented in detail in the following documents, with which each employee is provided:

- DAT Safety and Health Manual
- DAT Hazard Communication Plan
- DAT Chemical Hygiene Plan
- DAT Emergency Contingency Plan

XII. Facilities and Equipment

Physical Plant, Laboratories, Instrumentation and Maintenance

Facilities: 10,000 sq. ft. laboratory facility

Data/Analysis Technologies, Inc. location, mailing address and phone numbers are:

Data/Analysis Technologies, Inc.
7715 Corporate Blvd.
Plain City, OH 43064

800-733-8644
614-873-0710
614-873-0810 (Facsimile)

Data/Analysis Technologies, Inc. is a **female owned closely held S-corporation** and as such is not publicly traded.

A description of Data/Analysis Technologies, Inc. facilities and major equipment may be found in the SOQ.

Well maintained equipment is an essential ingredient in assuring the quality, completeness and timeliness of analytical data. DAT minimizes the risk of data incompleteness through the

performance of regular maintenance on all equipment, redundancy in equipment, provision of an ample stock of spare parts, and an inventory of specialized test equipment to support rapid repair, in the event unscheduled maintenance is required. The instrument laboratory is equipped with a UPS system which provides uninterruptable power to all the equipment.

XIII. Control Systems

Data/Analysis Technologies, Inc. provides a secure environment for our clients samples, employees and guests. Our standard procedures require that all doors remain locked at all times except the main entrance, which is open during regular business hours. Once access is gained there are secondary combination punch lock mechanisms providing authorized entrance into the laboratory. The lock combination is changed regularly.

Laboratory Access

Access to the analytical laboratory is normally restricted to employees. Exceptions will be made in the case of tours of the laboratory by prospective clients and clients who have a need to be present when their samples are analyzed. Visitors are to sign the Visitor Log and to be accompanied by a DAT employee at all times. The following rules are applied regarding punch lock entrances:

New punch lock combinations are obtained from an employee's supervisor or the receptionist at the beginning of each month.

Combinations are to be given only to active employees of Data/Analysis Technologies, Inc.

Laboratory personnel are trained to protect the lock code in the presence of visitors.

Security

Security is maintained by a key inventory system and an electronic alarm system which provides active access to only those individuals actively employed at Data/Analysis Technologies, Inc. A security check is performed nightly by supervisory personnel to ensure all entrances are secure. Locks on laboratory entrances, files and refrigerators ensure client confidentiality and personal safety. A punch lock system allowing selected entry into the laboratory and archive is in place.

An electronic fire alarm system provides early warning in case of a fire.

XIV. Document Control

Logbooks are maintained using a sign out system recorded in a designated logbook. The logbook must have documented on the front inside cover the assigned owner, the date issued, and the name and subject of the logbook. Logbooks must be maintained by initialing and dating every entry and striking all blank areas of the logbook.

The following is a list of all laboratory notebooks maintained at the Laboratory:

Area	Laboratory Notebook(s)
Sampling Receiving	Sample Receipt Logbook Refrigerator Logbook
Standards Control	Standards Logbook
Lab/Organics	Instrument Run Logbook Maintenance Logbook
GC	Instrument Run Logbook Maintenance Logbook
Archive	Archive Logbook for addition and removal of archived data
Shipping Administration	Sample Shipping Logbook DAT - Register Logbook
Personal	Phone Logs Personnel Logs

When complete, the log books are archived.

The laboratory maintains records of all original observations, calculations and final test results on either hard copy or magnetic media. These data are archived in accordance with GLP. The archive is locked at all times. All authorized personnel access the archive in the presence of the archivist and sign in. Any materials removed must be signed out by both.

XV. Sample Tracking/Custody Procedures

Sample Receipt

The Sample Custodian or designated assistant will receive all deliveries. The general condition of the package and the time/date of receipt will be recorded. Any documentation external to the package will be examined for consistency. In the case of samples delivered by a customer, the name of the person making the delivery will also be recorded. The package will be taken to the sample receiving area for examination by the Sample Custodian.

The Sample Custodian will examine all shipping containers and record the following information on the DAT Chain-of-Custody Form:

A. Presence/absence of the custody seal(s) on the shipping container(s).

B. Condition of the custody seal(s) (i.e., intact or broken.)

C. Temperature of the samples

Note: The front inside cover of each Sample Receiving Log book should indicate that samples are recorded.

The Sample Custodian will open the sample container(s) and record for each, the following information on the DAT Chain-of-Custody Form:

A. Presence/absence of chain-of-custody record(s).

B. Presence/absence of any customer Traffic Reports or Chronicles (SMO forms for EPA).

C. Presence/absence of airbills and/or bills of lading documenting the shipment of samples.

Note: Only the Sample Custodian may open external containers.

The Sample Custodian will remove the sample containers and record the following information on the DAT Chain-of-Custody Form:

A. Condition of the samples (intact, broken, leaking, etc.).

B. Presence/absence of sample tags/labels.

C. Compare with chain-of-custody record(s) -- if tag numbers are listed, do they match the sample tag numbers received?

1. Document either the fact that these numbers agree, or any discrepancy between the tag numbers received and those listed on the chain-of-custody record.

2. If sample tag numbers are not listed on the chain-of-custody record, this fact is recorded.

The Sample Custodian will compare the following documents to verify agreement among the information contained in them:

1. Chain-of-custody records

2. Sample tags

3. Traffic Reports or Chronicles (SMO forms)

4. Airbills or bills of lading

If any discrepancies are found, the appropriate official from the requesting agency or other client must be contacted (SMO in the case of EPA).

If all samples recorded on the chain-of-custody record were received by the lab and there were no problems observed with the sample shipment, the Sample Custodian will sign the chain-of-custody record in the "received for laboratory by:" box on the document, if such document exists. If problems are noted, the Sample Custodian will sign for the shipment and note problems in the remarks section of the DAT Chain-of-Custody Form or reference any additional documentation describing the problems.

The Sample Custodian will assign the DAT reference number to the sample and record this number, the client sample identification, the sample matrix, and storage location on the DAT Chain-of-Custody Form.

1. A copy of the DAT Chain-of-Custody Form will be maintained in the Sample Custodian's file.

2. The original of the DAT Chain-of-Custody Form and the original signed chain of custody will be filed in the master project file located in the archive.

3. A copy of the DAT Chain-of-Custody Form will accompany the other paperwork to the project manager. If samples requiring different storage locations are received on the same project, they will be logged onto separate DAT Chain-of-Custody Forms so that the original Chain-of-Custody Form can accompany each sample set. This may require several pages for a given project containing such samples. This will be documented under the receiving remarks of the DAT Chain-of-Custody Form. The DAT Chain-of-Custody Forms will be numbered with increasing book and page numbers.

5. Sample Storage. The Sample Custodian is responsible for the storage of samples.

6. Order Log-in. The Sample Custodian is responsible for initiation of the order log-in

Sample Security

Samples are to be stored in an appropriate, secured area. Samples requiring refrigeration will be stored in a refrigerator, those requiring freezing, in a freezer, and all others in a cabinet. Access to samples brought inside the punch locked doors and stored inside a freezer, refrigerator, or cabinet will therefore require the opening of the punch lock.

Temperature monitoring and corrective actions

1. Temperature of all the refrigerators and freezers will be taken daily and recorded in a Refrigerator and Freezer Temperature Logbook.

2. Refrigerator temperature must be kept at 4(±4)°C.

3. Freezer temperature must be kept at -10°C to -25°C.

4. A thermometer must be placed in every refrigerator and cooler.

5. The thermostat will be adjusted if the measured temperature is outside the acceptable range.

6. Outside service will be called if thermostat adjustment is not successful. If the samples must be removed for the refrigerators during their repairs, they will be stored in a cooler at 4°C ± 4°C. The corrective action taken will be entered into the Temperature Logbook under Corrective Action Taken.

Internal Chain-of-Custody Documentation

A sample is within chain-of-custody when it is in a secure area, such as, behind the punch lock doors of the laboratory. It is defined to be in a person's custody when:

- It is in his actual possession, or

- It is in his view, after being in his possession, or

- It was in his possession and then locked or sealed up to prevent tampering.

Anytime a sample is removed from a secured storage area, the person handling the sample must document the transfer of sample custody. To provide this documentation, the person removing the sample must write on the DAT Chain-of-Custody Form, which is kept with the samples, his/her name, the date and time of sample removal, the DAT sample number, and the date and time the sample was returned. The person removing the samples will be responsible for following proper chain-of-custody procedures while the sample is in his/her possession.

Sampling Archiving

Only the Sample Custodian or his designated assistant will archive samples. Samples to be archived must meet the following:

- Analysis completed.

- Data pack sent to client.

- Acceptance of data by client.

- In case of fish or milk, client is called and asked if samples are to be returned. With client approval, they will be disposed.

Samples will be placed in storage in an upright position.

XVI. SOP Management Procedures

Each activity associated with analytical practices at DAT is to have an appropriately detailed SOP to follow.

New SOP's

The need for new SOPs can be determined when a new method is implemented, when the scope of the existing method is extended or when some activities are being performed without adequate SOPs. Such a need can be identified by the analyst involved in the production, someone from management, or the QA Unit can identify the need and request new SOPs, usually as a follow-up corrective action of the internal audit. SOPs are created to provide a clear, concise step-by-step description of the procedure with explanatory information to enable a person with the appropriate background to perform the procedure.

Revisions To Existing SOPs

Revisions are made to SOPs at any given time to reflect changes in procedures.

Responsibility and Approval

The employee is responsible for implementing or modifying SOPs. Management is responsible for approving SOPs. The QAO should be provided with an information copy.

Each new SOP or revision to existing SOPs must receive approval by the author's and the revisor's supervisor.

Appendix A

Product Schedule

DAT Method	Analyte	Matrix
EPA 350.1	Ammonia(colorimetric)	Liquid
EPA 9010B	Cyanide distillation	Liquid/Solid
RSK 175	Headspace (methane, ethane, ethene)	Liquid
608	Organochlorine pesticides and PCB's	Liquid/Solid
8010	Halogenated volatile organic compounds (GC/HSD)	Liquid/Solid
8011	EDB and DBCP by GC-ECD	Liquid/Solid
8015	Non-halogenated volatile organic compounds (GC/FID)	Liquid/Solid
8020	Volatile aromatic organic compounds (GC/PID)	Liquid/Solid
8021	Halogenated and volatile organic compounds (GC/PID/ECD)	Liquid/Solid
8030/8031	Acrolein, Acrylonitrile, Acetonitrile (GC/FID)	Liquid/Solid
8040/8041	Phenolic compounds (GC/FID)	Liquid/Solid
8080	Organochlorine pesticides and PCB's (GC/ECD/HSD)	Liquid/Solid
8090	Nitroaromatic compounds and cyclic ketones (GC/ECD/FID)	Liquid/Solid
8100	PNAs by GC-FID	
8260	Volatile organics by GC/MS	Liquid/Solid
8280	Dioxins Low Resolution for Land Ban	All
8270	Semivolatile organics by GC/MS	Liquid/Solid
8140/8141	Organo-Phosphorus Pesticides	Liquid/Solid
8150/8151	Chlorinated Herbicides	Liquid/Solid
8310	Polycyclic Aromatic Hydrocarbons (HPLC/Fluorescence)	Ground water/Wastes
8316	Acrylamide, acrylonitrile and acrolein by HPLC	Water/Soil
8318	N-Methylcarbamates by HPLC	Water/Soil
8330	Nitroaromatics and nitramine explosives by HPLC	Water/Soil/Grass/Tissue
8330M	Explosives plus NG and PETN(HPLC)	All
8332	Nitroglycerin by HPLC	Water/Soil/Grass/Tissue
9014	Hydrogen Cyanide	Water/Soil
9052	Chloride(colorimetric)	Water
9056	Inorganic anions by Ion Chromatography	Water/Soil/Tissue
9034	Hydrogen Sulfide	Water (titrimetric)
SW-8105/604	Phenols and substituted phenols	Assorted
Anthraquinone		Black liquor/Pulp/Filters/Waste Water
Catechols, Chlorinated Phenolic Compounds		Paper liquors/Waste Water
Methanol		Black liquor/Pulp/Filters/Waste
Mercaptains & Sulfides		Air/Water
Organotin Analysis		Water/Sediment/Soil
Nonylphenol		Water/Soil
Nonylphenol Ethoxylates		Water
Glycol Ethers		Water
Caprolactam		Water
Terpenes & Pinenes		Water
Sulfonyl Ureas		Water/Soil/Plants
Triazine Herbicides		Chemical Products
NIOSH and OSHA Methods		Assorted

Air Methods f[illegible] Ambient and Stack

Method 26	[illegible]ons by IC;CL;F;Br;I	Acid Catch
Method 9057	[illegible]oride from HCL/CL_2 train	Train
Cations	[illegible]ons by IC; inorganic or organic	DI water/other
Anions	[illegible]ons by IC; Method 9056	DI water/other
Method 0011	[illegible]ehydes and Ketones	Air/100 mL DNPH Impingers
SW-8315	[illegible]maldehyde and CAA Aldehydes and Ketones	Air/DNPH
CARB 430	[illegible]ehydes	Air/DNPH
NCASI	[illegible]maldehyde(colorimetric)	Air/chilled water
EPA 308	[illegible]maldehyde(colorimetric)	chilled water
CARB 426	[illegible]drogen Cyanide	Impinger catch(.1 N NaOH)
EPA 6	[illegible]lfur Dioxide	Impinger catch
EPA 7D	[illegible]itrogen Oxides (IC)	Impinger catch
EPA Draft	[illegible]osgene(GC/MS)	Impinger catch(toluene/DEA)
OSHA 61	[illegible]osgene (GC)	Adsorbent (XAD-2/2-HMP)
OSHA 50	[illegible]hylene Oxide	Adsorbent (HBR coated)
EPA 8	[illegible]fur Dioxide	Impinger catch
Draft EPA	[illegible]mmonia	.1 N H_2SO
NIOSH 6015	[illegible]mmonia	Acid Silica gel
SW-5041 VOST	[illegible]t	Tennax/Tennax charcoal
TO-1	[illegible]t	Tennax/Tennax charcoal
TO-2	[illegible]OCs by GC/MS	Carbon Molecular Seive
TO-3	[illegible]OCs by GC-FID/ECD	Trap
TO-4	[illegible]ganochlorine pesticides and PCB's	Air/Puf Cartridge
TO-5	[illegible]dehydes and Ketones	Air/10 mL DNPH Solution
TO-6	[illegible]osgene	Air/10 mL Aniline/toluene
TO-7	[illegible] Nitrosodimethylamine	Adsorption tube
TO-8	[illegible]enol and Methyl phenols	Air (NaOH impinger catch)
TO-10	[illegible]ganochlorine pesticides	PUF
TO-11	[illegible]rmaldehyde	Air/DNPH coated Silica Gel
TO-13	[illegible]olycyclic Aromatic Hydrocarbons	Air/XAD or Puf
TO-14A	Ambient/source Volatiles	Summa Canister
TO-15	Ambient/source/stack	Summa Canister
TO-17	Ambient/source/stack	Adsorbent Tubes
Method 305	Total HAPS	Process, waste
Method 308	Methanol	Air/Water
NIOSH 2000	Alcohols	Silica gel tube
NIOSH 2010	Amines	Silica gel tube
NIOSH 7904	Hydrogen Cyanide(ISE)	Catch .1 N NaOH
Method NCASI [illegible]1	Chloroform	Tennax
Method 18	VOCs	Bags/tubes/summa
Method 16	Mercaptans & Sulfides	Bags/filters/water
Method 25D, 25[illegible]	VOCs	Waste
All NIOSH/OSHA Methods		Tubes/impingers/cassettes

Appendix B

Equipment Listing

Data/Analysis Technologies, Inc.

Chromatograph - HPLC

a. High Pressure Liquid Chromatography, Hewlett Packard model 1050
b. High Pressure Liquid Chromatograph - Beckman- Isocratic
c. High Pressure Liquid Chromatograph - Dionex Model 500
Detectors include UV, VIS, Fluor, EC, Conductivity

Chromatograph - GC

a. Hewlett Packard Model 5890 Series II; PID; ELCD, FID
b. Hewlett Packard Model 5890 Series II; ECD
c. Hewlett Packard Model 6890 GC;FPD;FID
d. Hewlett Packard Model 5890 Series II; ECD; NPD
e. Hewlett Packard ChemStation GC Data System; 2
f. Varian model 3700: FID; FPD

GC/MS
a. Hewlett Packard 5973 GC/MS/DS with 6890 GC-Entech Concentration system
b.. Finnigan 4500 GC-MS/DS
Upgraded to Hewlett Packard Model 5890 Series II gas chromatograph
Upgraded to Galaxy data system
c. Hewlett Packard 5988 GC/MS/DS system with HP Model 5890 Series II and Galaxy data system upgrade.

Spectrophotometric

a. Beckman DU UV/Visible Spectrophotometer
b. Fluorometer LDC
c. Fluorometer Ames
d. Hatachi UV/Visible Spectrophotometer

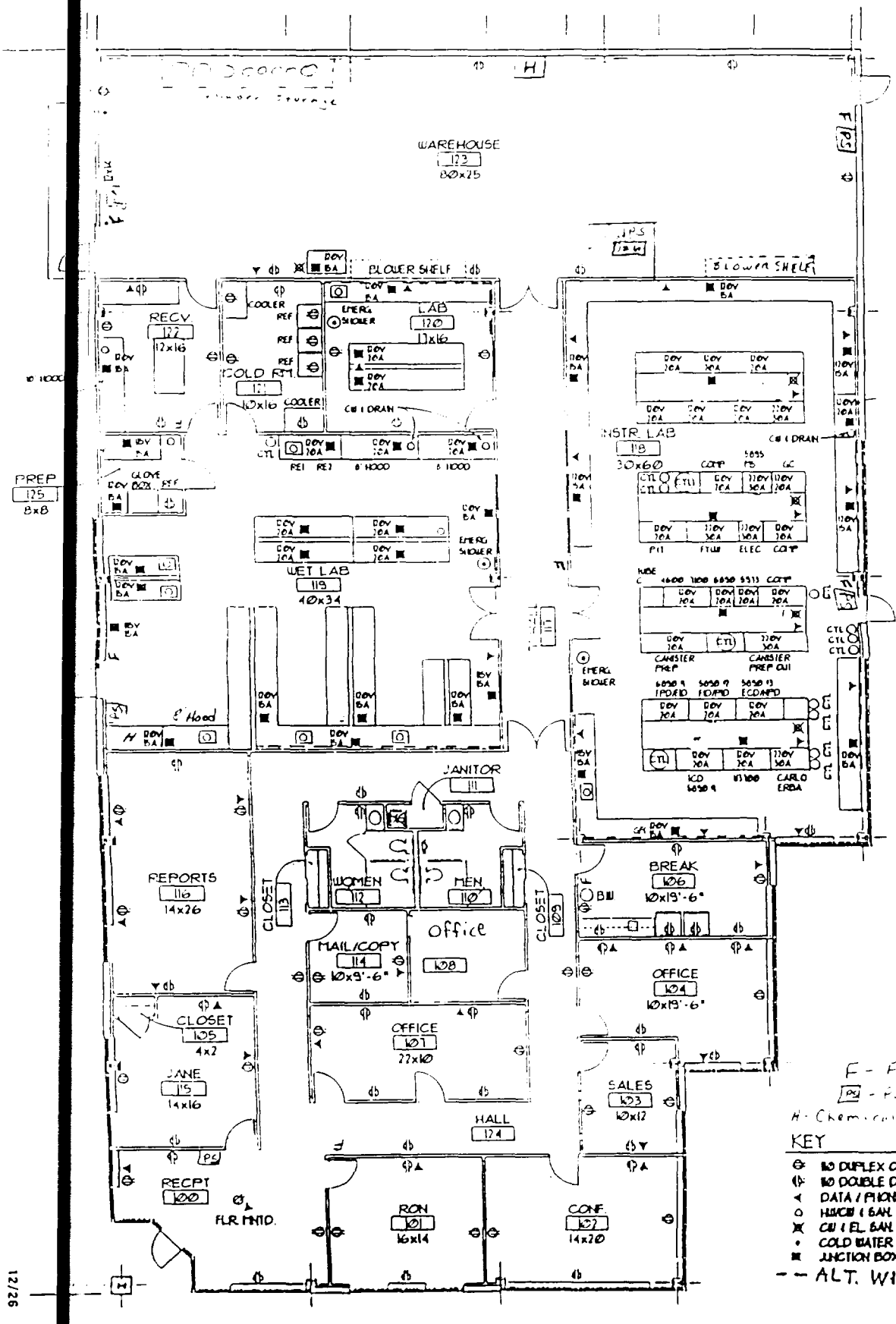
Other

a. 3- Hood; 1 ft chemical resistant, 1-5 hood ft chemical resistant
b. Hood; 5 ft laminar flow
c. Balance; Denver Instruments (0.00001 g.)
d . Balance; Denver Instruments (0.001 g.)
e. Balance Fisher model 711 (600 g.)

f. Oven; Fishe
g. 2-Rotary E aporator; Buchler;Buchii
h. 12-Soxhlet H ater; Precision
i. 2-Sonic Ba : Branson 3200
j. Centrifuge; n ernational UV
k. Furnace; T molyne 1400
l. Water Bath Precision
m. pH Meter: Orion 701 and 501
n. Calorimete Parr
o. Computers 14 Various (2-300;2-266;2-200;2-166;1-90;5-66)
p. Copy mach ne(3); Cannon 2020; Cannon 6650; Cannon 6551
q. Lab Bench 400 linear feet
r. Rotator; C le Palmer
s. Water Pur ication System; Barnsted
t. Refrigerat rs; 5
u. GPC - AB Autoprep
v. Labco o Glove Box
w. Entec TO-14 Concentrator
x. Entec Dynamic Diluter
y. Entec Canister Cleaner
z. Entec Automatic Sampler
aa. Entec Automatic Tube Sampler

Appendix C
Floor Plan

Data/Analysis Technologies, Inc. occupies a total of 10,000 sq ft of space. A sketch of the floor plan is on the following page.


WAREHOUSE
123
80x75
RECV
127
12x16
COLD RM.
121
10x16
COOLER
LAB
120
11x16
PLOWER SHELF
PREP
125
8x8
WET LAB
119
40x34
INSTR. LAB
118
30x60
JANITOR
111
REPORTS
116
14x26
CLOSET
113
WOMEN
112
MEN
110
CLOSET
109
MAIL/COPY
114
10x9'-6"
Office
108
BREAK
106
10x19'-6"
OFFICE
104
10x19'-6"
CLOSET
105
4x2
OFFICE
107
22x10
JANE
115
14x16
SALES
103
10x12
HALL
124
RECPT
100
FLR MNTD.
RON
101
16x14
CONF.
102
14x20
KEY
DUPLEX OUTLET
DOUBLE DUPLEX OUTLE
DATA / PHONE
COLD WATER
JUNCTION BOX
ALT. WIREMOL

12/26

Appendix D

Qualification Summary
(Key Personnel)

DAT, Inc.

Data/Analysis Technologies, Inc. (**DAT**) provides quality analytical products which are timely and cost effective. Our technical experience in applying EPA Methods to a variety of matrices is derived from the combined efforts of Dr. Mitchum, who as the lead scientist implements methods in the laboratory approves their use and reviews work products to assure method performance. Dr. Mitchum developed many of the EPA Methods in use today and implemented these methods in EPA. As an environmental scientist and quality assurance expert he also assures the methodology is applicable. He also brings to the company valuable skills which are used on each clients' program, through extraction, calibration and operation of the equipment necessry to perform the analysis requested.

DAT, Inc. is located in Plain City, Ohio. The laboratory capabilities include GC, GC/MS and HPLC in support of EPA, NIOSH, OSHA or DAT proprietary methodologies. The organizational structure allows our clients to obtain the services of a small laboratory providing that personal touch, fast turnaround and competitive pricing.

HPLC and GC analysis will be performed on the latest state of the art Hewlett Packard instrumentation. Our GC/MS facility is readily accessible and offers organic analytical and identification services.

DAT provides a complete analytical report containing all the QA/QC, chromatograms, blanks and standards necessary to completely validate the data package or a summary of analysis. A concise reduced format report is also available to support the needs of our clients. In the latter case the laboratory will retain the data package for later retrieval if needed by the client. The curriculum vitae of our key staff and their qualification statements are enclosed for your review.

QUALIFICATIONS STATEMENT
RONALD K. MITCHUM, Ph.D.

Summary of Qualifications:

Dr. Mitchum is an internationally recognized expert in the analytical chemistry and mass spectrometry of trace contaminants, EPA priority pollutants and Quality Assurance procedures. He has developed methods for RCRA, Superfund and Office of Toxic Substances, set regulatory limits for exposure while employed by the U.S. Food and Drug Administration, developed the U.S. EPA regulatory methods for analysis and was responsible for the quality of data produced by laboratories under contract to the U.S. Environmental Protection Agency. In his capacity as the Director of the Quality Assurance Division for the U.S. EPA Environmental Monitoring Systems Laboratory he directed audits of data, audits of laboratories, conducted a performance evaluation program and supplied the standard reference materials for the EPA monitoring efforts.

He was responsible for determining the quality of the data from the recent Love Canal Habitability Study, Denney Farms Dioxin Trial Burn, The National Dioxin Survey conducted by EPA and The Veterans Dioxin Study conducted by the Centers for Disease Control. He has developed innovative

methodologies fo he isolation and identification of halogenated and mixed halogenated dioxins and furans in car naceous and polymeric matrices. Developed regulations and reviewed industrial submissions of m t nods for commercial products for trace contaminants.

He has participat on international panels on environmental chemistry and organized international conferences and h is published over 100 research papers and presented numerous oral talks on environmental ch nistry.

Relevant Exper ence - 25 years

President of Dat Analysis Technologies, Inc., performs validation of environmental data, on site laboratory audi , environmental project management and coordination and environmental laboratory analy s

Dr. Mitchum w s president of Triangle Laboratories of Columbus, Inc and vice-president of Triangle Labor ories, RTP, Inc. He performed on site laboratory audits, validation of environmental ata and was directly involved in environmental project management and coordination an laboratory analysis.

Dr. Mitchum w s responsible for the environmental analytical chemistry program at Battelle. In this capacity h developed new market areas, introduced new technology for analysis and remediation and was responsible for the overall quality of the analytical chemistry research area at Battelle.

Dr. Mitchum c the development of regulatory methods and quality assurance presently in use today by the U S EPA for measuring toxic organic compounds in hazardous waste matrices, soil and tissue whil directing the Quality Assurance Division, EPA, Las Vegas, NV. These methods have served as he basis for U.S. EPA regulatory activities in quality assurance and the monitoring area. These me ods are presently being used for regulatory purposes under RCRA and SARA. The basic quan f cation and quality assurance parameters have been broadly used.

Also in this c acity he was responsible for the U.S. EPA contract laboratory program (CLP) quality assuran e program which provided quality assurance oversight to all analysis performed by contract labora ries throughout the United States. This included performing data audits, laboratory audits, standar s and performance evaluation programs.

He was respon ible for providing the quality assurance to the Love Canal Habitability Program. In this capacity h cted as an expert reviewing data, developing guidelines and certifying the overall quality of data He worked with Region II EPA and State environmental coordinators to complete the assignmen

He was respo ible for ensuring the quality of the National Dioxin Survey through quality assurance aud s and expert panel reviews. In this capacity all aspects of the National Dioxin Survey for the ational distribution of dioxins and furans from all sources were reviewed. An

expert panel was convened and Dr. Mitchum led the expert panel in discussions of the adequacy of testing methodologies, the data quality parameters and the review of the final report.

He was responsible for performing the assessment of the adequateness of the analytical and quality assurance procedures for the Veterans Dioxin in Blood study performed by the Center for Disease Control. In this capacity the quality assurance and methods were reviewed and audited. The adequateness of the procedures and testing methodology for dioxins and furans in human blood was assessed.

He was responsible for the organizing of international dioxin meetings while employed in the U.S. EPA and the U.S. FDA. He was responsible for major aspects of the 7th International Conference on Chlorinated Dioxins and Related Compounds, reviewing scientific submissions, organizing sessions and inviting speakers.

He is widely published in the analytical chemistry area having published over 40 abstracts and 70 peer reviewed papers and has given 71 oral presentations at scientific meetings.

Instrument Skills - MS Low Resolution - MS (HP-5970, Finnigan 4000, 4500, 5100, ITD, Kratos MS50, VG 70/70, VG TRIO, LKB-9000)

GC - HP, Varian, Perkin Elmer, AA - Perkin Elmer, ICP - Leaman, ICP - MS (VG, Siex)

SANDRA F. HEBENSTREIT

B.S., Chemistry

Summary Qualifications:

Performs Laboratory data validation of organic and inorganic data, ICP data and using EPA guidelines. Develops QC materials and QA methods. Trained in validation of dioxin/furan data and CLP organic and inorganic data using the USEPA National Functional Guidelines.

Instrumentation Skills: Perkin-Elmer spectrophotometer 603, Perkin-Elmer Spectrophotometer 5000, Perkin-Elmer ICP, Technician Auto Analyzer, Perkin-Elmer Zeeman, Conductivity bridge, PH meter, Spectrophotometer, Setaflash and Pensky-Martens closed cup flash point apparatus and turbidimeter.

Relevant Experience - 21 years

Chemist, Data Analysis Technologies, Inc. 1995-present.

Chemist, Triangle Laboratories, Inc., 1992-1995.

Ohio Department of Health, 1990-1991, Chemistry Lab Coordinator.

Ohio Department of Health, 1982-1990, Chemist II.

Ohio Department of Health, 1979-1982, Chemist I.

Chemical Abstracts Service, 1963-1969, Organic Index Editor.

D. JANE MITCHUM
B.S., Management Information Systems

Summary Qualifications:

Responsible for the management of finance and personnel. Operates computerized project management systems, accounting and inventory program. Manages data input and query for data validation staff. Trains data entry staff and supervises maintenance and retrieval operations.

As a Management Information Systems graduate possesses the skills to organize, interpret and query diverse databases. Computer programming skills in Cobal, Fortran, Basic, and Assembler languages. Managed unit operations in communications company for 12 years and was responsible for training and supervision.

Relevant Experience - 17 years

1995-present	Vice President/CFO, Data/Analysis Technologies, Inc., Dublin, OH.
1990-1995	Program Manager, Triangle Laboratories of Columbus, Inc., Dublin, OH.
1989-1990	Office Manager, E.P. Ferris & Associates, Inc., Columbus, OH.
1976-1978	Service Order Supervisor, Southwestern Bell Telephone Co., Little Rock, AR.
1965-1978	Service Order Supervisor, Southwestern Bell Telephone Co., Houston, TX.

QUALIFICATIONS STATEMENT

CURRICULUM VITAE

Ronald K. Mitchum, Ph.D.

Education:

B.S. Chemistry, 1968 Southwestern Oklahoma State University
Ph.D., Organic Chemistry, 1972 Oklahoma State University

Professional Experience

1995-present: Data/Analysis Technologies, Inc., President

1990-1995: Triangle Laboratories, Inc., Agrochemical Products Division., Columbus, OH, President

1990-1994: Triangle Laboratories, Inc., Columbus division, Dublin, OH, President

1988-1990: Department Manager: Methods Development Department, Battelle Memorial Institute, Columbus, Ohio.

1984-1988: U.S. EPA-Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. Director, Quality Assurance Division.

1982-1984: National Center for Toxicological Research, Department of Health and Human Services, Jefferson, Arkansas. Director, Division of Chemistry.

1980-1984: Consultant, Finnigan Corporation, Research, Development and Design.

1979-1984: Adjunct Associate Professor of Chemistry, University of Arkansas.

1983-1984: Adjunct Associate Professor, School of Medicine, University Medical Center, Little Rock, Arkansas.

1979-1982: National Center for Toxicological Research, Department of Health and Human Services, Jefferson, Arkansas. Supervisory Research Chemist, Chief, Spectroscopic Techniques Branch.

1976-1979: National Center for Toxicology Research, Department of Health and Human Services, Jefferson, Arkansas. Research Chemist.

1975-1976: University of Nebraska, Lincoln, Nebraska. Senior Postdoctoral Fellow; Kinetics of gas phase reactions.

1974-1975: University of Warwick, Coventry, England. Postdoctoral Research Fellow; Development of negative ion gas phase chemistry.

1972-1974: University of Houston, Houston, Texas. Postdoctoral Research Fellow; Theoretical and analytical chemical ionization mass spectrometry.

Selected Invitations and Achievements

(1) Invited to participate in WHO/IARC Methods Validation, Lyon, France, 1984.

(2) Invited to participate in workshop on Risk Assessment in Reproductive and Developmental Toxicology, Little Rock, Arkansas, 1987.

(3) Invited to present a lecture on the "Practical Implementation of the TCLP", California Water Pollution Control Association, Sacramento, California, 1988.

(4) Invited to organize a Symposium on Environmental Analysis, 9th Rocky Mountain ACS Meeting, 1988.

(5) Invited to organize and chair a session of the 7th International Symposium on Chlorinated Dioxins and Related Compounds, Las Vegas, Nevada, 1987.

(6) Invited to participate in a workshop on Chemical Measurements of Aerosols and Associated Vapors, UCLA, Los Angeles, California, 1988.

(7) Invited to author chapter in book "Instrumental Analysis of Pollutants Handbook," Elsevier, 1988.

(8) Invited to present plenary lecture, - 3rd International Conference on Analytical Environmental Chemistry, Chung Li, Taiwan, 1988.

(9) Invited to present Keynote lecture on "Analytical Chemistry and the Regulatory Agencies", Ohio Valley Chromatography Symposium, 1988.

(10) Invited to present lecture on "A Government Perspective" before the Analytical Instrumentation (SAMA), 1988.

(11) Invited to present a lecture before the EPA Region 9 Laboratory Directors Meeting, San Francisco, California, 1988.

(12) Invited to present a lecture before the 12th Symposium on Aquatic Toxicology and Hazard Assessment, Sparks, Nevada, 1988.

(13) Guest Editor, Chemosphere vol. 18, 1989.

(14) Invited to chair an EPA panel on TSCA Section 4 submissions, 1988.

(15) Invited to present lecture on Trace Analysis of Xenobiotics in Food Products, ACUPM/APHIS, Bethesda, MD, 1989.

(16) Invited to present lecture at HAZTECH-91 on "Lab Audits, Good Laboratory Practices and Data Validation", Pittsburgh, PA, 1991.

Membership in Professional or Honorary Societies

American Chemical Society, 1968 (Member)
American Society for Mass Spectrometry, 1975 (Member)
Association of Official Analytical Chemistry, 1989 (Member)

Offices, Committee Assignments Held in Professional and Honorary Societies

Chairman, Analytical Chemistry Session, Southwest Regional American Chemical Society meeting, 1979.

Chairman Elect (1983) of the Central Arkansas section of the American Chemical Society

Chairman (1984) of the Central Arkansas section of the American Chemical Society.
Editorial Board member, Journal Environmental and Biomedical Mass Spectrometry, 1985-1989.

Selected Oral Presentations

(1) R. K. Mitchum, "Advances in Analytical Methods for Dioxins and Furans," Presented before the Symposium on Advances in Analytical Methods for Monitoring Organic Chemicals in the Environment-Water and Hazardous Wastes, Cincinnati, Ohio, 1984.

(2) J. R. Donnelly, W. D. Munslow, T. L. Vonnahme, N. J. Nunn, G. W. Sovocool and R. K. Mitchum, "Extension of EPA RCRA Method 8280 to Analysis of Synthetic Bromo- and Bromochloro-Dioxins and Dibenzofurans," presented before the 7th International Symposium on Chlorinated Dioxins and Related Compounds", Las Vegas, NV, 1987.

(3) E. Nabb, D. Cardenes, J. R. Donnelly, L. C. Butler, R. F. Smiecinski, R. K. Mitchum, C. N. Rowland, "The Application of ICP/MS to Environmental Samples," presented before American Society for Mass Spectrometry, 35th Annual Conference, 1987.

(4) R. K. Mitchum, "Overview of EPA Methods and Quality Assurance," EPA Regional Laboratory Directors, San Francisco, California, 1987.

(5) R. K. Mitchum, "Practical Implementation of the TCLP," California Water Pollution Control Association, Sacramento, California, 1988.

(6) R. K. Mitchum, G. W. Sovocool, S. R. Donnelly, and W. D. Munslow, "Brominated Dioxins and Furans," The Engineering Foundation Conference, Santa Barbara, California, 1988.

(7) R. K. Mitchum, "Quality Assurance for Laboratory Analysis," Workshop on Environmental Chemistry, Taipei, Taiwan, 1988.

(8) R. K. Mitchum, "U.S. EPA Analytical Methodology, 3rd Annual Symposium on Analytical Chemistry, Chung Li, Taiwan, 1988.

(9) R. K. Mitchum, "Methods Development and Quality Assurance: EPAs Role," Ohio Valley Chromatography Symposium, Hueston Woods, Ohio, 1988.

(10) R. K. Mitchum, "Robotics: A Laboratory Perspective" Region 9 Laboratory Directors Conference, San Francisco, California, 1988.

(11) R. K. Mitchum, "Interagency Methods Harmonization: A Question of Need and Applicability", 12th Symposium on Aquatic Toxicology and Hazard Assessment, Sparks, Nevada, 1988.

(12) R. K. Mitchum, "Trace Analysis of Xenobiotics in Foods, Symposium 89 Epidemiology, Zoonoses, and Economics, Bethesda, MD, 1989.

(13) R. K. Mitchum, "Laboratory Audits, Data Validation and Good Laboratory Practices", HAZTECH-91, Pittsburgh, PA, 1991.

Selected Publications

(1) R. K. Mitchum and S. Pyle, "Environmental Applications of the Ion Trap Mass Spectrometer," Spectra, 11, 21 (1988).

(2) R. K. Mitchum, "Environmental Levels and Fate of Dioxins and Furans," Chemosphere, 16, 2193 (1987).

(3) W. A. Korfmacher, L. D. Betowski, C. L. Holder and R. K. Mitchum, "Characterization of Doxylamine and Pyrilamine Metabolites via TS/MS and TS/MS/MS," Biomed. Environ. Mass Spectrom., 15, 561 (1988).

(4) G. W. Sovocool, R. K. Mitchum, Y. Tondeur, W. D. Munslow, T. L. Vonnahme, and J. R. Donnelly, "Bromo- and Bromochloro-Polynuclear Aromatic Hydrocarbons, Dioxins and Dibenzofurans in Municipal Incinerator Fly Ash," Biomed. Environ. Mass Spectrom., 15, 669 (1988).

(5) G. W. Sovocool, J. R. Donnelly, W. D. Munslow, T. L. Vonnahme, N. J. Nunn, Y. Tondeur and R. K. Mitchum, "Analysis of Municipal Fly Ash for Br- and BrCl-dioxins and Dibenzofurans and Related Compounds," Chemosphere, 18, 193-200 (1989).

(6) J. R. Donnelly, W. D. Munslow, T. L. Vonnahme, N. J. Nunn, G. W. Sovocool, and R. K. Mitchum, "Preparation of BrCl Dioxins and Dibenzofurans and Analysis by RCRA 8280," Chemosphere, 18, 209-216 (1989).

(7) W. D. Munslow, J. R. Donnelly, R. K. Mitchum, and G. W. Sovocool, "Synthesis of Polyhalogenated Dibenzo-p-dioxins," Chemosphere, 18, 225-233 (1989).

(8) R. K. Mitchum, "Mass Spectrometry of Environmental Pollutants," invited chapter in Instrumental Analysis of Pollutants Handbook, C. N. Hewitt, ed., Elsevier Applied Science Pubs., LTD, Essex, England, in press (1991).

(9) R. K. Mitchum and J. R. Donnelly, "Quality Assurance/Quality Control Procedures for PCDD, PCDF and PCB Analysis", Chapter 9 in Environmental Carcinogens - Methods of Analysis and Exposure Measurement Volume 11 -Polychlorinated Dibenzodioxins, Dibenzofurans and Biphenyls, in press by International Agency for Research on Cancer, Lyon, France, 1990.

(10) J. R. Donnelly, A. H. Grange, N. J. Nunn, G. W. Sovocool, W. C. Brumley and R. K. Mitchum, "Analysis of Thermoplastic Resins for Brominated Dibenzofurans", Biomed. Environ. Mass Spectrom., 18, 884-896 (1989).

(11) Y. Tondeur, F. Varco, M. Chu, R. K. Mitchum, J. Hass, C. Mazac, D. McAllister, P. Rankin, R. Gorsich and M. Freiberg, "Ultra-Trace PBDD/PBDF in Retardant Chemicals: A New Methodology, Development Challenges and QA Guidelines", Kyoto Conference in Dioxins; MSW Incineration, 1991.

(12) T.W. Cochran and R.K. Mitchum, "Quality Assurance Plans: How, When, What and Why", Waste Management Symposium Proceedings, Durham, NC, 1993.

(13) R.K. Mitchum, R.L. Detra and G. Riley, "Suggestions for the analysis of Aldehydes and Ketones using EPA Method 0011", Waste Management Symposium Proceedings, Durham, NC, 1993.

CURRICULUM VITAE

Sandra F. Hebenstreit

Education:

B. S. Major Chemistry. Minor in Mathematics. St. Francis College in Loretto, PA. May 1963.

Professional Experience:

1995-present	Quality Assurance Specialist, Data/Analysis, Technologies, Inc.
1992-present	Chemist, Triangle Laboratories of Columbus, Inc., Dublin, OH.
1990-1991	Chemistry Lab Coordinator, The Ohio Department of Health- Columbus, OH.
1982-1990	Chemist II, The Ohio Department of Health- Columbus, OH.
1979-1982	Chemist I, The Ohio Department of Health- Columbus, OH.
1963-1969	Organic Index Editor, Chemical Abstracts Service- Columbus, OH.

Laboratory Skills:

EPA CLP, Non-CLP, Dioxin/Furan and Methods 8000, 500, and 600 Data Validation.

Atomic Absorption Spectroscopy. Flame, HGA and cold vapor Methods - analyzed potable and non-potable waters, leachates, oils, paint chips, filters and solids for metals.

Auto-Analysis. Analyzed potable and non-potable waters for total phosphorus, chloride, fluoride and sulfate.

Wet Chemistry. Analyzed potable and non-potable waters for Alkalinity, acidity, PH, hardness, turbidity, hexachrome, BOD, COD and conductivity.

Helped to initiate the hazardous waste program by preparing samples for analysis using the EP Toxicity method. Determined the flash points of various types of samples. Developed and implemented new and updated old methods of chemical analysis.

Helped Supervise and train other chemists.

Selected and structured chemical compounds from multi-language patents and edited names and information in abstracts for the annual and collective indexes.

CURRICULUM VITAE

D. Jane Mitchum

Education:

B.S. Management Information Systems, 1987 University of Nevada, Las Vegas, NV

Professional Experience:

1995-present	Vice President/CFO, Data/Analysis Technologies, Inc.
1990-1995	Triangle Laboratories, Inc., Agrochemical Products Division, Columbus, OH
	Program Manager - data entry specialist and management systems development. Trained and performed data entry for CLP and non-CLP validation. Developed information systems.
1989-1990	E. P. Ferris & Associates, Inc., Columbus, OH
	Office Manager - designed and installed computerized job costing and accounting system. Duties included accounting, payroll, job costing and invoicing.
1976-1978	Southwestern Bell Telephone Co., Little Rock, AR
	Service Order Supervisor - responsible for initial and continuation training of new employees in all aspects of computerized database entry. Supervised employee unit, being responsible for all aspects of the employees job performance.
1965-1978	Southwestern Bell Telephone Co., Houston, TX

Service Order Supervisor - converted Bell area to computerized database. After conversion complete, supervised employee unit performing database entry. Responsibilities were scheduling maintaining good working relationship with other departments and ensuring accurate and timely work completion.

CURRICULUM VITAE

Theresa Schwope

Education:

B.S., Chemistry	The Ohio State University, Columbus, OH, 1995

Professional Experience:

1995-present	Quality Assurance Specialist, Data/Analysis Technologies, Inc.
10/95-11/95	Quality Assurance Specialist, Triangle Laboratories of Columbus, Inc., Dublin, OH.
1991-1995	Medical Transcription, Self-employed.
1979-1991	Laboratory Technician, Ironsides Co., Columbus, OH.
1977-1979	Laboratory Technician, Defense Logistics agency, Philadelphia, PA.

Laboratory Skills:

Finnigan 4500 Series Mass Spectrometer, Hewlett Packard 5890 Series Gas Chromatography System, Hewlett Packard 1050 Series HPLC System, Hewlett Packard Fluorescence and UV Detectors, Dionex DX500 Series Ion Chromatography System.

Extraction Procedures for US EPA 8000 Series Methods, NIOSH Methods, OSHA Methods.

MISSOURI DEPARTMENT OF
HEALTH

Mel Carnahan
Governor

Coleen Kivlahan, M.D., M.S.P.H.
Director

P.O. Box 570, Jefferson City, MO 65102-0570 • 314-751-6400 • FAX 314-751-6010

April 6, 1995

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APR 10 1995

SPFD BRANCH
REGION VII

Mr. Robert L. Morby
Chief, Superfund Branch
Superfund Division
U. S. Environmental Protection Agency
726 Minnesota Ave.
Kansas City, KS 66101

Dear Mr. Morby:

Enclosed please find Preliminary Remediation Goals (PRGs) for the Arneson Timber Site in Steelville, Missouri. The U. S. Environmental Protection Agency (EPA) requested the Missouri Department of Health (DOH) calculate PRGs for pentachlorophenol in soil. Methodology presented in Risk Assessment Guidance for Superfund (RAGS), Volume I: Human Health Evaluation Manual, Parts A and B, were used to calculate PRGs. PRGs were calculated for residential, industrial and recreational visitor scenarios. EPA requested carcinogenic PRGs be calculated at 1×10^{-5} and 1×10^{-6} cancer risk levels and noncarcinogenic PRGs be calculated at a hazard index of 1.0.

Carcinogenic PRGs calculated for a resident adult were lower than those calculated for a resident child. This is due to the short exposure duration for the child (6 years) relative to a lifetime of 70 years. It is a function of the mathematics involved in making these calculations and may not be toxicologically appropriate.

Carcinogenic PRGs calculated for the resident adult, resident child and adult worker were lower than Missouri's Any-Use Soil Level (ASL) for pentachlorophenol (42 mg/kg). While the methodology used to calculate PRGs and ASLs is similar, different exposure variable values are used in the calculation of Missouri's ASLs. Additionally, Missouri's ASLs do not take dermal contact into consideration.

If you have any questions or need additional information, please feel free to contact Mr. Chuck Arnold or Ms. Cherri Baysinger-Daniel at (314) 751-6111.

Sincerely,

Daryl W. Roberts
Chief
Bureau of Environmental Epidemiology

RECEIVED
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SU

cc: Paul Doherty, Superfund Branch, EPA

Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, Missouri

Prepared by:

Missouri Department of Health
Bureau of Environmental Epidemiology
210 El Mercado Plaza, P. O. Box 570
Jefferson City, Missouri 65102

The Arneson Timber Site is located in Crawford County, in the vicinity of Steelville, MO. Activities at the site included treatment of wood products with pentachlorophenol (PCP). As a result, surface and near-surface soils at the site are contaminated with PCP. The U. S. Environmental Protection Agency (EPA) requested the Missouri Department of Health (DOH) calculate preliminary remediation goals (PRGs) for pentachlorophenol in soil.

Presently, the site is abandoned. Current human exposure is limited to individuals visiting the site. Thus, PRGs were calculated for a recreational visitor scenario. This scenario was defined as a juvenile (7 to 18 years of age) visiting the site 48 days/year ingesting and directly contacting PCP-contaminated soil. During the time the site was operational, land use was industrial. Future land use may remain industrial; however, there is nothing which prohibits future land use at the site from becoming residential. Therefore, PRGs were calculated for residential and industrial scenarios. The residential scenario was defined as an adult and child living on the site 350 days/year. The industrial scenario was defined as an adult working on the site 250 days/year. These individuals would ingest and directly contact soil contaminated with PCP.

PRGs were calculated for carcinogenic and noncarcinogenic effects using the following formulae:

Carcinogenic:

$$PRG = \frac{TR \times BW \times ATc \times 365\ days/year}{EF \times ED \times CF \times [(SFo \times IR \times FI) + (SFabs \times SA \times AF \times ABS)]}$$

Noncarcinogenic:

$$PRG = \frac{THI \times BW \times ATn \times 365\ days/year}{EF \times ED \times CF \times [(1/RfDo \times IR \times FI) + (1/RfDabs \times SA \times AF \times ABS)]}$$

Exposure variable definitions and values used in the equations are shown in Table 1.

Table 1.
Variable Values Used to Calculate Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, MO

ıriable	Adult Resident	Child Resident	Adult Worker	Juvenile Visitor
W = Body Weight (kg)	70 [a]	15 [a]	70 [a]	44 [b]
Tc = Averaging Time - carcinogenic (years)	70 [a]	70 [a]	70 [a]	70 [a]
Tn = Averaging Time - noncarcinogenic (years)	30 [a]	6 [a]	25 [a]	11 [c]
= Exposure Frequency (days/year)	350 [a]	350 [a]	250 [a]	48 [c]
) = Exposure Duration (years)	30 [a]	6 [a]	25 [a]	11 [c]
= Conversion factor (kg/mg)	1×10^{-6} [d]	1×10^{-6} [d]	1×10^{-6} [d]	1×10^{-6} [d]
= Soil Ingestion Rate (mg/day)	100 [a]	200 [a]	50 [a]	100 [a]
= Fraction Ingested (unitless)	1.0 [c]	1.0 [c]	1.0 [c]	1.0 [c]
A = Skin Surface Area (cm2)	5000 [f]	1850 [b]	2000 [f]	3325 [b]
= Skin Adherence Factor (mg/cm2)	1 [f]	1 [f]	1 [f]	1 [f]
BS = Absorption Factor (unitless)	0.25 [g]	0.25 [g]	0.25 [g]	0.25 [g]
o = Oral Slope Factor (kg-day/mg)	See Table 2.			
abs = Absorbed Slope Factor (kg-day/mg)	See Table 2.			
Do = Oral Reference Dose (mg/kg-day)	See Table 2.			
Dabs = Absorbed Reference Dose (mg/kg-day)	See Table 2.			
= Target Cancer Risk (unitless)	Defined by EPA.			
II = Target Hazard Index (unitless)	Defined by EPA.			

EPA 1991b.
EPA 1990.
Scenario-specific assumption.
EPA 1991a.
EPA 1989.
EPA 1992.
CAL EPA 1994.

PRG formulae were modified from equations found in EPA's Risk Assessment Guidance for Superfund (RAGS), Volume I: Human Health Evaluation Manual (Part A, EPA 1989) and Part B: Development of Risk-Based Preliminary Remediation Goals (EPA 1991a).

Oral Slope Factor (SFo) and Reference Dose (RfDo) values for pentachlorophenol used in assessing carcinogenic and noncarcinogenic effects from ingestion of soil were obtained from EPA's Integrated Risk Information System (IRIS). These values are presented in Table 2. In order to assess carcinogenic and noncarcinogenic effects from dermal exposure, SFo and RfDo toxicity values were adjusted from the administered doses found in IRIS to absorbed doses. Absorbed Slope Factor (SFabs) and Reference Dose (RfDabs) values were calculated using the following formulae (EPA 1989):

$$SF_{abs} = SF_o / \text{Oral Absorption Efficiency}$$

$$RfD_{abs} = RfD_o \times \text{Oral Absorption Efficiency}$$

The oral absorption efficiency used in making this adjustment was 86 percent (0.86). This value was provided by EPA's Environmental Criteria and Assessment Office (ECAO, Appendix B).

EPA requested carcinogenic PRGs be calculated at 1×10^{-5} and 1×10^{-6} target cancer risk (TR) levels and noncarcinogenic PRGs be calculated at a Target Hazard Index (THI) of 1.0. Carcinogenic and noncarcinogenic PRGs calculated for adult and child residents, an adult worker and a juvenile visitor are presented in Tables 3 and 4. Final PRGs calculated at each target cancer risk were determined by comparing calculated PRGs and selecting the lowest value.

Table 2.

Toxicity Values Used to Calculate Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, MO

Carcinogenic		
Slope Factor (kg-day/mg)		
Oral	Dermal	Target Organ
0.12*	0.14	Liver and vascular tumors.
Noncarcinogenic		
Reference Dose (mg/kg-day)		
Oral	Dermal	Critical Effect
0.03*	0.026	Liver/kidney pathology

*Value obtained from EPA's Integrated Risk Information System (IRIS).

Table 3.

Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, MO
1 x 10^{-5} Target Cancer Risk

Exposure Scenario	Carcinogenic (mg/kg)	Noncarcinogenic (mg/kg)	Final PRG (mg/kg)
Resident Adult	9.1	1420	9.1
Resident Child	20.6	639.7	20.6
Adult Worker	37.7	4891	37.7
Juvenile Visitor	165.9	21540	165.9

Table 4.

Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, MO
1 x 10^{-6} Target Cancer Risk

Exposure Scenario	Carcinogenic (mg/kg)	Noncarcinogenic (mg/kg)	Final PRG (mg/kg)
Resident Adult	0.9	1420	0.9
Resident Child	2.1	639.7	2.1
Adult Worker	3.8	4891	3.8
Juvenile Visitor	16.6	21540	16.6

References:

State of California Environmental Protection Agency (CAL EPA). 1994. Preliminary Endangerment Assessment Guidance Manual. Department of Toxic Substances Control. January 1994.

U. S. Environmental Protection Agency (EPA). 1989. Risk Assessment Guidance for Superfund. Volume I: Human Health Evaluation Manual (Part A). Office of Emergency and Remedial Response, Washington, D. C. EPA/540/1-89/002.

U. S. Environmental Protection Agency (EPA). 1990. Exposure Factors Handbook. Office of Health and Environmental Assessment, Washington, D. C. EPA/600/8-89/043.

U. S. Environmental Protection Agency (EPA). 1991a. Risk Assessment Guidance for Superfund. Volume I: Human Health Evaluation Manual, Part B: Development of Risk-Based Preliminary Remediation Goals. Office of Solid Waste and Emergency Response, Washington, D. C. OSWER Directive 9285.7-01B.

U. S. Environmental Protection Agency (EPA). 1991b. Standard Default Exposure Factors. Office of Solid Waste and Emergency Response, Washington, D. C. OSWER Directive 9284.6-03.

U. S. Environmental Protection Agency (EPA). 1992. Dermal Exposure Assessment: Principles and Applications. Office of Health and Environmental Assessment, Washington, D. C. EPA/600/8-91/011B.

U. S. Environmental Protection Agency (EPA). 1995. Integrated Risk Information System (IRIS) Database. March 1995.

Appendix A

Preliminary Remediation Goal Worksheets for the Arneson Timber Site, Steelville, MO

Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, MO

Residential Adult Exposure: 1 x 10-5 Cancer Risk Level

Carcinogenic PRG formula:

PRG (mg/kg) = (TR x BW x AT x 365 days/yr) / [EF x ED x CF x ((SFo x IR x FI) + (SFabs x SA x AF x ABS))] = **9.10873**

Where:	Value Used
TR = Target Risk (unitless)	0.00001
BW = Body Weight (kg)	70
AT = Averaging Time (years)	70
EF = Exposure Frequency (days/year)	350
ED = Exposure Duration (years)	30
CF = Conversion Factor (kg/mg)	0.000001
SFo = Oral Slope Factor (kg-day/mg)	0.12
SFabs = Absorbed Slope Factor (kg-day/mg)	0.14
IR = Ingestion Rate (mg/day)	100
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	5000
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Residential Adult Exposure: 1 x 10-6 Cancer Risk Level

Carcinogenic PRG formula:

PRG (mg/kg) = (TR x BW x AT x 365 days/yr) / [EF x ED x CF x ((SFo x IR x FI) + (SFabs x SA x AF x ABS))] = **0.91087**

Where:	Value Used
TR = Target Risk (unitless)	0.000001
BW = Body Weight (kg)	70
AT = Averaging Time (years)	70
EF = Exposure Frequency (days/year)	350
ED = Exposure Duration (years)	30
CF = Conversion Factor (kg/mg)	0.000001
SFo = Oral Slope Factor (kg-day/mg)	0.12
SFabs = Absorbed Slope Factor (kg-day/mg)	0.14
IR = Ingestion Rate (mg/day)	100
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	5000
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Residential Adult Exposure: Hazard Index = 1

Noncarcinogenic PRG formula:

PRG (mg/kg) = (THI x BW x AT x 365 days/yr) / [EF x ED x CF x ((1/RfDo x IR x FI) + (1/RfDabs x SA x AF x ABS))] = **1420**

Where:	Value Used
THI = Target Hazard Index (unitless)	1
BW = Body Weight (kg)	70
AT = Averaging Time (years)	30
EF = Exposure Frequency (days/year)	350
ED = Exposure Duration (years)	30
CF = Conversion Factor (kg/mg)	0.000001
RfDo = Oral Reference Dose (mg/kg-day)	0.03
1/RfDo = Inverse of Reference Dose (kg-day/mg)	33.3333
RfDabs = Absorbed Reference Dose (mg/kg-day)	0.026
1/RfDabs = Inverse of Absorbed Reference Dose (kg-day/mg)	38.46
IR = Ingestion Rate (mg/day)	100
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	5000
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, MO
Residential Child Exposure: 1 x 10-5 Cancer Risk Level

Carcinogenic PRG formula:

PRG (mg/kg) = (TR x BW x AT x 365 days/yr) / [EF x ED x CF x ((SFo x IR x FI) + (SFabs x SA x AF x ABS))] **20.5634**

Where:	Value Used
TR = Target Risk (unitless)	0.00001
BW = Body Weight (kg)	15
AT = Averaging Time (years)	70
EF = Exposure Frequency (days/year)	350
ED = Exposure Duration (years)	6
CF = Conversion Factor (kg/mg)	0.000001
SFo = Oral Slope Factor (kg-day/mg)	0.12
SFabs = Absorbed Slope Factor (kg-day/mg)	0.14
IR = Ingestion Rate (mg/day)	200
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	1850
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Residential Child Exposure: 1 x 10-6 Cancer Risk Level

Carcinogenic PRG formula:

PRG (mg/kg) = (TR x BW x AT x 365 days/yr) / [EF x ED x CF x ((SFo x IR x FI) + (SFabs x SA x AF x ABS))] = **2.05634**

Where:	Value Used
TR = Target Risk (unitless)	0.000001
BW = Body Weight (kg)	15
AT = Averaging Time (years)	70
EF = Exposure Frequency (days/year)	350
ED = Exposure Duration (years)	6
CF = Conversion Factor (kg/mg)	0.000001
SFo = Oral Slope Factor (kg-day/mg)	0.12
SFabs = Absorbed Slope Factor (kg-day/mg)	0.14
IR = Ingestion Rate (mg/day)	200
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	1850
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Residential Child Exposure: Hazard Index = 1

Noncarcinogenic PRG formula:

PRG (mg/kg) = (THI x BW x AT x 365 days/yr) / [EF x ED x CF x ((1/RfDo x IR x FI) + (1/RfDabs X SA x AF x ABS))] = **639.674**

Where:	Value Used
THI – Target Hazard Index (unitless)	1
BW = Body Weight (kg)	15
AT = Averaging Time (years)	6
EF = Exposure Frequency (days/year)	350
ED = Exposure Duration (years)	6
CF = Conversion Factor (kg/mg)	0.000001
RfDo = Oral Reference Dose (mg/kg-day)	0.03
1/RfDo = Inverse of Reference Dose (kg-day/mg)	33.3333
RfDabs = Absorbed Reference Dose (mg/kg-day)	0.026
1/RfDabs = Inverse of Absorbed Reference Dose (kg-day/mg)	38.46
IR – Ingestion Rate (mg/day)	200
FI – Fraction Ingested (unitless)	1
SA - Skin Surface Area (cm2)	1850
AF = Skin Adherence Factor (mg/cm2)	1
ABS - Absorption Factor (unitless)	0.25

Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, MO
Adult Worker Exposure: 1 x 10-5 Cancer Risk Level

Carcinogenic PRG formula:

PRG (mg/kg) = (TR x BW x AT x 365 days/yr) / [EF x ED x CF x ((SFo x IR x FI) + (SFabs x SA x AF x ABS))] = **37.6526**

Where:	Value Used
TR = Target Risk (unitless)	0.00001
BW = Body Weight (kg)	70
AT = Averaging Time (years)	70
EF = Exposure Frequency (days/year)	250
ED = Exposure Duration (years)	25
CF = Conversion Factor (kg/mg)	0.000001
SFo = Oral Slope Factor (kg-day/mg)	0.12
SFabs = Absorbed Slope Factor (kg-day/mg)	0.14
IR = Ingestion Rate (mg/day)	50
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	2000
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Adult Worker Exposure: 1 x 10-6 Cancer Risk Level

Carcinogenic PRG formula:

PRG (mg/kg) = (TR x BW x AT x 365 days/yr) / [EF x ED x CF x ((SFo x IR x FI) + (SFabs x SA x AF x ABS))] = **3.76526**

Where:	Value Used
TR = Target Risk (unitless)	0.000001
BW = Body Weight (kg)	70
AT = Averaging Time (years)	70
EF = Exposure Frequency (days/year)	250
ED = Exposure Duration (years)	25
CF = Conversion Factor (kg/mg)	0.000001
SFo = Oral Slope Factor (kg-day/mg)	0.12
SFabs = Absorbed Slope Factor (kg-day/mg)	0.14
IR = Ingestion Rate (mg/day)	50
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	2000
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Adult Worker Exposure: Hazard Index = 1

Noncarcinogenic PRG formula:

PRG (mg/kg) = (THI x BW x AT x 365 days/yr) / [EF x ED x CF x ((1/RfDo x IR x FI) + (1/RfDabs X SA x AF x ABS))] = **4890.73**

Where:	Value Used
THI = Target Hazard Index (unitless)	1
BW = Body Weight (kg)	70
AT = Averaging Time (years)	25
EF = Exposure Frequency (days/year)	250
ED = Exposure Duration (years)	25
CF = Conversion Factor (kg/mg)	0.000001
RfDo = Oral Reference Dose (mg/kg-day)	0.03
1/RfDo = Inverse of Reference Dose (kg-day/mg)	33.3333
RfDabs = Absorbed Reference Dose (mg/kg-day)	0.026
1/RfDabs = Inverse of Absorbed Reference Dose (kg-day/mg)	38.46
IR = Ingestion Rate (mg/day)	50
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	2000
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Preliminary Remediation Goals for Pentachlorophenol in Soil at the Arneson Timber Site, Steelville, MO
Trespasser Exposure: 1 x 10-5 Cancer Risk Level

Carcinogenic PRG formula:

PRG (mg/kg) = (TR x BW x AT x 365 days/yr) / [EF x ED x CF x ((SFo x IR x FI) + (SFabs x SA x AF x ABS))] = **165.855**

Where:	Value Used
TR = Target Risk (unitless)	0.00001
BW = Body Weight (kg)	44
AT = Averaging Time (years)	70
EF = Exposure Frequency (days/year)	48
ED = Exposure Duration (years)	11
CF = Conversion Factor (kg/mg)	0.000001
SFo = Oral Slope Factor (kg-day/mg)	0.12
SFabs = Absorbed Slope Factor (kg-day/mg)	0.14
IR = Ingestion Rate (mg/day)	100
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	3325
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Trespasser Exposure: 1x 10-6 Cancer Risk Level

Carcinogenic PRG formula:

PRG (mg/kg) = (TR x BW x AT x 365 days/yr) / [EF x ED x CF x ((SFo x IR x FI) + (SFabs x SA x AF x ABS))] = **16.5855**

Where:	Value Used
TR – Target Risk (unitless)	0.000001
BW – Body Weight (kg)	44
AT = Averaging Time (years)	70
EF = Exposure Frequency (days/year)	48
ED = Exposure Duration (years)	11
CF = Conversion Factor (kg/mg)	0.000001
SFo = Oral Slope Factor (kg-day/mg)	0.12
SFabs = Absorbed Slope Factor (kg-day/mg)	0.14
IR = Ingestion Rate (mg/day)	100
FI – Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	3325
AF = Skin Adherence Factor (mg/cm2)	1
ABS – Absorption Factor (unitless)	0.25

Trespasser Exposure: Hazard Index = 1

Noncarcinogenic PRG formula:

PRG (mg/kg) = (THI x BW x AT x 365 days/yr) / [EF x ED x CF x ((1/RfDo x IR x FI) + (1/RfDabs X SA x AF x ABS))] = **21539.6**

Where:	Value Used
THI = Target Hazard Index (unitless)	1
BW = Body Weight (kg)	44
AT = Averaging Time (years)	25
EF = Exposure Frequency (days/year)	48
ED = Exposure Duration (years)	11
CF = Conversion Factor (kg/mg)	0.000001
RfDo = Oral Reference Dose (mg/kg-day)	0.03
1/RfDo = Inverse of Reference Dose (kg-day/mg)	33.3333
RfDabs = Absorbed Reference Dose (mg/kg-day)	0.026
1/RfDabs = Inverse of Absorbed Reference Dose (kg-day/mg)	38.46
IR = Ingestion Rate (mg/day)	100
FI = Fraction Ingested (unitless)	1
SA = Skin Surface Area (cm2)	3325
AF = Skin Adherence Factor (mg/cm2)	1
ABS = Absorption Factor (unitless)	0.25

Appendix B

Contaminant-Specific Oral Absorption Efficiencies

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL CRITERIA AND ASSESSMENT OFFICE
CINCINNATI, OHIO 45268

NOV [illegible] 1991

SUBJECT: Review of the Oral Bioavailability Factors, Dermal Permeability Constants and Dermal Bioavailability Factors and Method used to Estimate Health Risks Due to Exposure of Volatile Organic Compounds Associated with Indoor Groundwater Use (Woodstock Landfill Site/Woodstock, IL)

FROM: Kenneth A. Poirier
Director
Superfund Health Risk Technology Support Center
Chemical Mixtures Assessment Branch

TO: Bill Bolen
U.S. EPA
Region V

This memorandum is in response to a request submitted by Mike Kierski of Warzyn Engineering for review of proposed oral bioavailability factors, dermal permeability constants and dermal bioavailability factors and review of the method used to estimate health risks due to exposure of volatile organic compounds associated with indoor groundwater use.

Attached please find the following information:

Attachment I: Contains a memorandum from John Schaum of the Exposure Assessment Branch to Cindy Sonich-Mullin, Acting Chief, Chemical Mixtures Assessment Branch. John Schaum has reviewed and provided comment on the following information:

1. Proposed procedure used to adjust oral dose-response factors for application to dermal doses
2. Proposed procedures used to assess dermal contact with water
3. Proposed procedures used to assess dermal contact with soil (organics and inorganic)

Attachment II: Table of Oral Absorption Values.

The provided oral absorption factors were obtained from available EPA documents and ATSDR Toxicological Profiles on the chemicals provided. Combined references are provided following the oral absorption table.

ORAL ABSORPTION FACTORS

Chemical	EPA Documents	ATSDR Profiles
4-methylphenol	HEEP 1985 Rats: 65-80%	1990 Public Comment Draft Humans: no data Rabbits: 65-84%
Naphthalene	HA 1987 Rats: 26-39% Updated HEA 1988 Rats: minimum of 26-39%	1989 draft Absorbed but no quantitati data available.
Nickel	HEA 1986 Humans: 1-10%	1988 Humans: 1-10% Rats, dogs and mice: 1-10%
Pentachlorophenol	HA 1987 Nearly complete absorption in humans and animals HEEP 1986 Humans: minimum of 86% Animals: > 70%	1989 Humans: readily absorbed but not quantified Rats and Monkeys: > 90%
Phenol	AWQCD 1980 and Updated HEA Readily absorbed but not quantified	1989 Humans: 85-98% Rats: 95% Squirrel monkey: 31%
Selenium	HEED 1989 Human: No data. Animal: Rats 72.7%± 14.2% by gavage. 9.5%± 1.7% when fed. HEA 1984 Human: ^{75}Se 44%-70% Animal: Rats, ^{75}Se > 90%	1989 Humans: 80-97% Animals: 80-100%

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION VII
726 MINNESOTA AVENUE
KANSAS CITY, KANSAS 66101

February 8, 1999

BY FEDERAL EXPRESS

Arneson Timber Company, Inc.
c/o N. Arne Arneson
1600 South Watson Road
St. Louis, Missouri 63124

Mr. Charles N. Leezy
625 N. Franklin
Cuba, Missouri 65453

Re: Arneson Timber Site, Crawford County, Missouri

Dear Messrs. Arneson and Leezy:

Re: Executed Copy of Administrative Order on Consent

Attached to this letter please find an executed copy of the Administrative Order on Consent (AOC) for the Arneson Timber Site. In the process of final approval, a few typographical errors were discovered and corrected prior to presentation of the AOC to the Director of Region VII's Superfund Division for his signature. The corrections made are as follows:

1. The caption on the first page of the AOC was corrected to include reference Sections 107 and 122 of CERCLA, and the docket number for the AOC was added.

2. In Paragraph 1, 3rd sentence, the words "Consent Order" were capitalized.

3. In Paragraph 16, 2nd sentence, the words "was removed" were added to the sentence after the phrase "("penta" or "PCP") sludge" in order to create a complete sentence.

4. In paragraph 21, last sentence, a closing right parenthesis was added after (ppb).

5. In the last sentence of Footnote 1 (to Paragraph 23), the repeated words "the most" were deleted.

6. In Paragraph 70, 1st sentence, the word "their" was changed to "its" to properly reflect the singular reference to Respondent ATC.

7. In Paragraph 81, 1st sentence, the word "their" was changed to "its" to properly reflect the singular reference to Respondent ATC.

8. In Paragraph 82, 1st sentence, the word "effected" was corrected to "affected."

9. In Paragraph 90, 2nd sentence, the singular reference to "Respondent" was corrected to state "Respondents."

10. In Paragraph 97, the run-on of the address and zip code for mailing payments to EPA into the following sentence was corrected.

As described above, all the corrections deal with minor grammatical errors such as mistakenly repeated words, singular/plural agreement, etc., and do not change the substance of the AOC. Accordingly, the AOC is now effective upon your receipt of this executed copy of the document. Thank you again for your cooperation in these matters. If you have any questions on the terms of the AOC, or your obligations under the AOC, please do not hesitate to call me at (913) 551-7879.

Sincerely,

Howard C. Bunch
Assistant Regional Counsel

Attachments:

cc:

For Arneson Timber Company, Inc.

Fred Lafser
Consulting Engineer
409 Greenfield Drive
St. Louis, Missouri 63132

For Mr. Charles Leezy

Paula Colman
Jenkins and Kling, P.C.
10 S. Brentwood Blvd., Suite 200
St. Louis, Missouri 63105-1694

THE UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION VII
726 MINNESOTA AVENUE
KANSAS CITY, KANSAS 66101

IN THE MATTER OF: THE ARNESON TIMBER SITE, City of Steelville, Missouri	ADMINISTRATIVE ORDER ON CONSENT
	CERCLA DOCKET NO: CERCLA-7-99-0006
ARNESON TIMBER COMPANY, INC., a corporation and CHARLES LEEZY, an individual Respondents	
Proceedings under Sections 104, 106(a)107 and 122 of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) as amended by the Superfund Amendments and Reauthorization Act of 1986,(SARA),42 U.S.C. Sections 9604, 9606(a) 9607 and 9622	

TABLE OF CONTENTS

I. Introduction

1. This Administrative Order on Consent (CONSENT ORDER), is entered into voluntarily by the United States Environmental Protection Agency (EPA) and Arneson Timber Company, Inc. (ATC), a corporation, and Charles Leezy, an individual(hereafter collectively referred to as "Respondents"). This CONSENT ORDER requires Respondent ATC to perform work at the Arneson Timber Site (Site), including the implementation of the response action chosen by EPA for the Site, pursuant to 40 C.F.R. § 300.415 of the National Oil and Hazardous Substances Pollution Contingency Plan (NCP). By this CONSENT ORDER, Respondent Leezy agrees and is required to grant access to Respondent ATC to perform the response action, and to implement institutional controls for the Site that will limit unauthorized access and exposure to the hazardous substances at the Site, during the implementation of the response action.

II. Jurisdiction

2. This CONSENT ORDER is issued by the Director, Superfund Division of Region VII of EPA to Respondents Arneson Timber Company, Inc. (ATC) and Charles Leezy pursuant to the authority vested in the President of the United States by Sections 104, 106, 107 and 122 of the Comprehensive Environmental Response,

Compensation, and Liability Act (CERCLA), as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA) (Public Law 99-499, 42 U.S.C. §§ 9604, 9606, 9607 and 9622). This authority was delegated to the Administrator of EPA by Executive Order 12580, 52 Fed. Reg. 2923 (dated January 23, 1987), and further delegated to the Regional Administrators by EPA Delegations Nos. 14-14-A and 14-14-C (dated April 16, 1984, and February 26, 1987, respectively). This authority was subsequently delegated to the Director, Superfund Division, by EPA Region VII Delegation No. R7-14-14C (dated January 1, 1995).

3. Respondents' participation in this CONSENT ORDER shall not constitute or be construed as an admission of liability or of the findings or determinations contained in this CONSENT ORDER. Respondents agree to comply with and be bound by the terms of this CONSENT ORDER. Respondents consent to and will not contest EPA's authority or jurisdiction to issue or to enforce this CONSENT ORDER. Respondents further agree not to contest the basis or validity of this CONSENT ORDER or any of its terms.

4. Pursuant to Section 106(a) of CERCLA, 42 U.S.C. Section 9606(a), the State of Missouri has previously been notified of this CONSENT ORDER and will be sent a copy of the executed CONSENT ORDER.

III. Definitions

5. Unless otherwise expressly provided herein, terms used in this CONSENT ORDER which are defined in CERCLA, or in regulations promulgated under CERCLA, shall have the meaning assigned to them in the statute or its implementing regulations. Whenever terms listed below are used in this CONSENT ORDER, or in the documents attached to this CONSENT ORDER or incorporated by reference into this CONSENT ORDER, the following definitions shall apply:

a. "CONSENT ORDER" shall mean this Administrative Order on Consent and all attachments hereto. In the event of conflict between this CONSENT ORDER and any provision of any other agreement, order or writing, the terms and conditions of this CONSENT ORDER shall control.

b. "Day" shall mean a calendar day unless expressly stated to be a working day. "Working day" shall mean a day other than a Saturday, Sunday, federal or state holiday. In computing any period of time under this CONSENT ORDER, where the last day would fall on a Saturday, Sunday, federal or state holiday, the period shall run until the end of the next Working day.

c. "National Contingency Plan" or "NCP" shall mean the National Oil and Hazardous Substances Pollution Contingency

Plan promulgated pursuant to Section 105 of CERCLA, 42 U.S.C. § 9605, codified at 40 C.F.R. Part 300 et seq., as amended.

d. "Paragraph" shall mean a portion of this CONSENT ORDER identified by an Arabic numeral, a letter of the alphabet or a lower case Roman numeral.

e. "Parties" shall mean the United States and the Respondents.

f. "Respondents" shall mean Arneson Timber Company, Inc. (ATC) and Mr. Charles Leezy. Arneson Timber Company, Inc., is a company incorporated in the State of Missouri with N. Arne Arneson as Registered Agent at 1600 South Warson Road, St. Louis, Missouri 63124. Charles Leezy is the current owner of the property, which he obtained by bequest and inheritance from his father, Mr. Leroy Leezy.

g. "RCRA" shall mean the Solid Waste Disposal Act as amended by the Resource Conservation and Recovery Act and the Hazardous and Solid Waste Amendments, 42 U.S.C. § 6901 et seq.

h. "Section" shall mean a portion of this CONSENT ORDER identified by a Roman numeral and includes one or more paragraphs, unless used to refer to a statutory or regulatory section.

i. "Site" shall include the former Arneson Timber wood treatment facility, located in an unincorporated wooded area between Cuba and Steelville, Crawford County, Missouri, bounded on the south-southeast by Lucky Clover Road, and located approximately 100 yards west-southwest of the intersection of that road and Missouri Route 19. The Site covers an area of approximately 1.07 acres (See Site Map, Attachment II) within a larger parcel of property consisting of approximately 110 acres, defined by the following approximate legal description: The NE 1/4 of the SW 1/4 of Section 20, T38N, R4W of Crawford County, Missouri.

j. "Statement of Work" or "SOW" shall mean the statement describing the Work to be implemented at the Site, as set forth in Attachment I to this CONSENT ORDER, and any and all substitutions, modifications, or revisions made to such document, in accordance with this CONSENT ORDER.

k. "United States" shall mean the United States of America, and any and all agencies and instrumentalities thereof.

l. "Work" shall mean all activities Respondents are required to perform under this CONSENT ORDER, including all activities required to be undertaken pursuant to the terms and conditions of this CONSENT ORDER and the SOW set forth in Attachment I.

IV. Statement of Purpose

6. By entering into this CONSENT ORDER, the mutual objectives of EPA and the Respondents for the former Arneson Timber wood treatment facility (hereinafter referred to as "Site") that is located near Steelville, Missouri are, inter alia, to set forth the terms and conditions under which Respondent ATC will perform work at the Site, including the implementation of the removal response action chosen by EPA for the Site, consistent with the NCP, 40 C.F.R. Part 300, and the implementation of institutional controls to limit unauthorized access and exposure to the hazardous substances at the Site.

V. Applicability

7. This CONSENT ORDER shall be binding upon Respondents, their successors, assigns, subsidiaries, and upon all persons, agents, contractors and consultants acting under or for the Respondents in carrying out the actions required by this CONSENT ORDER.

8. Respondents shall provide a copy of this CONSENT ORDER to each contractor, subcontractor, laboratory, and consultant retained to conduct any portion of the work performed pursuant to this CONSENT ORDER prior to such contractor's, subcontractor's, laboratory's or consultant's initiation of work conducted under this CONSENT ORDER.

9. Any contract entered by the Respondents for the purpose of carrying out this order shall incorporate the requirements of this CONSENT ORDER pertaining to the work to be performed or services or materials to be supplied.

10. No change in the ownership of the Site, or any portion thereof, shall alter Respondents' obligations under this Order.

11. Respondents shall give notice of this CONSENT ORDER to any successor in interest prior to transfer of ownership or operation of the Facility and shall provide EPA written

is located approximately 100 yards west-southwest of the intersection of that road and Missouri Route 19. There are currently eight residences within an estimated one quarter mile of the Site.

13. The potential contaminated area of the Site covers an area of approximately 1.07 acres, within a larger parcel of approximately 110 acres that is defined by the following approximate legal description: The NE 1/4 of the SW 1/4 of Section 20, T38N, R4W of Crawford County, Missouri.

14. While ATC operated at the Site it used pentachlorophenol in its timber milling and wood preservation operations. The Site currently contains one (1) wood frame buildings, various cement foundations (including a drip pad), remnants of a large sawdust pile, and a drinking water well that is no longer in use.

15. Based on information available to EPA, the relevant chronology of ownership and operation of the Site is as follows:

a. The Site was initially leased by W.H. Barnicle, as an individual, from about 1970-1972. In 1972, Arneson Timber Company purchased the treatment tanks and other equipment from Barnicle who was since died). The Site was subsequently leased by Respondent Arneson Timber Company, Inc., in 1972, as part of a larger 110 acre parcel of land,

then owned by a Mr. LeRoy Leezy. Respondent ATC operated its timber cutting and wood treatment/preservation business at the Site from 1972 to 1983.

b. In February, 1987, Charles Leezy inherited the property by bequest pursuant to a Quit Claim Deed transferred from his father LeRoy Leezy, who died in December, 1984.

c. The Site has not been in active use for approximately sixteen (16) years (1983-1999).

16. In January, 1983, Respondent ATC moved its wood treatment operations to an industrial park in Steelville, Missouri. During the movement of the facility, a 9,000 gallon capacity treatment tank containing pentachlorophenol ("penta" or "PCP") sludge was removed, during which penta sludge was removed from the tank and deposited in an unlined natural catch basin located near the tank. When the tank was removed, the hole left in the ground by the tank was partially filled with waste penta sludge, which was then drained to the catch basin.

17. By letter dated January 20, 1983, the Missouri Department of Natural Resources (MDNR) directed ATC to cleanup the area contaminated by the disposal of the penta sludge.

18. In response to MDNR's order, Respondent ATC attempted to remove the penta sludge from the surface tank hole and the catch

basin. Respondent directed the removal and placement of penta sludge and contaminated soils from the surface storage tank hole and catch basin into nine fifty-five gallon drums.

19. In February, 1983, MDNR performed an inspection of the cleanup efforts conducted by Respondent ATC, and found an oily layer visible on ground water about two feet below the surface, near an area of visible oily surface contamination. In response to this finding, MDNR requested that Respondent ATC conduct further cleanup of the Site.

20. Following Respondent ATC's additional cleanup activities, a total of eleven (11) fifty-gallon drums of contaminated sludges/soils were removed from the Site to a licensed hazardous waste disposal site in Sauget, Illinois. A bulldozer then filled in both the hole left by the removed tank and the nearby catch basin with soil from adajcent areas. The soil used to fill in the catch basin was from a berm which supported a trackway used to transfer treated lumber from the pressure tank to the drip pad. During normal daily operations, freshly treated lumber would drip penta solution to the earthern berm, which became

21. In December, 1983, EPA sampled at depths between 1.5 to 9 feet at the location of the former containment basin where Respondent ATC had disposed of the penta sludge. This sampling exposed areas of discolored soil. The results of a surface soil sample taken from the containment area demonstrated concentrations of PCP as high as 1,700 mg/kg (parts per million (ppm)). The results of samples taken between 4 to 6 feet demonstrated concentrations of PCP between 34 and 9,000 ppm. The results of samples taken from an on-site drinking well also revealed concentrations of PCP at 2.2 μg/l (parts per billion (ppb)).

22. EPA conducted further sampling at the Site in July, 1986, and August, 1986. The results of the 1986 sampling demonstrated concentrations of PCP between 6.5 and 340 ppm. EPA's 1986 investigation also indicated that some off-site migration of PCP had occurred, as surface sediment samples in intermittent channels near the Site demonstrated PCP at levels of 2.4 and 5.6 ppm.

23. Effective June 6, 1991, EPA listed residuals from chlorophenolic wood treatment processes as F032 hazardous waste pursuant to the Resource Conservation and Recovery Act (RCRA). The basis for this listing was the health risks posed by such

residuals and their typical by-products, such as dioxins[1] and furans (55 FR 50450 (December 6, 1990); 52 FR 53282 (December 30, 1988), Table 11).

24. In June, 1991, EPA conducted a Site Assessment in order to further characterize the extent of contamination on Site; Specifically, PCP's potential contaminants and/or byproducts, dioxins, and furans. The results of the 1991 sampling demonstrated concentrations as high as 710 ppm of PCP in soils.

25. Additionally, the June 1991 sampling detected the presence of tetra, penta and octa isomers of dioxin. The 1991 sample results were evaluated for potential health risks by comparison to the toxicity of 2,3,7,8-TCDD, and the calculation of a 2,3,7,8-TCDD "equivalent value." This analysis resulted in calculated 2,3,7,8-TCDD "equivalent values" ranging from 1.2 to

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[1]. Dioxins, or chlorinated dibenzo-p-dioxins, are divided into groups or isomers based on the number of chlorine atoms in the compound. The group with one chlorine atoms is called the mono-chlorinated dioxin(s). The groups with two through eight chlorine atoms are called di-, tri-,penta, hexa-,hepta- and octa-clorinated dioxin(s). The chlorine atom can be attached to the dioxin molecule at any one of eight positions. The name of the dioxin isomer indicates the position of the chlorine atoms. For example, the dioxin isomer with four chlorine atoms at positions 2,3,7 and 8 on the dioxin molecule is called 2,3,7,8-tetrachlorodibenzo-p-dioxin, or 2,3,7,8-TCDD. Different isomers of dioxin pose different health risks, with the most toxic being the 2,3,7,8-TCDD ("tetra") isomer.

10 ppb, with a strong correlation found between the locations of PCP and the 2,3,7,8-TCDD equivalents.

26. In May, 1996, EPA conducted additional sampling for PCP in soil at the Site (in response to plans by the Missouri Highway and Transportation Department (MHTD), which planned a realignment of Missouri Highway 19 and of Lucky Clover Road (which would take the new roadway through portions of the Arneson Timber Site), to determine the extent of PCP contamination on property that would be impacted by MHTD activities, and to allow for EPA and MHTD to develop options for dealing with the contaminated soil.

27. The results of the 1996 sampling demonstrated concentrations of PCP as high as 1,400 ppm in the area of the catch basin used to initially store the penta sludge prior to off-site removal. Additionally, 2,3,7,8-TCDD equivalent values were calculated at concentrations ranging from 0.8 to 2 ppb. The results of the 1996 investigation determined that an area of the Site, roughly 300 by 150 feet in size, was contaminated by PCP to levels greater than 5 ppm, from surface to depths of 4 feet.

28. The surface soil at the Site is a stony loam 8 to 15 inches deep that is classified within the Hobson-Coulstone-Clarksville series. The subsoil is a well-developed red clay containing iron

enrichment derived from the underlying dolomite and sandstone formation. Sandstone outcrops are typical surface features on the Site. The Site is in a karst area with the underlying aquifer approximately 160-180 feet below ground surface.

29. The Meramec River is 1,500 feet south (downgradient) of the Site. Heavy precipitation events have washed soil and sediments contaminated with PCP into swales draining off-site towards the Meramec River. Vehicles or persons trafficking on and off the Site may also spread the contaminants by entrainment. This possiblity exists due to the presence of nearby residences and road frontage. During the dry season the surface soils can become very dry, thereby increasing the potential for wind blown spreading of contaminants.

30. An ongoing threat of releases and migration of hazardous substances is posed by potential weather conditions at the Site, particularly heavy precipitation events which may wash contaminated soil and sediments into the drainageway leading off Site, as well as dry seasons which increase the potential for wind blown spread of contaminants.

31. Human and environmental exposure to the hazardous substances, pollutants and/or contaminants released at the Site may result in the following toxicological effects:

a. **Pentachlorophenol** - Pentachlorophenol is an herbicide, fungicide, slimicide, and algacide. The EPA Integrated Risk Information System lists PCP as a probable human carcinogen (B2). The probable lethal dose for a 70 kilogram person is 50-500 mg/kg (one teaspoon to one ounce). Potential routes of exposure include ingestion and inhalation. Ingestion of PCP has been shown to cause lung, liver, and kidney damage as well as contact dermatitis. Ingestion symptoms include increased then decreased respiration, blood pressure, and urinary output; fever; increased bowel action; motor weakness; collapse with convulsions; and death. Inhalation results in acute poisoning of the circulatory system with eventual heart failure. Exposures to dusts and mists containing PCP have also been shown to cause visual damage, scotoma, inflammation of conjunctiva, cornea opacity, cornea numbness, and slight pupil dilation. Persons with kidney and liver diseases are particularly susceptible to adverse health effects from exposures to PCP.

b. **Dioxin isomers** - EPA has determined that concentrations of 2,3,7,8-TCDD greater than 1 ppb, or other dioxin isomers with a cumulative toxicity equivalent to that of 1 ppb

2,3,7,8-TCDD, pose a risk to human health and the environment, Specifically:

i. 2,3,7,8-TCDD - EPA has determined that 2,3,7,8-TCDD is a possible human carcinogen when considered alone, and a probable human carcinogen when associated with chlorophenols, such as pentachlorophenol. Exposure to 2,3,7,8-TCDD may cause acne-like lesions ("chlorane"). Exposure to 2,3,7,8-TCDD has also been observed to cause skin rashes, discoloration, excessive body hair, and changes in blood and urine that may indicate liver damage. Alterations in the ability of the liver to metabolize (or breakdown) hemoglobin, lipids, sugar and protein have also been reported. An increase in the risk of diabetes and abnormal glucose tolerance tests have also been observed.

ii. Other Dioxin isomers - Some dioxin isomers are more harmful than others. For example, the "octa" isomer of dioxin is estimated as 1,000 times less toxic than 2,3,7,8-TCDD. However, dioxin isomers with five (penta, or six (hepta) chlorine atoms substituted in the 2,3,7,8 positions, are extremely toxic to animals.

EPA has also determined that the "hepta" dioxin isomer is a probable human carcinogen.

VII. EPA's Conclusions of Law

32. Respondents are "persons" as defined in Section 101(21) of CERCLA, 42 U.S.C. § 9601(21).

33. Respondent ATC was an "operator" of the Site at the time of "disposal" of "hazardous substances," as these terms are defined in Section 101 of CERCLA, 42 U.S.C. § 9601. Respondent Charles Leezy is the current "owner" of the Site, as this term is defined in Section 101(20) of CERCLA, 42 U.S.C. § 9601(20).

34. The Site is a "facility" within the meaning of Section 101(9) of CERCLA, 42 U.S.C. § 9601(9).

35. Respondent ATC is liable under Section 107(a)(2) of CERCLA, 42 U.S.C. § 9607(a)(2), as a "person" who was the operator of the Site at the time of disposal of hazardous substances. Further, Respondent ATC is liable under Section 107(a)(3) of CERCLA, 42 U.S.C. § 107(a)(3), as a "person" who arranged for the disposal of hazardous substances.

36. Pentachlorophenol and dioxins are "hazardous substances" within the meaning of Section 101(14) of CERCLA, 42 U.S.C. § 9601(14).

37. A "release" of hazardous substances into the environment has occurred at the Site, as defined by Sections 101(8) and 101(22) of CERCLA, 42 U.S.C. §§ 9601(8) and 9601(22).

VIII. EPA's Determinations

38. Based upon the foregoing Findings of Fact and Conclusions of Law, the Director of EPA Region VII's Superfund Division has determined that:

a. The conditions present at the Site may constitute an imminent and substantial endangerment to public health, welfare, or the environment, within the meaning of Section 106(a) of CERCLA, 42 U.S.C. § 9606(a);

b. EPA is authorized to act pursuant to Section 106(a) of CERCLA, 42 U.S.C. § 9606(a), to issue such orders as may be necessary to protect public health and the environment;

c. The actions required by this CONSENT ORDER are necessary to protect the public health and welfare and the environment; and

d. The actions ordered, and agreed to by Respondents, under the terms of this CONSENT ORDER are in the public interest and, if carried out in conformance with the requirements of this CONSENT ORDER, will be consistent with the National Contingency Plan, 40 C.F.R. Part 300, et seq.

IX. Work to be Performed

39. Based upon the foregoing Findings of Fact, Conclusions of Law and Determinations, Respondents are hereby ordered and agree to perform the work described hereafter;

40. Within ten (10) days of the effective date of this CONSENT ORDER, Respondent Charles Leezy shall establish Institutional Controls to control unauthorized access to the contaminated areas of the Site and shall apply to place the Site on the State Registry, in accordance with the requirements of Task I of the SOW. Respondent Charles Leezy shall also be responsible for providing access to the Site at all reasonable times, in accordance with Section XIII (Access) of this CONSENT ORDER. Respondent Charles Leezy's duties with regard to the Work to be performed at the Site are limited to those enumerated in this paragraph and referred to in Task I of the SOW for implementing Institutional Controls and in Section XIII (Access) of this CONSENT ORDER for providing access. Excluding the references to Respondent Charles Leezy in this paragraph, all references to Respondent in Section IX (Work to be Performed) of this CONSENT ORDER, are applicable only to Respondent Arneson Timber Company, Inc. (ATC).

41. Within thirty (30) days of the effective date of this CONSENT ORDER, Respondent ATC shall prepare and submit a draft Removal Action Work Plan (RAWP) to EPA for review and comment. Within forty-five (45) days of the effective date of this CONSENT ORDER Respondent ATC shall prepare and submit a final Removal Action Work Plan (RAWP) for EPA's review and approval, prepared in accordance with Tasks II-IV of the SOW, and that addresses any comments made by EPA on the draft RAWP. The RAWP shall describe the tasks and schedules required to accomplish the excavation and/or off-site disposal and/or bioremediation of soils at the Site, to achieve the goal of reducing contamination below EPA's "any use" action levels, and shall be prepared in accordance with the Statement of Work (SOW) to this CONSENT ORDER. (For surface soils between 0 to 48 inches in depth, EPA's "any use" action level for PCP is 9.1 mg/kg. For soils between 0 to 48 inches in depth, EPA's "industrial use" action level is 37.7 mg/kg of PCP. For soils between 0 to 12 inches in depth the "any use" action level is 1 ug/kg dioxin equivalents. For soils at depths between 0 and 48 inches, EPA's "industrial use" action level is 10.0 ug/kg dioxin equivalents).

42. The RAWP shall propose a removal action that includes any or all of the following components: (1) the removal and off-site

disposal of all soils contaminated above EPA's "any use" action levels; (2) the removal and off-site disposal of soils contaminated above EPA's "industrial use" action levels, combined with bioremediation to attempt to treat remaining contamination to EPA's "any use" action levels; (3) and/or the consolidation of soils contaminated above EPA's "industrial use" action levels and the use of bioremediation to reduce remaining soil contamination to EPA's "any use" action levels.

43. The RAWP shall state and require that if the approved removal action does not reduce on-site soil contamination, at depths less than four (4) feet, to at least EPA's "industrial use" levels, Respondent shall remove and properly dispose of off-site soils that remain contaminated above EPA's "industrial use" action levels. Respondent may propose alternate treatment methods to off-site disposal to EPA for review and approval, however, if these treatment methods are not approved by EPA, Respondent shall remain required to properly dispose of off-site those soils, at depths less than 4 feet, that remain contaminated above EPA's "industrial use" levels.

44. As components of the RAWP, Respondent ATC shall develop and submit a Sampling and Analysis Plan (SAP) and a Health and Safety

Plan (HSP), in accordance with Task II of the SOW. The RAWP shall be prepared in accordance with Tasks II and IV of the SOW.

45. The RAWP shall require the Removal Action be performed in accordance with the performance standards set forth in Task IV of the SOW.

46. If the RAWP proposes the use of bioremediation as a removal alternative to achieve "any use" action levels, the RAWP shall describe the tasks and schedules associated with excavation, construction of on-site in-situ treatment cells, a Bench scale Treatability Study, and if necessary, the off-site disposal of soils that exceed EPA's "industrial use" action levels for the Site. If the RAWP proposes the use of bioremediation to only treat soils contaminated at levels between EPA's "any use" and "industrial use" action levels, the Bench Scale Treatability Study may consist of the submission of the results of a literature review that demonstrates "passive" bioremediation and/or natural biological mechanisms may result in the reduction of contamination to EPA's "any use" action levels.

47. Upon EPA approval of the RAWP, Respondent ATC shall construct and implement the Removal Action in accordance with the approved designs, schedules and plans contained therein. Further, Respondent ATC shall conduct the Removal Action in

accordance with the performance standards outlined in Task IV of the SOW, as set forth in the approved RAWP.

48. Respondent ATC shall document that the purpose of the Removal Action has been accomplished, in accordance with the following procedures:

a. A Post-Construction/Removal Inspection and Report: Upon preliminary completion of the construction of the treatment cells, or completion of off-site removal of contaminated soils, Respondent ATC shall contact EPA for the purpose of conducting a Site inspection with EPA;

b. Post-Construction/Removal Monitoring: If EPA approves a removal alternative that includes some form of bioremediation to achieve "any use" action levels, Respondent shall perform Post-construction/Removal monitoring to determine the effectiveness of bioremediation to achieve EPA's "any use" and/or "industrial use" action levels.

c. A Final Inspection: Upon completion of any outstanding removal work and/or required monitoring, Respondent ATC shall notify EPA for the purpose of scheduling a final Site inspection, which shall consist of a walk-through inspection by EPA and Respondents of the project Site; and

d. Certification of Completion: Upon confirmation that the purpose, intent and requirements of the RAWP have been satisfied, EPA shall provide Respondents a written certification of completion of the Removal Action, in accordance with Task IV of the SOW.

49. Respondent ATC shall prepare the Work Plans and Reports set forth in Tasks I through IV of the SOW in a manner that accomplishes the design, construction, operation and maintenance, and monitoring requirements of the Removal Action.

50. Respondent ATC shall provide Progress Reports in accordance with Task V of the SOW. Respondent ATC shall provide EPA with signed, monthly progress reports by the 10th day of each calendar month following the effective date of this CONSENT ORDER until the date of EPA's approval of the Post-Construction/Removal Inspection Report. Thereafter, Progress Reports shall be submitted quarterly by the 10th day of each third calendar month.

51. Within thirty (30) days of the final Site inspection, Respondent ATC shall submit to EPA for review and approval a Removal Action Completion Report (Completion Report), in accordance with Task V of the SOW.

52. EPA may approve, approve with comments, disapprove, and/or require modifications to all Reports and/or Work plans, in

accordance with Section X of this CONSENT ORDER (Submissions Requiring EPA Approval). Once approved, or approved with modifications, the Reports and/or Work plans and all requirements and schedules contained therein shall become a part of and fully enforceable under this CONSENT ORDER. Respondent ATC shall implement the approved Reports and/or Work plans in accordance with the requirements and schedules contained therein.

X. Submissions Requiring EPA Approval

53. After review of any Report, Work plan, or other item which is required to be submitted for approval pursuant to this Consent Order, EPA may: (a) approve, in whole or in part, the submission; (b) approve the submission with modifications; (c) disapprove, in whole or in part, the submission, directing the Respondents to resubmit the document after modifications that address EPA's comments; (d) disapprove the submission and assume responsibility for performing all or any part of the response activities; or (e) any combination of the above.

54. In the event of approval, or approval with modifications by EPA, Respondent ATC shall proceed to take any action required by the plan, report, or other item, as approved or modified by EPA.

55. Upon receipt of a notice of disapproval Respondent ATC shall, within ten (10) working days, or such other time as

specified by EPA in such notice, correct the deficiencies and resubmit the plan, report, or other item for approval.

56. Notwithstanding the receipt of a notice of disapproval of a submission, Respondent ATC shall, at the direction of EPA, proceed to take any action required by any non-deficient portion of the submission. Implementation of any non-deficient portion of a submission shall not relieve Respondent ATC of any liability for stipulated penalties under Section XXIII (Stipulated Penalties) for the deficient portion of the submission.

57. In the event that a Work plan, Report, other item, or portion thereof, is resubmitted by Respondent ATC without adequately addressing EPA's comments, and EPA disapproves such a submittal, Respondent ATC shall be deemed to have failed to make the submittal in a timely and adequate manner and may be subject to stipulated penalties for such failure.

58. If Respondent ATC invokes dispute resolution over EPA's disapproval of a required submission, the provisions of Section XXII (Dispute Resolution) and Section XXIII (Stipulated Penalties) shall govern the terms of accrual and payment of any stipulated penalties for Respondent's failure to submit an approvable document and/or perform the required work during the period of dispute resolution. If EPA's disapproval of

Respondent's submission is upheld, stipulated penalties shall accrue for such violation from the date on which the disputed submission was required to have been submitted in an approvable form, as provided in Section XXIII (Stipulated Penalties).

59. All Reports, Work plans, and other items required to be submitted to EPA under this Consent Order shall, upon approval by EPA, be enforceable under this Consent Order. In the event EPA approves a portion of an item required to be submitted to EPA under this Consent Order, the approved portion shall be enforceable under this Consent Order.

XI. Contractors/Project Coordinators/Submittals

60. Within ten (10) days after the effective date of this CONSENT ORDER, Respondent ATC shall designate a Project Coordinator and shall submit the designated coordinator's name, address, telephone number, and qualifications to EPA. Respondent's Project Coordinator shall be responsible for administration of all the actions required of Respondents by the CONSENT ORDER. Respondent's Project Coordinator shall be present at the Site or readily available by telephone during Site Work.

61. EPA has designated Mr. Timothy Curry as its Project Coordinator. EPA's Project Coordinator shall have the

authority lawfully vested by the NCP in a Remedial Project Manager and On-Scene Coordinator.

62. All verbal notices and written documents, including, but not limited to written notices, reports, plans, and schedules, requested or required to be submitted to EPA pursuant to this CONSENT ORDER shall be directed to:

Mr. Timothy Curry
On-Scene Coordinator
ENSV, Superfund Division
U.S. Environmental Protection Agency
726 Minnesota Avenue
Kansas City, Kansas 66101

EPA and Respondent ATC shall direct all written submissions required by this CONSENT ORDER by certified or registered mail to each other's respective Project Coordinators.

63. For purposes of complying with a submission deadline, a submission shall be deemed delivered on the date indicated on the postmark or the certificate of service prepared by the sender. Response deadlines, however, shall be calculated from the date of actual receipt.

64. EPA and Respondent ATC shall have the right to change their designated Project Coordinators and contractors. Verbal notice of such change shall be provided to the other parties within

twenty-four (24) hours of such change and written notice shall follow within five (5) days of such change.

65. EPA retains the right to disapprove of any, or all, of the contractors, subcontractors or Project Coordinators proposed in the Work plans or subsequently selected by Respondents, pursuant to Section X (Submissions Requiring EPA Approval). If EPA disapproves of a proposed or selected contractor, subcontractor, or Project Coordinator, Respondent shall propose or retain a different person, and shall notify EPA of that person's name, address, telephone number, and qualifications within fifteen (15) days following EPA's disapproval.

XII. Periodic Reports and Meetings

66. Respondent ATC shall make presentations at, and participate in, meetings at the request of EPA during the initiation, conduct, and completion of the work to be performed under this CONSENT ORDER. In addition to discussion of the technical aspects of the work, topics may include anticipated problems or new issues.

67. Respondent ATC shall provide to EPA monthly written progress reports by the tenth (10th) day of each calendar month following the effective date of this CONSENT ORDER until the date of EPA's approval of the Post-Construction/Removal Inspection Report.

Thereafter, Respondent ATC shall submit Progress Reports quarterly by the 10th day of each third calendar month, in accordance with Task V of the SOW. Progress reports shall be submitted until Respondent ATC receive EPA's Certificate of Completion of the removal action, in accordance with Task IV of the SOW. The provisions of this paragraph shall not apply to Respondent Charles Leezy.

XIII. Access

68. At all reasonable times, Respondent Charles Leezy shall allow EPA, ATC and their authorized representatives to enter and freely move about all property at the Site and off-site areas where work required hereunder is being performed for the purposes of performing required work, inspecting conditions, activities, the results of activities, records, operating logs, and contracts related to the Site or Respondent ATC and its contractor(s) pursuant to this CONSENT ORDER; reviewing the progress of Respondents in carrying out the terms of this CONSENT ORDER; conducting tests as EPA or its authorized representatives deem necessary; using a camera, sound recording device or other documentary type equipment; and verifying the data submitted to EPA by Respondent ATC. Respondents shall allow these persons to inspect and copy all records, files, photographs, documents,

sampling and monitoring data and other writings related to the work undertaken in carrying out this Order. Nothing herein shall be interpreted as limiting or affecting EPA's right of entry or inspection authority under federal law. EPA shall be solely responsible for assuring compliance by its personnel and consultants with EPA's health and safety requirements during inspections.

69. If the Site, or any off-site area to which access is needed to carry out the terms of this CONSENT ORDER, is owned in whole or in part by a party other than Respondents, Respondent ATC shall obtain, or use its best efforts to obtain, site access agreements from the owner(s) of such property within fifteen (15) days of approval of any work plans for which site access is required. Such agreements shall provide access for EPA, its contractors and oversight officials, the State of Missouri and its contractors, and Respondents and their authorized representatives. Such agreements shall specify that Respondents are not EPA's representative(s) with respect to liability associated with Site activities. Copies of such agreements shall be provided to EPA prior to Respondents' initiation of field activities. "Best efforts" as used in this Section shall include

providing reasonable compensation to such property owner(s) in consideration of access.

70. If access agreements are not obtained within the time period referenced above, Respondent ATC shall immediately notify EPA of its failure to obtain access and shall provide documentation to EPA of its "best efforts" to obtain access. Additionally, Respondent ATC shall perform all other activities not requiring access to such off-site property and, if EPA performs the work, Respondent ATC shall reimburse EPA for all costs incurred in performing such activities. Respondent ATC shall integrate the results of any such tasks undertaken by EPA into its reports and deliverables.

71. In the event that Respondent ATC cannot obtain access agreements, EPA may obtain access for Respondent.

72. Nothing herein shall be construed as restricting the inspection or access authority of EPA under federal law.

XIV. Confidential Business Information

73. Respondents may assert a business confidentiality claim covering part or all of the information submitted pursuant to the terms of this CONSENT ORDER in the manner set out in 40 C.F.R. § 2.203(b). The information covered by such a claim will be disclosed by EPA only to the extent, and by the means of the

procedures, set forth in 40 C.F.R. Part 2, Subpart B. If no such claim accompanies the information when it is received by EPA, it may be made available to the public by EPA without further notice to the Respondents.

XV. Additional Work

74. During the course of the work performed pursuant to this CONSENT ORDER, EPA may determine that sampling, analysis, reporting or other tasks in addition to those specifically set forth herein are necessary to satisfy the purposes of this Order. If EPA so determines, it will advise Respondent ATC in writing of the nature of the additional tasks and the basis for EPA's determination. Respondent ATC shall undertake, perform, and complete all such additional tasks and provide such documents and reports required by EPA in addition to those provided for herein. The additional work shall be completed in accordance with the standards, specifications, and schedules determined or approved by EPA.

XVI. EPA Oversight

75. EPA may designate an On-Scene Coordinator (OSC) that shall have the authority vested in an On-Scene Coordinator by the NCP at 40 C.F.R. § 300.120. This includes the authority to halt, conduct, or direct any activities required by this CONSENT ORDER

and/or any response actions or portions thereof when conditions present an imminent and substantial endangerment to the public health or welfare or the environment or when necessary to assure that such activities or actions are consistent with the National Contingency Plan. The OSC, or any person designated by the OSC, shall have the right to move freely about the Site at all times when work is being carried out pursuant to this CONSENT ORDER and the attached Work Plan. In the event an OSC is not appointed, EPA's Project Coordinator or his designee shall be vested with the oversight authority described above.

XVII. Other Applicable Law

76. All actions required pursuant to this CONSENT ORDER shall be performed in accordance with all applicable local, state, and federal laws and regulations except as provided in CERCLA Section 121(e), 42 U.S.C. § 9621, and 40 C.F.R. § 300.415(j). In accordance with 40 C.F.R. § 300.415(j), all work required pursuant to this CONSENT ORDER shall, to the extent practicable, as determined by EPA, attain applicable or relevant and appropriate requirements ("ARARs") under federal environmental, state environmental, or facility siting laws. All ARARs shall be identified in the RAWP.

77. Any hazardous substances, pollutants or contaminants removed off-site pursuant to this CONSENT ORDER for treatment, storage or disposal shall be treated, stored, or disposed of at a facility in compliance, as determined by EPA, with the EPA Off-site Rule, 40 C.F.R. § 300.440, promulgated pursuant to 42 U.S.C. § 9621(d)(3).

XVIII. Force Majeure

78. Respondents shall perform the requirements of this CONSENT ORDER within the time limits set forth herein, unless the performance is prevented or delayed by events which constitute a force majeure. A force majeure is defined as any event arising from causes not foreseeable and beyond the control of Respondents, and their consultants and contractors, which could not be overcome by due diligence and which delays or prevents performance by a date required by this CONSENT ORDER. Such events do not include unanticipated or increased costs of performance, changed economic circumstances, normal precipitation events, or failure to obtain federal, state, or local permits.

79. Respondents shall notify EPA in writing ten (10) days after

demobilization and remobilization, a description of the cause of the delay and the measures taken or to be taken to minimize the delay, and an estimated timetable for implementation of these measures. Respondents shall adopt all reasonable measures to avoid and minimize the delay. Failure to comply with the notice provision of this Section shall constitute a waiver of Respondents' right to assert a force majeure.

80. If EPA determines that a delay has been or will be caused by a force majeure, the time for performance for that element of work may be extended, upon written approval of EPA, for a period equal to the delay resulting from such circumstances. Such an extension does not alter the schedule for performance or completion of other work required by this CONSENT ORDER unless specifically provided by EPA in its written approval.

XIX. Record Preservation

81. Respondent ATC shall preserve, during the pendency of this CONSENT ORDER and for a minimum of seven (7) years after its termination, all records and documents in its possession or in the possession of its divisions, employees, agents, accountants, or contractors which relate in any way to the work performed pursuant to this CONSENT ORDER, notwithstanding any internal document retention policy to the contrary.

XX. Subsequent Modification or Amendment

82. This CONSENT ORDER may be amended or modified by mutual agreement of EPA and Respondents. Such amendments or modifications shall be in writing, signed by the Respondent affected by the amendment and EPA, and shall become effective on the date on which a fully executed copy thereof is filed with the Regional Hearing Clerk, EPA Region VII.

83. No informal advice, guidance, suggestions, or comments by EPA regarding reports, plans, specifications, schedules, or any other writing submitted by Respondents shall relieve Respondents of the obligation to obtain such formal approval as may be required by this CONSENT ORDER, and to comply with all requirements of this CONSENT ORDER unless or until this CONSENT ORDER may be formally modified.

XXI. Reservation of Rights

84. EPA reserves the right to take any enforcement action against Respondent ATC pursuant to CERCLA and/or any other available legal authority, including the right to seek injunctive relief, monetary penalties and/or punitive damages for violations of law. EPA also reserves the right to bring an action in law or equity, pursuant to CERCLA and/or any other available legal

authority, against Respondent ATC and/or Respondent Leezy to enforce the terms of this CONSENT ORDER.

85. EPA expressly reserves all rights and defenses that it may have, including the right to require Respondents perform tasks in addition to those detailed herein. In addition, EPA reserves the right to undertake any response action at any time.

86. Nothing in this CONSENT ORDER shall constitute or be construed as a release by EPA or Respondents of any claim, cause of action, or demand in law or equity against Respondent ATC and/or any other party for any liability arising out or in any way relating to the generation, storage, treatment, handling, transportation, release, or disposal of any hazardous substances, pollutant, or contaminants at the Site. Specifically, EPA reserves its right to pursue any person deemed liable under this CONSENT ORDER for the releases of hazardous substances at the Site, pursuant to Section 107 of CERCLA, 42 U.S.C. § 9607, for costs incurred by EPA related to the Site.

87. This CONSENT ORDER does not constitute any decision on preauthorization of funds under Section 111(a)(2) of CERLCA, 42 U.S.C. § 9611(a)(2).

88. In entering this CONSENT ORDER, Respondents waive any right they may have to seek reimbursement from EPA under Section 106(b)(2) of CERCLA, 42 U.S.C. § 9606(b)(2).

89. If any judicial or administrative authority issues an order that invalidates any provision of this CONSENT ORDER, or finds that Respondents have sufficient cause not to comply with one or more provisions of this CONSENT ORDER, Respondents shall remain bound to comply with all other provisions of this CONSENT ORDER.

XXII. Dispute Resolution

90. If Respondents disagree, in whole or in part, with any EPA disapproval or other decision or directive (including any directive to perform additional work not specified in the Section IX (Work to be Performed) or the Statement of Work to this CONSENT ORDER) made by EPA pursuant to this CONSENT ORDER, Respondents shall notify EPA in writing of their objections and the basis for such objections, within ten (10) working days of receipt of EPA's disapproval, decision, or directive. Such notice shall set forth the specific points of the dispute, the position that Respondents maintain should be adopted as consistent with the requirements of this CONSENT ORDER, the factual and legal basis for Respondents' position, and all matters Respondents consider necessary for EPA's determination.

91. EPA and Respondents shall then have ten (10) working days from EPA's receipt of Respondents' objections to attempt to resolve the dispute. If agreement is reached, the resolution shall be reduced to writing, signed by each party, and incorporated into this CONSENT ORDER.

92. If the parties are unable to reach agreement within this ten (10) working-day period, the matter shall be referred to the EPA's Region VII Superfund Division Director. The Superfund Division Director shall then decide the matter and provide a written statement of his or her decision to both parties, which shall be incorporated into this CONSENT ORDER.

93. Notwithstanding any other provision of this CONSENT ORDER, no action or decision by EPA, including without limitation, decisions of the Superfund Division Director pursuant to this CONSENT ORDER, shall constitute final EPA action giving rise to any rights to judicial review prior to EPA's initiation of a judicial action to compel Respondents' compliance with the requirements of this CONSENT ORDER.

XXIII. Stipulated Penalties for Noncompliance

94. In the event Respondent ATC fails to comply with any requirement of this CONSENT ORDER, Respondent ATC shall pay stipulated penalties as set forth below. Such non-compliance by

Respondent ATC shall include completion of any activity required by this CONSENT ORDER, any plan or design approved under this Order or compliance with any other requirement of this CONSENT ORDER in an acceptable manner and within the time schedules specified and set forth in and approved under this CONSENT ORDER.

a. For failure to submit Progress Reports: One Hundred Fifty Dollars ($150.00) per day for the first through the seventh days of noncompliance and Two Hundred Fifty Dollars ($250.00) per day for the eighth day and each succeeding day of noncompliance thereafter;

b. For failure to comply with any due date for submission of any Report, Work plan or Design: Two-Hundred Fifty Dollars ($250.00) per day for the first through seventh days of noncompliance and Five Hundred Dollars ($500.00) per day for the eighth day and each succeeding day of noncompliance thereafter;

c. For failure to perform any work according to the schedules set forth in this CONSENT ORDER, the SOW, or according to the schedules set forth within any Report or Work plan approved pursuant to the terms of this CONSENT ORDER: Five Hundred Dollars ($500.00) per day for the first through seventh days of noncompliance and One Thousand

Dollars ($1,000.00) per day for the eighth day and each succeeding day of noncompliance thereafter.

95. All penalties for noncompliance shall begin to accrue on the date that performance is due and shall continue to accrue through the final day of correction of the noncompliance. Nothing herein shall prevent the simultaneous accrual of separate penalties for separate violations of this CONSENT ORDER.

96. All penalties owed to EPA under this Section shall be due within ten (10) days of notice from EPA that Respondent ATC is in noncompliance with this CONSENT ORDER. Interest shall begin to accrue on the unpaid balance at the end of this ten (10) day period. Interest shall accrue on the unpaid amount at the rate determined by the Secretary of the Treasury for that period for unpaid CERCLA claims (currently 4.53 percent per annum for the period January 1, 1999 through December 31, 1999) until such stipulated penalty and accrued interest are paid in full.

97. All penalties shall be paid by certified or cashier's check made payable to the Hazardous Substance Response Trust Fund and remitted to:

EPA - Region VII
Attn: Superfund Accounting
Post Office Box 360748M
Pittsburgh, Pennsylvania 15251

All payments shall reference the Site, Respondent ATC's name and address, and the EPA docket number of this action. A copy of the transmittal of payment shall be sent to the EPA contact specified herein.

98. The stipulated penalties set forth in this Section shall not preclude EPA from pursuing any other remedies or sanctions which may be available to EPA by reason of Respondent ATC's failure to comply with any of the requirements of this CONSENT ORDER, nor shall payment of stipulated penalties relieve Respondent of the responsibility to comply with this CONSENT ORDER.

99. The invocation of the dispute resolution process under Section XXII (Dispute Resolution), or a claim of force majeure under Section XVIII (Force Majeure), shall not stay the accrual of stipulated penalties, or extend or postpone any deadline or obligation of Respondents, including the obligation to pay stipulated penalties under this CONSENT ORDER with respect to a disputed issue unless EPA otherwise agrees in writing. Stipulated penalties shall continue to accrue during the dispute resolution process for all matters unrelated to the contested matters at issue in the dispute resolution process.

100. Pursuant to Section 106(b) of CERCLA, 42 U.S.C. §9606(b), Respondents' willful violation or failure or refusal to comply

with any provision of this CONSENT ORDER may subject Respondents to a civil penalty. Further, under Section 107(c)(3) of CERCLA, 42 U.S.C. 9607(c)(3), Respondent ATC's failure to comply with any portion of this CONSENT ORDER, without sufficient cause, may subject Respondent to punitive damages in an amount up to three times the amount of any costs incurred by the government as a result of Respondent's non-compliance.

XXIV. Indemnification

101. Neither the United States nor any agency or agents or employees thereof shall be liable for any injuries or damages to persons or property resulting from acts or omissions of a Respondent or their officers, directors, employees, agents, servants, receivers, trustees, successors, or assignees, or of any persons, including but not limited to firms, corporations, subsidiaries, contractors or consultants, in carrying out activities pursuant to this CONSENT ORDER, nor shall the United States or any agency or agents or employees thereof be represented to be a party to any contract entered into by a Respondent in carrying out activities pursuant to this CONSENT ORDER.

102. Respondent ATC agrees to indemnify and save and hold harmless the United States Government, its agencies, departments,

agents and employees from any and all claims or causes of action arising from or on account of acts or omissions of its officers, employees, receivers, trustees, agents, contractors, subcontractors or assigns, in carrying out any activities pursuant to this CONSENT ORDER. Respondent Leezy agrees to indemnify and save and hold harmless the United States Government, its agencies, departments, agents and employees from any and all claims or causes of action arising from or on account of acts or omissions of his officers, employees, receivers, trustees, agents, contractors, subcontractors or assigns, in carrying out any activities pursuant to this CONSENT ORDER. EPA is not and shall not be represented to be a party to any contract entered into by Respondents to carry out activities pursuant to this CONSENT ORDER.

XXV. Satisfaction and Completion

103. The requirements of this CONSENT ORDER shall be deemed satisfied upon written notice from EPA that the Respondents have demonstrated, to the satisfaction of EPA, that all of the Work required by this CONSENT ORDER has been completed.

XXVI. Signatures and Effective date

104. This CONSENT ORDER may be signed in part and counterpart by the Respondents and EPA.

105. The effective date of this CONSENT ORDER shall be the date on which Respondents receive a fully executed copy thereof.

IT IS SO ORDERED.

For Respondent, Arneson Timber Company, Inc.

BY: N Arvor Arneson President ATC DATE: 2-1-99
Name: ARNESON TIMBER CO. INC.
Title:

The representative of Respondent Arneson Timber Company who has signed the signature page above certifies that he/she is fully authorized to enter into the terms and conditions of this CONSENT ORDER and to bind the Respondent to the terms of this CONSENT ORDER.

Post-it® Fax Note 7671	Date 2-1-99	# of pages ▶ 1
To Howard Bunch	From Fred Lafser	
Co./Dept.	Co.	

In the Matter of the Arneson Timber Site

Signature Page:

For Respondent Charles Leezy:

Name: _____ DATE: 2-1-99

In the Matter of the Arneson Timber Site

Signature Page:

For the United States Environmental Protection Agency

BY: _____ DATE: 2/1/99
Howard C. Bunch
Assistant Regional Counsel, Region VII
United States Environmental Protection Agency

BY: _____ DATE: 2/2/99
Michael J. Sanderson, Director
Superfund Division, Region VII
United States Environmental Protection Agency

ATTACHMENT I:

STATEMENT OF WORK

SCOPE OF WORK
FOR THE REMOVAL ACTION
AT THE
ARNESON TIMBER SITE
CRAWFORD COUNTY, MISSOURI

PURPOSE

1. The purpose of this Scope of Work (SOW) for the Arneson Timber Site (Site) is to define the tasks, schedules, standards and guidelines which shall be followed by the Respondents to conduct a removal action to either (1) perform in-situ bioremediation of contaminated soils to levels protective of human health and the environment and/or (2) remove contaminated soils from the Site. In accomplishing the above purpose, the Respondents shall comply with the provisions of the corresponding Administrative Order on Consent (CONSENT ORDER) between the United States Environmental Protection Agency (EPA), Arneson Timber Company (ATC), and Charles Leezy (Respondents), this SOW, CERCLA, the National Contingency Plan (NCP) and EPA guidance (including, but not limited to the guidance documents referenced in this SOW).

WORK TO BE PERFORMED

2. Respondents shall perform the tasks set forth below in designing and implementing the work required for the Site. The work required shall consist of the following three tasks:

TASK I: Institutional Controls

TASK II: Removal Action Work Plan

TASK III: Bench scale biodegradation treatability test

TASK IV: Removal Action:

TASK V: Reports:

TASK I:
Institutional Controls

3. Within ten (10) days of the effective date of this Order, Respondent Charles Leezy shall minimize the possibility of unknowing, unauthorized entry and exposure to the contaminated areas of the site. These actions shall include but not be limited to:

A. Establishment and maintenance of a physical barrier (fence) around the perimeter of the contaminated soils area that is adequately sized to control entry.

B. Posting of signs to warn the public of a hazardous condition. These signs shall be posted at all entrances to the fenced area and along the sides of the fencing at intervals no greater than 40 feet. The signage shall state "Danger-Hazardous Substances, Unauthorized Persons Keep Out.;"

C. An application to the State of Missouri for placement of the Site on the state's Registry of Abandoned, Uncontrolled Hazardous Waste Disposal Sites; and

D. A restrictive covenant pertaining to the Site that shall restrict the use of the Site as described below. The restrictive covenant shall be prepared in conformance with the requirements under state law for a covenant running with the land, and the use restrictions contained therein shall be designed to insure protection of public health and the environment at the Site. and shall include but not be limited to prohibition of the following at the Site, until all contaminated soils are bio-remediated or removed so that soils at the Site are above EPA's any "use action" levels:

1. The use of the Site for any thing other than industrial uses. The restrictive covenant shall specifically prohibit residential occupation of the Site;
2. Wells for the use of ground-water; and
3. Construction of underground facilities (e.g., water, gas and other utilities).

Task II:
Removal Action Work Plan

6. Within thirty (30) days of the effective date of this CONSENT ORDER, Respondent ATC shall prepare and submit a draft Removal Action Work Plan (RAWP) to EPA for review and comment. Within forty-five (45) days of the effective date of this CONSENT ORDER, Respondent ATC shall prepare and submit a final Removal Action Work Plan (RAWP) for EPA's review and approval, prepared in accordance with Tasks II-IV of the SOW. and that addresses any comments made by EPA on the draft RAWP. If the RAWP proposes the use of bioremediation as a part of the removal alternative, the RAWP shall be prepared to describe the tasks and schedules associated with bench scale treatability testing, excavation, construction of on-site in-situ treatment cells and/or off-site disposal of soils that exceed EPA's cleanup levels for the Site. If the RAWP proposes the excavation and off-site disposal of contaminated soils as a removal alternative, the RAWP shall be prepared to describe the tasks and schedules

associated with excavation and off-site disposal of soils contaminated above EPA's "any use" or "industrial use" action levels, as appropriate. If the RAWP proposes to consolidate and bioremediate soils at the Site contaminated above EPA's "industrial use" levels, the RAWP shall be prepared to describe the tasks and schedules associated with such bioremediation. The RAWP shall also be prepared so as to require the response action to be performed in accordance with standards set forth in Task IV, below, and shall include the following information and meet the following requirements:

A. A brief background of the Site, including physical location;

B. Proposed methods of contaminated soil excavation, stockpiling, consolidation, processing and/or off-site disposal. Respondents shall describe the general method of how the contaminated soils will be handled to complete proposed bioremediation treatment actions;

C. Actions to be implemented at closure of the bio-treatment activities to ensure long-term effectiveness and permanence of the removal action, including collection of groundwater samples from the current on-site well and near site existing wells;

D. A clear and concise description of roles, relationships and assignment of responsibilities among the Respondents, Project Coordinator, Quality Assurance Officer, Construction Supervisor and Construction Personnel;

E. A proposed schedule for the removal action that will require commencement of on-site activity within thirty (30) days of approval of the RAWP;

F. A detailed description of site preparation activities, including establishment of security and control, definition of clearing and grubbing limits, establishment of work and support areas, and definition of decontamination areas;

G. A proposal for specific sampling criteria to define areas to be excavated, based upon EPA's soil cleanup action levels;

H. A proposed design of an air monitoring program to be used during site excavation and contaminated material handling activities;

I. A proposal for sampling and analytical procedures, including field screening and laboratory methods, to be conducted on soil samples collected during excavation activities to verify attainment of EPA's "any use" and/or

"industrial use" action levels;

J. A description of the methods proposed to be used to control fugitive dust generated from excavation at the Site;

K. A description of the method of transportation to be used for all contaminated materials, manifesting requirements in accordance with federal and state Department of Transportation (DOT) regulations, and material quantity accounting procedures. In addition, Respondent ATC shall provide written notice prior to any out-of-state shipment of waste material;

L. A description of Site restoration requirements after completion of the removal action, including the placement of clean fill over excavated areas and/or bioremediation cells. This description shall include all work necessary to restore property to its original pre-removal condition, including but not limited to the placement of clean fill, replacement of plantings and/or cover.

M. A plan for identifying and complying with applicable permitting requirements and environmental statutes.

7. As components of the Removal Action Work Plan, Respondent ATC shall develop and submit the following project plans to support field activities:

A. A Quality Assurance Project Plan (QAPP),

B. Health and Safety Plan (HSP)

8. The Quality Assurance Project Plan (QAPP) shall provide a process for obtaining data of sufficient quality and quantity to satisfy data needs to characterize the site (As described in EPA QA/R-5, EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, 11/97). No field activities shall take place until EPA has reviewed and approved the QAPP. The QAPP shall consist of two parts:

A. A **Sampling Analysis Plan (SAP)** which describes the estimated number, type, and location of samples to be collected at the site to further define the extent of contamination or to confirm when certain actions have achieved the desired results, or to conduct tests on the material for treatability. Respondents ATC shall include provisions for split samples provided to EPA, its contractors, or the State of Missouri as appropriate. No fewer than five (5) working days notice must be given to EPA prior to collecting samples. The plan shall also include the types of analyses to be conducted for each sample and a brief rationale for collecting the sample and performing the analysis (e.g., soil sample submitted for Multiple Extraction Procedure Analysis to confirm cleanup criteria have been met).

B. A **Field Sampling Plan (FSP)**, which describes policy, organization, and functional activities and the data quality objectives and measures necessary to achieve adequate data for use in planning and documenting the removal action. The FSP shall be written in accordance with a category III project as described in "Preparing Perfect Project Plans", U.S. EPA, Office of Research and Development, EPA/600/9-89/087, October 1989.

9. The QAPP shall require that all sample collection and analysis be performed in compliance with EPA approved methods, including timing of analysis and documentation of sample collection, handling, and analysis. Any proposed sampling scheme must be capable of producing representative and statistically valid samples, and generally conform to the following EPA guidance documents:

A. Compendium of ERT Field Analytical Procedures - Office of Emergency and Remedial Response, Publication 9360.4-04, May 1992.

B. Compendium of ERT Waste Sampling Procedures - Office of Solid Waste and Emergency Response, EPA/540/P-91/008, January 1991.

C. QA/QC Guidance for Removal Activities: Sampling QA/QC Plan and Data Validation Procedures - Office of Emergency and Remedial Response, EPA/540/G-90/004, April 1990.

D. Removal Program Representative Sampling Guidance, Volume 1: Soil - Office of Emergency and Remedial Response, publication 9360.4-10, November 1991.

E. Compendium of ERT Soil Sampling and Surface Geophysics Procedures - Office of Solid Waste and Emergency Response, EPA/540/P-91/006, January 1991.

10. The QAPP shall require that all samples shall be analyzed by a laboratory that participates in a quality assurance/quality control program equivalent to that specified in "USEPA Contract Laboratory Program Statement of Work for Organic Analysis", Exhibit E, EPA SOW No. 788, July 1994, which may be found on the Internet at "http://www.epa.gov/superfund/oerr/aoc/methods.htm."

11. The **Health and Safety Plan,** shall be prepared in accordance with 40 C.F.R. Part 300.150 and all applicable OSHA requirements at 29 C.F.R. 1910. In addition to the requirements addressed in these regulations, this plan shall generally follow the guidelines established in EPA's "Standard Operating Safety Guides", Office of Emergency and Remedial Response, publication 9285.1-03, June 1992.

12. Respondent ATC shall establish subcontracts for the appropriate analyses, treatment, transport and disposal vendors.

TASK III
Bench scale biodegradation treatability test

13. If approved within the RAWP, Respondent ATC shall perform bioremediation to address soils contaminated above "industrial use" levels that are not removed from the Site. Accordingly, bioremediation may be performed either as the primary component of the removal alternative, or if an initial removal of contaminated soils leaves soils contaminated above "industrial use" levels at the Site. If the approved RAWP requires bioremediation as the primary component of the removal action (i.e. no removal of soils for off-site disposal), within ninety (90) days of the effective date of this Order, Respondent ATC shall develop, conduct and document the results of a bench scale Treatability Test for proposed treatment method(s) for microbial biodegradation that are designed to remediate the contaminated areas of the site. If the approved RAWP allows bioremediation to address soils that remain contaminated above EPA's "industrial use" levels after an initial removal of soils for off-site disposal, within forty-five (45) days after completion of the initial soil removal, Respondent shall develop, conduct and document the results of a bench scale Treatability Test for proposed treatment method(s) for microbial biodegradation that are designed to remediate the contaminated areas of the site. This bench scale Treatability Test shall be prepared and conducted in a manner consistent with EPA guidance "Guide for Conducting Treatability Studies Under CERCLA, Biodegradation Remedy Selection, Interim Guidance" EPA/540/R-93/519a, August 1993.

If, after an initial soil removal, bioremediation is required to address soils remaining contaminated at levels between EPA's "any use" and "industrial use" levels, Respondent shall submit a Treatability Test within forty-five (45) days after completion of Respondent's initial soil removal. This Treatability Test may consist of the submission of the results of a literature review that demonstrates "passive" bioremediation and/or natural biological mechanisms may result in the reduction of contamination to EPA's "any use" action levels.

14. Within thirty (30) days after completion of the Treatability Test, Respondent shall submit to EPA for review and approval, a Treatability Test Report that summarizes the results of the described in Paragraph 13, above. Based on the data in this Report, EPA may approve the use of in-situ bioremediation as a component of the removal action for this site, or may require Respondents ATC to conduct removal and off-site disposal of soils contaminated above EPA's "industrial use" action levels (For surface soils between 0 to 4 feet in depth, 37.7 mg/kg of PCP is the "industrial use" action level. For soils between 0 to to 4 feet, EPA's "industrial use" action level is 10.0 ug/kg dioxin

equivalents). This Treatability Test Report shall include, at a minimum, the following information and satisfy the following requirements:

A. A written description of the parameters measured (soil pH, contaminant concentrations, percent contamination removal), the methods used to quantify the parameters, and the results so that the information described below will be available from the data generated by the Treatability Test;

B. Initial and final contaminant concentrations from batch testing conducted to measure the microbial degradation activity of the proposed treatment method(s);

C. A calculation of the percent of contaminant removal achieved through microbial biodegradation;

D. A determination of the optimal site specific method for implementing a biodegradation treatment on site;

E. An evaluation of the steps needed during the full-scale treatment phase to stimulate or maximize the biodegradation process;

F. An assessment of the treatment method's ability to meet the cleanup criteria;

G. An evaluation of the area needed to conduct the full-scale treatment with respect to the current area of existing contamination; and

H. An estimate of the time and costs to complete the bioremediation removal action.

TASK IV:
Removal Action

15. The Removal Action shall be conducted in accordance with the following performance standards, as set forth in the approved RAWP:

A. Utilizing EPA's previous sampling data, Respondent ATC shall prepare (1) a map that identifies all areas on-site where any soils up to a depth of four (4) feet in depth were found to exceed EPA's "any use" action level of 9.1 mg/kg for pentachlorophenol (PCP) and (2) a map that identifies all areas on site where any soils in the depth of surface to one foot in depth were found to exceed the "any use" soil cleanup level of 1 ug/kg dioxin equivalents;

B. Utilizing EPA's previous sampling data, Respondent ATC shall prepare (1) a map that identifies all areas on-site where any soils up to a depth of four (4) feet were found to exceed EPA's "industrial use" action levels of 37.7 mg/kg for pentachlorophenol (PCP) and (2) a map that identifies all areas on site where any soils in the depth of surface to four (4) foot in depth were found to exceed the "industrial use" soil cleanup level of 10 ug/kg dioxin equivalents;

C. If the approved RAWP requires bioremedation as a component of the removal action to address soils contaminated above "industrial use" levels. Respondent shall identify the locations of treatment cells for in-situ bioremediation;

D. In accordance with the removal action set forth in the approved RAWP, Respondent ATC shall excavate contaminated soils to depths no greater than four (4) feet and properly dispose of contaminated soils off-site, and/or consolidate into the treatment cells all excavated soils contaminated above "industrial use" action levels for dioxin equivalents (10 ug/kg), as appropriate to the approved removal action;

E. In accordance with the removal action set forth in the approved RAWP, Respondent ATC shall excavate contaminated soils to a depth no greater than four (4) feet and properly dispose of contaminated soils off-site, and/or consolidate into the treatment cells all soils contaminated above "industrial use" action levels for pentachlorophenol (37.7 mg/kg), as appropriate to the approved removal action;

F. Respondent shall sample the base of all excavated areas to determine whether levels of contamination above "industrial use" levels remain after excavation. If the results of demonstrate that soils at depths less than four (4) feet are contaminated above EPA's "industrial use" action levels, Respondent shall either excavate and properly dispose of such soils off-site, or place such soils into the bioremediation treatment cells. If at depths of four (4) feet or greater, sample results show contamination at levels greater than EPA's "industrial" action levels, EPA will evaluate the need for any further action to address such contamination outside the scope of this CONSENT ORDER and SOW;

G. Respondent ATC shall implement soil excavation and removal and other cleanup techniques that minimize the release of contaminants via airborne emissions and/or surface runoff;

H. Respondent ATC shall monitor the ambient air during excavation and soil loading activities. Ambient air monitoring during excavation and removal shall be

designed and implemented to determine compliance with National Primary and Secondary Ambient Air Quality Standards and/or levels protective of human health as determined by EPA. If these standards are exceeded, Respondent ATC shall utilize containment, chemical dust suppressants and/or water during excavation and removal activities to minimize generation of airborne emissions;

I. Respondent Charles Leezy shall maintain institutional controls around the site and the bioremediation cells until notified by EPA that the removal action has achieved the "any use" action levels;

J. Respondent ATC shall implement the bioremediation of all soils contaminated above EPA's "industrial use" levels in accordance with the requirements of the approved RAWP. Respondent ATC shall sample and monitor the effectiveness of the bioremediation at least bi-annually for five (5) years from the commencement of bioremediation. If, during this five (5) year period, EPA determines that the bioremediation has reduced contamination below EPA's "any use" levels, bioremediation may cease and all institutional controls may be removed. If, after five years, the bioremediation has not proven effective in reducing the levels of pentachlorophenol below "37.7 mg/kg and dioxin equivalents below 10 ug/kg (EPA's "Industrial use" action levels), Respondent ATC shall excavate and dispose of such soils offsite as RCRA hazardous waste. Respondent may propose alternate treatment methods to off-site disposal to EPA for review and approval, however, if these treatment methods are not approved by EPA, Respondent shall remain required to properly dispose of off-site soils at depths less than 4 feet that are contaminated above EPA's industrial use levels;

K. If, after five years from placement of soils within the bioremediation treatment cells, soils remain contaminated with PCP at levels between 9.1 mg/kg and 37.7 mg/kg, and with dioxin equivalents at levels between 1.0 ug/kg and 10.0 ug/kg, both the restrictive covenant and listing of the Site on the State of Missouri's registry for restricted future use shall be maintained. However, if the effectiveness of bioremediation has not been exhausted and may further reduce the levels of hazardous substances to below EPA's "any use" action levels, Respondent ATC shall continue bioremediation within the treatment cells until three bi-annual consecutive sampling events show no further significant decline in the levels of pentachlorophenol or dioxin equivalents;

L. If Respondent ATC removes and disposes off all soils at depths less than 4 feet that are contaminated above EPA's "industrial use" action levels, Respondent ATC shall sample and monitor the effectiveness of the passive bioremediation to address such remaining contamination at least annually for five (5) years from the commencement of bioremediation. If, during this five (5) year

period. EPA determines that the passive bioremediation has reduced contamination below EPA's "any use" levels, institutional controls may be removed from the Site.

M. All excavated soils contaminated with pentachlorophenol and dioxin equivalents above the "any use" levels of 9.1 mg/kg and 1.0 ug/kg, respectively, and not placed into the in-situ bioremediation treatment cells, shall be considered to be RCRA hazardous waste and shall be managed and taken off-site for disposal pursuant to applicable RCRA transportation and storage standards

11. Upon EPA approval of the RAWP, Respondent ATC shall construct and implement the Removal Action in accordance with the approved designs, schedules and plans contained therein. Thereafter, Respondent ATC shall document that the purpose of the Removal Action has been accomplished, in accordance with the following procedures:

A. Post-construction/Removal Inspection and Report: Upon preliminary completion of the construction of the treatment cells, or completion of off-site removal of contaminated soils, Respondent ATC shall contact EPA for the purpose of scheduling and conducting a site inspection with EPA. The inspection shall consist of a walk-through inspection of the entire project site. The purpose of the prefinal inspection will be to determine whether the removal action has been completed consistent with the approved RAWP.

A. Within thirty (30) days of the Post-construction/Removal inspection, Respondent ATC shall submit to EPA, for review and approval, a Post-construction/Removal Inspection Report that will document all unfinished removal action work discovered during the prefinal inspection.

B. Within the Inspection Report, Respondent ATC shall outline the actions required to resolve all outstanding removal action work and shall propose a schedule to complete these items.

C. Within the Inspection Report, Respondent ATC shall also certify that all other removal work has been completed in a manner sufficient to meet the requirements of the approved RAWP.

4. Respondent ATC shall provide a copy of the Inspection Report to all property owners on whose property access was required to complete the Removal Action.

B. Final Inspection: Upon completion of any outstanding removal action work, Respondent ATC shall notify EPA for the purpose of scheduling a final site inspection.

1. The final inspection shall consist of a walk-through inspection by EPA and Respondent ATC of the project site.

2. The Post-Construction/Removal Inspection Report shall be used as a checklist with the final inspection focusing on the outstanding removal work items identified in the prefinal inspection.

C. Certification of Completion: Upon confirmation that the purpose, intent and requirements of the Removal Action Work Plan have been satisfied, in conformance with engineering practice, EPA shall provide Respondents a written certification of completion of the Removal Action.

TASK V: REPORTS

12. Respondent ATC shall prepare the work plans and reports set forth in Task I through Task IV to accomplish the design, construction, operation and maintenance, and monitoring of the Removal Action. In addition, the Respondent ATC shall provide the following documentation:

A. Progress Reports: Respondent ATC shall provide the EPA with signed, monthly progress reports by the 10th day of each calendar month following the effective date of the Order until the date of EPA's approval of the Post-Construction/Removal Inspection Report. Thereafter, progress reports shall be submitted quarterly by the 10th day of each third calendar month. The monthly progress reports shall be of similar content as a Pollution Report (POLREP) as described in "Superfund Removal Procedures, Removal Response Reporting: POLREPS and OSC Reports", U.S. EPA, Office of Solid Waste and Emergency Response, Publication 9360.3-03, June 1994. At a minimum, the progress reports shall include the following information:

1. A summary of actions which have been taken to comply with the Consent Order during the reporting period;

2. Copies of results of sampling and tests and all other raw data received by Respondent ATC during the reporting period;

3. A description of work planned for the next reporting perod with scheduling related to such work;

4. A summary of problems encountered and any anticipated problems, any actual delays, and solutions developed and implemented to address any actual or anticipated problems or delays; and

5 Summaries of all contacts with representatives of the local community, public interest groups, and state and federal governments during the reporting period.

B. Completion Report: Within thirty (30) days of the final site inspection, Respondent ATC shall submit to EPA for review and approval a Removal Action Completion Report (Completion Report). The Completion Report shall include the following information:

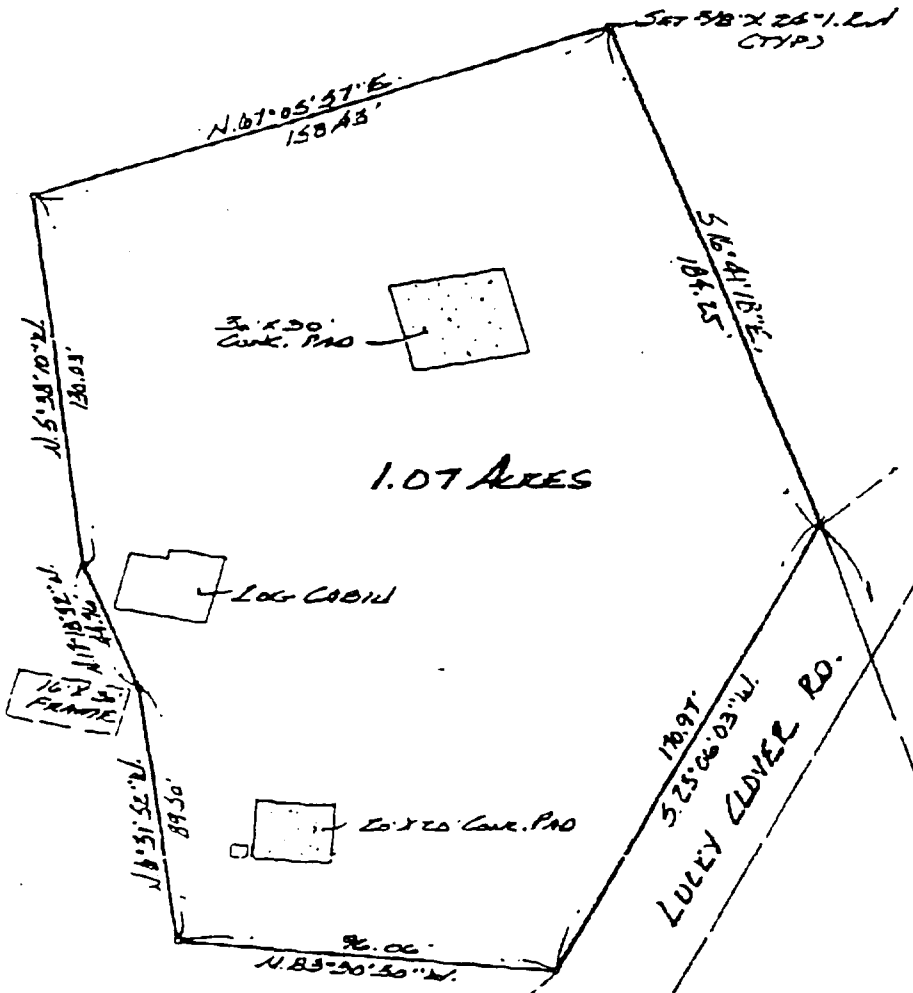
1. An activity summary of all removal actions;

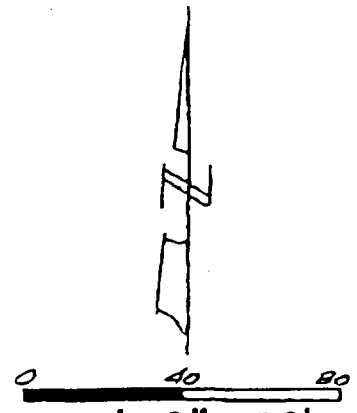
2, Copies of all sample results; and

3. A statement certifying the completion of the removal activities consistent with this Consent Order and plans approved hereunder.

ATTACHMENT II:

MAP OF SITE





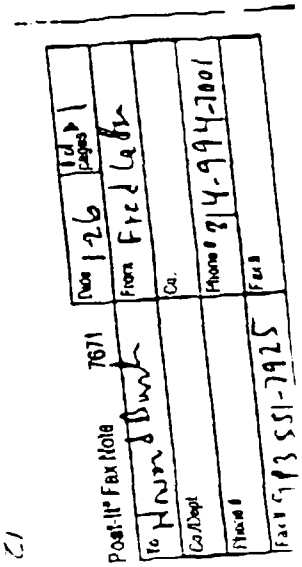
Scale 1"= 40'

T38N-R4W of the 5th P.M.

Class "B" Property

Survey #121-5298

Title Ref.: 337-638



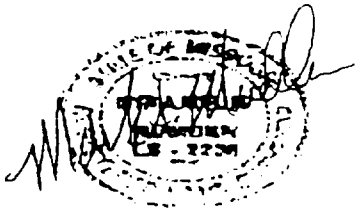
TRACT DESCRIPTION

Part of the NE ¼ of the SE ¼ of Section 20, T38N-R4W of the 5th P.M. in Crawford County, Missouri more fully described as follows:

Commencing at the SE Corner of said ¼ ¼ Section; thence N.15°20'38"W. 360.42 feet to the point of beginning; thence S.23°06'03"W. 170.97 feet; thence N.83°30'30"W. 96.06 feet; thence N.6°31'52"W. 89.50 feet; thence N.17°18'52"W. 44.96 feet; thence N.5°38'10"W. 130.03 feet; thence N.67°05'57"E. 158.43 feet; thence S.16°41'18"E. 184.25 feet to the point of beginning. CONTAINING 1.07 acres.

SURVEYORS DECLARATION

This is to declare that I, Mark A. Mueller have during the month of November, 1998 by order of Charles N. Leezy executed a survey of the tract of land platted hereon in accordance with the current Missouri Minimum Standards for Property Boundary Surveys.



11-8-98

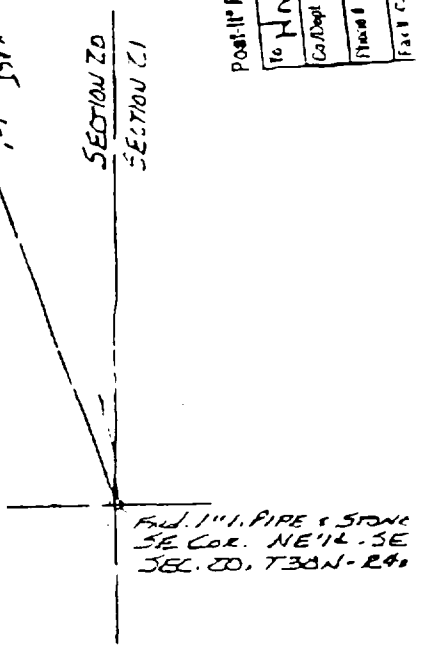


Exhibit A

NOTICE OF PLACEMENT ON *REGISTRY OF CONFIRMED ABANDONED OR UNCONTROLLED HAZARDOUS WASTE DISPOSAL SITES IN MISSOURI (REGISTRY)*

Owner of Record: Charles Leezy

Site Description: The Arneson Timber site is a former lumber mill and wood treating facility. Soil and groundwater at the site have been found to be contaminated with pentachlorophenol from wood treating processes at levels that exceed the Missouri Any-Use Soil Level and EPA's drinking water standard, the Maximum Contaminant Level. In addition, "dioxin equivalents" were detected on-site.

The legal description of the property being placed on the *Registry* is:

All of the Northeast Quarter of the Southeast Quarter, and the right to use a road right-of way across the South side of a 30 acre tract located in the Southeast corner of the Northwest Quarter of the Southeast Quarter as reserved by LeRoy Leezy and Carrie Leezy, his wife, in Warranty Deed recorded in Book 191, at page 133 of the Deed Records of Crawford County, Missouri, all in Section 20, Township 38 North, Range 4 West.

All that part of the Northwest Quarter of the Southwest Quarter, lying West of the right-of-way of Highway #19, in Section 21, Township 38 North, Range 4 West.

All of the Southwest Quarter of the Southwest Quarter of Section 21, Township 38 North, Range 4 West, EXCEPT a strip of land 25 feet wide off the North side of the Southwest Quarter of the Southwest Quarter, running East and parallel with the Northern boundary line of said Southwest Quarter of the Southwest Quarter from Highway 19, all in Section 21, Township 38 North, Range 4 West, to be used as a private road by William Diehl and Rae Diehl, his wife, their heirs or assigns or the public on their way to Leezy Cemetery.

A strip of land 25 feet wide off of the North side of the Southeast Quarter of the Southwest Quarter, running parallel with the Northern boundary line of said Southeast Quarter of the Southwest Quarter to the Leezy Cemetery, all in Section 21, Township 38 North, Range 4 West, to be used by LeRoy Leezy and Carrie Leezy, his wife, their heirs or assigns or the public, to enter the Leezy Cemetery (private road only). This is a continuation of the road deeded by LeRoy Leezy and Carrie Leezy, his wife, to William Diehl and Rae Diehl, his wife

All of the West Half of the Northwest Quarter of the Northwest Quarter of Section 28, Township 38 North, Range 4 West, lying North of the Meramec River and directly West of a tract of land heretofore deeded by

Preston Halbert and his wife to Charles Creek, making the top of the bluff the dividing line between the tract heretofore conveyed to Charles Creek and the tract herein conveyed.

The Missouri Department of Natural Resources has placed the above-described real property, owned by Charles Leezy, and located in the county of Crawford and state of Missouri, on the *Registry*. The site was the location of a lumber mill and wood treating facility between 1972 and 1983. During closure of the facility, Ameson Timber Company personnel spilled an unknown amount of spent pentachlorophenol wood preservative solution from an aboveground storage tank. Ameson Timber Company performed a partial cleanup of the pentachlorophenol spill in 1983. Soil and groundwater at the site have been found to be contaminated with pentachlorophenol from wood treating processes at levels that exceed the Missouri Any-Use Soil Level and the EPA's drinking water standard, the Maximum Contaminant Level. In addition, "dioxin equivalents" were detected on-site.

The *Registry* is maintained by the Missouri Department of Natural Resources pursuant to the Missouri Hazardous Waste Management Law, Section 260.440, RSMo. When the Director of the Department of Natural Resources (director) places a site on the *Registry*, he shall record with the Recorder of Deeds the period during which the site was used as a hazardous waste disposal area, Section 260.470, RSMo. In accordance with Section 260.465(1), RSMo, the property owner must obtain approval from the director prior to moving any contaminated material, or changing the use of the site in any way. Title 10, Division 25, Chapter 10 of the Code of State Regulations [10 CSR 25-10.010(3)(A)] requires change in use requests to be evaluated by the director to determine if the change results in: spread of contamination; increased human exposure to the hazardous materials; increased adverse environmental impacts;

or, making potential remedial actions to correct the problems at the site more difficult to undertake or complete. Questions regarding the *Registry* or this site should be directed to the Missouri Department of Natural Resources, Hazardous Waste Program, P.O. Box 176, Jefferson City, Missouri.

The Recorder of Deeds is directed to record this information so that any potential purchaser of the property will be given notice that the site has been placed on the *Registry* and that specific restrictions apply to ownership of this property while it is listed on the *Registry*.

In witness whereof I hereunto set my hand this 28TH of June, 1999.

DIVISION OF ENVIRONMENTAL QUALITY

John A. Young
Director

000174

STATE OF MISSOURI)

) SS.

COUNTY OF COLE)

On this 28th day of June, in the year 1999, before me Bonnie VanHook a Notary Public in and for said state, personally appeared John A. Young, Director of the Division of Environmental Quality, Missouri Department of Natural Resources, known to me to be the person who executed the within Notice of Placement on the *Registry of Confirmed Abandoned or Uncontrolled Hazardous Waste Disposal Sites in Missouri* on behalf of the Missouri Department of Natural Resources, and acknowledged to me that he executed the same for the purpose therein stated.

Bonnie VanHook

Notary Public

My commission expires:

BONNIE VAN HOOK
Notary Public – Notary Seal
STATE OF MISSOURI
Cole County
My Commission Expires: Sept. 4, 2001

Book 449 Page 172
FILED FOR RECORD
At 11 o'clock 10 Min. A M

JUL 02 1999

KAREN A. McPETERS
EX-OFFICIO RECORDER CRAWFORD COUNTY, MO
Sandra K Cook DEPUTY

No Charge St of Mo.
Dept of Natural Resources
Jefferson City, Mo
Attn: John A Young

000175

END OF DOCUMENT

INDIAN SPRINGS QUADRANGLE
MISSOURI-CRAWFORD CO
7.5 MINUTE SERIES (TOPOGRAPHIC)