Characterization and Monitoring of 300 Area Facility Liquid Waste Streams: Status Report

R. G. Riley
M. Y. Ballinger
E. G. Damberg
J. C. Evans
A. S. Ikenberry
K. B. Olsen
R. M. Ozanich
C. J. Thompson

Technical Editor:
K. L. Manke

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Pacific Northwest Laboratory
Richland, Washington 99352

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Abstract

The Facility Effluent Monitoring Program characterizes and monitors effluent streams (liquid waste streams) from 300 Area facilities owned by the U.S. Department of Energy and managed by Pacific Northwest Laboratory.^(a) This program is managed by the Pacific Northwest Laboratory. This report summarizes the results of characterizing and monitoring the following sources during a portion of this year: liquid waste streams from Buildings 331, 320, and 3720; treated and untreated Columbia River water; and water at the confluence of the waste streams (that is, end-of-pipe).

Characterization and monitoring data were evaluated for samples collected between March 22 and June 21, 1994, and subsequently analyzed for hazardous chemicals, radioactivity, and general parameters. Except for bis(2-ethylhexyl)phthalate, concentrations of chemicals detected and parameters measured at end-of-pipe were below the U.S. Environmental Protection Agency existing and proposed drinking water standards. The source of the chemicals, except bis(2-ethylhexyl)phthalate, is not currently known. The bis(2-ethylhexyl)phthalate is probably an artifact of the plastic tubing used in the early stages of the sampling program. This practice was stopped.

Concentrations and clearance times for contaminants at end-of-pipe depended strongly on source concentration at the facility release point, waste stream flow rates, dispersion, and the mechanical action of sumps. When present, the action of sumps had the greatest impact on contaminant clearance times. In the absence of sump activity, dispersion and flow rate were the controlling factors.

Future plans call for the use of facility monitoring and waste stream dynamics data (that is, transit times, clearance times, and dilution factors) to do the following:

- Track sources in the liquid waste stream.
- Determine what must be done in a facility to avoid undesirable releases at end-of-pipe.
- Help Treated Effluent Disposal Facility operators determine how to minimize treatment and disposal impacts if an excursion occurs in the liquid waste stream.

Initial field evaluations of a transportable gas chromatograph/mass spectrometer, adsorptive stripping voltametry, and solid phase extraction were favorable.

(a) The Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

Executive Summary

Introduction. Pacific Northwest Laboratory's^(a) Facility Effluent Monitoring Program is responsible for monitoring the effluent streams from U.S. Department of Energy-owned and Pacific Northwest Laboratory-operated facilities in the Hanford Site's 300 Area. For this report, effluent stream is defined as liquid waste, released from a building, that may contain radioactive and/or chemical contaminants. The program is funded by the U.S. Department of Energy's Office of the Deputy Assistant Secretary of Waste Management (EM-30) in response to U.S. Department of Energy Orders 5400.1 and 5400.5. Elements within these orders address responsible management of facility waste streams. The program was initiated at the beginning of fiscal year 1994.

Scope. This report summarizes the initial results from the liquid monitoring component of the program. Chemical and radiological characterization of liquid waste streams at Buildings 331, 320, and 3720; source water (Columbia River treated and untreated); and water at the confluence of the waste streams (that is, end-of-pipe) are discussed. Also, the influences of waste stream physical properties (for example, flow rate, dilution, dispersion, and sump operation) on contaminant behavior during transport from facility to end-of-pipe are assessed. Selected emerging characterization technologies are evaluated for incorporation into the program to enhance cost effectiveness and data quality.

Results. Chlorination of Columbia River water introduces volatile chlorination by-products (that is, chloroform and bromodichloromethane) into the facility source water at levels comparable to those observed in facility liquid waste streams. Observed concentration levels for these constituents were in the range of 1.1 to 32 $\mu\text{g/L}$ in facility waste streams and concentrations of 0.96 to 14 $\mu\text{g/L}$ at end-of-pipe. Chloroform and bromodichloromethane were not detected in Columbia River water; however, these constituents were found in chlorinated river water at concentrations ranging from 0.91 to 25 $\mu\text{g/L}$. While these two constituents are introduced into the waste stream, their observed concentration range at end-of-pipe was below the U.S. Environmental Protection Agency maximum contaminant level for trihalomethanes of 100 $\mu\text{g/L}$.

In selected facility liquid waste effluents, radioactivity and various chemical constituents were detected; however, with one exception, sources contributing to these releases are unknown at this time. Specific findings were as follows:

- Gross alpha (no uranium-235) and gross beta activity at Building 3720 ranged from 7.9 to 44.6 pCi/L and 2.0 to 17.6 pCi/L, respectively.
- Traces of xylenes (1.2 to 12.0 $\mu\text{g/L}$) were found at Building 320 and at end-of-pipe.

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- Ethanol, 2-(2-butoxyethoxy)acetate, was detected at Buildings 320 and 331 in the range of 67 to 510 $\mu\text{g/L}$.
- Bis(2-ethylhexyl)phthalate was found at Buildings 331 and 3720 (1.7 to 6.4 $\mu\text{g/L}$ and 1.2 to 7.6 $\mu\text{g/L}$, respectively) and at end-of-pipe (16 to 38 $\mu\text{g/L}$).
- Acetone was found within the range of 22 to 1,800 $\mu\text{g/L}$ at Buildings 320, 331, and 3720.
- Pesticides were found in the low parts-per-trillion level at Building 331.
- Nitrate levels ranged from 100 to 2,500 $\mu\text{g/L}$ at Buildings 320, 331, and 3720 and at end-of-pipe.
- Cyanide was frequently observed at Building 331 (1 to 70 $\mu\text{g/L}$) and occasionally at Building 3720 (2 to 10 $\mu\text{g/L}$).
- None of the chemicals listed above were detected in untreated or treated Columbia River water.

Based on a very limited data set, the concentrations of contaminants have not changed significantly when compared to previously published historical data. Many constituents have decreased in concentration.

Except for bis(2-ethylhexyl)phthalate, concentrations of chemicals detected and parameters measured at end-of-pipe between March 22 and June 21, 1994, were below the U.S. Environmental Protection Agency existing and proposed drinking water standards. In many cases, the measured values were 10 to 10,000 times lower than regulatory guidelines. Bis(2-ethylhexyl)phthalate, a chemical commonly found in plastic formulations and ubiquitous in the environment, was detected in end-of-pipe waste water at levels (16 to 38 $\mu\text{g/L}$) that were approximately 3 to 6 times the U.S. Environmental Protection Agency drinking water standard (6 $\mu\text{g/L}$). The increased levels are likely artifacts introduced by the use of plastic tubing in the early stages of the sampling program. This practice was stopped.

Occasional elevated levels of sodium, chloride, phosphate, and pH were observed in facility waste streams and/or at end-of-pipe.

Concentrations and clearance times for contaminants at end-of-pipe were highly dependent on waste stream flow rates, dispersion, and the mechanical action of sumps. When present, the action of sumps had the greatest impact on contaminant clearance times. In the absence of sump activity, dispersion and flow rate were the controlling factors. Dye tests performed at Buildings 331 and 3720 showed the following results:

- Average transit times for Buildings 3720 and 331 were 49 minutes, and 4 hours, 21 minutes, respectively.

- The mechanical action of a sump intercepting the Building 331 waste stream resulted in the splitting of the dye into discrete fractions that led to sequential dye discharges at end-of-pipe. The total number of dye pulses at end-of-pipe depended on the initial dye concentration.
- Clearance times (at the building and at end-of-pipe) for Building 3720 were less than 15 minutes in all cases. The generation of three discrete dye fractions in the Building 331 waste stream by an intercepting sump led to an average end-of-pipe clearance time of 4 hours, 23 minutes. This is 13 times greater than the estimated change from dispersion alone.
- Average dilution factors (concentration measured at a building divided by the estimated end-of-pipe concentration) for the dye as it passed from Buildings 3720 and 331 were 30 and 525, respectively.

Future plans call for the use of facility monitoring and waste stream dynamics data (that is, transit times, clearance times, and dilution factors) to do the following:

- Track sources in the liquid waste stream.
- Determine what must be done in a facility to avoid undesirable releases at end-of-pipe.
- Help Treated Effluent Disposal Facility operators determine how to minimize treatment and disposal impacts if an excursion occurs in the liquid waste stream.

Initial field evaluations of a transportable gas chromatograph/mass spectrometer, adsorptive stripping voltametry, and solid phase extraction were favorable.

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Acronym and Abbreviation List

ASV	adsorptive stripping voltametry
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
FEMP	Facility Effluent Monitoring Program
FY	fiscal year
GC/MS	gas chromatograph/mass spectrometer
MCL	maximum contaminant level
NPDES	National Pollutant Discharge Elimination System
PCB	polychlorinated biphenyl
PNL	Pacific Northwest Laboratory
SPE	solid phase extraction
SVOC	semivolatile organic compound
TEDF	Treated Effluent Disposal Facility
Tri-Party Agreement	<i>Hanford Federal Facility Agreement and Consent Order</i>
VOC	volatile organic compound
WHC	Westinghouse Hanford Company

Trademarks Used

Teflon is a registered trademark of duPont Company, Wilmington, Delaware.

Tygon is a registered trademark of Norton Company, Worcester, Massachusetts.

1.0 Introduction

Physical and chemical data are being gathered by the Pacific Northwest Laboratory (PNL) on the liquid waste streams released by 300 Area buildings owned by the U.S. Department of Energy (DOE) and operated by PNL (see Figure 1.1, map). A waste stream is defined as liquid waste, released from a building, that may contain radioactive and/or chemical contaminants. The 300 Area is located on the Hanford Site, a nuclear reservation, in southeastern Washington State. These buildings are involved in basic and applied research, and in evaluating and deploying technology in support of environmental restoration and waste management activities across the DOE complex. Samples from the liquid waste streams have been taken and analyzed for radiological and chemical constituents. The three buildings being studied this fiscal year (FY) are

- Building 320
- Building 331
- Building 3720.

PNL is also studying end-of-pipe, which is where liquid waste streams from all of the 300 Area buildings come together and drain into the 300 Area process leaching trenches. Building 315 effluent (treated Columbia River water) is the source water for the buildings. Untreated and treated water associated with this facility are also being studied as part of this program. Building 315 is operated by Westinghouse Hanford Company (WHC).

Five more buildings are scheduled for baseline characterization and monitoring

as sampling stations come online during FY 1995. The five additional buildings are

- Building 324
- Building 325
- Building 326
- Building 327
- Building 329.

The retention process sewer effluents from these five buildings are sent to 307 Basins where the combined streams are sampled for gross alpha and beta radioactivity before discharge to the process trenches. The process sewer effluent is sent directly to the process trenches. In the future, these streams will be sent to the Treated Effluent Disposal Facility (TEDF). This facility, which is managed by WHC, is scheduled to begin processing effluent in December 1994. The startup of TEDF is a milestone in the *Hanford Federal Facility Agreement and Consent Order*, also called the Tri-Party Agreement (Ecology et al. 1989). Facility liquid waste streams routed to TEDF will be treated and discharged to the Columbia River according to the requirements of a pending National Pollutant Discharge Elimination System (NPDES) permit.

Purpose. The purpose of this report is to summarize the data collected to characterize and monitor the effluents from buildings where radioactive and/or hazardous materials are used. The data are collected to meet the requirements of DOE Orders 5400.1 and 5400.5 and are reported to the Office of the Deputy

Figure 1.1. Location of Pacific Northwest Laboratory-Operated Buildings in the 300 Area

Assistant Secretary of Waste Management (EM-30); DOE, Richland Operations Office; PNL; and WHC. The report summarizes 1) the history of liquid waste disposal from target facilities and their chemistry, 2) the characterization and monitoring work performed from March to August 1994 on three buildings, 3) the waste stream dynamics tests, and 4) the program development.

Objectives. Three objectives that are directed toward meeting the intent of DOE Orders 5400.1 and 5400.5 for the liquid monitoring component of the Facility Effluent Monitoring Program (FEMP) have been identified. They are as follows:

- Establish a basis for evaluating proposed waste acceptance criteria for TEDF

Waste acceptance criteria need to be established on a technically rational basis such that protection of the public and the environment is maximized while minimizing impacts to operations at the 300 Area.

- Incorporate capabilities for proactive detection and response to events

Proactive detection and response to events will reduce the likelihood of having to respond to unusual occurrences and noncompliance problems that negatively impact 300 Area programmatic schedules.

- Enhance quality and cost effectiveness.

Traditional approaches to characterization and monitoring activities are costly and time consuming. Deploy-

ment of advanced characterization and monitoring technology will improve information quality and program cost effectiveness over the long term.

Organization. The 300 Area liquid waste streams are being characterized through the liquid monitoring component of the FEMP. The program is managed by the Facilities and Operations Directorate of PNL. The analytical support task is the liquid monitoring component of the FEMP and is managed by PNL's Earth and Environmental Sciences Center. The technical and management organization of the FEMP is shown in Figure 1.2. The analytical support task has two technical components: 1) routine monitoring of liquid waste streams and 2) program development.

Routine Monitoring. Under this component, samples of source water and liquid waste streams are taken. The samples are analyzed by analytical laboratories under contract with PNL. Samples are analyzed for a broad range of chemical constituents, gross radioactivity, and other general parameters. Data from the analyses have been evaluated to accomplish the following:

- Identify trends and interrelationships in waste stream chemistry.
- Describe chemical contributions made to end-of-pipe chemistry by untreated and treated influent water from Building 315 and facility process and retention process sewers.
- Determine the influence of transport in the waste stream (that is, contaminant dilution and dispersion) on waste stream chemistry at end-of-pipe.

Figure 1.2. Facility Effluent Monitoring Program Organization Chart

Data are stored in a database consisting of modular, commercially available hardware and software. The database structure grows as data needs and decision-making requirements demand.

Program Development. Waste stream dynamics are studied under this component to relate the effects of various contaminant release scenarios on

contaminant residence times and end-of-pipe concentrations. Under program development, promising emerging characterization technologies are evaluated. Over time, the technologies are deployed within the routine monitoring component to improve the cost effectiveness, data quality, and/or responsiveness of monitoring.

2.0 Background

Facilities in the 300 Area started up in February 1943 when the Hanford Site was created by the War Department to produce plutonium. The plutonium was used in the first atomic bombs. Activities in the 300 Area have included fabricating reactor fuel and evaluating pilot-scale separations processes before full-scale application in 200 Area processing plants.

For over four decades, chemically and radiologically contaminated waste waters from these activities were discharged to ponds, trenches, and cribs in the 300 Area. The waste waters were discharged using an intricate system of sewer lines linking facilities to waste disposal areas (Riley and Zachara 1992). The sewer system consisted of sanitary and process components. Most of the information describing the sewer system (past, present, and future) and 300 Area liquid waste disposal practices in this report was obtained from *Past Practices Technical Characterization Study-300 Area-Hanford Site* (Gerber 1992).

Sanitary Sewer and Radioactive Liquid Waste Systems.

The sanitary sewer and radioactive liquid waste systems are not monitored by the effluent monitoring program. These systems are described briefly here to provide a complete picture of the liquid effluent systems.

The sanitary sewer system serves the sanitary (lunchroom and bathroom) needs of the 300 Area buildings. The original 300 Area sanitary sewer system was composed of concrete and vitrified clay pipes that fed into a large septic tank near

the northeast edge of the 300 Area. A tile field led out of the septic tank to allow percolation of liquid wastes into the soil. Two more septic tanks were added in 1951 as new buildings were constructed in the 300 Area. At the same time, north and south leaching trenches replaced the tile field. WHC currently performs the required monitoring of the sanitary sewer system.

The radioactive liquid waste system was built to accept radioactive effluents from new research and development laboratories generating radioactive liquid waste. Originally, the system was a network of single-walled stainless steel pipes that connected several laboratories to Building 340. The original piping system was replaced with double-contained stainless steel pipes and a leak detection system in 1978 and 1979.

Future Plans for the Sanitary Sewer and Radioactive Liquid Waste Systems.

Plans are in place to connect the sanitary sewer with the Richland city publicly owned treatment works and retire the septic tanks in 1995. This is WHC project V-784.

Radioactive liquid waste from the radioactive liquid waste system is transferred from Building 340 to double-shell tanks via a 75,700-L (20,000-gal) rail car every 90 days. A project to upgrade Building 340 is in the initial phase (WHC project W-302).

Process Sewer. The original 300 Area process sewer system was primarily made of vitrified clay pipes with

acid-proof joints that connected the buildings to a process pond. Large quantities of uranium were released via the process sewer to the process pond in the first years of operation. From 1948 to 1956, liquids containing uranyl nitrate hexahydrate, ammonium nitrate, hexone, and other solvents from Building 321 operations were passed through the process sewer to cribs, which were built 8 km (5 mi) north of the 300 Area. Some process waste buildup (including uranium) may be found in process sewer drains, pipes, trenches, and sewer access holes.

As a result of the requirements in the Tri-Party Agreement (Ecology et al. 1989), flow to the process sewer was reduced significantly by installing closed-loop cooling water systems and routing cooling-water streams from the process sewer to the sanitary sewer. As a result, the process sewer system currently handles an average of 760 L/min (200 gal/min) (CH2M Hill and IT Corporation 1994); this is down 79.8% from the previous level of approximately 3,800 L/min (990 gal/min).

Future Plans for the Process

Sewer. Process sewer effluent is scheduled for diversion to TEDF in December 1994 for treatment and subsequent release to the Columbia River. The piping system for the 300 Area process sewer will be replaced in the next few years (WHC project L-070). Improvements will include changing the size and configuration of the process sewer pipes and incorporating a combination of vacuum transfer, gravity transfer, and pump stations for transport of waste water to TEDF.

Retention Process Sewer. In 1953, the retention process sewer was built for facilities with the potential for releasing radioactive material into the waste stream. Liquid wastes, considered clean but having the potential to be radiologically contaminated, were fed via the retention process sewer piping to the 307 Basins. At the 307 Basins, the liquids are screened for radiological activity. If radioactivity was not detected above release limits, the wastes were discharged to the trenches. If levels were above release limits, the wastes would have been pumped into the Building 340 radioactive liquid waste system tanks.

Future Plans for the Retention Process Sewer.

The piping system for the retention process sewer will be improved as part of WHC project L-070. This upgrade will change the configuration and size of the pipes, and incorporate new lift stations. In addition, the 300 Area building diverter stations will be upgraded to provide more sensitive radiological monitors. An upgrade to the 307 Basin system is currently ongoing.

Historical Information on 300 Area Waste Streams.

During the more than four decades of facility operation in the 300 Area, disposal practices have been modified significantly, and flow-reduction efforts have reduced the total volume of liquid effluent discharged by a factor of 5 to 10. In addition, because the research activities in the 300 Area are always changing, the chemical and radiological nature of the waste stream is always changing. Thus,

unequivocally determining what is in the waste stream at any given point in time is difficult, if not impossible. Furthermore, comparisons between past data and current data can only be made on a qualitative basis.

The *Preliminary 300 Area Process Wastewater Stream-Specific Report* (Millikin et al. 1990) published by WHC describes the process sewer network as being subdivided into major contributing branches. Based on the network design, approximately 20 strategic locations were selected within the process sewer for sampling. Several of the delineated branches of the process sewer corresponded to specific points of release from PNL-operated buildings. However, many of the branches reflected contributions from multiple facilities and, therefore, did not represent any one building's effluent.

Characterization data from the stream-specific report and other published reports (McKenney 1989; WHC 1992) are summarized in Table 2.1. Numerous organic compounds and heavy metals were detected at upstream locations in the process sewer but not at end-of-pipe. The data are considered of limited use because all of the samples were taken as grab samples and, therefore, represent, at any given time, a "snapshot" of the liquid waste stream composition.

Table 2.1. Historical Summary of 300 Area Liquid Effluent

Parameter	Concentration Range (µg/L)
General Chemical Parameter	
Alkalinity	40,000-60,000
Ammonia	100-400
Cyanide	10-15
Total carbon	10,000-16,000
Anions	
Chloride	3,000-4,000
Fluoride	200-250
Nitrate	1,000-3,500
Phosphate	1,800-8,500
Sulfate	17,000-20,000
Cations	
Aluminum	150-300
Barium	30-40
Cadmium	2-30
Calcium	~ 20,000
Copper	10-125
Iron	100-550
Lead	5-15
Magnesium	4,000-5,000
Manganese	5-35
Mercury	0.1-2
Nickel	10-15
Potassium	800-1,500
Sodium	2,500-5,000
Zinc	15-500
Organic Compounds	
Acetone	30-110
Chloroform	12-55
Gross Radioactivity	
Alpha activity	1-900 ^(a)
Beta activity	1-380 ^(a)
(a) Units are picocuries/liter.	

3.0 FY 1994 Liquid Waste Stream Characterization and Monitoring

Routine sampling of liquid waste streams is performed by PNL's Field Sampling Team. Collected samples are screened for radioactivity. Radiological screening is performed by PNL's Analytical Chemistry Laboratory with backup support from WHC's 222S Laboratory. After screening, samples are sent offsite for radiological and chemical analysis. IT Corporation (Richland, Washington) performs radiochemical analysis, and DataChem (Salt Lake City, Utah) performs chemical and general parameter analyses. Received data are evaluated by the project team for completeness and adherence to the statement of work requirements. Then, the data are stored in the database for subsequent evaluation relative to the liquid waste characterization and monitoring purpose and objectives.

The analytical contract is overseen through PNL's Analytical Support Services Program. Through the program, existing PNL contracts with DataChem Laboratories and IT Corporation were used to initiate characterization activities. This strategy saved the time and effort that would have been needed to choose, validate, and contract with a laboratory.

PNL's field sampling team, with a history of successful deployment of field sampling activities for Hanford groundwater monitoring programs, was recruited to perform the liquid waste field sampling for this program. This decision reduced the amount of time before information was available to begin assessing waste water quality.

A total of eight facilities, which includes process and retention (radiological) process sewers, were scheduled for routine sampling in FY 1994. Of these eight facilities, three (Buildings 320, 331, and 3720) were both sampled and analyzed. Sampling at other facilities will start when installation of the sampling equipment is completed. In addition to the three facilities, untreated Columbia River water, treated Columbia River water from Building 315, and water from end-of-pipe were sampled. The locations of the facilities and end-of-pipe considered as part of the scope of this program are shown in Figure 3.1.

Materials and Properties Being Studied. Materials and properties targeted for study were based on current and historical facility activities and chemical and radiological inventories. Activity and inventory information was acquired by interviewing researchers and studying the results of inventory surveys. The samples were analyzed for the following:

- alkalinity
- ammonium
- anions
- chemical oxygen demand
- cyanide
- gross alpha radioactivity
- gross beta radioactivity
- metals
- organic compounds (volatile and semivolatile)
- sulfides
- total carbon
- total dissolved solids
- total organic carbon.

Figure 3.1. Location of Facilities and End-of-Pipe

Based on known facility activities, chemical and radiological inventories, or monitoring results, samples from selected locations were analyzed for additional species [for example, other metals, pesticides, polychlorinated biphenyls (PCBs), tritium, and other radionuclides]. All samples were analyzed using methods in EPA SW-846 (EPA 1986) or other approved methods.

Sampling Stations. Sampling stations are access points at facilities for collecting process sewer and retention process sewer waste water samples. Also, the stations contain equipment for monitoring waste stream flow and chemistry.

Dedicated Sampling Stations. Each sampling station consists of a refrigerated sampler, flow meter, pH meter, and conductivity meter. The samplers collect flow-proportional composite samples using a peristaltic pump and 9.5-mm (3/8-in.) Teflon®-lined tubing. Flow, pH, and conductivity data are collected centrally via the Facility Management Control System at nine of the stations. Data collection at non-dedicated locations (Buildings 331 and 3720) is provided via standalone data acquisition equipment.

Status of Station Installations. Sampling stations have been installed at Buildings 320, 331, and 3720. These locations were chosen based on their higher potential to discharge hazardous constituents into the 300 Area sewer systems (see Figure 3.1). Also, three sample ports were built for Buildings 3730, 318, and 324 (High Bay), which have lower discharge potential. The sample ports allow the use of portable sampling equipment but have no continuous monitoring capabilities. By the end of calendar year 1994, sampling stations should be installed at Buildings 324, 326, 327, and 329 (PNL facilities within the

300 Area). Also, a sampling station will be installed at Building 325 when access is granted.

Sampling Limitations and

Schedule. Because of the time needed to report analytical results, the routine liquid monitoring data in this document only reflect samples obtained from March 22 through June 21, 1994. Untreated and treated Columbia River water sampling began on May 11, 1994. The small number of samples collected at the source water locations requires that this data be considered strictly tentative. The schedule and number of 24-hour composite liquid samples collected from the facilities, source water, and end-of-pipe are described in Table 3.1. Grab samples were collected for volatile organic compound (VOC) analysis using a U.S. Environmental Protection Agency (EPA) standard protocol. In addition, facility liquid effluents were sampled during off-work hours (for example, weekends, evenings, and holidays) approximately once a month to obtain estimates of background chemical and radiological discharge characteristics.

Sampling. The majority of the samples were acquired using refrigerated, flow-proportional composite samplers at dedicated sampling locations. The lack of an integrated sampler and flow-meter at end-of-pipe and at the source water (treated and untreated Columbia River water) sampling locations made time-proportional composite sampling necessary. Each sample was composited for approximately 24 hours. Samplers were typically programmed to begin sampling in the morning (between 7 a.m. and 9 a.m.) and were ready for sample collection the following morning.

Sample Blanks. Throughout the sampling campaigns, blank samples consisting

Table 3.1. Samples Collected from March to July 1994^(a)

Location	March	April	May	June	July
Building 331 process sewer ^(b)	2	4/2	4/2	4/2	4/2
Building 3720 process sewer	1	1	4/2	4/2	4/2
Building 320 process sewer	1	3	4/2	4/2	4/2
End-of-pipe	2	4/2	4/2	4/2	4/2
Untreated Columbia River water	0	0	2	2	2
Treated Columbia River water from Building 315	0	0	2	2	2
<p>(a) Numbers are samples collected as part of routine monitoring. All samples were 24-hour flow-proportional composites. The first number indicates samples taken during normal work hours, the second number indicates samples taken off-shift hours.</p> <p>(b) Pesticide and polychlorinated biphenyl analyses were also done on samples collected from this facility.</p>					

of laboratory purified water (including trip blanks and equipment blanks) were subjected to the same handling procedures as real samples and sent to the laboratories for analysis.

Results. The results of the blank samples (Table 3.2) were typically well below real sample constituent concentrations. Thus, artifact introduction during sample handling was minimal and did not appear to influence the analytical results.

The mean concentration and standard deviation of detected analytes and parameters are reported in Tables 3.3 through 3.9. However, only constituents with at least three positive detects are listed. This criterion combined with the fact that many results were near the laboratory method detection limits means the tables do not report some analytes. In the text, however,

analytes detected less than three times are discussed.

Source Water. A few general observations can be made regarding Columbia River source water. Untreated and treated source water had very similar cation concentrations. This is not clear from Tables 3.3 and 3.4 because of the requirement for data in the tables to have at least three positive detects. Selected anion concentrations were significantly higher in the treated source water than in the untreated source water. Chloride was approximately four times higher; fluoride was approximately two times higher; sulfate was 50 to 70% higher. Although 10 to 20% of total carbon and total organic carbon are removed in the treatment process, trihalomethanes (for example, chloroform) are introduced, increasing their concentrations above source background levels. The most

Table 3.2. Constituents in Liquid Effluent Blanks

Constituent	Frequency ^(a)	Concentration (µg/L)		
		Range	Average	Standard Deviation
General Chemical Parameters				
Total carbon	5/5	500-1,000	760	195
Total organic carbon	3/5	400-500	433	58
Anions				
Nitrate	4/8	80-200	140	69
Sulfate	6/8	70-80	73	5
Cations				
Calcium	3/5	37-52	45	7.5
Lead	3/3	1.0-1.8	1.4	0.4
Sodium	3/5	52-60	56	4
Zinc	3/5	2.3-5.5	4.2	1.7
Organic Compounds				
Chloroform	4/16	3.1-4.6	3.9	0.6
Tetrahydrofuran	6/13	2.8-6.7	4.7	1.6
(a) Number of samples with detectable constituent/total number of samples analyzed.				

common chlorination byproduct (chloroform) was found at similar or lower concentrations at all other sampling locations (other than the untreated source water, which contained no detectable chloroform).

Facility and End-of-Pipe Chemistry.

Data for Buildings 320, 331, and 3720, and end-of-pipe are summarized in Tables 3.5, 3.6, 3.7, and 3.8 respectively. Comparisons of constituent concentrations among these four locations, as well as with source water, allow each facility to be evaluated for potential discharges. The presence of a constituent in a facility's effluent is not necessarily a result of current disposal practices. The presence of a constituent may

be the result of other factors (for example, past disposal practices, untreated source water characteristics, source water treatment, outdoor activities such as pest control, or sampling and/or laboratory error).

General Chemical Parameters. General chemical parameter concentrations were similar among all locations with the following exceptions. One high chemical oxygen demand result was found at end-of-pipe (Table 3.8). Slightly higher levels of total carbon, total organic carbon, and ammonia were found at Building 331 (Table 3.6). Cyanide was frequently detected at Building 331 (Table 3.6),

Table 3.3. Constituents in Untreated Source (River) Water

Constituent	Frequency ^(a)	Concentration (µg/L)		
		Range	Average	Standard Deviation
General Chemical Parameters				
Alkalinity	3/3	48,000-60,000	52,667	6,429
Total carbon	3/3	18,000-21,000	19,667	1,527
Total organic carbon	3/3	4,000-7,000	5,000	1,732
Cations				
Aluminum	3/3	36-260	115	125
Barium	3/3	28-33	30	3
Calcium	3/3	18,000-21,000	19,333	1,528
Iron	3/3	67-530	223	266
Lead	3/3	0.62-8.1	3.2	4.3
Magnesium	3/3	4,300-5,100	4,633	416
Manganese	3/3	4.5-16	8.8	6.3
Potassium	3/3	1,200-1,400	1,267	115
Sodium	3/3	2,400-3,000	2,633	321
(a) Number of samples with detectable constituent/total number of samples analyzed.				

and trace levels were also found at Building 3720 (Table 3.7).

Anions. Many effluent anion concentrations were similar to the source water (chloride, fluoride, phosphate, and sulfate). Exceptions include a single large chloride excursion at end-of-pipe (Table 3.8), elevated chloride levels (approximately 50%) at Building 331, and high phosphate concentrations at Building 331. Nitrate levels were approximately three times higher at all facilities relative to the source water, although Building 331 had levels approximately five times higher.

Cations. Many effluent cation concentrations were similar to the source water (barium, calcium, magnesium, manganese, and potassium), although potassium was almost twice as high at Building 331 (Table 3.6). Sodium concentrations were two to three times higher at Buildings 331 and 3720 relative to the source water. A single large excursion of sodium was also detected at end-of-pipe (Table 3.8). Iron, copper, lead, and zinc were almost always detected in all effluents, while tin was detected in about half. Trace levels of cadmium were occasionally detected in Building 320 effluent (Table 3.5), and trace levels of mercury were typical in Building 3720 effluent (Table 3.7).

Table 3.4. Constituents in Treated Source Water

Constituent	Frequency ^(a)	Concentration (µg/L)		
		Range	Average	Standard Deviation
General Chemical Parameters				
Alkalinity	3/3	40,000-60,000	46,667	11,547
Total carbon	3/3	15,000-19,000	17,333	2,082
Total organic carbon	3/3	3,000-5,000	4,000	1,000
Anions				
Chloride	3/3	4,000-5,100	4,400	608
Fluoride	3/3	500-600	533	71
Sulfate	3/3	16,000-17,000	16,333	577
Organic Compounds				
Bromodichloromethane	5/5	0.91-1.4	1.1	0.2
Chloroform	6/6	15-25	19.2	3.5
(a) Number of samples with detectable constituent/total number of samples analyzed.				

Volatile Organic Compounds. To varying degrees, VOCs were found at all locations, and concentrations were typically very low. Chloroform and bromodichloromethane (both chlorination byproducts) were found at all facilities, although the concentrations were similar to or less than the concentration in the treated source water (Table 3.4). Xylenes (reported as the sum of ortho-, meta-, and para-xylene) were found intermittently at very low concentrations at several locations (Tables 3.5 and 3.8).

Acetone was also found intermittently at several locations. The concentration of acetone averaged 27 to 482 µg/L (Tables 3.5, 3.6, and 3.7). Single releases of moderate concentrations of acetone were detected at Buildings 331 and 3720, although no acetone was ever detected at

end-of-pipe. Factors that can influence the difference observed include dilution, volatilization, and decomposition in the waste stream.

Semivolatile Organic Compounds.

The most common semivolatile organic compound (SVOC) detected was bis(2-ethylhexyl)phthalate, which is known to leach from plastic tubing. The use of a Tygon® sampling line at end-of-pipe is a likely source (Table 3.8). A second SVOC [ethanol, 2-(2-butoxyethoxy)acetate] of unknown origin was also found at Buildings 320 and 331 (Tables 3.5 and 3.6).

Pesticides. Extremely low levels (2 to 33 parts per trillion) of pesticides (Heptachlor, delta-BHC, Endosulfan I, and gamma-BHC) were occasionally found at Building 331 (Table 3.6) and end-of-pipe

Table 3.5. Building 320: Constituents in Liquid Effluent

Constituent	Frequency ^(a)	Concentration (µg/L)		
		Range	Average	Standard Deviation
General Chemical Parameters				
Alkalinity	14/14	40,000-70,000	55,000	10,000
Sulfides	13/17	200-300	270	60
Total carbon	12/12	14,000-17,000	15,900	1,200
Total dissolved solids	16/16	80,000-170,000	99,000	22,000
Total organic carbon	14/14	2,000-3,000	2,200	400
Anions				
Chloride	12/12	3,800-4,700	4,250	261
Fluoride	12/12	400-600	492	79
Nitrate	12/12	100-2,000	600	564
Sulfate	12/12	15,000-18,000	16,417	793
Cations				
Aluminum	12/12	42-170	92	42
Barium	12/12	28-33	30	1.5
Cadmium	3/12	3.5-4.3	3.8	0.4
Calcium	12/12	19,000-23,000	20,250	1,357
Copper	12/12	4.9-8.2	6.5	1.1
Iron	11/12	19-42	27.1	7.8
Lead	8/12	0.6-1.8	1.0	0.4
Magnesium	12/12	4,200-5,400	4,700	374
Manganese	9/12	1.0-1.8	1.3	0.2
Potassium	9/12	990-1,600	1,288	205
Sodium	12/12	2,600-3,200	2,817	185
Tin	4/12	37-82	56	22
Zinc	12/12	15-33	21.5	6.2
Organic Compounds				
Acetone	3/18	25-31	27	3.5
Bromodichloromethane	7/9	1.1-2.1	1.3	0.4
Chloroform	18/18	10-32	19.9	5.6
Ethanol, 2-(2-butoxyethoxy) acetate	4/4	67-510	277	182
Xylenes (total)	3/18	3.1-12.0	8.7	4.9
(a) Number of samples with detectable constituent/total number of samples analyzed.				

Table 3.6. Building 331: Constituents in Liquid Effluent

Constituent	Frequency ^(a)	Concentration (µg/L)		
		Range	Average	Standard Deviation
General Chemical Parameters				
Alkalinity	16/16	40,000-70,000	58,594	8,562
Ammonia	11/14	30-1,000	231	381
Chemical oxygen demand	14/16	7,000-50,000	19,714	11,841
Cyanide	13/16	1.0-70	27.4	27.0
Sulfides	5/16	200-400	260	89
Total carbon	13/13	14,000-30,000	20,538	4,033
Total dissolved solids	16/16	90,000-140,000	114,375	14,127
Total organic carbon	14/14	2,000-14,000	6,000	3,013
Anions				
Chloride	14/14	3,600-13,000	6,471	2,215
Fluoride	14/14	400-700	586	86
Nitrate	14/14	300-2,500	1,071	662
Phosphate	7/14	500-6,100	2,429	1,902
Sulfate	14/14	14,000-19,000	17,000	1,710
Cations				
Aluminum	14/14	70-630	223	199
Barium	14/14	24-34	28.6	2.8
Calcium	14/14	19,000-23,000	20,357	1,277
Copper	14/14	12-110	34.7	28.4
Iron	14/14	23-260	90	59
Lead	15/15	0.66-5.0	2.0	1.2
Magnesium	14/14	4,500-5,300	4,871	261
Manganese	13/14	1.3-7.8	3.1	1.7
Potassium	12/14	940-7,500	2,270	1,863
Sodium	14/14	3,400-17,000	6,893	3,587
Tin	11/14	72-110	85	25.5
Zinc	14/14	37-140	71	27
Organic Compounds				
Acetone	4/21	29-1,800	481.5	879
Bis(2-ethylhexyl)phthalate	6/7	1.7-6.4	3.7	2.1
Bromodichloromethane	5/10	0.76-2.0	1.4	0.47
Chloroform	21/21	5.8-23	11.8	4.9
Delta-BHC	5/14	0.006-0.044	0.016	0.011
Endosulfan I	6/14	0.002-0.006	0.004	0.002
Ethanol, 2-(2-butoxyethoxy) acetate	4/4	150-190	175	17
Gamma-BHC (Lindane)	4/14	0.002-0.004	0.003	0.001
(a) Number of samples with detectable constituent/total number of samples analyzed.				

Table 3.7. Building 3720: Constituents in Liquid Effluent

Constituent	Frequency ^(a)	Concentration (µg/L)		
		Range	Average	Standard Deviation
General Chemical Parameters				
Alkalinity	11/11	40,000-80,000	59,100	11,362
Chemical oxygen demand	9/13	7,000-51,000	14,322	13,906
Cyanide	4/10	2-10	5	3.5
Sulfides	4/12	200-300	225	50
Total carbon	9/9	14,000-30,000	20,778	4,494
Total dissolved solids	12/12	80,000-150,000	108,333	18,990
Total organic carbon	10/10	2,000-16,000	4,400	4,195
Anions				
Chloride	12/12	3,800-4,700	4,250	261
Fluoride	12/12	400-600	492	79
Nitrate	12/12	100-2,000	600	564
Sulfate	12/12	15,000-18,000	16,417	793
Cations				
Aluminum	9/9	50-190	105	42
Barium	9/9	24-66	33.7	12.9
Calcium	9/9	20,000-29,000	22,000	2,828
Copper	9/9	6.8-130	26.1	39.1
Iron	9/9	53-110	82.7	18.6
Lead	9/9	1.1-7.1	3.4	1.8
Magnesium	9/9	4,300-6,400	5,000	620
Manganese	9/9	2-10	4.2	2.5
Mercury	11/12	0.10-0.87	0.24	0.23
Potassium	8/9	960-1,700	1,270	256
Sodium	9/9	3,100-13,000	6,456	2,878
Tin	4/9	34-65	52.8	14.1
Zinc	9/9	31-280	73.3	79.1
Organic Compounds				
Acetone	5/10	22-530	133	223
Bis(2-ethylhexyl)phthalate	5/8	1.2-7.6	3.8	2.5
Bromodichloromethane	4/6	0.75-1.3	0.98	0.2
Chloroform	10/10	5-18	11.9	3.9
Trichloroethene	10/10	1.0-1.8	1.3	0.2
(a) Number of samples with detectable constituent/total number of samples analyzed.				

Table 3.8. End-of-Pipe: Constituents in Liquid Effluent

Constituent	Frequency ^(a)	Concentration (µg/L)		
		Range	Average	Standard Deviation
General Chemical Parameters				
Alkalinity	10/10	40,000-60,000	48,400	5,700
Chemical oxygen demand	7/12	8,000-84,000	21,857	27,733
Sulfides	5/12	200-300	240	55
Total carbon	10/10	14,000-22,000	17,300	2,751
Total dissolved solids	11/11	80,000-110,000	93,636	8,090
Total organic carbon	11/11	2,000-8,000	3,091	1,700
Anions				
Chloride	10/10	4,000-190,000	23,480	58,515
Fluoride	10/10	300-800	550	135
Nitrate	10/10	400-1,500	680	371
Sulfate	10/10	14,000-19,000	17,000	1,333
Cations				
Aluminum	10/10	58-150	103	29
Barium	10/10	28-64	33	11
Calcium	10/10	19,000-45,000	22,600	7,919
Copper	10/10	9.0-15	11.5	1.8
Iron	10/10	51-110	80	17
Lead	9/9	1.0-3.8	2.2	1.1
Magnesium	10/10	4,400-10,000	5,300	1,677
Manganese	10/10	2.0-5.1	3.2	1.0
Potassium	10/10	950-1,600	1,234	204
Sodium	10/10	3,200-95,000	12,790	28,888
Tin	5/10	35-100	62	25
Zinc	10/10	24-60	38	11
Organic Compounds				
Bis(2-ethylhexyl)phthalate	7/7	16-38	25.4	7.5
Bromodichloromethane	5/9	0.96-2.6	1.7	0.7
Chloroform	14/14	5.2-14	10.2	2.5
Heptachlor	3/7	0.008-0.01	0.009	0.001
Trichloromonofluoromethane	3/9	1.5-4.4	3.4	1.6
Xylenes (total)	4/14	1.2-2.1	1.7	0.4
(a) Number of samples with detectable constituent/total number of samples analyzed.				

(Table 3.8). In addition, delta-BHC was found twice in untreated source water and once in treated source water (data not shown). This pesticide could be the result of analytical laboratory error. Analytical error is very likely at these ultra-trace concentrations. Currently, the reason for the higher frequency of detection of these constituents at Building 331 (Table 3.6) is unknown.

Radiological Parameters. Because of the sensitive analysis methods used, very low levels of gross alpha and gross beta radiation were detected. Because of errors associated with counting radioactive decay, clean samples may exhibit false positive results as seen in the apparent positive readings in the blanks (Table 3.9). Building 3720 effluent exhibited continuous low-level detection of alpha and beta radioactivity (Table 3.9).

Comparison to Historical Data. The concentrations of the majority of these chemicals have not changed significantly from historically reported levels. However, general decreases have been seen in the following: heavy metals (cadmium, lead, mercury, and nickel), some anions (nitrate and phosphate), individual organic compounds, and radioactivity (gross alpha and beta). Fluoride, on the other hand, appears to have increased by a factor of 2 to 3.

Compliance at End-of-Pipe.

Table 3.10 compares concentrations at end-of-pipe with maximum contaminant level (MCL) parameters. The following EPA-regulated constituents were detected at end-of-pipe:

- xylenes
- bis(2-ethylhexyl)phthalate
- Heptachlor (a pesticide)
- total trihalomethanes
- barium
- copper
- lead
- fluoride
- nitrate
- gross alpha radioactivity
- gross beta radioactivity
- tritium.

Except for bis(2-ethylhexyl)phthalate, all organic chemicals were below EPA MCLs. The reported concentration range for bis(2-ethylhexyl)phthalate was above the EPA MCL by a factor of 3 to 6.

Barium and lead were below the EPA MCL by factors of 100 and 10, respectively. Copper was a factor of 100 below a proposed EPA MCL. Fluoride and nitrate were within a factor of 10 of the EPA MCL. Tritium was a factor of 100 below the EPA MCL while gross alpha and gross beta radioactivity levels were within a factor of 10 of EPA MCLs.

Table 3.9. Radiological Parameters of 300 Area Facility Liquid Effluent

Constituent	Frequency ^(a)	Concentration (pCi/L)		
		Range ^(b)	Mean	Standard Error of Mean
Building 320				
Gross alpha activity	1/17	-0.2 to 1.0	0.3	0.1
Gross beta activity	5/17	0 to 3.9	1.1	0.3
Building 331				
Gross alpha activity	4/17	-0.2 to 12.8	1.3	0.8
Gross beta activity	6/17	-0.6 to 7.1	2.0	0.4
Building 3720				
Gross alpha activity	13/13	7.9 to 44.6	17.7	2.7
Gross beta activity	12/13	2.0 to 17.6	5.6	1.2
End-of-Pipe				
Gross alpha activity	12/12	1.8 to 5.2	3.2	0.3
Gross beta activity	3/12	-0.1 to 3.6	1.6	0.3
Tritium	1/13	-244 to 423	39	46
Blanks				
Gross alpha activity	0/5	-0.3 to 0.2	-0.1	0.1
Gross beta activity	1/5	-1.4 to 2.3	0.2	0.6
Tritium	2/3	127 to 279	212	45
(a) Number of samples with counts greater than the total counting error/total number of samples analyzed.				
(b) Negative numbers indicate less than background values measured.				

Table 3.10. End-of-Pipe Chemistry Compared to Regulatory Compliance

Constituent or Parameter	Concentration ^(a)	
	End-of-Pipe ^(b)	EPA Maximum Contaminant Level
Xylenes	1.2 to 2.1	10,000
Bis(2-ethylhexyl)phthalate	16 to 38	6
Total trihalomethanes	0.96 to 14	100
Heptachlor	0.008 to 0.01	0.4
Barium	28 to 64	1,000
Copper	9.0 to 15	1,300
Lead	1.0 to 3.8	50
Fluoride	300 to 800	4,000 ^(c)
Nitrate	400 to 1,500	10,000
Gross alpha radioactivity	1.8 to 5.2 ^(d)	15 ^(d)
Gross beta radioactivity	-0.1 to 3.6 ^(d)	8 ^(d)
Tritium	-244 to 423 ^(d)	20,000 ^(d)
(a) Concentrations are in micrograms/liter unless otherwise noted.		
(b) Samples collected between March 22 and June 21, 1994.		
(c) Proposed maximum contamination level.		
(d) Concentrations in picocuries/liter.		

4.0 Waste Stream Dynamics Tests

Waste stream dynamics tests involve the release of a known concentration and volume of a nonhazardous, fluorescent dye (Acid Red #52) into a facility waste stream. The dye's concentration is then monitored using a fluorometer at the facility sampling point and at the end-of-pipe. Based on the fluorescence readings (which are directly proportional to concentration), important stream flow parameters such as contaminant transit times and dilution factors can be assessed.

Two facilities were chosen for an initial appraisal of 300 Area stream dynamics:

- Building 331, the PNL facility furthest from the end-of-pipe
- Building 3720, the PNL facility closest to the end-of-pipe.

In each experiment, continuous, real-time data collection was facilitated by diverting a portion of the stream through a flow cell mounted inside each fluorometer. The temperature and pH of the stream were also continuously monitored for later correction of the fluorescence measurements.

Test Schedule. The schedule for conducting waste stream dynamics tests is summarized in Table 3.1. Initial tests at Buildings 331 and 3720 in February 1994, 1) generated information to refine test design, 2) evaluated field logistics, and 3) developed and tested the dye measurement system.

Table 4.1 summarizes the test activity during July. A total of 12 dye releases were made at Building 3720, while 6 releases were made at Building 331. Unfortunately,

seven of the releases were not useful for quantitative dilution estimates, because the dye concentration at the end-of-pipe was too high for the limited dynamic range of the fluorometer. However, the off-scale data were useful for determining transit and clearance times.

Results. Figures 4.1 and 4.2 display representative end-of-pipe fluorescence profiles associated with dye releases at Buildings 3720 and 331, respectively. Normally, each dye release at Building 3720 resulted in one predominant peak (observed initially at the facility and later at the end-of-pipe) from which timing parameters and dilution factors were calculated. The data from Building 331 were more complex. Multiple peaks with decreasing intensities were observed at the end-of-pipe following each dye release (Figure 4.2). This behavior appears to result from incomplete purging of the 3906 lift-station sump located southeast of Building 315. Clearly, the multiple peaks from Building 331 releases will be important in event-response activities at this facility.

Three types of time measurements were collected in each waste stream dynamics test: arrival time, transit time, and clearance time (Figure 4.3). Arrival time is the time at which a contaminant is first detected at the end-of-pipe. Transit time is the time at which the maximum concentration passes a given point (for example, end-of-pipe). Clearance time is the time required for a discrete stream constituent (that is, a contaminant which has been released into a stream) to completely flow past a given point. Clearance times were determined at both facilities and at end-of-pipe.

Figure 4.1. End-of-Pipe Fluorescence From Two Dye Releases at Building 3720

Table 4.1. Stream Dynamics: July Test Dates and Number of Dye Releases

Building	Number of Dye Releases					
	July 1	July 7	July 8	July 11	July 14	July 21
3720	1 ^(a,b) 2 ^(a)	1 1 ^(a) 1 ^(b)	2 ^(a)	1 3 ^(b)	—	—
331	—	1 ^(a)	—	1 ^(a)	2	2 ^(b)
(a) Detection at end-of-pipe only.						
(b) Off-scale measurement(s) at end-of-pipe (data not useful for dilution estimates).						

Arrival Times and End-of-Pipe Transit Times. The average and range of arrival times and end-of-pipe transit times for Buildings 3720 and 331 are listed in

Tables 4.2 and 4.3. Generally, the transit times were about 5 to 8 minutes greater than the arrival times. For Building 3720, the average transit time was 49 minutes;

Figure 4.2. End-of-Pipe Fluorescence at Building 331

the corresponding time for Building 331 was 4 hours and 21 minutes (for the primary dye peak). The longer time required to travel from Building 331 is consistent with the longer flow path from Building 331 to the end-of-pipe (Table 3.1). In principle, the average transit times in Tables 4.2 and 4.3 serve as a range that should encompass the transit times of the other 300 Area facilities. However, low flow rates at some facilities and the unknown retention properties of sumps could place other facilities outside this range.

Each facility showed a large range in transit times (Tables 4.2 and 4.3). Indeed, the ranges are greater than the corresponding average transit times. These variations are primarily caused by the activation of sump pumps and fluctuations in the process sewer flow rates.

Clearance Times. The average clearance times at the two buildings and the end-of-pipe are listed in Table 4.4. For both buildings, the band(s) of dye broadened by the time they reached the end-of-pipe. This broadening was attributed to normal dilution and dispersion of the dye as it passed through the process sewer. However, for Building 331, the primary influence on the clearance time was the action of a sump, which resulted in an average clearance time of 4 hours, 23 minutes. This sump-enhanced increase is approximately 13 times greater than the estimated change from dispersion alone. An additional factor that may lengthen clearance times at both facilities is chemical interaction by some contaminants (for example, adsorption of metals on a pipe).

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A = arrival time. B = transit time. C = clearance time.

Figure 4.3. Time Measurements From Fluorescence Data. The lower trace corresponds to a flow path with an intercepting sump.

Table 4.2. Building 3720 Arrival and Transit Times

	Average	Range
Arrival Time	42 min	18 min to 1 h 12 min
Transit Time	49 min	19 min to 1 h 25 min

Because the fluorescent dye is relatively inert, such interactions have not influenced these results.

Dilution Factors. Dilution factors for the two buildings are summarized in Table 4.5. On average, the dye was diluted by a factor of 30 as it passed from Building 3720 to the end-of-pipe; the corresponding dilution

factor for Building 331 was 524. These values are consistent with the observed dispersion (peak-width) changes associated with the two facilities.

For Building 331, each pulse of dye created by the action of the 3906 sump was approximately two to three times more dilute than its predecessor (Table 4.5).

Table 4.3. Building 331 Arrival and Transit Times

	Primary Dye Peak		Secondary Dye Peak		Tertiary Dye Peak ^(a)	
	Average	Range	Average	Range	Average	Range
Arrival Time	4 h 13 min	2 h 38 min to 7 h 30 min	6 h 16 min	3 h 35 min to 10 h 53 min	7 h 59 min	6 h 6 min to 10 h 34 min
Transit Time	4 h 21 min	2 h 51 min to 7 h 36 min	6 h 20 min	3 h 39 min to 10 h 55 min	8 h 1 min	6 h 7 min to 10 h 36 min
(a) A third peak was not observed in every test.						

Table 4.4. Average Clearance Times

Building	At the Building	At the End-of-Pipe	Relative Increase (%)
3720	6 min 36 s	8 min 30 s	29
331	9 min 12 s	4 h 23 min	2,760

Table 4.5. Dilution Factors

Building	Primary		Secondary		Tertiary ^(a)	
	Average	Range	Average	Range	Average	Range
3720	30	8 - 54	—	—	—	—
331	524	471 - 616	980	638 - 1561	3,092	2,893 - 3,290
(a) A third peak was not observed in every test at Building 331.						

Thus, for an event involving the release of a contaminant from Building 331, the number of significant pulses at the end-of-pipe will depend largely on the initial concentration of the contaminant. Consequently, the initial concentration will have a major influence on the end-of-pipe clearance time.

As these results indicate, clearance times, sump effects, and contaminant concentration will have to be considered seriously along with flow rates in determining the overall impacts of facility releases on the chemistry at the end-of-pipe.

5.0 Technology Evaluation

Technologies under development (during late FY 1993 and early FY 1994) in DOE's Office of Technology Development (EM-50) were screened for potential use in the FEMP. Also screened were technologies on display at the U.S.'s largest show of modern laboratory equipment sponsored by the Spectroscopy Society of Pittsburgh and the Society for Analytical Chemists of Pittsburgh (called the Pittsburgh Conference) (February 27 through March 4, 1994, in Chicago, Illinois). Through the task planning process, three technologies were selected for evaluation and possible future deployment, beginning in FY 1994. The three technologies were selected and evaluated based on the potential to improve near and intermediate-term cost effectiveness for effluent characterization and monitoring. The technologies are as follows:

- field transportable gas chromatograph/mass spectrometer (GC/MS) to measure VOCs
- adsorptive stripping voltametry (ASV) for field measurement of chemical uranium
- solid phase extraction (SPE) for pre-concentration and cleanup of liquid samples in the laboratory or the field.

The schedule of FY 1994 activities for each of these three technologies is summarized in Figure 5.1.

Gas Chromatograph/Mass Spectrometer.

Of the field transportable GC/MS systems available, the SpectraTrak GC/MS was chosen and

purchased because it is capable of powerful analyses in both field or laboratory use (McDonald et al. 1994). To provide complete remote capability for the GC/MS, a small 2.4-KVA generator was also purchased. The SpectraTrak GC/MS has been used to measure VOCs at Sandia National Laboratory's Chemical Waste Landfill (as part of DOE's Office of Technology Development program) and in Department of Defense remediation activities. Moreover, the SpectraTrak GC/MS has been used in the commercial sector addressing remediation and health and safety issues.

Methods Development and Analysis of Water Samples.

The primary emphasis in FY 1994 has been water analysis. The GC/MS can analyze either VOCs or SVOCs in water or gaseous matrices. However, because sample preparation is more laboratory intensive for SVOCs, the GC/MS will be used primarily to analyze the VOCs using standard methods (for example, EPA Method 502.2) or methods developed in our laboratory.

Purge-and-trap gas chromatography, using EPA Method 502.2, has very good sensitivity for water analysis and a reasonably high degree of selectivity for known or expected components. However, it is not well suited for identification of unknowns and may be somewhat prone to occasional misidentification. The analysis time is very long on a per sample basis (approximately 75 minutes per sample). Because effluent samples may potentially contain a wide range of unknown or unexpected water soluble

Figure 5.1. FY 1994 Schedule

soluble organic compounds, this method is used primarily in this project for quality control of samples sent to the analytical services laboratory.

To augment EPA Method 502.2, procedure development has concentrated on testing a headspace method for analysis of VOCs in waste water samples.

The method chosen has adequate sensitivity, excellent identification capability, and high sample throughput (20 minutes per sample). The detection levels attainable by this method are quite respectable, typically around 1 $\mu\text{g/L}$ for most VOC species of interest. Thus, the headspace method can be used with confidence down to and somewhat below drinking water standards for all major VOC species of interest, except vinyl chloride. Quantitative comparisons between the headspace method and purge-and-trap gas chromatography, where possible, have been uniformly favorable as expected because the same calibration standard is used on both instruments. The procedure has been successfully implemented both in the laboratory and in the field (300 Area) on samples from several PNL facilities.

Deployment for Air Sampling and Analysis. The GC/MS can collect and analyze air samples. To do this, a sorbent trap method will be required to obtain adequate sensitivity. After extensive evaluation, commercially available carbon-based sorbent traps were chosen. Preliminary experiments show the traps have a detection sensitivity in the range of less than part per billion by volume with 1-L samples. Procedures for trap preparation and use have been obtained from Dr. Roger Jenkins and co-workers at Oak Ridge National Laboratory. Materials and supplies for the traps are currently on order from the commercial supplier. Further testing based on the Oak Ridge National Laboratory recommendations will begin when the equipment arrives. Decisions on the best way to deploy samplers and analytical instrumentation will require a more extensive evaluation of site conditions and access to sampling points.

Results. To date, two major VOC liquid sampling and analysis campaigns have been carried out in the 300 Area. These two events are described in the following text.

The first sampling exercise was performed at Building 3720 during the week of June 13, 1994. The PNL mobile laboratory containing the GC/MS was moved to the back parking lot of Building 3720. Water samples for VOC analysis were collected from the building sewer pipe outfall through a 4.6-m (15-ft) length of 0.6-cm (1/4-in.) stainless steel line connected to a positive displacement pump at street level. The VOC vials were filled at periodic intervals through the pump.

The principal VOC found routinely in the samples was chloroform, presumably associated with the chlorination of source water. Chloroform levels in Building 3720 were somewhat higher than in past measurements and were moderately variable, ranging from 37 to 95 $\mu\text{g/L}$. No measurable levels of the brominated trihalomethanes were observed. Very minor amounts of methylene chloride ranging from 1 to 9 $\mu\text{g/L}$ were also observed in some samples.

After the baseline measurements, a tracer release was performed using a test point at the far corner of the building. Extremely small quantities of methylene chloride, a mixture of xylenes, and tetrabromoethene were released in a manner designed to simulate a glassware cleanup. Based on conservative tracers such as pH, the transit time to the sampling point was estimated to be 5 to 10 minutes. A significantly longer transit time was observed for all species. Xylene arrived first after about a 30-minute

delay, then methylene chloride about 30 to 45 minutes later. The tetrabromoethene was not observed as a sharp peak; it was observed as a broad peak of tribromoethene (an apparent degradation product or impurity) after significant delay. These results show that the building plumbing does have significant storage capacity for a slow release of even moderately water-soluble organic compounds (see Figure 5.2).

A second sampling activity was performed during the week of July 18, 1994, focusing on sampling points at Building 320, Building 331, and near the end-of-pipe. Samples were collected in duplicate at approximately 1-hour intervals from each sampling point over 3 days. Composite samples collected over 24 hours were taken on one occasion from Building 331 for comparison with results from the commercial laboratory. Results of this work are still under analysis and will be included in a forthcoming detailed report summarizing FY 1994 characterization and monitoring results.

Adsorptive Stripping

Voltametry. Adsorptive stripping voltametry (ASV) is an analytical technique that pre-concentrates selected trace metals (for example, uranium, chromium, and iron) from a ligand-treated (for example, cupferron) sample solution. The ligand forms an electrolytically stable complex that is adsorbed into the mercury during the deposition process (also called pre-concentration). After pre-concentration, the analyte is measured using advanced electrochemical methods. The pre-concentration process allows detection limits at the parts-per-billion level for most metals. Because of the potential attributes of ASV for effluent monitoring

of uranium, a flow system was laboratory and field tested to measure the concentration of uranium in 300 Area process sewer water.

The uranium monitoring flow system (see Figure 5.3) was evaluated through a sub-contract with New Mexico State University to detect uranium in process sewer water in near-real-time (4- to 5-minute intervals) with a detection limit of 5 $\mu\text{g/L}$ uranium(VI) in solution. The system included a two-channel, variable speed peristaltic pump. The first channel supplied a sample flow, and the second channel supplied the cupferron/acetate buffer solution flow to a plastic "Y" where the two flows merge. The uranium and cupferron were allowed to react in a glass reaction spiral (reaction residence time of approximately 1 minute) before analysis. The reaction solution was then focused upwards onto a mercury drop suspended from the base of a dropping mercury electrode. The lower portion of the dropping mercury electrode and the glass tube used to focus the flow stream onto the mercury drop were contained in a glass reservoir below the liquid surface.

The static liquid level in the reservoir was maintained by a drain spout near the top of the reservoir.

Laboratory and Field Testing. Flow injection analysis was initially evaluated as a sample introduction method. However, this approach failed to provide consistent results. Subsequent work focused exclusively on a continuous flow system.

Figure 5.4 shows the system response versus uranium concentration. The uranium monitoring flow system was found to be linear from 0 to 200 $\mu\text{g/L}$

Figure 5.2. Building 3720 Volatile Organic Compound Tracer Tests

uranium. From 0 to 40 µg/L, there appears to be a nonlinear effect, possibly from kinetic effects of the uranium-cupferron interaction.

Figure 5.5 demonstrates the reproducibility of the uranium flow monitoring system at 50 µg/L uranium using a single mercury drop. The relative standard deviation of 15 measurements using a single mercury drop was 4.7%. Subsequent work suggested a more realistic relative standard deviation of approximately 10%.

After initial testing with distilled water, the system was used testing 300 Area process sewer water. The process sewer

water was found to contain a potential interferant to the uranium measurement, believed to be a surfactant (detergent) (Figure 5.6). This caused a significant error with the integration on the uranium peak. To minimize the effect of the interferant on the uranium peak, the mercury drop was manually replaced after every three or four analyses. This procedure was used for subsequent laboratory analyses using process sewer water and during field testing.

Field testing for near-real-time monitoring of uranium in process sewer water was conducted June 14 through 16, 1994, by PNL and New Mexico State University personnel at Building 3720 process sewer

Figure 5.3. Uranium Monitoring Flow System

line concurrently with the GC/MS test. The purpose of the field test was to prove that the fundamental ideas behind the uranium monitoring flow system would work (Figure 5.3). During testing, diverted flow from the sewer to the uranium monitoring flow system was provided by a variable flow pump. The flow rate ranged from 5 to 20 mL/min. The uranium monitoring flow system was "Teed" into the sampling line after the sampling pump and sampled at a flow rate of 1 to 2 mL/min. Excess flow was discarded into a drain. During the course of testing, five integrated samples--spanning 5 to 55 minutes of monitoring--were collected for uranium analysis by an inductively coupled argon plasma-mass spectrometer (ICAP-MS).

These results were compared to the ASV field measurements.

Figure 5.4. Cupferron Flow System

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Figure 5.5. Uranium-Cupferron Flow System

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Figure 5.6. Uranium (VI) and Surfactant

Results. The ICAP-MS analysis of integrated grab samples measured the background concentration at approximately $30 \mu\text{g/L}$. Values measured by ASV during online monitoring ranged from 10 to $20 \mu\text{g/L}$ during the same period. The results of the field testing suggest the ASV uranium monitoring flow system can measure uranium at a detection limit of $5 \mu\text{g/L}$. The system response to uranium was found to depend on the pH of the process sewer water sample. Increasing the acetate buffer solution from 0.1 to 1 M is expected to significantly decrease or eliminate the affects of pH on the uranium measurement. Near real-time tests also showed optimal replacement of the mercury drop after every second or third measurement. These refinements should increase the accuracy and precision of the ASV measurement. Another refinement will include configuring the system to

routinely calibrate and zero check itself for adaptation to long-term unattended monitoring of process effluents.

Solid Phase Extraction. Solid phase extraction (SPE), which is strongly supported by the EPA's Office of Solid Waste, will eliminate some of the sample preparation activities that are performed in the field and/or the laboratory. Operation in the field will enhance sample stability during transport from the field to the laboratory. In turn, sample quality and, thus, data quality will be increased. Characterization cost effectiveness will also be enhanced. As seen at the Pittsburgh Conference, SPE technology is readily available from manufacturers for application to a broad suite of semivolatile analytes and liquid matrices. Favorable results down to very low levels (nanogram/liter) have been reported for these filter disks in the open literature (Tompkins et al. 1992).

Methods Development. Because the methods typically used to analyze SVOCs are slow, impractical, and proliferate waste, a solid disk extraction method was evaluated for separation and concentration of SVOCs in water. SPE disks were evaluated and had recoveries in the range of 50 to 90%. Recovered extracts were subjected to

one of two analysis procedures; gas chromatography with flame ionization detection was used for routine samples, and GC/MS was used on any samples showing unknown peaks. This analytical method selection process should provide the most cost-effective approach while assuring that no peaks are missed. Because this approach is now available for routine use, a more aggressive sampling program is anticipated in the near future.

Field Application. This analytical selection process has been used successfully in the FEMP. An end-of-pipe sample that showed a medium-volatility unknown peak was reanalyzed by GC/MS. The peak was identified as 2-(2-butoxyethoxy)ethanol acetate by comparison with a National Institute of Standards and Technology library mass spectrum with a very high degree of certainty. The concentration was estimated to be 1 to 2 mg/L based on an estimated response factor. As is frequently encountered in the analysis of uncommon compounds, no reference standard is currently available for the material. While the origin of 2-(2-butoxyethoxy)ethanol acetate is still under investigation, preliminary information suggests that it is associated with cleaning materials.

6.0 Conclusions

PNL's FEMP is responsible for monitoring the effluent streams from DOE-owned and PNL-operated facilities in the 300 Area of the Hanford Site. The program is just beginning. Waste streams have been sampled and analyzed at Buildings 331, 320, and 3720.

In selected facility liquid waste effluents, radioactivity and various chemical constituents were detected; however, with one exception, sources contributing to these releases are unknown at this time. Specific findings were as follows:

- Gross alpha (no uranium-235) and gross beta radioactivity at Building 3720 ranged from 7.9 to 44.6 pCi/L and 2.0 to 17.6 pCi/L, respectively.
- Traces of xylenes (1.2 to 12.0 $\mu\text{g/L}$) were found at Building 320 and at end-of-pipe.
- Ethanol, 2-(2-butoxyethoxy)acetate, was detected at Buildings 320 and 331 in the range of 67 to 510 $\mu\text{g/L}$.
- Bis(2-ethylhexyl)phthalate was found at Buildings 331 and 3720 (1.7 to 6.4 $\mu\text{g/L}$ and 1.2 to 7.6 $\mu\text{g/L}$, respectively) and at end-of-pipe (16 to 38 $\mu\text{g/L}$).
- Acetone was found within the range of 22 to 1,800 $\mu\text{g/L}$ at Buildings 320, 331, and 3720.
- Pesticides were found in the low parts-per-trillion level at Building 331.
- Nitrate levels ranged from 100 to 2,500 $\mu\text{g/L}$ at Buildings 320, 331, and 3720 and at end-of-pipe.
- Cyanide was frequently observed at Building 331 (1 to 70 $\mu\text{g/L}$) and occasionally at Building 3720 (2 to 10 $\mu\text{g/L}$).
- None of the chemicals listed above were detected in raw or treated Columbia River water.

Based on a very limited data set, the concentrations of contaminants have not changed significantly when compared to previously published historical data. Many constituents have decreased in concentration.

Except for bis(2-ethylhexyl)phthalate, concentrations of chemicals detected and parameters measured at end-of-pipe between March 22 and June 21, 1994, were below the EPA existing and proposed drinking water standards. In many cases, the measured values were 10 to 10,000 times lower than regulatory guidelines. Bis(2-ethylhexyl)phthalate, a chemical commonly found in plastic formulations and ubiquitous in the environment, was detected in end-of-pipe waste water at levels (16 to 38 $\mu\text{g/L}$) that were approximately 3 to 6 times the EPA drinking water standard (6 $\mu\text{g/L}$). The source of this chemical is probably a result of the Tygon sampling line.

Occasional elevated levels of sodium, chloride, phosphate, and pH were observed in facility waste streams or at end-of-pipe. Concentrations and clearance times for contaminants at end-of-pipe were highly dependent on waste stream flow rates, dispersion, and the mechanical action of sumps. When present, the action of sumps had the greatest impact on contaminant clearance times. In the absence of sump

activity, dispersion and flow rate were the controlling factors. Dye tests performed at Buildings 331 and 3720 also showed the following results:

- Average transit times for Buildings 3720 and 331 were 49 minutes, and 4 hours, 21 minutes, respectively.
- The mechanical action of a sump intercepting the Building 331 waste stream resulted in the splitting of the dye into discrete fractions that led to subsequent dye discharges at end-of-pipe. The total number of dye pulses at end-of-pipe depended on the initial dye concentration.
- Clearance times (at the building and at end-of-pipe) for Building 3720 were less than 15 minutes in all cases. The generation of three discrete dye fractions in the Building 331 waste stream by an intercepting sump led to an average end-of-pipe clearance time of 4 hours, 23 minutes. This is 13 times greater than the estimated change from dispersion alone.

- Average dilution factors (concentration measured at a building divided by the estimated end-of-pipe concentration) for the dye as it passed from Buildings 3720 and 331 were 30 and 525, respectively.

Future plans call for the use of facility monitoring and waste stream dynamics data to do the following:

- Track sources in the liquid waste stream.
- Determine what must be done in a facility to avoid undesirable releases at end-of-pipe.
- Help TEDF operators determine how to minimize treatment and disposed impacts if an excursion occurs in the liquid waste stream.

Initial field evaluations of transportable GC/MS, ASV, and SPE were favorable.

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