NORTHERN LAKE SERVICE, INC.
QUALITY ASSURANCE PROJECT PLAN

WISCONSIN DNR
SAMPLE COLLECTION AND ANALYSIS PROGRAM
ONALASKA TOWN LANDFILL SUPERFUND SITE

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LABORATORY QUALITY ASSURANCE PROJECT PLAN

for

State of Wisconsin
Department of Natural Resources
Sample Collection and Analysis Program
Bullet Shoot Section - Black River
Onalaska Town Landfill Superfund Site
LaCrosse County, Wisconsin
October 12, 1995

NLS - QAPP - 95/2 (Revision 0)

WDNR Project Manager	Date
WDNR Project QA Officer	Date
Steven R. Cupp	10/12/95
Northern Lake Service Project Manager	Date
W J Mroczka Jr.	10/12/95
Northern Lake Service QA Officer	Date

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3 PROJECT DESCRIPTION

This quality assurance project plan (QAPP) outlines specific quality assurance (QA) and quality control (QC) procedures to be followed by Northern Lake Service, Inc. for sample collection and chemical analyses related to the State of Wisconsin, Department of Natural Resources, Sample Collection and Analysis Program for the Bullet Shoot Section of the Black River near the Onalaska Town Landfill Superfund Site in LaCrosse County, Wisconsin. The plan calls for the sampling and analyses of Sediment and Surface Water samples and reporting the results in a spreadsheet format.

3.1 Purpose

The purpose of this QAPP is to provide a detailed description of all elements involved in the sampling and generation of data of acceptable quality and completeness for the sampling and analysis of the parameters referenced in this document. Data Quality Objectives for this project are specified in the sampling plans and objectives contained in Part I of this document. Guidelines for this plan have been obtained from USEPA Content Requirements for QAPPs, January 1993, USEPA Region V Document #R5-QAS-93-001.

3.2 Scope

The scope of this QAPP is to outline QC requirements for all data generated during the project based on quality judgments using the following three types of information.

* Overall qualifications of the data which includes internal and external performance and systems audits to ensure that there are adequate facilities and equipment, qualified personnel, documented laboratory procedures, accurate data reduction, proper validation, and complete reporting.

* Data that measure the daily performance of the laboratory according to the specific method performed. This includes data from calibration procedures and instrument performance.

* Data that evaluate the overall quality of the package that is used to determine precision, accuracy, representativeness, completeness, and comparability which is in compliance with the data quality objectives listed. Such data includes laboratory method blanks, and duplicate control samples.

3.3 Analytical Procedures

Analyses of the sediment and surface water samples will be performed in accordance with approved EPA, Methods of Soil Analysis and Standard Methods methodologies. Refer to Tables 1 through 7 for a listing of the specific parameters / compounds and analysis methods.

TABLE 1
SEDIMENT AND SURFACE WATER SAMPLING AND ANALYSIS SUMMARY
BULLET SHOOT SECTION - BLACK RIVER, ONALASKA TOWN LANDFILL SITE
GENERAL WET CHEMISTRY - PARAMETERS AND METHODS

PARAMETER	SURFACE WATER METHODS	SEDIMENT METHODS
pH	EPA 150.1	EPA 9045C
BOD, 5-Day	SM 5210B	Not Applicable
Temperature (° F)	EPA 170.1	EPA 170.1
Ammonia	EPA 350.1	MSA 33
Iron, Total	EPA 200.7	Not Applicable
Chloride	EPA 325.2	Not Applicable

TABLE 2
SEDIMENT AND SURFACE WATER SAMPLING AND ANALYSIS SUMMARY
BULLET SHOOT SECTION - BLACK RIVER, ONALASKA TOWN LANDFILL SITE
ACID EXTRACT COMPOUNDS - PARAMETERS AND METHODS

COMPOUND	SURFACE WATER METHODS	SEDIMENT METHODS
Parachlorometa cresol	EPA 604	EPA 8270
2-Chlorophenol	EPA 604	EPA 8270
2,4-Dichlorophenol	EPA 604	EPA 8270
2,4-Dimethylphenol	EPA 604	EPA 8270
4,6-Dinitro-o-cresol	EPA 604	EPA 8270
2,4-Dinitrophenol	EPA 604	EPA 8270
2-Nitrophenol	EPA 604	EPA 8270
4-Nitrophenol	EPA 604	EPA 8270
Pentachlorophenol	EPA 604	EPA 8270
Phenol	EPA 604	EPA 8270
2,4,6-Trichlorophenol	EPA 604	EPA 8270

TABLE 3
SEDIMENT AND SURFACE WATER SAMPLING AND ANALYSIS SUMMARY
BULLET SHOOT SECTION - BLACK RIVER, ONALASKA TOWN LANDFILL SITE
VOLATILE COMPOUNDS - PARAMETERS AND METHODS

COMPOUND	SURFACE WATER METHODS	SEDIMENT METHODS
Acrolein	EPA 624	EPA 8260
Acrylonitrile	EPA 624	EPA 8260
Benzene	EPA 624	EPA 8260
Bromoform	EPA 624	EPA 8260
Carbon tetrachloride	EPA 624	EPA 8260
Chlorobenzene	EPA 624	EPA 8260
Chlorodibromomethane	EPA 624	EPA 8260
Chloroethane	EPA 624	EPA 8260
Chloroform	EPA 624	EPA 8260
Dichlorobomomethane	EPA 624	EPA 8260
1,1-Dichloroethane	EPA 624	EPA 8260
1,2-Dichloroethane	EPA 624	EPA 8260
1,1-Dichloroethylene	EPA 624	EPA 8260
Trans-1,2-Dichloroethylene	EPA 624	EPA 8260
1,2-Dichloropropane	EPA 624	EPA 8260
Cis-1,3-Dichloropropylene	EPA 624	EPA 8260
Trans-1,3-Dichloropropylene	EPA 624	EPA 8260
Ethylbenzene	EPA 624	EPA 8260
Methyl bromide	EPA 624	EPA 8260
Methyl chloride	EPA 624	EPA 8260
Methylene chloride	EPA 624	EPA 8260
1,1,2,2-Tetrachloroethane	EPA 624	EPA 8260
Tetrachloroethylene	EPA 624	EPA 8260
Toluene	EPA 624	EPA 8260
1,1,1-Trichloroethane	EPA 624	EPA 8260
1,1,2-Trichloroethane	EPA 624	EPA 8260
Trichloroethylene	EPA 624	EPA 8260
Vinyl chloride	EPA 624	EPA 8260
Xylene	EPA 624	EPA 8260

TABLE 4
SEDIMENT AND SURFACE WATER SAMPLING AND ANALYSIS SUMMARY
BULLET SHOOT SECTION - BLACK RIVER, ONALASKA TOWN LANDFILL SITE
BASE / NEUTRAL COMPOUNDS - PARAMETERS AND METHODS

COMPOUND	SURFACE WATER METHODS	SEDIMENT METHODS
Acenaphthene	EPA 625	EPA 8270
Acenaphthylene	EPA 625	EPA 8270
Anthracene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Benzidine	EPA 625	EPA 8270
Benzo (a) anthracene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Benzo (a) pyrene	EPA 610 (HPLC)	EPA 8310 (HPLC)
3,4-Benzofluoranthene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Benzo (ghi) perylene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Benzo (k) fluoranthene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Bis (2-chlorethoxy) methane	EPA 625	EPA 8270
Bis (2-chloroethyl) ether	EPA 625	EPA 8270
Bis (2-chloroisopropyl) ethene	EPA 625	EPA 8270
Bis (2-ethylhexyl) phthalate	EPA 625	EPA 8270
4-Bromophenyl phenyl ether	EPA 625	EPA 8270
Butyl benzyl phthalate	EPA 625	EPA 8270
2-Chloronapthalene	EPA 625	EPA 8270
4-Chlorophenyl phenyl ether	EPA 625	EPA 8270
Chrysene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Dibenzo (a,h) anthracene	EPA 610 (HPLC)	EPA 8310 (HPLC)
1,2-Dichlorobenzene	EPA 625	EPA 8270
1,3-Dichlorobenzene	EPA 625	EPA 8270
1,4-Dichlorobenzene	EPA 625	EPA 8270
3,3'-Dichlorobenzidine	EPA 625	EPA 8270
(CONTINUE ON NEXT PAGE)		

TABLE 4 (Continued)
SEDIMENT AND SURFACE WATER SAMPLING AND ANALYSIS SUMMARY
BULLET SHOOT SECTION - BLACK RIVER, ONALASKA TOWN LANDFILL SITE
BASE / NEUTRAL COMPOUNDS - PARAMETERS AND METHODS

COMPOUND	SURFACE WATER METHODS	SEDIMENT METHODS
Diethyl phthalate	EPA 625	EPA 8270
Dimethyl phthalate	EPA 625	EPA 8270
Di-n-butyl phthalate	EPA 625	EPA 8270
2,4-Dinitrotoluene	EPA 625	EPA 8270
2,6-Dinitrotoluene	EPA 625	EPA 8270
Di-n-octyl phthalate	EPA 625	EPA 8270
1,2-Diphenylhydrazine	EPA 625	EPA 8270
Fluoranthene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Fluorene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Hexachlorobenzene	EPA 612	EPA 8270
Hexachlorobutadiene	EPA 612	EPA 8270
Hexachlorocyclopentadiene	EPA 612	EPA 8270
Hexachloroethane	EPA 625	EPA 8270
Indeno (1,2,3-cd) pyrene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Isophorone	EPA 625	EPA 8270
Naphthalene	EPA 625	EPA 8270
Nitrobenzene	EPA 625	EPA 8270
N-nitrosodimethylamine	EPA 625	EPA 8270
N-nitrosodi-n-propylamine	EPA 625	EPA 8270
N-nitrosodiphenylamine	EPA 625	EPA 8270
Phenanthrene	EPA 610 (HPLC)	EPA 8310 (HPLC)
Pyrene	EPA 610 (HPLC)	EPA 8310 (HPLC)
1,2,4-Trichlorobenzene	EPA 625	EPA 8270

TABLE 5
SEDIMENT AND SURFACE WATER SAMPLING AND ANALYSIS SUMMARY
BULLET SHOOT SECTION - BLACK RIVER, ONALASKA TOWN LANDFILL SITE
PESTICIDE COMPOUNDS - PARAMETERS AND METHODS

COMPOUND	SURFACE WATER METHODS	SEDIMENT METHODS
Aldrin	EPA 608	EPA 8081
BHC-hexachlorocyclohexane	EPA 608	EPA 8081
alpha-BHC	EPA 608	EPA 8081
beta-BHC	EPA 608	EPA 8081
delta-BHC	EPA 608	EPA 8081
gamma-BHC	EPA 608	EPA 8081
Chlordane	EPA 608	EPA 8081
4,4'-DDT	EPA 608	EPA 8081
4,4'-DDE	EPA 608	EPA 8081
4,4'-DDD	EPA 608	EPA 8081
Dieldrin	EPA 608	EPA 8081
alpha - Endosulfan	EPA 608	EPA 8081
beta - Endosulfan	EPA 608	EPA 8081
Endosulfan sulfate	EPA 608	EPA 8081
Endrin	EPA 608	EPA 8081
Endrin aldehyde	EPA 608	EPA 8081
Heptachlor	EPA 608	EPA 8081
Heptachlor epoxide	EPA 608	EPA 8081
PCB - 1016	EPA 608	EPA 8081
PCB - 1221	EPA 608	EPA 8081
PCB - 1232	EPA 608	EPA 8081
PCB - 1242	EPA 608	EPA 8081
PCB - 1248	EPA 608	EPA 8081
PCB - 1254	EPA 608	EPA 8081
PCB - 1260	EPA 608	EPA 8081
Toxaphene	EPA 608	EPA 8081

TABLE 6
SEDIMENT AND SURFACE WATER SAMPLING AND ANALYSIS SUMMARY
BULLET SHOOT SECTION - BLACK RIVER, ONALASKA TOWN LANDFILL SITE
METALS, HARDNESS & OTHER ANALYTES - PARAMETERS AND METHODS

PARAMETER / ANALYTE	SURFACE WATER METHODS *	SEDIMENT METHODS *
Aluminum (total)	EPA 200.7 (MDL = 41 μg/L)	EPA 6010 (MDL = 4.1 mg/Kg)
Antimony (total)	EPA 204.2	EPA 7041
Arsenic (total)	EPA 206.2	EPA 7060
Beryllium (total)	EPA 200.7 (MDL=0.28 μg/L)	EPA 6010 (MDL=0.03 mg/Kg)
Cadmium (total)	EPA 213.2	EPA 7131
Chromium (total)	EPA 200.7 (MDL=5.8 μg/L)	EPA 6010 (MDL=0.58 mg/Kg)
Chromium (Hexavalent)	EPA 218.4	Not Applicable
Copper (total)	EPA 200.7 (MDL=3.8 μg/L)	EPA 6010 (MDL=0.32 mg/Kg)
Cyanide (amenable)	EPA 335.1	Not Applicable
Lead (total)	EPA 239.2	EPA 7421
Mercury (total)	EPA 245.1	EPA 7471
Nickel (total)	EPA 200.7	EPA 6010 (MDL= 0.59 mg/Kg)
Selenium (total)	EPA 270.2	EPA 7740
Silver (total)	EPA 272.2	EPA 7761
Thallium (total)	EPA 279.2	EPA 7841
Zinc (total)	EPA 200.7 (MDL=12 μg/L)	EPA 6010 (MDL=1.2 mg/Kg)
Hardness	EPA 130.1	Not Applicable

* Method Detection Limits (MDLs) are dependent upon sample size, percent solids concentrations, dilution requirements, and interfering element correction factors.

TABLE 7
SEDIMENT AND SURFACE WATER SAMPLING AND ANALYSIS SUMMARY
BULLET SHOOT SECTION - BLACK RIVER, ONALASKA TOWN LANDFILL SITE
NON-PRIORITY POLLUTANTS & DIOXIN - PARAMETERS AND METHODS

COMPOUND / ANALYTE	SURFACE WATER METHODS	SEDIMENT METHODS
Cis-1,2-Dichloroethylene	EPA 624	EPA 8260
1,1-Dichloropropylene	EPA 624	EPA 8260
2,3-Dichloropropylene	EPA 624	EPA 8260
Pentachlorobenzene	EPA 625	EPA 8270
1,2,4,5-Tetrachlorobenzene	EPA 625	EPA 8270
N-Nitrosodiethylamine	EPA 625	EPA 8270
N-Nitrosodi-N-Butylamine	EPA 625	EPA 8270
N-Nitrosopyrrolidine	EPA 625	EPA 8270
2,5-Dinitrophenol	EPA 604	EPA 8270
2,4,5-Trichlorophenol	EPA 604	EPA 8270
BHC- Technical Grade	EPA 608	EPA 8081
Parathion (ethyl plus methyl)	EPA 608	EPA 8081
2,3,7,8-Tetrachloro-dibenzo-p-dioxin	EPA 1613	EPA 8290

4 Project Organization and Responsibilities

In order to ensure that all QA/QC procedures are strictly adhered to, specific responsibilities must be assigned to each individual involved in the project.

Steven R. Crupi is the designated Northern Lake Service Project Manager (PM). The responsibility for day to day management of the project rests with the PM and management team. These responsibilities include, but are not limited to, coordinating bottle shipments to the field, monitoring the project within the laboratory, ensuring proper login of the samples, communicating progress and/or anomalies encountered in the laboratory to the client, and approving the final report issued to the client.

The QA officer, Mr. W. Joe Nosek, Jr., will oversee and be responsible for all QA/QC activities including audits, preparation of QA specifications, and corrective action. Mr. Nosek, reports directly to Mr. Ron K. Krueger, Northern Lake Service's President and CEO.

Laboratory supervisors are responsible for producing fully documented data of acceptable quality from their respective departments. Figure 4.1 illustrates the Northern Lake Service organizational structure.

NORTHERN LAKE SERVICE, INC. - ORGANIZATIONAL CHART

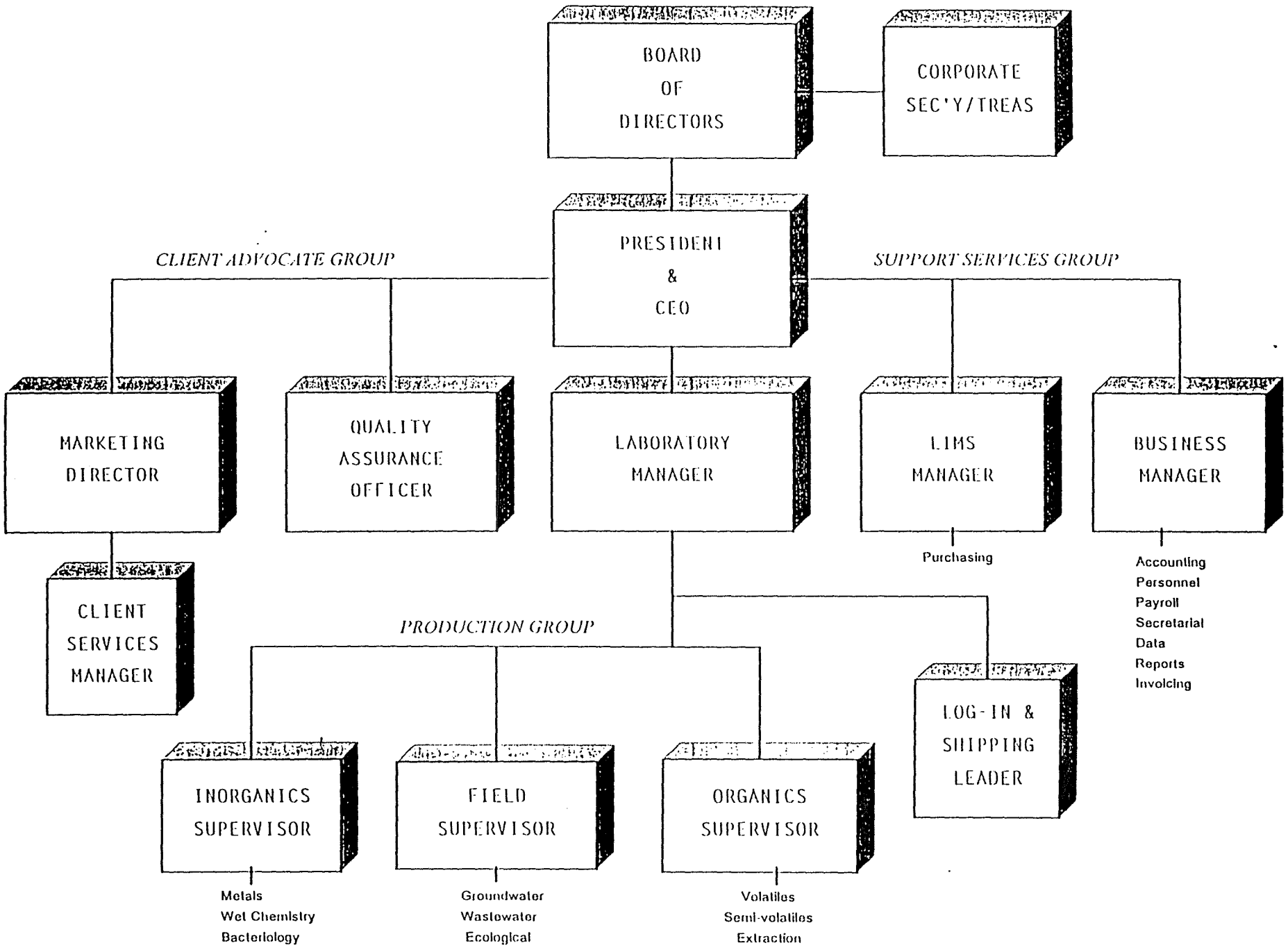


Figure 4.1

5 Quality Assurance Objectives

5.1 Quality Assurance Objectives

Quality assurance objectives can be expressed in terms of precision, accuracy, representativeness, comparability, and completeness.

Adherence to the data quality objectives will be quantitatively measured by comparing the results of the matrix spike (MS) and matrix spike duplicate (MSD) analyses to control limits. MS consist of a sample which is spiked with target compounds representative of the method analytes. MS/MSD is analyzed for every 20 samples within an analytical batch. The MS/MSD sample will be collected along with the other samples from the Onalaska Town Landfill project site.

The MS/MSD is used to monitor both the precision and accuracy of the analytical method. MS/MSD are monitored for accuracy (average percent recovery) of each spiked analyte and precision (relative percent difference - RPD) between each analyte duplicate. Section 11 defines Calculations of Data Quality Indicators.

5.2 Representativeness

Representativeness is a qualitative element related to the ability to collect a sample that reflects the characteristics of that part of the environment being assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project site.

5.3 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions. It is expected that laboratories should provide data, meeting QC acceptance criteria, for 95% or more of the requested determinations. It is necessary for data users to identify any sample types which requires 100% completeness.

5.4 Comparability

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. For example, the use of EPA-approved methods and procedures ensure comparability with other data from previous or following studies using the same methods.

5.5 Control Limits

Control limits for accuracy and precision are generated for each analyte per method per matrix for all analyses. These limits are based upon Northern Lake Service historical sample data. Control limits represent the 99% confidence interval and are equal to the mean values of the control samples, plus or minus three standard deviations of the values. All results of quality control samples must fall within these limits or corrective action must be addressed.

5.6 Quality Control Samples and Quality Assurance Objectives

Precision is the degree to which the measurement is reproducible. Precision can be assessed by duplicate measurements of a laboratory control sample (LCS) or environmental samples. Precision is routinely monitored by comparing the relative percent difference (RPD) between duplicate measurements with control limits established at plus three standard deviations from the mean RPD of historical duplicate data. If the calculated RPD is outside of the control limits, the samples associated with that analytical batch are subject to corrective action measures.

Accuracy measures the degree of difference between observed and true values. The actual test result is compared to the theoretical result of 100% recovery and the percent recovery calculated. The accuracy of sample data can be assessed using the LCS, environmental samples spiked with target analytes or surrogate spiking. Accuracy data are evaluated against control limits established as plus or minus three standard deviations of the mean recovery of historical accuracy data.

Occasionally, it is apparent that although a MS/MSD is out of control, the samples associated with this MS/MSD are unaffected. If all other QC criteria are acceptable, the data are flagged. The laboratory may report the data with a narrative supporting the decision.

5.7 Matrix Specific Quality Control

For organics analyses, the percent recovery and relative percent difference (RPD) of the matrix spike (MS) and matrix spike duplicate (MSD) pair will be calculated. This allows for demonstration of the effect of the matrix on the method performance. Reextraction and reanalysis decisions are made based on the MS/MSD, Method Blanks, and QC requirements of the methods.

Matrix spikes on project specific samples are analyzed when requested on the chain of custody. The performance of matrix spike analyses for aqueous matrices requires additional sample volume which must be collected and submitted at the same time as the original routine sample.

Typical frequencies include one pair of matrix spikes for each sample type per batch of twenty or fewer samples extracted on one day.

5.8 Surrogates

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not an analyte of interest. Surrogates are added to samples to monitor the effect of the matrix on the accuracy of analysis. Results are reported in terms of percent recovery. Acceptable control limits for surrogate recovery are specified in the methods or laboratory limits generated from historical data.

5.9 Method Blanks

Method blanks, also known as reagent, analytical or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

As a part of the standard Northern Lake Service QC program a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, cleanup, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. The concentration of target analytes in the blank should be below the reporting limit for each analyte.

If the blank does not meet acceptance criteria, the source of contamination must be investigated and appropriate corrective action must be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on the sample results. Corrective actions may include reanalysis of the blank, and/or repreparation and reanalysis of the blank and all associated samples.

6 Sampling, Sample Preservation, Receipt, Custody and Documentation

6.1 Sampling

Northern Lake Service will provide trained sampling personnel to obtain representative samples of the sediment and surface water at the midpoint of the river section approximately 100 feet up river and 100 feet down river from the point where the groundwater treatment system discharges into the Bullet Shoot Section of the Black River. Sediment samples will be collected from the river bottom using an hand operated Ekman sampling dredge. Surface water samples will be collected in the water column at a point which is greater than one foot above the river bottom and which is below the surface of the river.

6.2 Sample Preservation

Northern Lake Service will supply all the appropriate containers and preservatives along with coolers and chain of custody records for this project. The primary purpose of the chain of custody procedure is to document the possession of the samples from collection through storage and analysis to reporting. Upon completion of sampling, samples will be appropriately labeled on the Field Sampling Record form (Figure 6.0) and Chain-Of-Custody form (Figure 6.1) completed by the appropriate field personnel. Appropriate sample containers and recommended holding times are listed in Table 8. The sample containers will be packed in shipping coolers, packed in ice and shipped to the laboratory.

6.3 Sample Receipt and Custody

Upon receipt by Northern Lake Service, samples will be processed through an orderly sequence specifically designed to ensure continuous integrity of both the sample and its documentation. The contents of coolers are removed, inspected, and coordinated with the chain of custody records. All samples are carefully checked for label identification and proper preservation, sufficient sample volume, and accurate chain of custody records. Each sample is then assigned a unique laboratory identification number assigned by the Laboratory Information Management System (LIMS) that stores all identifications and essential information.

Any discrepancies involving sample integrity, sample volume, sample breakage, cooler temperature, holding time expiration, appropriate container use, preservatives, and missing or incorrect documentation are immediately noted. These discrepancies are noted on the NLS "Noncompliance for Environmental Samples" report form shown in (Figure 6.2). The project manager will be notified and resolve any discrepancies. These anomalies will be noted on the NLS Sample Track form shown in (Figure 6.3).

When all of the log-in procedures have been completed, the sample custodian stores the samples in the appropriate refrigerator. Access to all areas of Northern Lake Service is monitored to prevent any unauthorized contacts with samples, extracts, or documentation. All samples will be retained for 30 days from the issuance date of the analytical report.

6.4 Documentation

Complete and accurate documentation of analytical and procedural information is an important part of the project. These activities will be documented with the use of Standard Operating Procedures (SOPs), a laboratory data management system, laboratory benchsheets, laboratory notebooks and orderly project files.

Details of analytical and QC protocols are contained in laboratory-specific SOPs. SOPs are documents which contain detailed information on the equipment, procedures and control measures for the correct performance of a laboratory procedure or analysis. All SOPs are approved by the laboratory QA Department before being implemented.

Laboratory benchsheets are used to document information from routine laboratory operations, including sample preparation and analysis. Benchsheets are used to ensure that the information is recorded in a complete and organized manner and that the analysis can be reconstructed, if necessary.

A project file will be created by the laboratory for the project. The project file will contain documents associated with the project including correspondence from the client, copies of chain of custody records, project events, any anomalies, and a copy of the final report.

Figure 6.0

NORTHERN LAKE SERVICE, INC.
FIELD SAMPLING RECORD

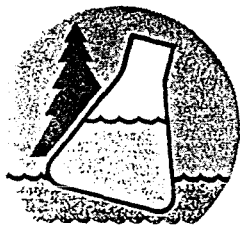
FIELD SAMPLER: _____ *C-O-C Number:* _____

_____

(Project Name / Description)

NLS SAMPLE NUMBER				
SAMPLE IDENTIFICATION				
LOCATION OF SAMPLE				
DESCRIPTION OF SAMPLE				
pH				
CONDUCTIVITY				
TEMPERATURE				
TIME				
DATE				
COMMENTS				

Figure 6.1



NORTHERN LAKE SERVICE, INC.

Analytical Laboratory and Environmental Services

400 North Lake Avenue • Crandon, WI 54520
Tel: (715) 478-2777 • Fax: (715) 478-3060

NO. 14563

SAMPLE COLLECTION AND CHAIN OF CUSTODY RECORD

Wisconsin Lab Cert. No. 721026460

RETURN THIS FORM WITH SAMPLES.

CLIENT		PROJECT TITLE	
ADDRESS		PROJECT NO.	P.O. NO.
CITY	STATE ZIP	CONTACT	PHONE

ITEM NO.	NLS LAB NO.	SAMPLE ID	COLLECTION DATE	COLLECTION TIME	SAMPLE TYPE	GRAB/ COMP.	CONTAINER/PRESERVATIVE					COLLECTION REMARKS
1.												
2.												
3.												
4.												
5.												
6.												
7.												
8.												
9.												
10.												
11.												
12.												

SAMPLE TYPE:
SW = surface water; WW = wastewater; GW = groundwater; DW = drinking water; TIS = tissue; AIR = air; PROD = product; SOIL = soil; SED = sediment; describe others

CONTAINER: P = plastic; G = glass; V = glass vial; B = plastic bag; describe others

PRESERVATIVES & PREPARATION: NP = nothing added; S = sulfuric acid; N = nitric acid; Z = zinc acetate; OH = sodium hydroxide; HA = hydrochloric & ascorbic acid; H = hydrochloric acid; F = field filtered

COLLECTED BY (signatures)	CUSTODY SEAL NO. (IF ANY)	DATE/TIME
RELINQUISHED BY (signature)	RECEIVED BY (signature)	DATE/TIME
RELINQUISHED BY (signature)	RECEIVED BY (signature)	DATE/TIME
DISPATCHED BY (signature)	METHOD OF TRANSPORT	DATE/TIME

RECEIVED AT NLS BY (signature)	DATE/TIME	CONDITION	TEMP.
SEAL INTACT? ☐ YES ☐ NO	SEAL #	REMARKS & OTHER INFORMATION	

IMPORTANT:
1. TO MEET REGULATORY REQUIREMENTS, THIS FORM MUST BE COMPLETED IN DETAIL AND INCLUDED IN THE SHIPPER CONTAINING THE SAMPLES DESCRIBED.
2. PLEASE USE ONE LINE PER SAMPLE, NOT PER BOTTLE.
3. RETURN THIS FORM WITH SAMPLES - CLIENT MAY KEEP PINK COPY.

ORIGINAL COPY

Figure 6.2

NORTHERN LAKE SERVICE, INC.

NONCOMPLIANCE FOR ENVIRONMENTAL SAMPLES
(FOR COMPLIANCE MONITORING SAMPLES)

Client: _____ Date: _____

Address: _____ NLS Project Number: _____

NC REFERENCE NUMBER	DESCRIPTION OF NON COMPLIANCE
1	Sample(s) received at _____ °C which is above EPA protocol of 4 °C.
2	Sample(s) received frozen or partially frozen.
3	Sample(s) not properly preserved per EPA protocol for: (List Analytes): →
4	Sample(s) received in bottles not furnished by NLS. Chemical preservation methods, if used, are unknown.
5	Sample(s) received beyond EPA holding time for: ANALYTE / EPA HOLD TIME / PAST EPA HOLD TIME _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____
6	Sampling Date / Time not supplied by client. The actual holding time is unknown to NLS.
7	Sample(s) received without proper paperwork. (Explain): →
8	Sample(s) not field filtered for dissolved metals (Including hardness). Lab filtered upon sample receipt, if analyzed.
9	VOC vial(s) received with headspace which does not conform to EPA protocol. Explanation, if any: →
10	Insufficient sample size to complete analysis. (List Samples): →

NLS Representative's Signature: _____

Figure 6.3

NLS SAMPLE TRACK

√ Quick Turn	Due Date

CLIENT:	ORDERED BY:	DATE:
	PHONE:	ORDER REC.BY:
	DESCRIPTION/CLIENT PROJ:	DATE TO BE SAMPLED:

DATA TO:	EXTRA COPIES:	BILLING TO:

PRICING:	QUOTE #:
	PO #:
	MOB:
	PURG: TAD/DISK:

C.O.C.#	Date Collected	Date Rec. at NLS	Sample Matrix	NLS Sample #s	NLS Proj.#	Bottles Rec'd

LAB INSTRUCTIONS:	REPORTING INSTRUCTIONS:	SAMPLE CONDIT/COMMENTS:

CLIENT CONTACT AFTER SAMPLE RECEIPT:

WHO	FOR _	INIT	DATE
	Delivery		
	Log-in		
	Log-in Ck.		
	Log-in Ck.		
	Subbed Out		
	Work Review		
	Work Review		
	Data Review-1		
	Data Review-2		
	Special Report		
	TAD/Disk		
	Pkg. Review		
	Billing Ck.		

TABLE 8

Sample Containers, Volumes, Preservation and Holding Times (Surface Waters)

Analyte	Container / Volume	Preservation	Holding Time
Volatile Organic Compounds	3 X 40 mL GlassVials	HCl to pH <2.0	14 days
Acid ExtractCompounds	2 X 1 L iter Glass Amber	Refrigerate at 4° C (On Ice)	7 days
Base / Neutral Compounds	2 X 1 Liter Glass Amber	Refrigerate at 4 ° C (On Ice)	7 days
Pesticide Compounds	2 X 1 Liter Glass Amber	Refrigerate at 4° C (On Ice)	7 days
Non - Priority Pollutants (Phenols)	2 X 1 Liter Glass Amber	Refrigerate at 4° C (On Ice)	7 days
Non - Priority Pollutants (Method 608 compounds)	2 X1 Liter Glass Amber	Refrigerate at 4° C (On Ice)	7 days
Dioxin	2 X 1 Liter Glass Amber	Refrigerate at 4° C (On Ice)	7 days
Ammonia - Nitrogen	1 X 60 mL Plastic	H_2SO_4 to pH <2.0	28 days
pH	1 X 60 mL Plastic	Refrigerate at 4° C (On Ice)	Immediately
Cyanide	1 X 250 mL Plastic	NaOH to pH >12.0	14 days
BOD (5-day)	1 X 500 mL Plastic	Refrigerate at 4° C (On Ice)	48 hours
Chloride	1 X 125 mL Plastic	Refrigerate at 4° C (On Ice)	28 days
Metals	1 X 960 mL Plastic	HNO_3 to pH < 2.0	6 months
Total Hardness	1 X 500 mL Plastic	HNO_3 to pH < 2.0	6 months
Mercury	1 X 960 mL Plastic	HNO_3 to pH < 2.0	28 days
Chromium (Hexavalent)	1 X 500 mL Plastic	Refrigerate at 4° C (On Ice)	24 hours

TABLE 8 (Continued)
Sample Containers, Volumes, Preservation and Holding Times (Sediments)

Analyte	Container / Volume	Preservation	Holding Time
Volatile Organic Compounds	3 X 40 mL GlassVials	Refrigerate at 4° C (On Ice)	14 days
Acid ExtractCompounds	2 X 125 mL Glass Wide - mouth	Refrigerate at 4° C (On Ice)	7 days
Base / Neutral Compounds	2 X 125 mL Glass Wide - mouth	Refrigerate at 4 ° C (On Ice)	7 days
Pesticide Compounds	2 X 60 mL Glass (Soil Jar)	Refrigerate at 4° C (On Ice)	7 days
Non - Priority Pollutants (Phenols)	2 X 125 mL Glass Wide - mouth	Refrigerate at 4° C (On Ice)	7 days
Non - Priority Pollutants (Method 8081 compounds)	2 X 60 mL Glass (Soil Jar)	Refrigerate at 4° C (On Ice)	7 days
Dioxin	2 X 125 mL Glass Wide - mouth	Refrigerate at 4° C (On Ice)	7 days
Ammonia - Nitrogen	1 X 60 mL Plastic	Refrigerate at 4° C (On Ice)	28 days
pH	1 X 60 mL Glass (Soil Jar)	Refrigerate at 4° C (On Ice)	As Soon As Possible
Metals	2 X 125 mL Glass Wide - mouth	Refrigerate at 4° C (On Ice)	6 months
Mercury	2 X 125 mL Glass Wide - mouth	Refrigerate at 4° C (On Ice)	28 days

7 Analytical Procedures and Calibration

7.1 Analytical Procedures

In accordance with the objectives of the QAPP, samples will be analyzed for designated analytes listed in Tables 1 through 7.. These tables include the approved EPA, Methods of Soil Analysis, and Standard Methods methodologies from the following authoratative sources.

- Methods of Chemical Analysis of Water and Wastes, EPA-600/4-79-020
- Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057
- Test Methods For Evaluating Solid Waste, EPA - SW846 third edition
- Methods of Soil Analysis, second edition, Part 2, No. 9, ASA/SSSA - Madison

Quality control samples to be analyzed with each type of analysis are outlined in Section 9.

7.2 Calibration Procedures and Frequency

Northern Lake Service will perform analytical methods found in EPA approved methods (SW-846 EPA 600 series, etc). Methods contained in these manuals cite the specific calibration and check procedures that are required to conduct the analyses and produce quality data.

7.2.1 GC Analysis

GC analyses calibration criteria will consist of a five point calibration curve. Calibration factors are calculated and a determination of the linearity of the curve is calculated. The calibration is checked on an ongoing basis (generally every 10 samples) or daily whichever is more frequent. If the percent difference exceeds that which is required in the method, the system is recalibrated and all samples analyzed after the last acceptable calibration check are reanalyzed.

7.2.2 GC/MS Analysis

GC/MS analyses calibration criteria will consist of a five point calibration curve. Relative responce factors are calculated and the percent Relative Standard Deviation (RSD) is determined for each compound. Each method specifies the allowable % RSD for the relative responce factors. Continuing Calibration Check (CCC) compounds must have a % RSD $< 30\%$, while SPCC compounds must have a minimum responce of 0.05. A mid-level calibration standard is analyzed every 12 hours. If the acceptance criteria are not met recalibration is necessary and all samples analyzed after the last acceptable calibration check standard are reanalyzed.

7.2.3 HPLC Analysis

HPLC analyses calibration criteria will consist of a five point calibration curve. Calibration factors are calculated and a determination of the linearity of the curve is calculated. The calibration is checked on an on-going basis (generally every 10 samples) or daily whichever is more frequent. If the percent difference exceeds that which is required in the method, the system is recalibrated and all samples analyzed after the last acceptable calibration check are reanalyzed.

7.2.4 Metals

For Metals analyses, three types of analytical methodology are performed; inductively coupled argon plasma emission spectroscopy (ICAP-ES), cold vapor atomic absorption (CVAA) and atomic absorption spectroscopy graphite furnace (GFAA).

Each ICAP calibration curve is established daily by analyzing a blank and a standard. The concentration of the standard is dependent on the element and the sample type. Immediately following calibration an initial calibration blank (ICB) and an initial calibration verification (ICV) are analyzed. The ICV must agree within ± 10% of its true value for the analysis to proceed. The calibration is monitored throughout the run by analyzing a continuing calibration verification standard (CCV) and a continuing calibration blank every ten samples and at the end of an analysis run. The CCV must agree within ± 10% of its true value for the data to be deemed acceptable. If this criterion is not met, all samples analyzed after the last acceptable CCV will be reanalyzed.

An inter-element interference check standard (ICS) is analyzed at the beginning and end of each analytical run to verify that the inter-element and background correction factors have remained constant. Results outside of established (±20%) criteria require reanalysis of the last set of samples.

Each GFAA Unit is calibrated utilizing a blank and three standards prior to analyses being conducted. A calibration curve is prepared for a specific analyte at detectable concentrations, followed by an ICV and an ICB.. The calibration is monitored throughout the run by analyzing the CCV and CCB every ten samples and at the end of the analysis sequence. The CCV must agree within ± 10% of its true value for the data to be deemed acceptable. If this criterion is not met, all samples analyzed after the last acceptable CCV will be reanalyzed.

The CVAA unit is calibrated by the analysis of a blank and five standards prior to sample analysis. A calibration curve is calculated and verified by the analysis of an ICV standard. The ICV must agree within ± 10% of its true value before the analysis of samples is allowed to proceed. The ICB is analyzed to monitor for any system contamination. The calibration is monitored throughout the run by the analysis of a CCV and a CCB after every 10 samples and at the end of an analysis sequence. The CCV must agree within ± 10% of its true value for the data to be deemed acceptable. If this criterion is not met, all samples analyzed after the last acceptable CCV will be reanalyzed.

7.2.5 Wet Chemistry Analyses

Wet chemistry methods have varying calibration procedures. Each system is calibrated prior to sample analyses being conducted. A description of one common calibration approach starts with a five-point curve. A correlation coefficient is determined and must be greater than 0.995. The calibration is checked every ten samples and must agree within ± 10%, or the ten samples analyzed prior to the unacceptable calibration check are reanalyzed.

8 Data Reduction, Validation and Reporting

8.1 Data Reduction and Validation

All analytical data generated are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction and three levels of review shown below.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory standard operating procedures (SOPs). Each analyst reviews the quality of his work based on an established set of guidelines. The analyst reviews the data package to ensure that:

* Sample preparation information is correct and complete.
* Analysis information is correct and complete.
* The appropriate SOPs have been followed.
* Analytical results are correct and complete.
* QC samples are within established control limits.
* Special sample preparation and analytical requirements have been met.
* Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented; holding times are documented, etc.)

The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step is designated as the level I review. The analyst then passes the data package to an independent reviewer who performs a level II review.

The level II review is performed by a laboratory supervisor, or peer whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

* Calibration data are scientifically sound, appropriate to the method, and completely documented.
* QC samples are within established guidelines.
* Qualitative identification of sample components are correct.
* Quantitative results are correct.
* Documentation is complete and correct.
* The data is ready for incorporation into the final report.

The level II review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the benchsheet. If no problems are found with the data package, the review is complete.

An important element of the level II review is the documentation of any errors that have been identified and corrected during the review process. Northern Lake Service believes that the data package submitted for a level II review should be free of errors. The level II data review is also documented with the signature of the reviewer and the date. The project is then approved and a final report is prepared. Before the report is released to the client, the project manager or laboratory supervisor(s) review(s) the report to ensure that the data meets the overall objectives of the project. This is the level III review.

8.2 Data Reporting

Northern Lake Service will provide a standard analytical report containing the following items:

* Analytical Data: concentration of target analyte, reporting limits, method performed, date of collection, extraction and analysis.

* Comments Section: addresses sample integrity upon receipt, and data qualifiers, i.e., matrix interferences, unacceptable Quality Control, elevated reporting limits.

* In addition to a written report NLS will provide the analytica report data in a spreadsheet format.

8.3 Project Files

Project files are created for each project handled within the laboratory. These files contain documents associated with the project. This includes correspondence to and from the client, chain of custody records, copies of laboratory notebook entries where appropriate to the project, and a copy of the final report. When a project is complete, all records are filed. Raw data and all pertinent records are retained for a minimum of three years, unless the scope of work requires a longer time period.

9 Internal Quality Control Checks

9.1 Laboratory QC Checks

Northern Lake Service's general QC protocol for analytical analyses include the following items:

* A minimum of one method blank is analyzed per sample batch to detect contamination during preparation and/or analysis.

* Matrix spike and matrix spike duplicates for organic analyses and matrix spike and duplicates for inorganic analyses will be analyzed for every 20 samples to determine the effect of the matrix on the method performed.

* Internal and Surrogate standards will be added where appropriate to determine recoveries and to account for sample-to-sample variation(s).

* Calibration of instrumentation will be determined according to the appropriate methods.

10 Performance and System Audits

10.1 External Audits of Northern Lake Service

Northern Lake Service participates in a wide variety of certifications, state and federal programs and contracts, and is subjected to rigorous external audits by many government agencies and private clients.

Northern Lake Service presently holds certifications from the States of Wisconsin and Minnesota, and is audited on a regular basis by the WDNR - Office of Technical Services auditors. Quarterly internal blind and performance evaluations analyses are also performed under these certification requirements, in addition to our participation in U.S. EPA WS and WP series performance evaluation samples.

10.2 Northern Lake Service Internal Audits

Northern Lake Service is periodically audited by the QA department. The frequency of these audits is not to be less than annually. These audits are intended to serve two purposes:

1.) To ensure that laboratory staff are complying with the procedures defined in laboratory SOPs, QAPPs, and contracts.

2.) To determine any sample flow or analytical problems.

11 Calculation of Data Quality Indicators

11.1 Precision

Precision is determined by the comparison of duplicate control samples. The RPD of duplicate control samples will be used to estimate the precision. The following equation will be used to determine this:

$$RPD = \frac{(D_1 - D_2)}{(D_1 + D_2)/2} X\ 100$$

Where:

RPD = Relative Percent Difference
D_1 = First Sample Value
D_2 = Second Sample Value

11.2 Accuracy

The determination of accuracy of a measurement requires a knowledge of the true or accepted value for the analyte being measured. The average percent recovery of duplicate control samples will be used to estimate accuracy. Accuracy will be calculated in terms of average percent recovery in the following equation:

$$\text{Average percent recovery} = \frac{X}{T} \times 100$$

Where:

X = Observed valued
T = True value

11.3 Analytical Completeness

Determining whether a database is complete or incomplete is a subjective evaluation. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for all of the analytical protocols. Less obvious is whether data are sufficient to achieve the goals of the project. All data are reviewed in terms of goals in order to determine if the database is sufficient.

Percent completeness is calculated as follows:

$$\text{Completeness} = \frac{\text{Valid data obtained}}{\text{Total data needed}} X\ 100$$

11.4 Detection Limits

The sensitivity of an analytical method is related to the detection limit (i.e., the lowest concentration of an analyte that can be detected at a specific confidence level). Definitions of method detection limit (MDL), and limit of quantitation (LOQ), follow in this session.

MDL - This is the minimum signal level required to qualitatively identify a specific analyte by a specific procedure at a greater than 99% confidence interval. An MDL is measured by analyzing a minimum of seven replicates spiked at 1-10 times the expected method detection limit. It is calculated by the standard deviation of the replicate readings times the student T - value at the desired confidence level. Northern Lake Service uses a 99% confidence interval and seven spiked replicates of a control matrix in the determination of method detection limits.

LOQ - This is the minimum level that can be reliably quantitated by a method within specified limits of precision and accuracy. Northern Lake Service's LOQs are derived from the evaluation of intralaboratory method detection limit studies.

All data are evaluated and reported relative to the MDL. Concentrations between the MDL and LOQ are considered positive detections and estimated values of less certain quantitation than values obtained that are above the LOQ.

12 Corrective Action

Corrective actions for laboratory problems are specified in Northern Lake Service's SOPs. Specific QC procedures are designed to help analysts determine the need for corrective action. Often, personal experience is most valuable in alerting the analyst to suspicious data or malfunctioning equipment.

The essential steps in the corrective action systems are as follows:

1.) Identify and define the problem.
2.) Investigate the problem.
3.) Determine the cause of the problem.
4.) Determine a corrective action to eliminate the problem.
5.) Assign and/or accept responsibility for implementing the corrective action.
6.) Establish effectiveness of the corrective action and incorporate it into the day-to-day procedures.
7.) Verify that the corrective action has eliminated the problem.

This scheme is generally accomplished with the assistance of the QA department. Any laboratory analyst may notify the QA officer of a problem. The QA officer initiates the corrective action scheme by relating the problem to the appropriate laboratory supervisors who investigate or assign responsibility for investigating the problem and its cause. Once determined, an appropriate corrective action is approved by the QA officer. Its implementation is later verified through an audit.

Close scrutiny is paid to the quality and validity of the analytical data for any given analysis. Data acceptability is judged utilizing precision and accuracy information in the QC samples. Corrective action at the bench level is generally initiated by an out-of-control QC sample result. The nature of each corrective action is determined by the method performed, the matrix type and the end-use of the data. In some instances, a re-analysis, re-extraction, or recalibration may be necessary to correct the problem.

Northern Lake Service, Inc. (NLS) will notify the Wisconsin Department of Natural Resources (WDNR) as soon as possible for all problems that will impact data useability. At this time NLS and WDNR will together determine the appropriate corrective action steps so that all samples and data will be valid. WDNR and NLS may at this time elect to resample to obtain completely valid data rather than perform other corrective action.

13 Preventive Maintenance

Laboratory personnel are trained in routine monitoring and maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by either trained staff or trained service engineers employed by the instrument manufacturer.

Each laboratory has detailed SOPs on file that describe preventive maintenance procedures and conditions necessitating maintenance. The laboratories also maintain detailed logbooks documenting the preventive maintenance and repairs performed on each analytical instrument.

14 References

"Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", Office of Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. EPA, EPA-600/4-83-004, February, 1983.

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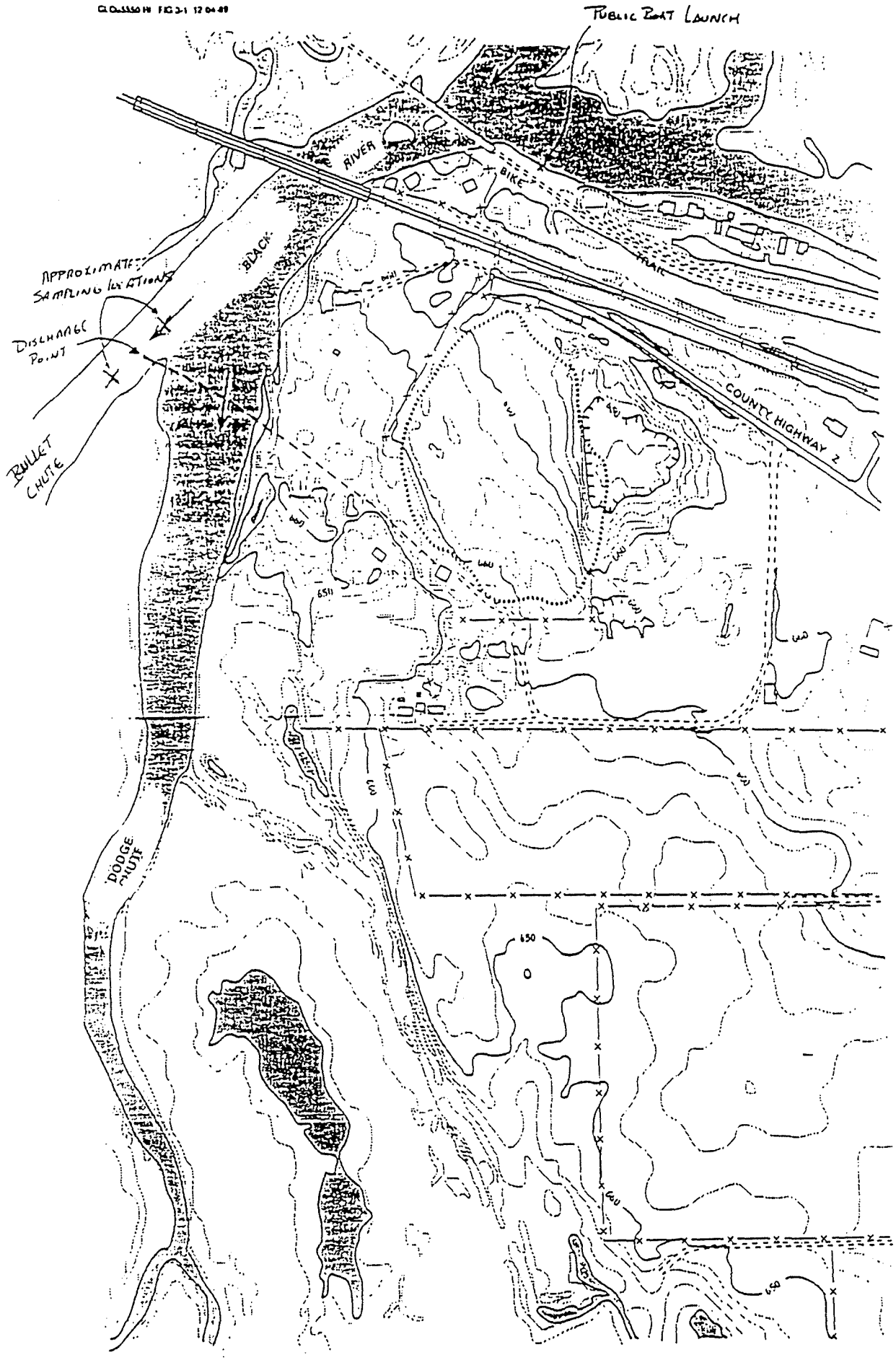
"Methods of Soil Analysis", Second edition, Part 2, Number 9, American Society of Agronomy, Inc. and Soil Science Society of America, Inc. - Madison, Wisconsin, 1982



SAMPLE COLLECTION SITE MAP
BLACK RIVER, WISCONSIN - BULLET SHOOT SECTION
ONALASKA TOWN LANDFILL - SUPERFUND SITE
LACROSSE COUNTY, WISCONSIN

SITE TOPOGRAPHY