MINNESOTA POLLUTION CONTROL AGENCY
SITE REMEDIATION SECTION

DRAFT GUIDELINES
RISK BASED SITE CHARACTERIZATION AND SAMPLING GUIDANCE

WORKING DRAFT, September 16, 1998

Comment Period Ends December 31, 1998

Send Written Comments to:
Guidance Coordination Team
Minnesota Pollution Control Agency
Site Remediation Section
520 Lafayette Road
St. Paul, Minnesota 55155-4194
Fax (651) 296-9707

NOTICE

THIS DOCUMENT IS A WORKING DRAFT. The Site Remediation Section of MPCA is developing guidelines for evaluating risks to human health and the environment at sites that may require investigation or response actions pursuant to the Minnesota Environmental Response and Liability Act, Minn. Stat. § 115B.01 to 115B.24 (MERLA)**.**

DEVELOPMENT OF A SITE REMEDIATION SECTION SITE EVALUATION MANUAL. The attached document and other documents will be incorporated into a Site Remediation Risk-Based Site Evaluation Manual which will contain guidelines for conducting MERLA-related evaluations, including risk evaluations under the State Superfund program and the MPCA Voluntary Investigation and Cleanup (VIC) Program.

MPCA staff intend to use the policies and procedures in the manual as guidelines to evaluate the need for investigation or remedial actions to address releases and threatened releases of hazardous substances or pollutants or contaminants under MERLA, and the scope and nature of such actions. These policies and procedures are not exclusive and do not have the force and effect of law. MPCA staff may use other policies or procedures to evaluate the need for or adequacy of response actions under MERLA, including procedures set forth in outstanding MPCA Requests for Response Action and Consent Orders. The final standard for all such evaluations is the MERLA statutory requirement that such actions must be reasonable and necessary to protect the public health and welfare and the environment.

The Minnesota state Superfund program, governed by the Minnesota Environmental Response and Liability Act (MERLA) and the supplementary rules, and the federal Superfund program, governed by the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) and the federal regulations in the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), work together to clean up various types of sites.

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~ Continuation ~

Under CERCLA, failure to act consistently with the NCP can result in a party not recovering its response costs from a RP. There is no NCP consistency requirement in MERLA, although under MERLA the costs must be reasonable and necessary. The guidance documents are intended to function in a similar manner to the NCP. However, because the guidance documents do not require every procedural specification of the NCP, parties are advised to consult an attorney early in the cleanup process if they intend cost recovery under CERCLA, which specifically states that the party seeking reimbursement must show that its costs are "consistent" with the NCP.

For removals, investigations and National Priority List sites, the federal and state governments must act consistently with the NCP. Note that CERCLA requires "consistency," or "accordance," as distinguished from "compliance," with the NCP. This infers some flexibility in selecting the appropriate remedy while following the basic requirements of the NCP. The extent of flexibility is still debated in courts. The NCP provides that a party does not have to comply with every single requirement of the NCP verbatim, but that the response action, when evaluated as a whole, be in "substantial compliance" with the NCP and result in a CERCLA-quality cleanup. The courts have emphasized that the community relations aspects are a part of the NCP response action, including the right of the public to participate in the remedial action selection process.

The preamble to the NCP recognizes government programs, like the Minnesota program under MERLA which has similarities to the NCP, that achieve the same objectives, but are not congruent with the NCP in every respect. EPA believes that these governmental bodies, consistent with CERCLA intent, should have flexibility to implement response actions and bring cost recovery actions for those response actions as long as the response actions are not inconsistent with the NCP, even if achieved by different methods. EPA believes that it is not necessary to define what actions are "not inconsistent with the NCP," and will make determinations on a case-by-case basis.

EXPLANATION:

[NOTE TO WORK GROUP: Include qualifying remarks specific to your document in this "explanation" box.]

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Users of this document are responsible for confirming with the MPCA Site staff the version of the working draft to be used.

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(NOTE TO WORK GROUPS: Copies distributed to non-MPCA staff must be accompanied by instructions to the recipient and where to focus their review. A specific list of questions may be appropriate.)

(NOTE TO PERSONS OTHER THAN THE SITE REMEDIATION SECTION (SRS) STAFF: As necessary, please distribute this draft document to selected members of your staff for review and comment. Based on past interactions with SRS, suggested staff are indicated in parenthesis)

@SRS

Jim Warner (MD/MO)
Gary Pulford (CO)
Cathy Moeger (MD/OP)
Duane Anderson (EO/ED) (Dann White, Dave Maschwitz)
Bruce Brott (MD/SR) (Beth Gawrys, Bryon Adams)
Mike Kanner (MD/SR) (Chris Zadak, Mike Bares)
Dave Thornton (PP/MF) (Cynthia Hollerbach)
Alan Williams (AG)
Rita Messing (MDH)
Larry Gust (MDH)
Dan Stoddard (Dept. Ag) (Cathy Villas-Horn, John Moeger)

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Note: For definitions of terms and concepts, please refer to the RBSE Glossary document.

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EXECUTIVE SUMMARY

The Site Remediation Section (SRS) of the Minnesota Pollution Control Agency (MPCA) developed this Risk Based Site Characterization and Sampling Guidance as part of a program wide guidance development effort. The document is intended to be used for developing sampling and analysis plans at Voluntary Investigation and Cleanup (VIC) and Superfund sites.

The overall objective of this document is to provide guidance on the sampling and analysis requirements for site activities from investigation through closure at sites under the MPCA jurisdiction. This document is organized by media (soil, ground water, air, and sediments), with media-independent topics such as Quality Assurance/Quality Control (QA/QC) and target analytes discussed separately.

This document presents guidelines for collecting data of sufficient quality and quantity to facilitate risk-based site evaluation and remedy selection, as well as remedy verification. The emphasis is on collecting adequate data, while keeping in mind cost effectiveness and rapid progress in site investigation. Since sites vary widely in their size and complexity, it is not possible to present a cookbook on how to sample every site. This guidance is general and is intended to be used, with some flexibility, by competent environmental professionals in their evaluation of sites.

Generally, it is desirable to identify the overall objectives of any environmental investigation prior to collecting samples. Identifying these objectives makes it possible to design a sample plan tailored to the specific needs of a site, resulting in more efficient and cost effective investigations. The data quality objectives (DQOs) should reflect the overall sampling objectives.

In order to make appropriate remedial action decisions, it is usually necessary to evaluate the nature and extent of soil, ground water, and sediment contamination attributable to releases at the site. In addition, the air pathway may need to be evaluated, data for all chemicals of potential concern collected, and hydrogeologic conditions at the site established. The evaluation of all media should include a discussion of the concentrations of contaminants, the physical and chemical nature of the contaminants, and the lateral and vertical distribution of contaminants at the site.

Large amounts of data can be generated at relatively low cost by field screening and field analytical methods. As a result, data produced through field screening and field analytical techniques are becoming increasingly significant in many site decisions. Therefore, it is critical to implement consistent and appropriate QA/QC measures for field techniques. Many of these field methods can be supplemented with laboratory analyses to achieve higher DQOs. In the interest of cost-effectiveness and timeliness, the MPCA is now advocating the use of field labs where appropriate and where an adequate level of QA/QC can be attained. Mobile labs must have a current QA/QC plan on file with the MPCA.

The three main reasons for conducting soil sampling are to evaluate potential human health and ecological risks on the site and in the vicinity of the property in question, to determine the potential for soil contaminants to leach into ground water, and to assess the need and extent of potential remedial actions. The choice of a soil sampling method is based on many factors, including accessibility, cost, soil conditions, and type of data desired.

Verification sampling strategies for soil remediation depend on the type of remediation -- excavation or in-situ treatment. The minimum number of samples and sampling locations are different for each type. While the

minimum number of samples required is easily determined for both situations, determining the sampling locations is more complex and requires some professional judgment. The sampling strategies are outlined in the guidance document.

Ground water quality data have traditionally been obtained at permanent monitoring wells constructed to MDH well code specifications. Properly constructed permanent wells produce the highest quality data, and multiple sampling events from the same sampling point are the best way to track temporal changes in water quality. However, ground water monitoring using direct-push techniques may be more appropriate than permanent monitoring wells at some sites. Regardless of the method used, two rounds of ground water samples will generally be required. Detailed guidance on tools and methods for sampling wells is given in the MPCA Example Ground Water Sampling Protocol, which is an Appendix to this guidance document.

Sampling to demonstrate aerobic or anaerobic natural attenuation of contaminants can be accomplished through laboratory and field data collection. Field collection of many parameters can be accomplished without laboratory analysis using field titration or colorimetric kits. The conditions that must be demonstrated before approval of a natural attenuation remedy for ground water are a stable plume, and an appropriate aquifer environment for (bio)chemical degradation. Natural attenuation must be clearly demonstrated on a site-by site basis.

To assure that air sampling efforts provide adequate data for the risk assessment, the sampling and analysis plan should be developed in consultation with the MPCA Risk Assessor. It is recommended that air sampling be planned and conducted by specialists who have a thorough understanding of air sampling theory and technology.

When conducting sediment sampling, the top two- to six inches of sediment is generally considered to be the portion of the sediment column which is available for exposure to ecological receptors. Samples from deeper in the sediment column are also collected for estimates of contaminant volume for remediation or sediment management.

1.0 INTRODUCTION

1.1 Overview

This document presents guidelines for collecting data of sufficient quality and quantity to facilitate risk-based site evaluation and remedy selection, as well as remedy verification. The emphasis is on collecting adequate data rather than perfect data in the interests of cost effectiveness and rapid progress in site investigation. Since sites vary widely in their size and complexity, it is not possible to present a cookbook on how to sample every site. This guidance is general and is intended to be used, with some flexibility, by competent environmental professionals in their evaluation of sites.

This document is organized by media, with media-independent topics such as Quality Assurance/Quality Control (QA/QC) and analytic methods discussed separately. The media discussed include soil, ground water, surface water, air, and sediments. The overall objective of this document is to provide concise guidance on the sampling requirements for site activities from investigation through closure at sites under MPCA jurisdiction.

2.0 TARGET ANALYTES

The selection of appropriate analytical parameters is necessary to meet the objectives of identifying and verifying chemicals of potential concern (COPC) present at a site, quantifying the extent and level of contamination at the site, determining the level of exposure to site contaminants, selection of a remedy, and, following site remediation, verifying that remediation objectives have been achieved. Analyte selection should be based on the premise that a reasonable potential exists for a particular contaminant to be present on a site. This document reflects the recommendations made in other MPCA guidance documents with respect to analytes and timing of sampling.

Some sources to consider when selecting analytes include: historical data, land use data, visual evidence of contamination, field screening instrument readings, olfactory evidence, hearsay evidence, contaminant breakdown products, and analytic data collected during earlier phases of site investigations.

2.1 Identification of Chemicals of Potential Concern (COPC)

2.1.1 Historical and land use data

Prior to conducting a sampling program, it is recommended that the site history be evaluated. This may take the form of a Phase I environmental site assessment, a Preliminary Assessment conducted for Superfund site listing, or an environmental site investigation conducted for another purpose. Guidelines for conducting a Phase I site assessment can be found in MPCA Voluntary Investigation and Cleanup Guidance Document # 8 entitled, “Phase I Investigation.” A site sampling and analysis plan should not be formulated until there is a reasonable understanding of the site history. Generally, the less historical information available, the more analytic data is necessary to adequately evaluate a site. The historical evaluation portion of the Phase I should detail the current and historic operations and practices at the site. Table 2A contains recommended soil and ground water analytical parameters to be used for site characterization based on various types of operations.

Minnesota Pollution Control Agency
Site Remediation Section
520 Lafayette Road
St. Paul, Minnesota 55155-4194

Table 2A Property Use and Target Analytes

OPERATIONS	COPCs for soil	COPCs for Ground Water
metal plating facility	Metals, VOCs, cyanide	Metals, VOCs, cyanide, pH
dry cleaning	VOCs	VOCs
rail yards	DRO, PAHs, Metals, VOCs, Semivolatiles, asbestos	DRO, PAHs , Metals, VOCs, Semivolatiles
dumps	VOCs, DRO, PAHs, Metals, Pesticides, PCBs, Semivolatiles, asbestos	VOCs, DROs, PAHs, Metals, Pesticides, Semivolatiles
coal gasification	PAHs, Metals, Cyanide	PAHs, Metals, Cyanide
wood treating	PAHs, Metals, PCBs, Semivolatiles, Dioxins/Furans, DRO	Semivolatiles, Metals, PAHs, DRO
tank farms	VOCs, DRO, GRO, PAHs	VOCs, DRO, GRO, PAHs
transformer refurbishing	PCBs, DRO	PCBs, DRO, VOCs
machine shops	VOCs, Semivolatiles, Metals	VOCs, Semivolatiles, Metals
semi conductor manufacturing	VOCs, Metals	VOCs, Metals
scrap yards	PAHs, Metals, DRO, VOCs, PCBs	PAHs, Metals, DRO, VOCs, PCBs
foundries	VOCs, PAHs, Metals	VOCs, PAHs, Metals

As the remedial investigation progresses, a review of the analytic data may indicate that the list of chemicals of potential concern should be expanded or narrowed.

2.1.2 Visual, olfactory, and screening data

Analytes may also be selected based on visual, olfactory, or field screening data. Many types of contamination are readily visible in surface soils, sediments, ground water, and surface water. For example, some indicators of surface soil contamination are stained soils, stressed vegetation, or lack of vegetation and presence of debris. Sediments may show unusual textures, colors or odors, while ground water and surface water may exhibit discoloration, surface sheens, or chemical odors. If the site history indicates that wastes were likely disposed of at the surface, visual and olfactory evidence may indicate which areas are likely to have been impacted and what types of compounds are likely to be present. For many compounds, visual and olfactory evidence may be corroborated by field screening techniques or laboratory data.

2.1.3 Data from earlier site investigations

Many sites have been investigated earlier under the jurisdiction of other MPCA programs or independently outside of MPCA jurisdiction. To determine the COPC for the sampling and analysis plan, analytical data generated in earlier investigations should be evaluated for evidence of compounds not expected to be present at the site, as well as for evidence of compounds that are expected to be present at the site, but were not directly detected (e.g., contaminant found in one medium, but not looked for in another). These data should only be used if they have been evaluated and determined to be of acceptable quality, based on the QA/QC requirements outlined in Section 3.0.

2.1.4 Hearsay evidence

Sites often come to the attention of the MPCA due to allegations made by neighbors, interested citizens, or employees at a site. This evidence is often verbal only and may or may not be accurate. Any allegations made concerning specific contaminants should be evaluated and a decision made as to whether those compounds should be added to the target analyte list. When in doubt, it is advisable to analyze for the alleged contaminants.

2.1.5 Breakdown products

When identifying COPCs, consideration must be given to the possible existence of contaminants created through the degradation of "parent" compounds. Some compounds break down to other compounds under common vadose zone and aquifer conditions; these breakdown products may be more toxic or mobile than their parent compounds. Table 2B is a listing of some parent compounds and their common breakdown products. Parent compounds and their breakdown products may or may not be a part of the same analyte suite. Some common breakdown products such as vinyl chloride (derived from many chlorinated solvents) pose greater health risks than their parent compounds. The presence of aerobic or anaerobic conditions in or near ground water can be established by using the methods mentioned or referenced in Section 4.4.

Table 2B Common Breakdown Products

Parent Compound	Breakdown Product(s)	Required Conditions / Pathway
tetrachloroethylene	trichloroethylene, dichloroethylene, vinyl chloride	anaerobic
trichloroethylene	dichloroethylene, vinyl chloride	anaerobic
dichloroethylene	vinyl chloride	anaerobic
pentachlorophenol	mono-, di-, and trichlorophenols anisoles	aerobic anaerobic
chlorinated volatiles and semivolatiles	dioxins and furans	combustion

2.1.6 Other Parameters

In addition to analyzing for COPCs, it is desirable in many cases to analyze for other parameters which may aid in risk assessment, remedy selection, fate and transport modeling, evaluating the soil leaching pathway, and natural attenuation. Examples of additional parameters and their utility are presented in Table 2C. A table outlining the specific parameters for evaluating the soil leaching pathway can be found in Appendix 3. Some commonly evaluated natural attenuation parameters are listed in Section 7.5. A detailed discussion regarding natural attenuation can be found in the MPCA guidance document entitled "Natural Attenuation of Chlorinated Solvents in Ground Water."

Table 2C Other Parameters

Soil Physical Properties	Utility
permeability	fate and transport
porosity	contaminant fate and transport
volumetric water content	fate and transport studies
Soil Chemical Properties	
cation exchange capacity (CEC)	metals soil retention
distribution coefficient (Kd)	organic compound retention
total organic carbon (TOC)	fate and transport studies
microbial consortia	bioremediation studies
contaminant specific TCLP	hazardous waste determination
Ground Water Chemistry	
hardness	treatability
iron	treatability
tritium	ground water dating
pH	metals transport
oxygen	bioremediation studies
nitrate	bioremediation studies
phosphorous	bioremediation studies
TOC	fate and transport studies
acid volatile sulfide (AVS)	bioavailability/toxicity studies
particle size analysis	treatability

3.0 DATA QUALITY OBJECTIVES AND QUALITY ASSURANCE/QUALITY CONTROL

3.1 Data Quality Objectives (DQOs)

The EPA has published a guidance document for DQOs, entitled "Guidance for the Data Quality Objectives Process," EPA QA/G-4. The EPA guidance document describes what a DQO is, and what the process is and does. The EPA guidance should be used to develop the DQOs for data and site work, and is the basis for the seven-step process provided below. The DQO process is iterative, and may take several iterations to determine the best course of action.

DQOs are qualitative and quantitative statements derived from the outputs of the first six steps of the DQO process. These statements clarify the study objectives, define the most appropriate type of data to collect, determine the appropriate conditions from which to collect the data, and specify tolerable limits on decision errors which will be used as the basis for establishing the quantity and quality of data needed to support the decision.

The seven steps listed below are used in the DQO process, and are used to generate an optimal design for sampling and analytical work. Each step must be used to complete the process, but depending upon the site, each step may or may not involve the same degree of depth. These steps must be clearly described in the site Quality Assurance Program Plan (QAPP) or Work Plan. The QAPP must be approved prior to any work being done on the site.

1. State the Problem - The purpose is to clearly define the problem so the focus will be unambiguous. The following steps need to be completed to begin the process:

a. Identify the members of the planning team. This will be driven by the size and complexity of the problem. There should be representation from all stakeholders. Potential individuals include; samplers, chemists, engineers, modelers, hydrogeologists, etc..

b. Identify the decision maker. The decision maker is the leader of the team. This person has ultimate authority for making final decisions.

c. Give a concise description of the problem. Describe the conditions and circumstances that are causing the problem. Discuss how they affect human health or the environment, and potential non-compliance with regulations. Describe how the problem is currently understood. If the problem is complex, consider breaking it into smaller components.

d. Specify the available resources and relevant deadlines for the study. Discuss budget, personnel, vehicles, and any other resources that may be needed.

2. Identify the Decision - The purpose of this step is to define the decision statement that drives the work being done on a site. A decision statement links the principal study question to possible actions that will solve the problem.

a. The principal study question is: what is the reason for doing the work? The principle reason for doing the study is to answer a question, such as “Is the site contributing to groundwater degradation due to on-site pollution?”.

b. After defining the principal study question, the different actions that could be taken to answer the question need to be written down, such as “Buried solvent barrels on site are leaking and causing ground water degradation”.

c. The last step is to combine the principle study question with the possible actions to form a decision statement. An example from above would be “Determine if solvent barrels buried on site are causing degradation of ground water”. This is a simplified example, but shows the logic involved in developing the decision statement.

d. When several separate decisions are needed, list them, and define what the sequence is in which they must be solved for the site. A flow chart is a possible method of tracking the levels of decisions to be made.

3. Identify the Inputs to the Decision - Identify the inputs that are needed to resolve the decision statement and determine which inputs require environmental measurements.

a. Determine the variables or other information that is needed. Answer questions on whether chemical or physical properties need be studied to answer the decision statement.

b. Determine the source of the information needed (e.g. historical records, previous data, regulatory guidance, etc.).

c. Identify the information needed to establish the action level. This could be driven by risk assessment or regulatory thresholds or limits.

d. Confirm that the appropriate analytical methods exist to provide the necessary data. Consider the chemicals of concern and limits that are required to find methods that would be appropriate for measurement.

4. Define the Boundaries of the Study - Define the spatial and temporal boundaries that are covered by the decision statement.

a. Give a detailed description of the boundaries of the problem. This would be physical and temporal (the time-frame when the data is taken).

b. When appropriate, divide the population into strata that have homogeneous characteristics.

c. Determine when to collect data. Weather, humidity, time of year, etc. can affect the data.

d. Define the scale of decision-making. This deals with focusing sampling and analysis in an area that is most affected by the study. For example the top six inches of soil in children's play area.

e. Identify any practical constraints on data collection. This includes weather, consent to sample on someone's property, equipment, time, and personnel.

5. Develop a Decision Rule - Define the parameter of interest, specify the action level and integrate previous DQO outputs into a single statement that describes a logical basis for choosing among alternative actions.

a. Specify the statistical parameter that characterizes the parameter of interest. This means what is to be measured must be defined. Is the mean of the concentrations driving the cleanup? This may be defined by regulatory guidance.

b. Specify the action level for the study. Choose what level would cause a different action. For example if the soil has < 1 mg/kg of a contaminant, the soil is left on site, vs. >1 mg/kg the soil goes to a landfill vs. >50 mg/kg the soil goes to a hazardous waste landfill. Confirm that all action levels can be met by the laboratory analytical methods.

c. Develop a decision rule. This takes into account the prior steps in the following example "If the soil (average value from sampling a grid) within the site (defined by the boundaries) is less than the action level (e.g. 1 mg/kg) then take action A (leave the soil in place); otherwise, take action B (remove the soil).

6. Specify Tolerable Limits on Decision Errors - This step allows the tolerance to be set up for decision errors. This is based upon the consideration of the consequences of making an incorrect decision. This step involves setting the limits on a false positive or false negative, and realizing the impacts that these errors could cause. There will also be a gray region that is defined as the region where the consequences of decision errors are relatively minor. Part of the gray region also deals with the error built into any analytical or sampling method that is used. A planner can go as far in depth as they wish in this step to determine what level of data and number of samples they need to feel confident they have a site picture that is "good enough". It is recommended that, at a

minimum, this step should generate the level of analytical data needed, the number of samples needed to have a statistically valid set of data, and the type of sampling needed (e.g. how precise).

7. Optimize the Design for Obtaining Data - This step identifies the most resource-effective data collection design for generating data that satisfies the DQO requirements of the site.

a. Review the outputs from the prior six steps and determine if they are consistent with the requirements of the site.

b. Develop the general data collection design alternatives. These could be simple random sampling, stratified random sampling, systematic sampling, etc.

c. Select the optimal sample size that satisfies the DQOs for each data collection design. This includes a cost function of number of samples to total cost of sampling and analysis. If the cost or other constraint compared to the needed data do not match, then one of the constraints will need to be relaxed. These include: increasing the budget, increasing the tolerable error rates, easing the schedule, changing the boundaries, etc..

d. Select the most resource-effective data collection design that satisfies all of the DQOs. Choose the option that gives the best balance of costs and ability to meet project requirements.

e. Document the details, theory, and assumptions behind choosing the option to be used in the sampling and analysis plan and in the QAPP. An experienced statistician can review the results of the work and make suggestions that could reduce the sample load or costs in other areas.

The old DQOs, as known from EPA Region V, had five levels for analytical data. The new DQOs only use two. These are: qualitative data and court-defensible/quantitative data. The major differences between the two are the method by which the data is gathered and analyzed. A rush sample is normally not considered a court-defensible data point, unless standardized methods are used to generate the data (i.e., full quality assurance is utilized). Examples of qualitative data include: mobile gas chromatographs, immunoassay kits, colormetric field tests, etc.. Court-defensible data will nearly always be performed in a certified, fixed-base laboratory. The planner must be aware of what the eventual use of the data will be, as there are numerous analytical methods, some of which are not as dependable as others. Examples of method choices include: Method 8310 vs. 8270 for Polyaromatic Hydrocarbons (PAHs), 8121 vs. 8260 for Volatiles analysis (VOCs), 8040 vs. 8270 for phenols, etc. Each of these methods will produce “good” data, but an analytical method to be used on-site must be chosen that will give data of sufficient quality to meet the DQOs.

3.2 Quality Assurance/Quality Control

3.2.1 Field Methods and QA/QC

Large amounts of data can be generated at relatively low cost by field screening and field analytical methods. As a result, data produced in the field through field screening and field analytical techniques is becoming increasingly significant in many site decisions. Therefore, it is critical to implement consistent and appropriate QA/AC measures for these techniques. Table 3A presents some basic QA/QC considerations for some common filed screening and field analytical techniques. The uses of some common field screening and field analytical methods is presented in Section 5.4.

Table 3A Field Methods and QA/QC

METHOD	BASIC QA/QC CONSIDERATIONS
PID AND FID	• Follow MPCA bag headspace procedure (Appendix 4) for soil samples. • Zero instrument gauge to ambient air, however calibration to a standard such as O_2 is probably better since "dirty" ambient air could mask low VOC concentrations in a headspace sample. • If taking readings in a vehicle, zero to a standard and then measure ambient air. VOCs can volatilize from vinyl dashboards (especially new cars). • In humid and/ or cold conditions, keep instrument in heated vehicle to prevent moisture condensation due to extreme temperature fluctuations. • Avoid anything that can cause interference with instrument readings such as smoking, insect repellent, auto exhaust, markers, etc. • Document everything: wind, temperature, humidity, calibration data, problems encountered, locations, field observations, etc.
XRF	• Should have 10 - 20% lab duplicates. • Should have separate calibration curves for each analyte and matrix type. • Ex-situ is the preferred analysis method. • Use dry samples or normalize for water content. • Analyze uniformly sized material (use sieves if possible). • Maintain proper incidence angle.
COLORIMETRIC	• Should have 10 - 25% lab duplicates. • Check vendor literature for interferences, false positives, etc. • Generally applicable as screening tool.
IMMUNOASSAY	• Should have 10 - 25% lab duplicates. • Only targets small number of compounds. • Interference by non-target compounds can obscure results.

3.2.2 Mobil Lab QA/QC

Significant cost savings can often be realized through the use of on-site mobile labs. Mobile labs usually contain a gas chromatograph and may also contain an XRF or other instruments. Mobile labs can provide almost immediate results which facilitates decisions regarding the site investigation during implementation of field work. Use of mobile lab can significantly reduce sample holding times, leading to potentially more accurate analyses of volatile compounds. QA/QC requirements for mobile labs can be much less stringent that those for fixed labs, but the MPCA advocates the use of field lab where appropriate and where an adequate level of QA/QC can be attained. Mobile labs must have an approved QA/QC plan on file with the MPCA. The detailed requirements for the QA/QC plan are attached as Appendix 1 with the highlights listed below. Questions on QA/QC requirements are best answered by the MPCA QA/QC Coordinator.

Generally the minimum Level of QA/QC required for mobile labs is:

- Quality Assurance Program Plan (QAPP) including Standard Operating Procedures (SOPs) on file with the MPCA and present on site.
- A second source calibration blank may be required by the MPCA QA/QC coordinator

- Initial on-site calibration using 3 to 5 standards with acceptance criteria of <25% difference for target compounds, then twice daily continuing calibration standards analysis.
- For VOCs the following requirements must also be met:
  - each sample spiked with surrogate compound
  - matrix spike (MS)/ matrix spike duplicate (MSD): 1 per 10 analyses
  - Method blank: 1 per 10 samples
- A minimum of 1 method blank per day
- reporting limits (RLs) must be less than action levels for site
- Documentation of work performed should be available

Reporting requirements for a mobile lab are:

- RLs or practical quantitation limits (PQLs)
- chain of custody (COC) for each sample set must be present
- % surrogate recovery for all samples
- results of all blanks must be reported
- MS/MSD/Relative Percent Difference (RPD)/% Recovery must be reported
- all raw data must always be submitted in the report of analyses
- analysis date, and date of receipt of each sample must be reported
- any data qualifiers must be noted and their meaning described
- analytic method, compound list and concentration found must be reported
- any dilutions performed must be noted
- narrative on any problems including initial and continuing calibrations

3.2.3 Fixed Lab QA/QC

Fixed laboratories have been used almost exclusively in the past for soil and ground water analyses at MPCA sites. The level of QA/QC attained by the fixed labs has varied widely. In an effort to standardize the QA/QC requirements and assure that consistently high quality analytic data are used, the State of Minnesota has established a program of laboratory certification. Laboratories must either be certified by the State of Minnesota, be directly certified by a state which has reciprocity with Minnesota, or be a current Certified Laboratory Program (CLP) lab. Consult the MPCA QA/QC Coordinator for laboratory certification information. A brief discussion of MPCA QA/QC requirements follows:

Fixed Lab QA/QC Requirements

- MN Laboratory Certification, direct certification by state with reciprocity (WI, ND, WV, WA) or current CLP lab
- calibration specific to methods used and calibration checks done
- 10% method blanks
- surrogate recoveries must be within range specified by method or 30% to 150% if not specified by method
- matrix spikes/ matrix spike duplicates - 5% for organics, 10% for inorganics
- reporting limits: RLs or PQLs should be 2 to 10 times method detection limit (MDL), RL must be less than action levels for site

Reporting Requirements

- all raw data must always be available for review
- date sample received
- date sample extracted/ digested
- date sample analyzed
- analytic method (digestion/extraction/analysis)
- RL/PQLs
- concentration found
- compound list
- signature of lab officer
- surrogate recoveries
- MS/MSD/Duplicate/RPDs/% Recovery
- data qualifiers or flags
- project #
- sample #
- COC
- narrative of any modifications made to method or problems encountered

Field QA/QC documentation

- COC with signature
- date
- sampler's name and affiliation
- location
- project # and/or name
- objective of sampling event
- weather conditions
- time each sample collected
- problems encountered
- equipment used
- site sketch with sample locations
- descriptions of material sampled
- olfactory or visual evidence of contamination
- PID or other instrument readings
- well construction if applicable
- blanks: how and where collected
- personnel on site

4.0 GROUND WATER SAMPLING

4.1 Introduction

This section presents guidelines for collecting ground water data of sufficient quality and quantity to facilitate risk-based site evaluation and remedy selection. The emphasis is on collecting adequate data, keeping in mind cost effectiveness and rapid progress in site investigation. Since sites vary widely in their size and complexity, it is not possible to present a cookbook on how to sample every site. This guidance is general and is intended to be used, with some flexibility, by environmental professionals in the evaluation of sites.

4.2 Ground Water Standards

The *Ground Water Policy Document* contains a description of the different standards which apply to ground water as well as a risk-based approach to applying those standards. Briefly, the standards are:

1. HRLs - Health Risk Limits, a state standard set by the MDH.
2. MCLs - Maximum Contaminant Levels, a federal standard which applies to municipal water supplies and is applied at the point of use.
3. HBVs - Health Based Values, state standards for contaminants for which HRLs do not exist.
4. Surface Water Standards - state values which apply where ground water discharges to surface water; these standards vary depending on the receiving surface water body.

4.3 Ground Water Investigations

4.3.1 Preliminary Investigations

In a preliminary ground water investigation, an unrestricted property use scenario is assumed. Because potable use of all ground water is assumed, sample location or number is not receptor dependent. Generally, a minimum of three monitoring wells is required for a preliminary investigation to determine the nature of ground water contamination and ground water flow direction. The number and location of the wells is site specific depending on the nature and location of the release.

However, a preliminary ground water evaluation does not necessarily require permanent monitoring wells. A ground water investigation resulting in site closure can be accomplished with direct push technology as long as the DQOs are met. The following guidelines or concepts are to be followed:

The extent of ground water contamination must be adequately defined. Adequate spatial coverage will generally require at least 1 sample per identified area of soil contamination or other release, with additional push probes to define the extent and magnitude of the release. If soil contamination or another type of release is not evident, 6 equally spaced direct push samples per 0.5 acres should be collected and submitted for laboratory analysis.

With either approach (i.e., push probes or permanent wells), a minimum of two rounds of ground water samples will generally be required. When using push probes, exact duplication of the first round of push probe point locations is not required for the second round of ground water samples. If push probes are used, it is necessary to evaluate the vertical profile of a plume at potential monitoring well locations by collecting ground water samples every 5 feet in a plume until two sequential non-detect samples are collected. If permanent monitoring wells are placed to evaluate the plume, nested wells with different depths for screened intervals may be necessary, especially if NAPLs are likely to be present. Given this information, the zone of highest contamination can be identified. This evaluation will be very site-specific, depending on the nature of the contaminants present and geologic conditions at the site. Samples may be required at additional depths if vertical migration of contamination is a concern.

In the event that contaminants exceed the appropriate standards, additional wells will generally be required to demonstrate that the plume is not migrating and that it does not exceed the appropriate remediation goal at the applicable compliance point. In addition, it must be determined whether the plume is stable or unstable. For guidance on the topic of plume stability, please refer to the MPCA *Ground Water Policy Document*.

4.3.2 Extensive Investigations

An extensive site-specific ground water investigation may be required in situations where ground water flow is affected by pumping wells or where complex geologic conditions make a relatively sophisticated hydrogeologic evaluation necessary. Treatability or biotreatability studies may require additional sampling data depending upon the specific remedy selected.

Site specific data to be used for modeling purposes, such as rate of advective transport, retardation, dispersivity, and decay must be collected using scientific practices approved by the MPCA staff. Site specific values for contaminant fate and transport calculations such as K_d, soil bulk density and effective porosity must also be used.

4.4 Sampling for Natural Attenuation

Natural attenuation is the reduction in concentration of contaminants by natural (without human intervention) methods. If natural attenuation is being considered as a potential remedy, there are specific tasks that are considered essential for each phase of the evaluation. The screening tasks are intended to provide a basis for deciding whether contaminant attenuation is occurring and whether further sampling and analysis of this remedy is worthwhile.

Natural attenuation must be clearly demonstrated on a site-by site basis. The issues that must be demonstrated are plume stability and appropriate aquifer conditions for degradation of the contaminant. For example, a determination must be made whether oxidizing or reducing conditions are present. Sampling to demonstrate aerobic or anaerobic natural attenuation of contaminants can be accomplished through *screening*, which involves laboratory and field data collection. Parameters necessary for the demonstration of natural attenuation through *screening* and *verification* are provided in Appendix 3.

For further discussion of natural attenuation screening or verification, see the MPCA document entitled *Natural Attenuation of Chlorinated Solvents in Ground Water*.

4.5 Ground Water Sample Collection Methods

Two primary means of installing ground water monitoring points exist: direct-push methods and monitoring wells. Either method is suitable for most sites, although each method has its strengths and weaknesses and suitability to particular situations. Sections 4.3.2 and 4.3.2.3 discuss the primary methods of ground water sampling from installed monitoring points. Sampling ground water via residential wells is discussed in section 4.3.3.

A secondary method of collecting ground water samples from installed monitoring points is through a screened auger. In order to insure that the desired portion of the aquifer is sampled, the auger joints should be properly sealed with O-rings or teflon tape. For this type of temporary well, the screened auger should be properly purged (i.e., at least three volumes of the screened interval removed). It is important to note that any boring from which a water sample is collected is considered a "well" by MDH, and a monitoring well permit or sealing permit (for a temporary well) is required under Minnesota Rules, Chapter 4725.

4.5.1 Direct-push Methods

Direct-push sampling uses hydraulically and hammer-operated rams to push a screened sampling tube into the subsurface to a water bearing zone. A ground water sample is then withdrawn either manually or using a small sampling pump. Direct-push sampling is generally limited to aquifers occurring in unconsolidated deposits and has a maximum practical depth of about 50 feet. Direct-push sampling is generally much less expensive than installing and sampling permanent monitoring wells and a large number of samples can be collected in a relatively short period of time. It is typically used to characterize the extent of ground water contamination and choose locations for permanent wells, rather than for long-term monitoring. Direct-push sampling is very useful in determining the vertical extent of contamination as several discrete samples can be collected at different depths. The discrete nature of the samples is due to the short length (1 or 2 feet) which is screened in the sampling probe. These discrete samples are less representative of the aquifer as a whole than are samples collected from a monitoring well with a longer screened length. Care must be taken that clean materials at depth are not contaminated by shallower contamination through smearing when samples are taken at more than one depth or from beneath contaminated soils or portions of an aquifer. Direct-push tools are generally rented by the day, including operators, and may or may not include an on-site lab. Ground water data collected with these technologies may be less accurate than permanent monitoring well data. However, these technologies may be appropriate, particularly for plume delineation, if applied correctly with prudent QA/QC. When the highest level of data quality is necessary, permanent wells may be a better choice than direct-push sampling points. Push-points must be abandoned per MDH well code and points left in place for more than 48 hours must be permitted as monitoring wells.

4.5.1.1 Direct-push Ground Water Sampling

Generally speaking there are three tools available for sampling ground water using direct-push tools:

1. Riser and screen are placed inside probe hole - probe is pulled back and sample is collected through the screen. The riser and screen (generally PVC) may be left in place for up to 48 hours and used as a temporary well. It must be decontaminated before reuse or disposed of after one use. Samples may be taken using :
   a. Small diameter bailer

b. Peristaltic pump - if probe hole is < 25' deep. The vacuum generated by the peristaltic pump may cause problems for VOC samples as it may cause volatilization of the sample. The volatilization can be minimized by using the pump to draw the sample up into the tubing, *without running the water through the pump*. Then the pump can be reversed to dispense the sample into the vials.

c. A length of Teflon or plastic tubing may be used with a check valve to retrieve a sample directly or the tubing may be inserted into the probe hole and pinched and withdrawn to retrieve the sample.

d. A length of tubing may be used in combination with a vacuum pump to withdraw the sample. The vacuum pump may be inappropriate for VOC sampling as it may cause volatilization of the sample.

2. For probe tools, samples are taken through an extendible point with a screen or slots - the point is lost after one use. The tools are generally too small in diameter to allow the use of a bailer for sampling this method. Sampling methods which may be acceptable include:

   a. Peristaltic pump (See 1.b. above)

   b. Tubing methods (See 1.c. and 1.d. above)

3. Some push-probe rigs utilize an inner and an outer rod, the inner rod is screened. The outer rod is pulled back to expose the screen on the inner rod, through which the sample is withdrawn. Sampling methods which may be acceptable for these types of tools include:

   a. Small diameter bailer inserted inside inner rod.

   b. Peristaltic pump - some tools have a fitting which attaches to the screened section so a sample can be pulled without contacting the outer rod.

   c. Tubing methods

4.5.2 Monitoring Wells

Ground water quality data have traditionally been obtained at permanent monitoring wells constructed to MDH well code specifications. Properly constructed permanent wells produce the highest quality data, and multiple sampling events from the same sampling point are the best way to track temporal changes in water quality. Wells are thus most appropriate for long-term monitoring, for example when changes in water quality over time must be evaluated in order to judge the effectiveness of a remedy. Decisions on the number of wells necessary and well placement must be made on a site-specific basis. The well network usually is designed to adequately delineate the contaminant plume, provide necessary water level data, and delineate background water quality. Wells may be screened at or below the water table, depending on aquifer characteristics, the physical characteristics of the contaminants being monitored, and on whether NAPLs are present.

4.5.2.1 Well Construction and Development

Construction of all water wells, including temporary and permanent monitoring wells, is regulated by the Minnesota Department of Health (MDH). Wells must be installed by a licensed well driller. Definitions, monitoring well construction standards, and specific requirements for

monitoring wells are found in Minnesota Rules, Chapter 4725 which are periodically revised. A rules Handbook is available from the MDH. Details of monitoring well construction must be made on a site-specific basis and should be a part of the site Work Plan.

4.5.2.2 Well Drilling and Construction Logs

Well logs must be submitted to MDH for each well installed. The logs should include, as a minimum: a description of geologic materials encountered and the depths of upper and lower contacts of each geologic unit, this description must be performed by a qualified geologist; a description of well construction materials including well casing and size, drilling method, measured water levels, screen type and slot size, nature of sand pack and grout materials; surveyed elevation of the top of casing with depths of screen top and bottom, other well materials, and land surface referenced to the surveyed top of casing elevation; the location of each well must be provided with a precision of ±15 feet, the geographic location may be surveyed or located by global positioning system (GPS) or any other method which gives sufficient precision and may be reported in state plane coordinates, universal transverse mercator units (UTMs) or latitude/longitude.

4.5.2.3 Sampling Monitoring Wells

Wells may be sampled with bailers or several different types of sampling pumps. Detailed guidance on tools and methods for sampling wells is given in the MPCA Example Ground Water Sampling Protocol (Appendix 2). Procedures for monitoring well sampling should be included in a site-specific sampling and analysis plan which should be submitted as a part of the Work Plan or Monitoring Plan. Site specific DQOs should be developed for each site and approved in the appropriate Work Plan. Table 4A outlines ground water sampling components and the various methods for each component.

Table 4A Ground Water Sampling Components

GROUND WATER SAMPLING COMPONENTS	
	METHODS (IN DESCENDING ORDER OF QUALITY)
PURGING	Bladder pump
	Low flow-rate centrifugal electric submersible pump (e.g., Grundfos® "Redi-Flo 2")
	Low flow-rate helical rotor electric submersible pump (e.g., Keck)
	Peristaltic suction lift pump
	bailer (many different types and materials)
	open faucet or tap (e.g., residential wells)
STABILIZATION	Flow cell with temperature, specific conductance, pH, dissolved oxygen, and oxidation-reduction potential probes - continuous monitoring
	Probes placed into separate aliquots (grab samples) of water - periodic monitoring
	Three well volumes removed before sampling
SAMPLING	Bladder pump
	Low flow-rate centrifugal electric submersible pump (e.g., Grundfos® "Redi-Flo 2")
	Low flow-rate helical rotor electric submersible pump (e.g., Keck)
	Peristaltic suction lift pump
	bailer (many different types and materials)
	grab sample from faucet or tap (e.g., residential wells)

4.6 Residential Wells

Residential wells require special consideration during an investigation, due to the fact that different procedures must often be used to access, purge, stabilize, and sample the well. Unfortunately, information about the well depth and construction may be unknown or severely limited. Ideally, a residential well sample should be collected at the tap nearest the actual wellhead, and should be taken before the water has passed through any pressurized holding tank, water softener, or filtration system. It is advisable to take the time to select the location that will permit the collection of the *least-disturbed* water sample.

If possible, before sampling, the residential well should be purged until the pH, temperature, specific conductivity, and other parameters (see Appendix 2) have stabilized, before a sample is collected. However, this is rarely possible, given the plumbing and other limitations that exist in most residences. Table 4A lists the various methods for sampling residential wells, and provides recommendations for obtaining the highest quality sample. Additional information regarding residential well sampling is provided in the MPCA Ground Water Monitoring and Assessment Program Field Guidance Manual.

Table 4B Residential Well Sampling Methods

CONDITION	PURGE	STABILIZE	SAMPLE	NOTES
Well depth and diameter known; no pressure tank or softener ahead of tap; drain available for purge water	Until pH, temp, cond, are stable	Utilize flow-through cell and probes with multi-parameter meter	"Trickle" flow with minimum aeration of water; remove aerator if present in tap	If well cannot be stabilized, purge 3 volumes before sampling
Well depth unknown; may be pressure tank ahead of tap; drain available for purge water	Until pH, temp, cond, are stable	Utilize separate probes and meters; use aliquots to purge water to determine stability	"Trickle" flow with minimum aeration of water; remove aerator if present in tap	If pressure tank in-line, note on sampling forms; if well cannot be stabilized, purge at least one volume of pressure (holding) tank
Well depth unknown; pressure tank or softener ahead of tap; no drain available for purge water; no significant volume recently purged (e.g., laundry done, etc.)	Ten minutes at full discharge rate, or until temperature stable; Use bucket to collect purge water	Use thermometer or hand test	"Trickle" flow with minimum aeration of water; remove aerator if present in tap	If pressure tank or softener in-line, note on sampling forms; If temp. cannot be stabilized, purge at least one volume of pressure (holding) tank
Well depth unknown; pressure tank or softener ahead of tap; no drain available for purge water; significant volume recently purged (e.g., laundry done, etc.)	One minute at full discharge rate, or until temperature stable; Use bucket to collect purge water	Use thermometer or hand test	"Trickle" flow with minimum aeration of water; remove aerator if present in tap	If pressure tank or softener in-line, note on sampling forms

5.0 SOIL SAMPLING

5.1 Introduction

The purpose of this section is to describe and discuss recommended procedures for collecting soil samples from potentially contaminated properties. The two main reasons for conducting soil sampling are to evaluate potential human health and ecological risks on the site and in the vicinity of the property in question, and to determine the potential for soil contaminants to leach into ground water. A consistent sampling approach, as described in this document, should ideally evaluate the nature and extent of soil contamination. This approach

allows investigators to determine actual and potential exposure scenarios, assess risks, and evaluate leaching potential. Once these parameters have been identified and evaluated, cost-effective remedies can then be selected and implemented

Sampling and analysis of soils are important yet expensive aspects of the site investigation process. The accuracy of the conclusions based on sampling information is often viewed as directly proportional to the amount of analytical data generated. This document attempts to present guidance which strikes a balance between collecting a sufficient volume of data for drawing accurate conclusions and collecting a limited volume of data to allow cost-effective decision-making.

5.2 Establishing Soil Background Concentrations

When the site-specific risk assessment procedure call for background concentrations, they should be established based on-site specific waste constituents, specific chemicals used in various processes, facility operations, or remedial investigation results. Sample analyses may include metals, organic constituents, or other site specific waste constituents. Analyses should be conducted in accordance with EPA or other approved methods.

Regional or local factors can play a part in the background concentrations of a chemical in soil so selection of sample location may warrant careful consideration. For example, the geologic origin (e.g., the parent rock) of glacial drift may have been high in copper, lead, or other metals that may be potential contaminants. Additionally, the hydrogeologic situation can alter the quantity of these elements. Ground water recharge areas (e.g., highlands) are frequently leached of metals while ground water discharge areas (e.g., swamps, floodplain) are the recipients of leached metals. Thus, sites in low areas will usually have higher background concentrations than upland areas. Other conditions, such as precipitation and atmospheric fallout from widely dispersed human and natural activities, also affect soil concentrations.

Prior to conducting background sampling, coordinate with MPCA staff to develop a sampling strategy that will allow a reasonable statistical evaluation of the data to be conducted. Generally, a minimum of four samples must be used to establish "background" in soils. This will help account for natural constituent occurrences and inherent variability within each distinctive soil horizon. Background samples must be collected in an area which has not been impacted by environmental contamination from the site and must be representative of natural background conditions. Based on waste type, contaminant mobility, operation practices, and soil type (sand, silty sand, clay), an estimate of contamination depth should be made and background samples taken at comparable depths for the particular soil type. Multiple soil horizons should have "background" established separately (e.g., minimum of four samples per each soil unit).

EXAMPLE:

Brown medium coarse SAND	4 samples
Lt. brown silty fine SAND	4 samples
Gray silty CLAY w/trace of fine-med sand	4 samples

For additional information regarding background concentrations, please refer to the MPCA *Risk-Based Evaluation for Soil - Human Health Pathway Guidance*. Sections 3.2.4 and 6.3 of this document contain information pertaining to the establishment of background concentrations.

5.3 Soil Reference Values, Soil Leaching Values, and Other Soil Criteria

The Soil Reference Values (SRVs) and the Soil Leaching Values (SLVs) are tools to be used to assist in determining whether further investigation and possible cleanup is needed for a particular exposure area due to potential risks to human health and/or the environment. The SRVs and SLVs represent contaminant levels in the media above which unacceptable risks could occur under general exposure conditions. Residential exposure scenarios are assumed for all sites unless more site specific property use information is provided. For complete guidance on assessing human and ecological risk and the ramifications of current and future property use, refer to the following documents:

1. Guidance on Incorporation of Planned Property Use Into Site Decisions
2. Risk-Based Evaluation for Soil - Human Health Pathway Guidance
3. Ground Water Policy Guidance
4. Risk-Based Guidance for the Soil Leaching Pathway
5. Risk-Based Evaluation for Ecological Receptors - Soil Pathway Guidance
6. Sediment Assessment Guidance

5.3.1 SRVs

SRVs are risk-based soil concentrations based on specific property use scenarios and specific target risk levels. Residential SRVs allow for both adult and child receptors and combined direct exposure pathways. The SRVs reflect the most common direct exposure pathways to help determine when additional investigation and/or remediation is necessary.

If multiple contaminants are present, the cumulative risk must be evaluated on a site-specific basis. Sites which potentially include additional exposure pathways, particularly pathways involving food production and consumption, also require site-specific evaluations. Assume risks to be additive for carcinogens and for noncarcinogens with similar toxic endpoints in order to ensure that total site risk remains at or below the acceptable risk limits.

Industrial and recreational SRVs are available for a site if the proposed property use scenario for the site fits the criteria for these uses. Sites using industrial and recreational SRVs are subject to institutional controls such as affidavits or restrictive covenants in order to ensure that the future property use remains as industrial or recreational unless the site is cleaned up to the residential SRVs.

A detailed discussion as well as a table of the SRVs can be found in the MPCA *Risk-Based Evaluation for Soil - Human Health Pathway Guidance*. Contact the MPCA project team if a contaminant does not have an SRV. If toxicity values exist for a contaminant, an SRV can be derived.

5.3.2 SLVs

Exposure of humans to contaminants in soil may occur from the ingestion of ground water contaminants resulting from the leaching of contaminants from soil. The SLVs are soil screening values

which are protective of ground water as a drinking water source. SLVs represent soil contaminant concentrations which have the potential to leach to ground water, thereby causing adverse health effects to human receptors via ingestion of drinking water. SLVs are designed for use during early phases of a site investigation in evaluating whether further soil investigation is warranted. The SLVs are meant to be used in conjunction with the SRVs. A detailed discussion, as well as a table of the SLVs, can be found in the MPCA Risk Based Guidance for the Soil Leaching Pathway

When site soil contaminant concentrations exceed either or both the SRV and/or SLV, further soil investigation is warranted. For sites where ground water liability assurances are requested, and where soil concentrations do not exceed screening values, both the soil leaching and ground water pathways may proceed to the next phase of investigation. However, the ground water pathway must be screened using ground water data.

Ecological receptors are not likely to be exposed directly to ground water, but may be exposed indirectly if soil contaminants leach to ground water and then flow to a surface water body (e.g., a stream, lake, or wetland). If exposure through this pathway may reasonably occur, then SLVs which are protective of surface water must be used.

5.3.3 Ecological Soil Screening

Ecological receptors may be exposed to soil contaminants through dermal (or root) contact, by incidental ingestion of soil particles, by eating plants or soil invertebrates contaminated via bioaccumulation from soil, or by inhalation of soil vapors/particles. Direct soil contact benchmark values were derived for terrestrial plants, soil invertebrates, and soil microorganisms because these organisms represent important components of terrestrial ecosystems. If soil contaminant concentrations are below effects levels for these receptors, it is reasonable to expect that impacts from direct soil exposure will be minimal for other receptors. Ecological Soil Screening criteria apply to the uppermost four feet of soil. The direct soil contact benchmarks are provided in the Ecological Soil Screening Table #1. Tables containing the Ecological Soil Screening criteria can be found in the MPCA *Risk-Based Evaluation for Ecological Receptors - Soil Pathway Guidance.*

Soil contaminants can also bioaccumulate in plants and soil invertebrates, and be passed to higher animals which feed on them (food chain exposure). Because this can be an important exposure route for certain chemicals, such as mercury and PCBs, screening criteria were also developed for bioaccumulative contaminants for the food chain pathway. The bioaccumulation screening criteria can be found in the Ecological Soil Screening Table #2.

Note that the ecological soil screening criteria do not address inhalation exposure so in some cases they may not be adequately protective for exposure to volatile contaminants. If inhalation exposure is likely to be an important pathway, consult the Ecological Risk Assessor.

In general, ecological soil screening criteria are to be applied in areas that provide wildlife habitat (i.e., vegetated areas such as grassy, brushy, or wooded areas) or may do so in the future. Areas which are or will be covered with impervious materials (e.g., pavement) do not need the application of ecological soil screening criteria.

5.4 Field Screening and Field Analytical Methods for Soils

Two important aspects of soil sampling are field screening and field analytical methods. The results of field screening of soils are useful to determine not only the extent of contamination, but also the number of samples that require laboratory analysis.

Field screening methods involve the use of portable devices that do not provide contaminant-specific information. On the other hand, field analytical methods can often identify the contaminant species. Table 5A lists some examples of equipment that are commonly used for field screening and field analytical methods, and provides information about using the equipment. In order to screen soils for VOCs, MPCA staff recommends the use of the "Soil Sample Collection and Analysis Procedures" as described in the July 1996 Fact Sheet #3.22 by the MPCA Tanks and Emergency Response Section, which is found in Appendix 4.

Regardless of which type of equipment is used, a thorough utilities check (i.e., Gopher State One Call) must be completed before any subsurface sampling is conducted. It is important to have the appropriate training and to wear the proper personal protective equipment (OSHA - 29 CFR 1910.120 Appendix B) during soil sampling.

Table 5A Field Methods for Soils

Methods	Uses	Advantages	Disadvantages
PID	General screening of double bonded organic vapors; does not detect methane; must use appropriate lamp size based on contaminants expected to be present. Often used with Jar Headspace procedure.	fast, cheap; can make in-the-field decisions	not contaminant specific; can severely under or overestimate contaminant concentrations due to several factors, such as number of contaminants present, heterogeneity of matrix, contaminant response factors and ionization potentials
FID	General screening of any organic vapors uses hydrogen flame to ionize compounds up to 15.4 eV, including methane	fast, cheap; not limited to double -bonded compounds; can use with small column and identify chemicals; can detect methane; can make in-the-field decisions	can be more cumbersome than PID
Immunoassay	can target and quantify specific groups of compounds, including PAHs, PCBs and semivolatiles	can reduce number of analytical samples; can obtain large data sets; can make in-the-field decisions; available in kit form, and is relatively inexpensive	only targets small number of analytes; need good calibration curves
Colorimetric	can evaluate wide variety of organic and inorganic contaminants	some types available in kit form; can get real-time data	sensitivity and detection limits may be inadequate for some applications
XRF	can target and quantify specific metals	can reduce number of analytical samples; can obtain large data sets; can make in-the-field decisions	is expensive to purchase; need reliable standards for setup and calibration; requires highly trained professionals to operate; can have matrix problems
Mobile Lab/ On-site GC	extremely versatile; can target and quantify many compounds	can reduce number of fixed-base laboratory samples; can obtain large data sets; can make in-the-field decisions; generally data is fairly accurate	lower level of DQO than a fixed lab; need 10-20% duplicates at fixed lab to achieve higher level DQO; small sample size may not be representative; require highly trained professionals to operate

5.5 Soil Sample Collection Methods

The choice of a soil sampling method is based on many factors, including accessibility, cost, soil conditions, and type of data desired. The presence and location of buried and overhead utility lines may rule out the use of certain equipment. Other obstacles at the site, such as buildings, paved areas, trees, or debris may also dictate which type of sampling equipment may be used. Table 5B lists the most common methods used for soil sampling purposes. The table shows the advantages and disadvantages of each method, and lists options for VOC sampling. These methods are intended to be used to collect soil samples for field screening, field analytical, and laboratory analytical purposes. A good summary of other field analytical methods can be found in the EPA publication entitled Field Analytical and Site Characterization Technologies (Reference 11).

Table 5B Sample Collection Methods for Soils

METHOD	ADVANTAGES	DISADVANTAGES	OPTIONS (FOR VOCS)	COMMENTS
Hand Auger	Allows access when use of drillrig may be impossible due to location of utilities, etc.	Sampling depths are limited, and auger may not penetrate some materials. Sample is disrupted and aerated.	Inner sleeves Subcoring	Stainless steel augers preferred. Highest QA/QC when sleeves are utilized (for VOCs).
Subcoring Samplers	Allow collection of headspace-free samples	Sample may not be representative of interval. Rocks and debris can cause problems.	Methanol preservation Biocide addition Purge and Trap (P & T) vial	Can be used to sample soil from ground, split-spoon, or soil pile. Some vendors offer efficient systems for collection and storage of samples. Soil may be easily extruded into P & T vial.
Split-spoon	Can be used with hollow-stem auger or direct push equipment. Allows collection of sample at specified depth.	Limited to use in areas with drillrig access. Certain soil types difficult to sample.	Inner sleeves Subcoring	Sleeves required if multiple samples needed for screening, characterization and analysis. Use of sealing caps on ends of sleeves is preferred for VOCs if sample can be analyzed within 48 hours. Subcoring of sleeve is excellent alternative.
Sonic Rotary Drilling	Collects relatively large, undisturbed, continuous sample.	Casing may heat up and cause sample to lose volatiles.	Subcoring	Excellent tool for gathering stratigraphic information. Subcoring may prevent potential loss of volatiles from soil.

5.5.1 Sampling Soils for VOC Analysis

Sampling soils for VOCs presents special problems for field and laboratory personnel. The most significant problem is the volatilization during the collection, storage, and analysis of soil samples. These guidelines cannot explicitly describe every condition which can cause losses of VOCs. In general, however, any sample exposure to air can cause a rapid release of VOCs. Because of soil mixing conducted during sample collection, composite samples should not be collected for VOC analysis. Similarly, separate samples should be collected for the purposes of jarheadspace screening, physical description of soil characteristics, laboratory testing of physical properties, and laboratory testing for chemical parameters. The site-specific sampling plan should discuss how samples will be collected from the sampling devices, and should describe which sample will be used for which purpose.

MPCA staff recommends that preparers of sampling plans consult SW 846, Method 5035 which describes EPA-approved methods for collecting soil samples for VOC analysis. Please refer to the MPCA memorandum attached as Appendix 5. The choice of a method is should be coordinated with the laboratory to ensure the use of proper containers, preservatives, storage and analyses.

Table 5C lists some recommended soil sampling options that minimize the loss of volatiles. Subcoring, methanol preservation, and biocide addition are described in greater detail in SW 846, Method 5035. The selection of one of these methods will depend upon site-specific parameters, such as site history, soil type, depth to ground water, location of utilities, and other factors (including cost).

Table 5C Recommended Soil Sampling Methods for VOCs

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES	COMMENTS
Inner sleeves	Use as liners in split-spoon or hand auger. Stainless steel or brass preferred. Use middle sleeve for VOC analysis of sample, and subcore or cap immediately after sample removed from ground. If capped, place sleeve on dry ice and analyze within 48 hrs.	Help minimize losses of volatiles from split-spoon or hand auger.	With some soil types, sleeves may not fill completely during sample collection.	If soil is coarse sand or gravel, use soil gas sampling for VOCs. If sample cannot be analyzed for VOCs within 48 hrs, use methanol preservation, or subcoring plus methanol or biocide and P & T vial.
Subcoring	Insert into center of sleeve and cap immediately after sample collected. Place sample on dry ice or extrude into P & T vial with methanol or biocide.	Allows several options with headspace-free sample.	Sample may be too small to represent entire interval. Rocks and debris may present physical and analytical problems.	If soil is coarse sand or gravel, use soil gas sampling for VOCs. If sample cannot be analyzed for VOCs within 48 hrs, freeze sample or store on dry ice for up to 1 week..
Methanol preservation	Use tared container. Amount of methanol depends on mass of soil . Can also be added to certain P & T vials.	Allows longer holding time for samples. Prevents further losses of volatiles.	Requires higher detection limit. Generates hazardous waste.	If soil is coarse sand or gravel, use soil gas sampling for VOCs. If possible, use with P & T vial. Clayey, cohesive soils can present problem.
Biocide addition	Use sodium bisulfate as alternative to methanol if lower detection limit desired. Store sample on dry ice.	Prevents losses of VOCs due to biodegradation. Allows lower detection limit.	Does not prevent VOC losses due to volatilization.	If soil is coarse sand or gravel, use soil gas sampling for VOCs. If possible, use with P & T vial.
Purge and trap vial	Vials with double septa and frit-sparging preferred. Choice of preservation methods and sample sizes. Most efficient when used with subcoring device. Store sample on dry ice.	After sample is collected, vial is never reopened. Avoids potential losses of VOCs during storage, preparation and analysis stages.	Vials are expensive and may not be widely available.	If soil is coarse sand or gravel, use soil gas sampling for VOCs. Can be combined with other options to yield high quality data; not currently used in Minnesota.

5.5.2 Soil Gas Sampling

Some sites or subsurface conditions may dictate that soil gas sampling for VOCs or methane gas be conducted instead of "traditional" soil sampling. For example, sites with abundant coarse sand or

gravel in the subsurface may yield little useful VOC data from "traditional" soil sampling, even if the recommended methods listed above are used. Under such conditions, soils may be too porous to retain VOCs, and may also present problems for some of the above-listed sample collection techniques.

Several different methods can be used to collect soil gas data, and the best method for a given site will depend upon the specific conditions at the site, the purpose of the sampling and the cost. Soil gas samples can be collected actively with the aid of direct-reading equipment (e.g., FIDs), or can be collected passively using carbon collectors . Table 5D lists some common methods for sampling soil gas.

Table 5D Soil Gas Sampling Methods

METHOD	EQUIPMENT	EXAMPLES	ADVANTAGES	DISADVANTAGES	COMMENTS
Active	PID, FID, Detector Tubes, Combustible Gas Indicators, etc.	OVM, OVA, Draeger, Gastech, etc.	Direct-reading equipment	PID not compound-specific. Detection limits for detector tubes may be too high.	Can be very effective if PID used in conjunction with detector tubes.
Passive	Activated Carbon Collectors	Petrex, EMFLUX, GORE-SORBER	Fairly simple installation. Collect compound-specific data	Interpretation of data may be difficult.	Useful for delineating limits of soil and/or ground water contamination.
Combination	Stainless Steel Canisters	Summa, Stabilizer	Portable system. Allows identification of contaminants	Does not provide actual concentration of contaminants in soil.	Good screening tool for determining contaminants of concern.

5.6 Soil Investigations

The general objectives of a soil investigation are to determine if a site poses a significant potential risk and whether or not a site requires further evaluation or remediation. Additional objectives are to minimize regulatory involvement and obtain quick regulatory closure. A preliminary soil evaluation should determine the concentrations of contaminants, the physical and chemical nature of the contaminants, the lateral and vertical distribution of contaminants, and the geologic conditions at the site. All soil exposure pathways (except food chain for human exposure and inhalation for ecological exposure) are assumed to exist. Site characterization of soil should incorporate three objectives: (1) the evaluation of human health and ecological exposure scenarios, (2) the evaluation of soil leaching potential, and (3) the estimation of contaminant volume needing remediation.

Prior to conducting a soil investigation, the following data should, if possible, be compiled:

- **Identification of COPCs** (see Section 2.0). These will be based on historic land use and site activities. Samples should be collected from areas of visibly contaminated soil, soil near sumps, pits, drains, sewer pipes, process areas, and any other suspect areas identified in the site use evaluation.
- **Quantification of COPCs.** Select appropriate MDH, EPA, or other lab methods which will quantify COPCs and meet DQOs.
- Identify suspect areas of concern based on site history, site inspection, and/or limited analytical data.

5.6.1 Preliminary Investigations

5.6.1.1 Evaluation of Human and Ecological Exposure to Soil

For preliminary soil investigations, it is assumed that all common human and ecological soil exposure pathways exist. These pathways include inhalation (for humans only), dermal contact and ingestion, but do not include indirect exposure via the foodchain (for humans). A residential property use scenario is assumed. A preliminary sampling plan must conservatively assess human and ecological exposure risk relative to the appropriate standards or criteria. To adequately assess human and ecological exposure risk, soil sampling needs to address both the lateral and vertical distribution of contaminants.

For a preliminary evaluation of soils, adequate lateral spatial coverage is required. As long as desired DQOs are met, it is recommended that field screening or field analytical methods be used (XRF, immunoassay, mobile lab). In conjunction with lab samples, these field methods can provide better spatial coverage of a site at a lower cost. The number of lateral soil sampling locations will be determined by the surface area of a site and the presence of discrete areas of contamination (i.e., source areas). Guidelines for determining the number of sampling locations for sites *with no apparent discrete areas of soil contamination* are listed in Table 5E. Additional information on exposure areas and concentrations can be found in the MPCA *Risk-Based Evaluation for Soil - Human Health Pathway Guidance*.

Table 5E Recommended Minimum Preliminary Soil Sampling Density

Surface Area of Site	Number of Lateral Sample Locations
less than 2 acres	6 sample locations per 0.5 acre (12/acre)
2-5 acres	sample locations placed on 75' centers (~ 8 /acre)
5-40 acres	sample locations placed on 100' centers (~ 4 /acre)
40+ acres	sample locations placed on 130' centers (~ 3 /acre)

The sample locations can be determined using a grid or can be randomly spaced to cover the suspected contaminated area. More samples, in addition to the numbers listed above, may be required due to site-specific contamination or geologic conditions.

The suspected contaminated area is often an exposure area which is defined as the location of potential contact between a human or environmental receptor and a release of contaminants. Hot spots should be identified using field screening, visual, olfactory, and past and present property use data to target areas where releases are likely to have occurred. If discrete source areas (hot spots) are known or suspected, samples shall be collected and analyzed from three separate lateral locations within each hot spot. Sample locations are sited to attempt to quantify the maximum contaminant concentrations and provide adequate definition of the extent of contamination. These sample locations should be biased towards visual, olfactory, and screening observations as well as suspect areas based on past and present land use. Compositing of lateral soil samples and averaging of lateral soil sample analytical results are *not acceptable* in preliminary investigations.

Direct exposure also needs to be assessed in the vertical direction. Generally, when assessing the direct exposure pathway, averaging vertical contaminant concentrations is not allowed, unless analytical data show that contaminant concentrations are fairly homogeneous. The most relevant

interval for the direct exposure pathway is the top two to six inches of soil. However, deeper soils must also be evaluated because they may become exposed at a later date. The key is to evaluate each horizon or layer of contaminated soil.

For human exposure, it is assumed that the top four feet of soil is accessible, and four to twelve feet below grade is potentially accessible. Refer to the MPCA Guidance on Incorporation of Planned Property Use Into Site Decisions. Ecological exposure is assumed to be limited to the uppermost four feet of soil. These assumptions may need to be adjusted on a site-specific basis. Compositing of vertical soil samples *is not acceptable except under site specific circumstances that have been reviewed and approved by MPCA staff.* Averaging of vertical soil sample analytical results is also *not acceptable* in preliminary investigations. The following soil sampling guidelines are suggested:

- Collect surface (upper two- to six inches) samples.
- Collect a separate sample for each distinct soil horizon.
- For the vertical profile for human exposure, two separate intervals will be considered: 0-4 feet, and 4-12 feet. These intervals correspond to accessible and potentially accessible soils, respectively. A *worst case* sample shall be collected from both the accessible and potentially accessible depth intervals and submitted for laboratory analysis. Selection of samples may be based on the following:

  1. If a field screening or field analytical instrument is used (See Table 5A) field results may be good overall indicators as to whether or not worst-case contamination has been encountered.

  2. Visual and olfactory observations as well as past and present site use may provide good indications as to where to collect worst case samples

  3. Geologic observations can also direct worst case sampling under some circumstances. If VOCs are the target compounds and field screening does not yield any indications of contamination, collecting a soil sample at the upper surface of low permeability units such as clay or silt may be acceptable. However, keep in mind that certain contaminants saturate low porosity zones before being deflected around them and often highest concentrations, although potentially trapped, are found here.

- For the vertical profile for ecological exposure, the soil screening values apply only to the top four feet. Selection of samples should be based on the same three criteria listed above for human exposure.

5.6.1.2 The Evaluation of Soil Leaching Potential

In a preliminary investigation , levels of soil contaminants are compared with the Soil Leaching Values (SLVs) as an estimate of the likelihood for leaching potential. SLVs can be found in the MPCA Risk Based Guidance for the Soil Leaching Pathway Users Guide. Evaluation of each specific hot spot must be conducted using samples meeting the following sampling guidelines:

- Worst case samples shall be collected as discussed in Section 5.6.1.
- Samples shall include those collected above the capillary fringe.
- Log soil borings to establish geologic conditions at the site. Continuous sampling of soil borings is generally recommended.

5.6.2 Extensive Investigations

If contaminant levels exceed the appropriate standards or criteria and a cleanup is not conducted, a more extensive investigation is necessary. In extensive investigations, site specific inputs are used rather than generic defaults.

Extensive investigations require detailed characterization of surface and subsurface soil horizons, contaminant chemistry, and actual and potential receptors *sufficient for a site specific risk assessment.* In addition, site specific inputs are used to calculate site specific (Tier 2) SLVs to estimate of the likelihood for leaching potential. Information regarding the calculation of Tier 2 SLVs can be found in the MPCA *Risk-Based Guidance for the Soil Leaching Pathway User's Guide* (May 1998).

More flexibility with respect to remediation goals may be available if it can be demonstrated that the default standards and criteria cannot be met, and that no receptors will be adversely impacted by utilizing alternate compliance points, remediation time, or remediation strategy. In this case, site specific standards and criteria are generated using site specific data.

Extensive investigations may be conducted when:

- remediation of the site based on preliminary investigation results is not practicably feasible, or cost-effective; or
- default parameters do not adequately represent geologic conditions or exposure pathways at the site, for example, if complexities at the site involve heterogeneous, discontinuous soils, multiple source areas or contaminant streams, or unusual terrain (e.g., shallow buried bedrock valleys); or
- contaminants without specific standards or criteria are present; or
- a regulated party (RP or VP) believes site-specific data will ultimately result in a more protective or cost-effective investigation or remedy.

Extensive investigations may require:

- actual sampling of biota to determine if bioaccumulation or direct effects are occurring. This situation requires evaluation and approval by the MPCA staff and the Risk Assessors. Refer to the Risk-Based Evaluation for Soil - Human Health Pathway Guidance and the Risk-Based Evaluation for Ecological Receptors - Soil Pathway Guidance for additional guidance. The document entitled "EPA Ecological Risk Assessment Guidance for Superfund - Appendix B" (EPA 540-R-97-006) is one source for information on biota sampling; and
- use of bioassays, determination of site-specific bioavailability, population and community analyses.

In some site specific situations, averaging and compositing of samples may be acceptable. It is important to note that averaging is not acceptable for acutely toxic contaminants (see the MPCA *Risk-Based Evaluation for Soil - Human Health Pathway Guidance*). For certain pathways, specifically for human soil exposure and ecological soil food chain exposure, the 95% upper confidence limit (UCL) of the arithmetic mean should be calculated for the data set. This is to ensure that the average concentration used in risk calculations does not underestimate the exposure potential. The 95% UCL of the mean requires a minimum of 10 data points. The appropriate standards and criteria are generally applied to these averaged soil concentrations.

If compositing of samples is conducted and grid sampling is used, each grid square should be divided into four sub-areas for composite sampling. The composite concentration can then be applied to the grid square. If composite sampling is conducted without a grid, assign the composite concentration to the centroid of the polygon formed by the individual sample locations (no more than four). Averaging of composite sample data is not acceptable, since the composite samples are already representative of a physical average of the sub-samples. For more information on this topic, please refer to Section 5 (Data Collection and Evaluation) of the MPCA *Risk-Based Evaluation for Soil - Human Health Pathway Guidance*.

Certain site-specific soil data are required for the assessment of human health risks. Parameters such as soil moisture and total organic carbon should be analyzed. See the MPCA *Risk-Based Evaluation for Soil - Human Health Pathway Guidance* for additional information.. All laboratory method detection limits should be low enough so data can be used for risk evaluation purposes. In order to be used to evaluate risk, the data should also be representative of potential exposure scenarios.

6.0 SURFACE WATER SAMPLING

(To be added at a later date)

7.0 SAMPLING FOR REMEDIATION VERIFICATION

7.1 Introduction

Information presented in this section is intended to guide the environmental professional in the recommended methods for verifying that soil contamination has been adequately remediated. Primarily, the minimum number and the location of required samples are addressed.

Verification sampling strategies for soil remediation depend on the type of remediation -- excavation or in-situ treatment. The minimum number of samples and sampling locations are different for each remediation type. While the minimum number of samples required is easily determined for both situations, determining the sampling locations is more complex and requires some professional judgment. The sampling strategies are outlined below.

Ex-situ remedies may be amenable to statistical sampling strategies or batch sampling. Any proposed sampling for ex-situ remedies should be developed on a site by site basis with the oversight of the MPCA project staff.

7.2 Excavations

Verifying that contaminated soil has been remediated by means of excavation requires samples from the excavation floors and sidewalls. The tables below provide the minimum number of samples necessary to verify cleanup for various sizes of excavations. Remediation verification is demonstrated by comparing the analytical results from each sampling point with the cleanup goals. If the cleanup goals are exceeded at any point, this verification methodology may require additional excavation at that point until the goals are met. Specifically, if less than ten samples are collected from either excavation floors or sidewalls, the calculated average concentrations will have very little meaning from a risk standpoint. In these situations, the appropriate risk/cleanup standards should be considered as numbers that are not to be exceeded in any sample.

A sampling strategy that uses bias to choose sample locations is recommended. This guidance document cannot dictate the exact locations for sample collection using this strategy. The location of the sample collection points relies on site specific information from the remedial investigation, analysis of the release or contaminant distribution and the soil types encountered in the excavation. Sampling and analyzing the soil samples from the locations most likely to have contaminants can minimize the number of samples needed to verify that remediation is complete. Since professional judgment and site specific knowledge are required for selecting sampling locations, the rationale used to select these locations must be well documented in the implementation report.

Analysis of data generated by prior investigations at the site should yield information for the verification analysis. The field personnel present during the remediation should be sufficiently familiar with the conditions on site to implement an appropriate verification sampling plan. Soil verification sampling should incorporate all pertinent biases of a site which may include, but are not limited to, the following:

- preferential pathways of contaminant migration
- source areas, stained soils, other site specific "clues" (e.g., fractures in clays)
- changes in soil characteristics (e.g., sand/clay interfaces)
- soil types and characteristics.

Compositing soil samples for verifying soil remediation may be acceptable for non-volatile parameters. Generally, when sampling for non-volatile parameters, each composite sample to be analyzed may be comprised of a maximum of four subsamples. However, please be aware that if contamination is indicated in a composited sample at levels above the cleanup goal, the entire area of the excavation comprising the composite sample may require additional excavation until the cleanup goals are met. Suspected contaminated areas discovered during verification sampling should not be sampled as part of a composite but should be sampled discretely.

The minimum required number of verification samples is determined by the subsequent tables. Confirmation sampling should generally be conducted on a grid.

7.2.1 Excavation Floor

The minimum acceptable number of floor samples to be analyzed is based on the area of the excavation floor as designated in Table 7A shown below.

Table 7A Excavation Floor Samples

Area of Floor (sq ft)	Number of Samples
<500	2
500-<1,000	3
1,000-<1,500	4
1,500-<2,500	5
2,500-<4,000	6
4,000-<6,000	7
6,000-<8,500	8
8,500-<10,890 (0.25 acres)	9
>10,890	Use Guidance Below

The following guidance is to be used when excavation floor areas exceed 10,890 square feet:

Floor Acreage	Square Feet	Grid Interval
0.25 - 3.0	10,890-130,680	15 - 30 Feet
3.0 and over	130,680 +	30 Feet plus

7.2.2 Excavation Sidewalls

Sidewall samples are required to verify that the horizontal extent of the soil contamination has been remediated. The number of sidewall samples shall be determined by Table 7B shown below. In no case is less than one sample on each sidewall acceptable. Known hot spots should be sampled separately. Once again, when sampling for non-volatile parameters, each sample to be analyzed may be comprised of four subsamples.

Table 7B Excavation Sidewall Samples

Area of Sidewall (sq ft)	Number of Samples
<500	4
500-1,000	5
1,000-1,500	6
1,500-2,000	7
2,000-3,000	8
3,000-4,000	9
>4,000	1 sample per 45 lineal feet of sidewall

When sampling the sidewalls of excavations that exceed five feet in depth, the sidewall sampling locations must be staggered in the vertical plane. This will ensure that lateral remediation has been adequate at all depths within the excavation.

7.3 Soil Stockpiles

Often times an excavation results in a contaminated soil stockpile that then needs to be treated (on- or off-site) or sent off-site for appropriate disposal. Sampling of the stockpile is necessary in order to characterize the contaminated or treated soil and to determine the appropriate final disposition. Landfills and the various types of treatment facilities (such as thermal treatment facilities or land farm sites) have permitted limits on the levels of contaminants they can accept. Sampling is necessary to ensure receiving facilities are operating within their permit limits. Additional samples beyond what is recommended here may be necessary based on each facility's specific permit requirements. TCLP and/or total analyses should be conducted for each type of contaminant suspected to be present. The detection limits for the total analyses should be determined based on the requirements of the receiving facilities permit, or on the cleanup level established for the site. The following table shall be used to determine the appropriate number of stockpile samples to be collected for analyses.

Table 7C Stockpile Samples

Cubic Yards of Soil in Pile	Number of Samples
0-500	1 per 100 cubic yards
501- 1000	1 per 250 cubic yards
1001 or more	1 per 500 cubic yards

If less than ten samples are collected from a stockpile, a calculated average concentration will have very little meaning from a risk standpoint. Therefore, in this type of situation, the appropriate risk/cleanup standards should be considered as numbers that are not to be exceeded in any sample. Compositing of stockpile samples is acceptable for the non-volatile parameters. Each sample may be comprised of four subsamples collected randomly from within the stockpile.

7.4 In-Situ Soil Remediation

When in-situ remedies are used, the effectiveness of the remedy must be verified by soil sampling. In these cases, three-dimensional sampling must be undertaken to verify that the soils have been adequately treated.

In instances of in-situ stabilization, the sampling should be conducted using a grid pattern with a vertical component added at each node. The number of samples collected for analyses should be determined using Tables 7A and 7B. The vertical extent of the remedy should be determined by compositing samples within each grid over 10 foot depth intervals extending to the bottom of the stabilization zone.

For in-situ treatment such as soil vapor extraction (SVE), the number of samples collected for analyses should be determined using Tables 7A and 7B, but should be biased toward the sampling points located remote from the SVE points. The vertical component must also be addressed and, therefore, the soil borings should be screened continuously using a PID, and any soils showing elevated organic vapors should be sampled. If no elevated PID readings are detected, discrete samples should be collected at 5 foot intervals over the depth of the treatment zone.

Compositing of remediation verification samples is acceptable for in-situ remediations for the non-volatile parameters. Each sample may be comprised of no more than 4 subsamples.

8.0 AIR MONITORING AND SAMPLING

8.1 Introduction

Air is an important medium to sample at many contaminated sites, and may be a cause for concern due to potential receptor exposure. Exposure to airborne contamination may occur as a result of migrating particulates or vapors. The presence of bare contaminated soil at a site can easily lead to the generation of airborne contaminated particulate material or vapors. Even subsurface soil (or ground water) contamination can generate vapors that may present risks to receptors.

Collection of air quality data may be required in two different ways at contaminated sites: monitoring and sampling. Monitoring of airborne contaminants is recommended in the early stages of site investigations, and may also be necessary during subsequent investigation and cleanup procedures. Monitoring involves the use of equipment that provides a direct readout of the results, although the results are usually not compound-specific. Examples of this type of equipment include: combustible gas indicators, oxygen monitors, and photoionization detectors. It is important to note that these types of monitoring devices are useful for directing sampling activities, and for evaluating potential exposure or unsafe situations for investigation/remediation personnel. These instruments are not sensitive enough to evaluate (long-term) exposure risks for the general public. Two general types of monitoring are often used during investigations: personal (breathing zone) and area (portion of site). In most cases, monitoring is conducted on or adjacent to the site perimeter.

Sampling involves the use of various types of sampling pumps, containers and other devices. Air samples collected in this way are sent to a laboratory for analysis, and are compound-specific. This type of sampling can be quite complicated, and often requires personnel to collect background (upwind) samples and miscellaneous meteorological data (temperature, wind speed, barometric pressure, etc.). It is recommended that air sampling be planned and conducted by specialists who have a thorough understanding of air sampling theory and technology.

In addition to air monitoring and sampling, modeling of airborne contaminants can also be used to help evaluate potential risks via this pathway. Modeling is generally used for evaluating indoor air concentrations. Before a model is used, it is necessary to establish the validity of the model for conditions similar to those that may be encountered at the site. EPA staff is currently working on an Excel-based spreadsheet for modeling vapor intrusion into buildings. A draft model is currently available, and it is anticipated that a final version will be released in 1999, which will be accessible via the EPA website.

To assure that site sampling efforts provide adequate data for the risk assessment, the sampling and analysis plan should be developed in consultation with the MPCA Risk Assessor. Analytical data should be collected during an investigation to fully characterize the nature, extent, and severity of the contamination. Some or all of the data obtained may be used for the risk assessment. These data must be representative of actual and foreseeable exposures to be useful in assessing risk.

8.2 Air Monitoring

Some of the main objectives for air monitoring are to:

1. Identify and quantify airborne contaminants;
2. Evaluate the potential risk posed by the presence of airborne contaminants;
3. Determine level of worker protection needed;
4. Track changes in airborne contaminants that occur during the investigation, cleanup or incident;
5. Assist in defining work zones; and
6. Provide data to be used to protect on-site workers or off-site populations from exposure during remediation activities.

An important aspect of air monitoring is to select the equipment that is appropriate for the site. As with other media, the COPC must be determined based on available information. The COPC will help the investigator determine the appropriate monitoring equipment. Other factors pertaining to the equipment, such as selectivity, sensitivity, accuracy and precision will also aid in the selection of the proper monitoring equipment.

At the majority of sites, monitoring equipment will usually include combustible gas indicators, oxygen monitors, and photoionization detectors. At other sites, detector tubes and certain compound specific field kits may be used, depending on the types of contaminants.

Action levels for on-site investigation/remediation workers that are used during air monitoring are generally based on standards established by NIOSH, OSHA, or ACGIH. However, these standards do not apply to other human or ecological receptors. Action levels for these other potential receptors depend upon the type of contaminant and type of receptor, and may be much lower than the above-listed standards.

8.3 Air Sampling

In planning a sampling program, both sampling time and sampling duration are important to consider in obtaining representative samples. Seasonal changes, such as the depth to ground water, as well as daily changes in ambient air pressure, for example, may have significant effects on the areas to be sampled. For a long-term exposure evaluation (as opposed to an imminent hazard evaluation) sampling should be conducted several times a year. Unfortunately, air sampling is time consuming and expensive, and it is not always possible to obtain samples that fully reflect temporal variations in concentration.

Whenever possible, standard EPA methods should be employed. Nevertheless, it is important to evaluate the utility of a standard method relative to site-specific data requirements before sampling is initiated. Descriptions of air sampling methods are beyond the scope of this guidance. However, EPA-approved air sampling methods are described in the Federal Register, and may be accessed through the Internet at: **http://134.67.104.12/html/emtic/cfrprom.htm**.

If sampling is only to be done once or twice, due to resource limitations, it must be demonstrated that the concentrations will be highest at those specified sampling times. Factors such as depth to ground water,

soil type, climate, distribution of buildings, and pavement should be carefully evaluated in order to select sampling times and locations.

The sampling duration should correspond as closely as possible to the duration of the exposure being evaluated. Since sampling results are often used to evaluate subchronic exposures (longer than a few months) and chronic exposures (longer than a few years), it is important to make sampling durations as long as possible.

Sampling locations should include areas where concentrations are likely to be highest and areas where the frequency and duration of the exposure is high. For example, trenches and basements of buildings are areas where high concentrations of contaminants would be expected. Samples within a given area are likely to show substantial variability, thus it is advisable to collect samples from several different areas in order to evaluate potential exposure risks at a site.

9.0 SEDIMENT SAMPLING

9.1 Introduction

Sediments are generally fine-grained materials (silt and/or clay) deposited in surface water bodies or wetlands. Sediments may contain almost any type of relatively non-volatile contaminants, depending on the source of contamination, although the most common contaminants are PAHs, PCBs, metals, and pesticides. Sediments are generally deposited by gravitational settling from the water column or direct depositions from a sludge, slurry, or liquid. Since sediments occur in a subaqueous environment, they may move after deposition due to gravitational settling or resuspension and subsequent redeposition in flowing water.

9.2 Sediment Criteria

There are no laws or rules in Minnesota which establish strict sediment quality standards. The water quality rules (Minn. Rules, Chapter 7050) have a provision that sediment quality should not be allowed to degrade below levels which will impact benthic invertebrates or other biota. Sediment and criteria are based on sediment quality guidelines or standards developed in other states and Canada. A Sediment Ecological Screening Criteria table is provided in the forthcoming MPCA Sediment Assessment Guidance. The values provided in the table are taken to be threshold levels for sediment contamination at the Tier I and Tier 2 levels of investigation.

9.3 Detection Levels

Sediment ecological screening criteria are often considerably lower than SRVs. This may necessitate using analytic methods with lower detection levels. At a minimum, the detection levels must be lower than the sediment screening criteria . For some compounds, this may prove difficult without the use of special analytic procedures.

9.4 Sediment Sampling Methods

Sediment samples may be collected using ordinary soil sampling equipment or dredges specially designed for sediment sampling. Two types of commonly used dredges are the Ponar and Eckman dredges. The dredges generally collect larger volumes of sediments than soil sampling tools or corers. These dredges are useful for surficial sediment sampling where large sample volumes are necessary, for example when bioassays

will be performed. A useful reference for sediment sampling methods is the EPA *Sampling Protocols for Collecting Surface Water, Bed Sediment, Bivalves, and Fish for Priority Pollutant Analysis* (1982).

9.5 Sampling Density and Depth

The top two- to six inches of sediment is generally considered to be the portion of the sediment column which is available for exposure to ecological receptors. Samples from deeper in the sediment column are generally collected for estimates of contaminant volume for remediation or sediment management.

Sediments are generally fairly homogeneous in the type of contamination present, but may vary greatly over very small areas in the levels of contamination present. As a result, the sampling density should be based, in part, upon the variability observed at a site. Generally, the following minimum sampling density is recommended for Tier 1 sampling of sediments:

3 samples per hot spot or as a minimum for an area of contamination
< 2 acre sites: 3 samples/acre
2 to 5 acre sites: sample on 150 foot centers
> 5 acre sites: sample on 200 foot centers

Grid sampling is generally recommended once the nature and approximate extent of contamination has been delineated through visual or field screening methods. One or more transects in and across the direction of sediment transport may be useful for preliminary estimation of the extent of contamination. It may also be necessary to identify the contaminant gradient and sample along it for Tier 3 bioassays or invertebrate community evaluations.

9.6 Field Screening Methods

Contaminated sediments are often readily identified visually. A preliminary estimate of the extent of sediment contamination can often be completed at low cost by visual examination of auger or dredge samples.

Ultraviolet fluorescence has been used experimentally as a semiquantitative method for PAH in sediment analysis. This method requires calibration against samples from the site analyzed through laboratory methods. This makes it more suitable for large sites where developing a calibration curve is practical and cost-effective. UV Fluorescence may also be used as a rough qualitative tool without a calibration curve.

Immunoassay kits similar to those used for soils are available for semiquantitative sediment analysis. Analytes for which these kits may be used include PCBs, PAHs, and pesticides.

XRF analysis may be suitable for field screening for metals in sediments. Ten percent lab duplicates are generally necessary for quality control. Sediment samples will generally require drying prior to XRF analysis.

Current research has shown that laboratory analysis of acid volatile sulfide (AVS) in sediments can be a valuable indicator of the bioavailability of metals when used in coordination with sediment toxicity studies. For further information consult the MPCA ecological risk assessor or current literature on the subject.

9.7 Analytes and Analytic Methods for Sediment Sampling

Since sediment contaminants are generally restricted to dense and persistent compounds, the number of potential contaminants is more restricted than for soils. The source of contamination is generally known before a sediment sampling program is initiated, helping reduce the list of analytes for most sites. Table 2A in Section 2.1.1 is a general list of contaminants present at different types of sites. This can be used a s preliminary list of analytes for sediment sampling. At the Tier 2 and 3 levels, nonionic organic contaminant concentrations are often normalized to TOC, to allow for site-specific bioavailability differences, necessitating TOC analyses for some sediment sampling programs.

Analytic methods for sediments are generally similar to those for soils. Sediments may require drying or ashing prior to analysis, depending on the analytes, and may require different extractions than the comparable soil method. Lower detection levels are also often necessary for sediment than the routine soil methods. Sediments may also be analyzed directly for their toxicity on ecological receptors through bioassays or other direct measures of toxicity rather than extrapolating from laboratory determined contaminant levels to theoretical toxicities.

Divalent metals bioavailability and toxicity may also be estimated using AVS and simultaneously extracted metals (SEM). Sediment particle size is another useful parameter to aid in interpretation of contaminant data.

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APPENDIX 1: MOBILE LABORATORY QA/QC REQUIREMENTS

GUIDANCE DOCUMENT

QUALITY ASSURANCE / QUALITY CONTROL PLAN

OUTLINE FOR A MOBILE LABORATORY

Prepared by the Minnesota Department of Agriculture

and

the Minnesota Pollution Control Agency

Use of this guidance document.

The project manager/project team assigned to the project, must determine the scope of the project and the data quality objectives for the project prior to deciding to use a mobile laboratory on a specific site. Consultants should be sure their use of the mobile laboratory meets the requirements of the state and federal programs/guidelines for which they are completing work. The level of Quality Assurance documentation (e.g. standard operating procedures, quality control plan) must at a minimum, be complete enough to give the project staff data of a quality that meets the data quality objectives (DQO) for the site. For some screening analyses somewhat less stringent quality assurance may be acceptable, whereas for some very involved analyses even more stringent quality assurance may be required.

This document lists the general standard that must be present in the quality assurance / quality control plan for all mobile laboratories submitting data to the Minnesota Pollution Control Agency and the Minnesota Department of Agriculture Agronomy and Plant Protection Division.

Table of Contents

1.0 INTRODUCTION TO MOBILE LABORATORY

This section should give a general introduction to the laboratory and the different kinds of analyses performed on the premises.

1.1 Mission statement of the mobile laboratory;
1.2 Quality assurance policy of the mobile laboratory;
1.3 Size of the laboratory;
  1.3.1 Dimensions and layout
  1.3.2 Equipment list (major items)
1.4 Definition of terms; and,
1.5 Lab certifications.

2.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

This section should introduce the reader to the different key personnel in the Laboratory.

2.1 Organization chart (This includes the company as well as the mobile lab);
2.2 Description of lines of communication;
2.3 Work units in laboratory; and,
2.4 Brief description of key positions

3.0 SAMPLE CUSTODY

This section should completely describe the procedure from the receipt of the samples until the samples are disposed of (cradle to grave). (Note: Standard Operating Procedures (SOPs) may be referenced where applicable.)

3.1 Sample receipt policies;
3.2 Sample log-in;
3.3 Example laboratory chain of custody (COC);
3.4 Condition of sample upon receipt;
3.5 Sample storage, preservation and contamination controls;
3.6 Tracking of samples;
3.7 Evidence files (for legal samples); and,
3.8 Sample disposal.

4.0 CALIBRATION PROCEDURES AND FREQUENCIES

This section shall describe the procedures used by the lab to calibrate instrumentation and equipment, including balances, in the lab. SOPs may be referred to (where appropriate). All applicable analyses to the site must be discussed.

4.1 Frequency of Calibration of All Instruments;
- 4.1.1 3 to 5 points for the calibration curves. This does not include the method blank.
- 4.1.2 Type of curve(s)

4.2 Criteria for acceptance of calibration;

4.3 Updating and verification of calibrations;
- 4.3.1 Continuing calibration verification standards
- 4.3.2 Continuing calibration blanks (frequency, required for continued analysis)
- 4.3.3 Frequency of updates of curves

4.4 Records of calibration for instruments; and,

4.5 Standards
- 4.5.1 Expiration dates
- 4.5.2 Testing for purity and validation
- 4.5.3 Records of receipt and tracking
- 4.5.4 Disposal of unused standards

5.0 INTERNAL QUALITY CONTROL CHECKS

The mobile laboratory shall describe in detail all Quality Assurance / Quality Control (QA/QC) practices that are used in the laboratory. It is recommended that a flow chart showing from sample receipt to report generation, be included to give a visual picture of the path taken by a sample and the QA/QC association with the sample. The items listed below describe some of the parameters associated with the Internal Quality in a laboratory. This list is not intended to be conclusive as to all the QA/QC a laboratory performs.

The limits associated with specific parameters and how they are developed must be described by the laboratory. Control Charting and any other method of tracking the limits must also be included. Refer to Section 9.

5.1 10% matrix spikes and matrix spike duplicates and/or duplicates with a minimum of once per sample set;

5.2 Lab control samples;

5.3 Surrogates and internal standards, (where appropriate);

5.4 Blanks (equipment, field, reagent, instrument and refrigerator);

5.5 10% method blanks;

5.6 Zero and span gases, mass spec tuning if an MS is used onsite;

5.7 Proficiency testing of analysts and method;

5.8 Sample preservation and holding times;

5.9 Reagent storage and purity; and,

5.10 Glassware cleaning.

6.0 DATA REDUCTION, VALIDATION, AND REPORTING

The laboratory will describe, in detail, the in-house data reduction and validation procedures. It is strongly recommended that someone besides the analyst review all raw data and final reports that are generated in the analytical process. Any SOPs associated with these procedures should be referenced. The review process taken by an analyst/filed chemist to report data must include:

6.1 Procedures for re-running a sample;
6.2 A description of the different flags and procedures for flagging data;
6.3 Use of spikes and duplicates in assessing data;
6.4 Use of blanks in assessing data;
6.5 Use of laboratory control samples in assessing data;
6.6 Use of surrogates in assessing data
6.7 Data reporting format;
6.8 Assessing if data meets reporting limits for project;
6.9 Questionable sample condition;
6.10 Holding times; and,
6.11 Practical quantitation limits.

7.0 PERFORMANCE AND SYSTEM AUDITS

The laboratory should have a policy of internal audits to verify the QA/QC plan is being followed. The audit should include an examination of the sample receipt documentation, sample log-in, sample storage, chain of custody procedures, sample preparation and analysis, instrument operating records, etc.

External audits performed on the mobile laboratory should be discussed as to who performs them, who has performed them, what will be and has been audited, and the results of these audits. Blind QC samples should be submitted to the lab to verify system performance.

8.0 PREVENTATIVE MAINTENANCE

The laboratory will describe routine preventative maintenance program used to minimize equipment failure and breakdown. Trained staff should be on the premises to repair equipment and/or a contract be in place with a vendor to do so in a timely manner. All maintenance performed on the equipment shall be recorded in individual books that are kept with the instrument. The lab shall submit a table of its instrumentation and all preventive maintenance regularly performed.

9.0 ROUTINE PROCEDURES TO ASSESS DATA QUALITY & DETERMINE REPORTING LIMITS

The procedures that are used by the laboratory to assess data shall be annotated.

9.1 Precision;
9.2 Accuracy;
9.3 Representativeness;
9.4 Completeness;
9.5 Reporting Limits; and, (These must be explained on how they were derived.)
  9.5.1 Instrument Detection Limits (IDLs)
  9.5.2 Method Detection Limits (MDLs)
  9.5.3 Practical Quantitation Limits (PQLs) or Reporting Limits (RLs)
9.6 A method detection level study will be run onsite prior to samples being run.

10.0 CORRECTIVE ACTION

Corrective action may be required for instruments or in the analytical process. The laboratory must list common problems associated with corrective action and corresponding actions taken by the analysts to correct the situation. If the corrective action of the analyst can not correct the problem there must be a procedure in place for informing management and the QA/QC Officer. The procedures that management will take must be listed. All corrective action taken must be documented on appropriate forms and in the maintenance book for the specific instrument (when applicable).

11.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

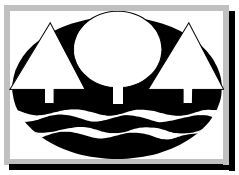
On long term projects, the laboratory will normally submit quality assurance reports to the Contractor's Project Manager (and to the state liaison, upon request). The report should include:

11.1 Any changes or modifications to the QA/QC Plan;
11.2 Any changes to any of the standard operating procedures;
11.3 Any significant QA/QC problems and recommended solutions;
11.4 Results of corrective action;
11.5 Any limits that shall be imposed on data;
11.6 Samples received in questionable condition;
11.7 Holding times that have been missed;
11.8 Management changes that affect work done for the State of Minnesota; and,
11.9 Any other issues that will affect the project.

12.0 FILE HANDLING AND STORAGE

This section shall describe the procedures used by the laboratory to file data for immediate and long term storage. Discussion of longevity of files and data, Laboratory Information Management System (LIMS) backups, and any other items applicable can be included.

APPENDIX 2: SAMPLING PROTOCOL TEMPLATE FOR MONITORING WELLS



Superfund and Voluntary Investigation and Cleanup Programs Minnesota Pollution Control Agency

Sampling Protocol Template For Monitoring Wells

FOREWORD

When to Use a Sampling Protocol

A sampling protocol is a description of the equipment and methodologies used in the collection of samples at a given site for a defined long term project. The goals of a protocol are to help maintain a high level of consistency in sampling methods for the duration of the project or operable unit. Protocols are not required at all sites. They are most useful at sites where:

- multiple sampling events take place over a long period of time (years);

- multiple consulting firms may be used during the duration of the project; and

- samples are used for monitoring changes in concentrations close to project action levels.

An example of such a site would be a project involving long term monitoring of monitoring wells located between drinking water wells and a release source where the concentrations in the monitoring wells are close to levels set for a response action (activation of a ground water containment system, initiate alternative water supply, etc.). The goal of the protocol would be to ensure that the sampling methods would produce a higher level of data consistency if the consultants or MPCA staff change during the project. Having a sampling protocol for a site does not limit the sampling options, but does require an informed decision be made when changing the sampling methods due to advances in technology or to reflect changes in the data.

Protocols do not take the place of sampling plans. For a site with a sampling protocol in force, sampling plans detail the schedule, specific parameters and changes in equipment or methodologies for a sampling event or short term series of sampling events. Sampling plans may reference the site sampling protocol for economy of preparation and review time of the plans.

USE OF TEMPLATE

The purpose of this sampling protocol template is to provide an example of the level of detail generally required by MPCA staff and to expedite preparation of a sampling protocol. Use of the template is recommended, but not required.

This Sampling Protocol Template has been designed to be used as a component of, and in concert with, the complete "MPCA Ground Water Sampling Guidance Document, Development of Sampling Plans, Protocols and Reports, 1995" (Guidance Document). This Sampling Protocol Template is a flexible template and requires modification to become suitable for application to a site. Refer to Chapter 3 of the Guidance Document and the "Instructions" that follow. Your site-specific *data quality objectives (DQOs)* should play a major role in determining how you modify the Sampling Protocol Template. DQOs are defined in the Guidance Document. *See Chapter 2, Table 1 from the Guidance Document for DQO criteria* . Topics in Chapters Four and Five of the Guidance Document are presented parallel to the structure of the Sampling Protocol Template The parallel structure is intended to assist the user in cross-referencing technical background information while customizing the Sampling Protocol Template.

TEMPLATE COPIES

This Sampling Protocol Template will be available for copying on diskette in the MPCA library to facilitate modification with word processing software. It is also available on diskette for site-specific application. Forward the request to MPCA, support staff by calling (651) 296-7291. The requester shall provide a formatted disk with a return self-addressed, stamped disk envelope for duplication of the protocol. Until further notice, there will be no charge for copying the protocol to the diskette. Initially, a copy will be available in Microsoft® Word for Windows version 6.0. In the future, copies may be available in other formats or available for electronic transfer via modem.

This Sampling Protocol Template must be edited before it can be applied to an actual site. This section provides instructions on how to customize the Sampling Protocol Template for site-specific needs. More general, but important guidance on using this Sampling Protocol Template can be found in Chapter Three of the main body of the Guidance Document.

This Sampling Protocol Template was designed to assist user's with the creation of scenario-specific protocols. Private organizations and individuals can use this template to create a site and/or event specific protocol.

HOW TO EDIT THE SAMPLING PROTOCOL TEMPLATE

Obtain a electronic copy of this Sampling Protocol Template from the sources listed previously. The Sampling Protocol Template is designed to be easily edited and customized into a scenario-specific protocol. Square brackets "**[ ]**" are used to indicate where scenario-specific choices must be made, while braces "**{ }**" are used to indicate editorial comments. Simply fill in the information requested or choose from the options listed within the brackets.

Refer to the technical guidance presented in Chapters Four and Five of the Guidance Document for assistance in evaluating proposed modifications.

[?]: When brackets appear around a question mark, enter the necessary site- or event-specific information, or enter additional detail if appropriate.

l. Begin editing the Sampling Protocol Template. The editing should reflect the sampling objectives stated in the Sampling Plan. Do not delete the Table of Contents; it should be retained in the final protocol. Do not delete the editorial comments in advance; they should be read as you are editing each section. It is normally best to begin by customizing only the tables and appendices that include the lists of analytes (parameters) to be analyzed. Then proceed to the main body of the protocol.

IMPORTANT: Changing text in one part of the document, even when it is associated with brackets "**[ ]**", may require additional changes in related text located in other parts of the document. To avoid overusing the brackets, (subsequent) related text is often unmarked. When any text is changed, use the Table of Contents or use the "search" (or "find") function of your word processor to locate other occurrences of key words for modification or deletion. For example, when changing the word "pump" to "bailer", use "search" to locate other places where obvious changes are required.

2. When you have finished customizing your program- or site-specific sampling protocol, use a word processing "search" function to look for all "**{ }**"s and "**[ ]**"s to ensure that all of them were found.

All brackets "**[ ]**", braces "**{ }**" and editorial comments must be removed from the actual (final) protocol before use in the field. Specific choices must be selected from the alternatives located within or adjacent to the "**[ ]**"s.

3. Next delete the "Foreword" and the "Instructions" (if included on the diskette).

4. Finish editing any of the appendices, tables and text that were not customized earlier.

REVIEW/APPROVAL

NOTE: MPCA Section staff may request that significant, proposed changes to protocols be marked to facilitate review and approval. Consider highlighting or otherwise marking all proposed changes or deletions for reviewers/approvers. Word-processing software can make this entire process very easy. Software such as Microsoft® Word includes "revision" features that can automatically mark all proposed deletions and additions to an original document. "Annotations" can also be imbedded within the text to provide explanations for the proposed changes.

Two types of alterations can be made that are not considered changes:

deleting an editorial comment along with the braces that enclose it after final approval

deleting alternate text within brackets.

Sampling Protocol Template For Monitoring Wells

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Superfund and Voluntary Investigation and Cleanup Programs

Sampling Protocol Template

For Monitoring Wells

1.0 INTRODUCTION

This document outlines procedures to be used for ground water quality measurements and for collecting and handling ground water samples obtained from monitoring wells at **[fill in site name]** during the time period **[specify sampling dates]**. Deviations from these procedures may be required by unforeseen circumstances that develop during the sampling event(s). Such deviations will be approved by the lead technical staff or the field crew leader as described below. When regulatory or lead technical staff approvals cannot be obtained in advance, deviations from the established procedures will be evaluated as soon as possible after sampling and the need for re-sampling will be evaluated. Deviations from the specified procedures will be clearly noted on the [**sampling information form (SIF) or field logbook**] used for the sampling of each well and will be included in the Sampling and Analysis Report.

2.0 ADVANCE PREPARATION FOR SAMPLING

{*For technical guidance, refer to page 37 of the MPCA Ground Water Sampling Guidance Document (Guidance Document): Chapter Four, Section 4.2:* "ADVANCE PREPARATION FOR SAMPLING"**}**

Selection of analytical parameters, laboratory arrangements, the order of sampling wells, field measurement and sampling techniques, equipment selection and other quality assurance measures are based on the sampling objectives presented in the main body of the Sampling and Analysis Plan.

2.1 Selection Of Analytical Parameters

Samples will be collected for analysis of the parameters shown in Appendix **[**1**]** to fulfill requirements of the MPCA Superfund and/or Voluntary Investigation and Cleanup (VIC) programs. ***{****In order to create this appendix, edit (customize) Appendix B from the main body of the Guidance Document or insert a table of parameters required by the Superfund or VIC Programs. . Indicate what analytical method and reporting limit will apply for each parameter.****}*** Samples will be collected and analyzed for the parameters from the wells listed in Table 3.

Analytical techniques for trace metals and organic compounds were selected primarily on the basis of ability to [detect potential contaminants at low levels; positively identify contaminants detected as opposed to achieving the lowest detection levels].

2.2 Detection Limits

Practical quantitation limits are listed in the project specific QAPP and in Appendix A.

2.3 Quality Assurance For Field Procedures

Particular care will be exercised to avoid the following common ways in which cross contamination or background contamination may compromise ground water samples:

- improper storage or transportation of equipment
- contaminating the equipment or sample bottles on site by setting them on or near or downwind of potential contamination sources such as uncovered ground, a contaminated vehicle, or vehicle or generator exhaust
- handling bottles or equipment with dirty hands or gloves
- inadequate cleaning of well purging or sampling devices

Field methods quality assurance verification procedures are described below in Section 4.4, "Field Blanks, Replicates and Split Samples". Field personnel should work under the assumption that contamination exists in land surface, soil and vegetation near sampling points, wash water, etc. Therefore, exposure to these media will be minimized by taking at least the following precautions:

- minimizing the amount of rinse water left on washed materials
- minimizing the time sampling containers are exposed to airborne dust or volatile contaminants in ambient air
- placing equipment on clean, ground-covering materials instead of on the land surface

Clean gloves made of appropriately inert material will be worn by all field crew. Gloves will be kept clean while handling sampling-related materials. The gloves will be replaced by a new pair between each sampling site.

2.4 Sampling Containers And Preservatives

[**Laboratory-supplied or contractor purchased**] sampling containers and preservatives to be used for samples from all wells are shown in Table 2. The Laboratory Quality Assurance Project Plan (Lab-QAPjP) includes specific procedures for the following: sample container cleaning, testing, labeling and storage; preparation and addition of preservatives. Preservatives for volatile organic samples are added to the sample container in the field. Chemical preservatives for all other parameters are added in the [**laboratory, field**] before samples are collected.

2.5 Purging And Sampling Equipment

Well purging and sampling equipment includes the following:

- [list the equipment used including the pump name and model, if applicable]]
- pump discharge lines, if applicable: [new or decontaminated tubing type]
- regulators and compressed nitrogen [air] tanks
- [{list other equipment such as}rope, other pumps, generators, air compressors (with air/oil filter), etc.]

2.6 Decontamination, Storage And Transport Of Equipment

{*When practical, the following alternatives can substantially reduce the time spent on field decontamination and may result in considerably less opportunity for cross-contamination: 1) permanently installing sampling pumps and tubing, 2) discarding bailer and bailer line or dedicating bailer and bailer line to individual wells, 3) discarding pump tubing or dedicating tubing to individual wells (place in labeled plastic bag for next sampling event), 4) for bladder pumps: discarding bladder or dedicating bladder to individual wells, 5)discarding other sampling related equipment such as filtration devices, personal protection gear and materials coming in contact with actual sampling equipment or personnel. If one of these approaches is to be used, specify such here. Delete field decontamination steps that have become unnecessary due to the change in approach. Replace these field decontamination steps by specifying "laboratory" or manufacturer's decontamination procedures used to prepare equipment for the field. For convenience, the "laboratory" or manufacturer's procedures may be specified by reference to an appendix attached to this protocol.****}***

[**(New or decontaminated) pump tubing will be used each time each well is sampled; Tubing will be dedicated to a single well for subsequent sampling events. Between sampling events, the tubing will be stored in a sealed, chemically inert plastic bag. The bag will be labeled with the well name and stored in a secure, clean location.]** Pump bladders will be **[discarded after use at each well; dedicated, labeled, and stored in the same manner as tubing; decontaminated by circulating decontamination fluids through the pump as described below].**

All sampling-related equipment including filtration devices, personal protection gear and materials coming into contact with actual sampling equipment or with sampling personnel will be decontaminated. ***{****If using bailers or sampling pumps and tubing that are permanently installed or dedicated to individual wells, state that they are exempt from <u>field</u> decontamination.****}*** Decontamination will be performed [**before, between or after**] working at each sampling point, ***{****Here specify where decontamination will be performed for all equipment:****}*** **[....in a lab or controlled "clean" room, at a decontamination station in the field; at each individual sampling point in the field**.] All equipment will be handled in a manner that will minimize cross-contamination between wells and avoid introducing surface contamination or ambient air contamination into a well.

Before mobilizing for field work or performing any decontamination, a source of "control" water and organic-free deionized water for decontamination will be selected and evaluated. The evaluation process will include sufficient laboratory analysis to assess the suitability of the proposed water. The proposed decontamination water will only be used for decontamination if analyses indicate it is appropriate for the complete set of target parameters. In the event that use of a desorbing agent is necessary, the desorbing agent will be **[*list type of desorbing agent*** made from reagent grade components and deionized water . Examples of organic desorbing agents are **isopropanol acetone methanol**. Examples of inorganic desorbing agents are a 10% nitric or hydrochloric acid solution made with reagent grade acid and deionized water. Equipment will be decontaminated in the following manner:

[**Modify decontamination procedure listed below to suit site specific needs**] {Additional examples of decontamination procedures can be found in Chapter Four, Section 4.2 Pages 57-59 of the Guidance Document.}

Equipment that does <u>not</u> contact sample water or the inside of the well
clean (inside and out where possible) with a hot water pressure washer filled with clean water
[clean (inside and out) with an Alconox/clean-water solution - applied with a scrub brush where practical**]**
rinse with clean control water

inspect for remaining particles or surface film and repeat cleaning and rinse procedures if necessary

Equipment that contacts sample water or the inside of the well
clean (inside and out where possible) with an Alconox/clean-water solution - applied with a scrub brush made of inert materials
rinse with clean "control" water
inspect for remaining particles or surface film and repeat cleaning and rinse procedures if necessary ***{If the sampling objective is only to obtain a gross, qualitative evaluation of contamination, the above procedures may be sufficient. If parameter-specific evaluation at trace level concentrations is necessary, addition of procedures #6 and #7 are suggested to meet high DQOs.}***

{The following procedure may be necessary when gross levels of contamination exist:} [rinse with an inorganic desorbing agent] ***{****delete if samples will* ***not*** *be analyzed for inorganic chemicals.}* *[*organic desorbing agent] ***{****delete if samples will* ***not*** *be analyzed for organic chemicals}* *{Note: use of desorbing agents requires pre-approval by MPCA Site Remediation staff.}*

rinse with clean "control" water
rinse thoroughly with laboratory controlled deionized water
shake off remaining water [and allow to air dry]

The internal surfaces of pumps **{***here specify any other equipment that should be decontaminated internally***}** and tubing that cannot be adequately cleaned by the above methods alone will also be cleaned by circulating decontamination fluids through them. The fluids will be circulated through this equipment in the order shown above under "B".

Wastewater from well purging and equipment cleaning will be [containerized on-site until analytical results are obtained to determine proper disposal][sewered on-site after sampling][disposed of on-site on the ground surface within the zone of contamination after sampling]. Disposable personal protective and sampling equipment will be containerized on-site [for disposal at a sanitary landfill].

When transporting or storing equipment after cleaning, the equipment will be protected in a manner that minimizes the potential for contamination. {Specify here how equipment will be protected.} The tubing will be placed in a clean, inert plastic bag. **{**Here specify other equipment to be wrapped in inert plastic or aluminum foil**}**.

2.7 Selection Of Sample Collection Techniques

Sample collection techniques outlined in this document have been tailored to the goals of this sampling event and the individual characteristics of this site. A summary of the sampling goals and the pertinent site, well and contaminant characteristics is given in the Sampling and Analysis Plan.

2.8 Order of Sampling

The ground water monitoring wells will be purged and sampled in the following order:

[**list the purging and sampling order here as well as on Table 2]**

{Refer to the Chapter Four, Section 4.3, Page 60 and Appendix A, Table A-2 of Guidance for a discussion of how to select a sampling sequence.}

3.0 PRELIMINARY FIELD WORK

{For technical guidance, refer to page 61 of the MPCA Ground Water Sampling Guidance Document: Chapter Four, Section 4.3: "PRELIMINARY FIELD WORK."**}**

[**List any necessary preliminary field work here**]

3.1 Field Inspections And Field Decisions

Before purging or sampling, all wells should be inspected to verify that:

- all sampling points are safely accessible;
- all wells are in satisfactory condition;
- current water levels indicate a gradient consistent with the preliminary order of well sampling;
- the existing health and safety plan procedures are appropriate for actual site conditions.

Any unusual conditions including the presence of wind-blown dust or odor in the ambient air should be recorded **[on a SIF or field log**].

- well depth and that the annular seal is intact at the surface.

3.2 Detection Of Immiscible Layers

Air inside a well suspected of significant contamination will be tested immediately with an organic vapor detecting device **[list type of device here]**. The measurement will be recorded on the [**SIF or field log book**]. If immiscible layers of contaminants (free product "floaters" or "sinkers") are suspected or if odors or an oil sheen are observed, procedures will be followed to characterize the distribution of contaminants in the water-yielding zone adjacent to the well screen. Because free product can accumulate anywhere from the top to the bottom of the water column, the normal sequence of purging and sampling will be preceded by a free-product evaluation step to allow for the best characterization of contamination. An attempt to measure the thickness of any free product will be made using the following equipment: **[list equipment here, e.g., an interface probe]**. General procedures for detection and sample collection of immiscible layers will be in accordance with guidance provided in U.S. EPA RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992, Section 7.2.3; specific detailed procedures actually used in response to site/well conditions will be recorded on the [**SIF or field logbook**] and included in the Sampling and Analysis Report. The presence of and characteristics of any detected immiscible layers will be noted on the [**SIF and field logbook**].

A bailer will be used to collect any pre-purging samples from the water table surface and a thief sampler will be used to collect any pre-purging discrete-interval samples from below the top of the water column. In addition to any discrete-interval samples collected, an additional sample will be collected from near the middle of the water column after normal purging. Analytical needs for these three samples will be reviewed with the [**Superfund or**

Voluntary Investigation and Cleanup Program technical representative] to determine which analyses are required for each sample. Visual screening or sequential analysis of samples may eliminate the need to analyze all samples collected in some circumstances.

3.3 Water-Level Measurements

Prior to any well evacuation or sampling, initial static water levels will be measured and recorded for all wells. This is done to facilitate selection of the proper pump intake depths for purging and sampling and calculation of the ground water flow direction.

During initial static water level measurement, a minimum of two water level measurements will be made at each well. The two water level measurements will be made in rapid succession. If there is poor agreement between the first and second static water level measurements (i.e., a difference of more than 0.01 feet), data will be re-evaluated for measurement errors, unsuspected pumping that may be causing transient changes in gradient, etc. If the discrepancy cannot be rectified, a third static water level measurement will be made at each questionable sampling point to assess the true water level, verify non-steady state conditions, etc.

The sampling crew will make water-level measurements at all appropriate monitoring wells and piezometers within the shortest time interval practical to provide comparable numbers by which to calculate the ground water gradient. A time limit exceeding **[list amount of time in hours]** will be considered a reportable protocol exception for this sampling event. An additional water level measurement will be taken immediately after sampling to evaluate potential cascading problems. These water levels will be entered on the [**SIF or field logbook**].

Water levels will be measured with a(n) [electronic water-level sensor probe; steel tape].

The depth-to-water should be referenced to the measuring point marked at the top of the innermost well casing. Where a measuring point has not been marked at the top of the casing, the measuring point will be assumed to be at the top of the innermost casing on the north side of the casing. When reporting absolute water level elevation, this measurement will be converted to water level elevation (MSL) from the surveyed elevation of the top of well casing. Water level measurement data will be recorded on the [**SIF or field logbook**

{Further information regarding water level measurements can be found in the Guidance, Chapter 4, Section 4.3, Page 62.}

3.4 Field Water-Quality Measurements

[**Specific conductance, pH, temperature, turbidity, or dissolved oxygen (redox potential)]**will be measured in the field immediately before sample collection. All measurements will be recorded on **[the SIF or fieldbook**]. Purging and stabilization information will also be noted on the [**SIF or logbook**].

{** Editorial note: Without use of a properly designed flow cell, procedures required to obtain meaningful field water quality data are much more complex. Acquisition of reliable data requires a thorough understanding of factors affecting field readings, extensive training, and a lot of care and patience. A limited amount of background information to facilitate the design of such procedures is provided in Chapter Four, Sec 4.3, page 64, "Measurements Without a Flow Cell". **If a flow cell is not used for any field water quality measurement, then the procedures outlined in Chapter. 4.3 must be detailed in this section.}

All measurements except for turbidity will be taken within a [**closed flow cell, other device**] designed to allow measurement of these parameters while minimizing changes in temperature, pressure, and dissolved gases from the in-situ aquifer environment. The flow cell has the following characteristics:

- Air tight fittings for installation of all probes.
- Intake is connected directly to the pump discharge line.
- Resides in a water bath kept at a temperature close to the in-situ ground water temperature.
- A discharge line at least 3 feet long that is connected to the flow cell with an air tight connection.
- A maximum volume of no greater than five times the per minute volumetric rate of inflow to the cell to maintain measurement sensitivity to temporal changes in water quality.
- A minimum volume of 500 ml to provide enough thermal mass to minimize external temperature effects.
- The flow cell will be shielded from strong winds and on hot days it will be shielded from direct sunlight.

The operation of the probes will be as follows:

1. The flow of extracted ground water through the flow cell will be maintained as continuous and steady as practical throughout the measurement period.
2. Discharge velocities through the flow cell are kept low enough to prevent streaming potential problems with probes.
3. All probes will be fully immersed without touching the sides of the air tight, non-metallic flow cell.
4. All probes will be allowed to equilibrate with fresh well water for five minutes before recording measurements.

Specific procedural details for measurement of individual field water quality parameters are outlined in the manufacturer's instruction/owner's manual. General care, maintenance, calibration procedures, and operation of each measurement device will also follow manufacturer's specifications as detailed in the instruction/owner's manual for each device.

3.5 Purging And Stabilization

Before a well is sampled for the dissolved phase, it will be evacuated to ensure that samples contain fresh formation water. While the well is being purged, water quality parameters described above in Section 3.4, "Field Water-Quality Measurements", and the quantity of water evacuated will be recorded on the [**SIF or field logbook**].

A purging rate that will minimize drawdown while still allowing the well to be purged in a reasonable length of time will be used and recorded on [**SIF** or logbook]. Care will be taken to avoid any significant amount of cascading or turbulence in the well.

Wells with extremely slow recharge rates due to tight formation materials, will require alternate purging and sampling methods. If normal purging is clearly impractical, the well will be pumped to near dryness and allowed to partially recover [insert estimate of time required] Sampling will then commence as soon as possible after evacuation. **{***The maximum reasonable time limit is one hour; however, data for sensitive parameters may be considered questionable unless sampling occurs even sooner after purging.***}**

Wells that do not have extremely slow recharge rates will be purged and sampled as described below. Purging will be conducted in a manner that, to the extent practical, removes all the "old" water in the well so it is replaced by fresh ground water from outside the well installation.

1. The well will be purged by withdrawing water from within [**list number of feet**] feet of the top of the water column.
2. Repeated vertical adjustment of the purging equipment intake may be necessary as the water level drops.
3. **[List type of equipment which will be used for purging and sampling].**
4. Sampling will immediately follow purging and stabilization .

Field water quality parameters will be measured for stabilization [**every 3 or 5 minutes**; after each water-column volume is purged]. The following target criteria for three consecutive measurements (every 3-5 minutes or one water-column volume apart) will be used to demonstrate stabilization:

- pH +/- 0.1 units
- temperature +/- 0.1 degrees Celsius
- specific conductance (temperature corrected EC) +/- 5%
- dissolved oxygen +/-0.5 mg/L **[**redox potential +/-20 mv**]**
- turbidity: less than or equal to 5 NTU **{**10 NTU may be acceptable when not sampling for sensitive parameters such as trace metals or trace organics.**}**

Samples for laboratory analysis will be collected only after a minimum of **[# of water column volumes**] water-column volumes have been purged and stabilization of field water-quality parameters has been demonstrated by meeting the target criteria defined in the preceding paragraph. If field parameters do not stabilize after approximately five water-column volumes, then field staff will check operator procedures, equipment functioning and well construction information for potential problems. In particular, field staff will re-evaluate whether or not water is being withdrawn from the appropriate depth to effectively evacuate the well.

If all the checks produce no new insight, a decision might be made to collect samples after five or more water-column volumes have been purged even if field measurements have not stabilized. Before authorizing the laboratory to analyze samples, the meaningfulness and value of completing laboratory analysis of the sampling suite will be evaluated by reviewing the results of field measurements, well construction data, site hydrogeology, etc. Where such data is presented, it will be clearly documented that stabilization was not achieved; at a minimum, this fact will be reported on the **[SIF or field logbook]** and in the Sampling and Analysis Report.

As with water from well development, purge water will be properly stored, tested, and disposed of in accordance with all applicable rules including Minnesota Rule 7060. Fifty-five gallon drums will be located at each of the wells to collect water removed from the wells during development or evacuation. No significant amount of well water will be emptied or discharged onto the ground surface unless analytical data are available and indicate that the water is not contaminated. After water analyses become available, and appropriate disposal alternatives are evaluated, the water will be disposed of in an environmentally safe manner that does not conflict with any applicable rules.

4.0 SAMPLE COLLECTION

{*For technical guidance, refer to page 75 of the MPCA Ground Water Sampling Guidance Document (Guidance Document): Chapter Four, Section 4.4:* "SAMPLE COLLECTION"**}**

This section describes procedures for setting the sampling pump and collecting ground water samples. Field data for these items will be recorded on the SIF for each sampling point.

4.1 Pump Setting

A **[insert actual sampling device to be used]** will be used as the default device for sample collection. If well recovery is so slow that a satisfactory water column height (for normal pump operation) is not reached in a reasonable amount of time, [**a zero submergence bladder pump or Teflon® bailer]** will be used for sample collection. The [**SIF or field logbook**] will show what type of **device** was used to sample each well. If any device other than the one described above is used, it should be reported as a protocol exception.

[In very slowly recharging wells, the pump intake will be set approximately two feet from the bottom of the well to minimize aeration problems] {*Note for alternate scenario where static water level is sufficiently above the top of the screen: the sampling pump intake should be set at approximately two feet above the top of the screen and at least two feet below the top of the water column.***}**

The same pump should be used for sampling as was used for purging. Pumping will be continuous and sampling will immediately follow purging. If pumping is not continuous it will be noted on the [**SIF or logbook**]. The sample collection pumping rate will be less than or equal to the purging rate. The sampling rate will be based on the purging and sampling rate test, and will not cause cascading or turbulence. The rate will be less than or equal to [**list rate here**].

4.2 Sample Filtration

Table 2 identifies which sample containers will be filled with sample water that has been filtered in the field. Sample filtration will be completed as follows:

1. The filter will be connected directly to the well sampling pump discharge line using positive pressure to force the sample through the filter.
2. From the filter, the flow will be routed directly into the sample collection container.
3. A **[insert pore size]** micron pore size filter will be used.
4. The flow rate will not exceed a rate that causes cascading or turbulence to occur.
5. Agitation and aeration of the sample will be minimized.
6. [**insert tubing type**] tubing will be used for the pump and filter discharge lines.

4.3 Filling Sample Containers

{*For technical guidance, refer to page 84 of the MPCA Ground Water Sampling Guidance Document: Chapter Four, Section 4.4 "Sample Collection"}*

Table 2 summarizes the sample container type, filling method, preservation method and holding time for each analytical parameter set. Individually prepared bottles will not be opened until they are to be filled with water samples.

1.

2. A clean and dry sheet of relatively inert plastic shall be placed on the ground surface in the wellhead area. If materials used in the sampling process must be put down, they will be placed on a clean portion of the plastic sheet instead of the ground surface.

3. A clean pair of **[ ]** ***{****specify glove type: appropriate gloves should be specified in the Health and Safety plan but should be made of material that will not contribute contaminants to sample containers****}*** gloves will be put on at the onset of sampling activities at each new sampling point.

4. Sampling personnel will keep their hands as clean as practical and replace gloves if they become soiled while performing sampling activities.

5.

Bottles will be labeled and chain-of-custody sections will be filled out by the field personnel according to procedures described below in Section 5: "Documentation of Sampling Event". To prevent a mix up with sample bottle identification, no sampling-point specific information such as "well name" will be filled out in advance of sampling. Chain of custody information will be completed before leaving the sampling point. Laboratory-prepared bottles will be used to assure quality control.

The order of filling bottles with water to be analyzed will be as follows:

1. major and minor ions
2. nitrates
3. cyanide
4. trace metals
5. chromium VI
6. "miscellaneous" parameters
7. volatile organics
8. non-volatile organics
9. dioxin and dibenzo furans
10. coliform bacteria
11. total organic carbon
12. total phosphorus

13. sulfide

14. radium, gross alpha, and gross beta

[This order will be reversed in very slowly recharging wells and will be noted on the SIF or field logbook.] Replicate samples will be collected sequentially as described in Section 4.4: "Field Blanks, Replicates and Split Samples". Methods for filling sample containers for individual analyses are described in Table 2.

The sample water discharge point at the end of the tube will be held as close as possible to the sample container without allowing the sample tubing to contact the container. **[The exception to this rule is for dissolved oxygen and chemical oxygen demand samples where the container is filled from the bottom up by inserting the tube into the bottom of the container.]** At a minimum, sampling personnel will use their body to shield the sampling container from wind and airborne dust while filling. When strong winds, heavy rain, or dusty conditions are present, additional measures will be implemented to guard against background interference.

4.4 Field Blanks and Replicates

Sample blanks will be collected to detect background or method contamination. Replicate samples will be collected to evaluate variability in analytical methods. QA/QC samples will be collected at sampling points suspected to have relatively higher levels of contamination to provide meaningful information for blank or duplicate sample evaluation. Field duplicate samples will be assigned identification aliases on the sample bottle label and on the chain of custody sheet to avoid alerting laboratories that the sample is a replicate sample. The true identity of the field duplicate samples will be recorded in the field sampling log.

The collection schedule for QA/QC samples will be as follows:

1. one trip blank (composed of three replicate vials) for each cooler of VOC samples
2. one field methods (equipment) blank each day by each field sampling crew (or one field blank for every tenth primary sample if it results in more blanks collected)
3. at least one replicate set for every **[# of samples**]samples collected
4. one field ambient air blank each day by each field sampling crew (or one field blank for every tenth primary sample if it results in more blanks collected) *{Ambient air filled blanks are appropriate on a site specific basis. Examples of appropriate situations are: where an automobile engine continues to run during the sampling event; where wind is mobilizing particulates; or when VOCs are being emitted from an operating facility during sampling.}*

For each type of QA/QC sample, containers will be prepared and submitted for the following analyses:

1. trip blank: **[purgeable halocarbons, purgeable aromatics]**

2. field methods (equipment) blank: **[purgeable halocarbons, purgeable aromatics, trace metals, non-volatile organics, dioxins and furans, and total coliform bacteria.]**

3. replicates: all analytical parameters
4. field ambient air blank: **[purgeable halocarbons, purgeable aromatics, trace metals]**

Field Blank Samples

Methods that will be used for preparing field blank samples are described below.

Trip blanks for VOCs consist of a set of three pre-filled 40 ml purge and trap vials that will be filled and sealed by **[the primary VOC analytical laboratory or specify another source]** with laboratory-controlled, **[HPLC-grade]**, organic-free water. The 40 ml, purge and trap, blank sample vials will travel with the actual sample vials to and from the field in the cooler, to the well head, etc., so that the blanks are exposed to precisely the same conditions as the actual samples. The bottle blanks will not be opened until they are analyzed in the laboratory along with the actual VOC samples they have accompanied.

Field equipment/methods blanks will be collected in the field for target parameters . Sample containers used for each blank will be the same as for the actual analysis of sample water for these parameter groups. All containers shall be pre-cleaned within the laboratory's QA/QC program in the same manner as primary sample bottles. The sample blank containers will be filled in the field. Laboratory-controlled, **[HPLC-grade]**, organic-free water will be used to fill all organic blank samples. Trace metals blanks will be filled with laboratory-prepared, triply distilled water. The same preservatives will be added to both the methods blank and the primary samples.

{*Collection of field equipment/methods blank samples should be conducted to simulate actual field sampling methods in a manner that would detect the presence of background or cross-contamination of samples from the ambient environment, preservatives or sampling equipment. An effort should be made to have the blank sample water contact all the interfaces and preservatives (where applicable) that the sample water will contact. These may include the sampling mechanism, ambient air, sample container and, when applicable, tubing, filtration membranes and preservatives.****}***

Laboratory-supplied blank water will be pumped out of a short section of (mock up) well casing by the sampling pump fitted with the same tubing used in the previously sampled well **[assuming there is not a permanent sampling pump installation]** and into the sample blank containers. Blanks for filtered samples will be collected by passing the blank sample water through the filtration device and the same type of filters used for collecting the primary samples.

Ambient air field blanks will be filled in the field. VOC vials will be filled with laboratory-controlled, **[HPLC-grade]**, organic-free water, while trace metal containers will be filled with laboratory-prepared **[triply distilled]** water. Empty vials will be opened and placed or held as closely as practical to the point (vertical positioning will be respected) at which actual sample containers are opened and filled. The sample blank containers will be filled with the laboratory-supplied water by the same personnel and at approximately the same time as the primary (actual) samples are being collected. The sample blank water in each container will be exposed to the air on site for an amount of time equivalent to that for filling and closing a primary sample container. A[**n ambient air field blank and a**] field equipment/methods blank sample will be collected sometime during the first day of each sampling event (round of sampling) and at every tenth sampling point. **[Sample documentation should indicate that the ambient air field blank samples need only be analyzed when the corresponding field equipment/methods blank detects contamination provided that no holding times would be exceeded.]**

Field Replicate Samples

Field replicate samples of actual ground water will be collected for the following parameters: **[**?**]** The **[state number of replicates to be collected]** replicate samples will be collected by sequentially filling all **[# of]** containers as close together in time as practical with a sampling stream that is as steady and continuous as practical. The sequence number (first, second, etc.) and time of sample filling will be listed in the field notebook. The time that each individual container was filled will be listed on the container and on the Sample Identification - field chain of Custody Record (SI-FCCR) in the same manner as primary samples. **[Here state which laboratory will receive which sequence numbers of each parameter type.]** One field replicate sample set will be collected for every ten primary sampling sets.

Field Split Samples

[**Field split samples of actual ground water will be collected for the following parameters: [list parameters here**] The [**state number (#) of split samples to be collected**] split samples will be collected by filling the **[# of]** subsample containers from a single homogeneous sample water **[stream (divided just before discharge into sample containers)][container**] at the same time. [**Here state which laboratory will receive which split samples.] [One field split sample will be collected for every ten primary sampling sets**]

{*Editorial note: Do not split VOC samples*.}]

5.0 DOCUMENTATION OF SAMPLING EVENT

{*For technical guidance, refer to page 92 of the MPCA Ground Water Sampling Guidance Document (Guidance Document) Chapter Four, Section 45:* "DOCUMENTATION OF SAMPLING EVENT"**}**

This sampling protocol template includes the use of forms shown in Appendix B; they are designed for documentation of field activities and collection of field data. They also provide a means to verify whether or not this protocol was followed during a number of key steps in the ground water sampling event. The forms include the following:

1. Sampling Information Form
2. Purging and Stabilization Form
3. Identification - Field Chain of Custody Record (SI-FCCR)

5.1 Sample Identification

The Sample Identification - Field Chain of Custody Record (SI-FCCR) in Appendix B will be completed as described above in Section 5.0, "Documentation of Sampling Event".

The SI-FCCR will be at least a two-part (carbonless copy) form.

Each sample container will be labeled with the following information:

- unique container ID #

- sample collection Date and Time
- initials of person collecting sample
- analyses required
- preservation method

Container information will be entered at the sampling point at the time of sample collection. However, for containers receiving preservatives in advance, "analyses required" and "preservation method" will be entered onto labels by laboratory staff. For containers receiving preservatives in the field, "preservation method" will be entered at the time individual containers are filled.

5.2 Chain Of Custody

A chain-of-custody record (SI-FCCR) will be initiated in the field at the time of sampling; a copy will accompany each set of samples (cooler) shipped to any laboratory.

Each time responsibility for custody of the samples changes, the new and previous custodians will sign the record and denote the date and time. A copy of the signed record will be made by the receiving laboratory. The final signed SI-FCCR will be submitted with analytical results in the Sampling and Analysis Report.

Field Chain of Custody Documentation

All signatures related to sample custody will be made in indelible ink on the SI-FCCR in a timely fashion. One or more signatures will be entered to identify the person or persons who are collecting the samples. Each time the custody of a sample or group of samples is transferred, a signature, date and time will be entered to document the transfer. The signatures, date and time will be entered at the time of transfer. A sample will be considered to be in custody if it is in any one of the following states:

1. in actual physical possession
2. in view, after being in physical possession
3. in physical possession and locked up so that no one can tamper with it
4. in a secured area, restricted to authorized personnel

A secured area such as a locked storage shed or locked vehicle specified in the "comments" column, may be used for temporary storage. When using such an area, the time, date, and location of the secured area will be recorded in the "relinquished by" space. The time at which an individual regains custody will then be recorded in the "received by" space.

Chain of Custody During Shipping and Transfer of Samples

When samples are shipped, the person sealing the shipping container will enter the time, date and their signature on the SI-FCCR. The laboratory part of the SI-FCCR will be enclosed in the container; the top page (first part) will be retained for the project manager's file. A post office receipt, bill of lading, or similar document from the shipper will be retained as part of the permanent chain-of-custody documentation.

One or more custody seals will be affixed over the opening of the shipping container in a manner that precludes opening the container without breaking the seal(s). The container seal(s) will be inscribed with the signature of the person sealing the container and the date and time sealed.

The receiving laboratory will be notified in advance of chain-of-custody procedures that must be followed for a group of samples. The laboratory will be instructed to note whether or not the container seal(s) are intact and sign in the appropriate blank on the SI-FCCR at the time of receipt. They will also be instructed to keep a copy and return the original form to their client's quality assurance officer.

5.3 Field Sampling Log

A daily field log of sampling activities will be kept by the leader of the field sampling crew. At a minimum, the log will contain a record of the following items:

- list of field personnel present
- field conditions(see Section 5.5)
- description of exceptions to this protocol including specification of which samples may have been impacted by exception(s)
- For each well sampled:
    1) Well Name and unique SI-FCCR # used to identify samples,
    2) equipment used for evacuation and stabilization,
    3) date and time that purging and sampling began and ended,
    4) a list of all samples sent to each laboratory

{For field duplicates, include an alias cross reference list for QA/QC samples}

5.4 Exceptions To Sampling Protocol

- This protocol defines the procedures to be followed during this sampling event. Exceptions to this protocol will be noted on the [**SIF or field logbook**].

If there has been any potentially significant impact on sample integrity, then the potential impact for each parameter for each sample affected will be footnoted whenever the results are reported or referred to in the Sampling and Analysis Report.

5.5 Field Conditions

Field conditions during the sampling event will be recorded on the [SIF or field logbook]. The Sampling and Analysis Report will include a statement regarding the likelihood that any unusual field conditions had a significant impact on the integrity of results. Field conditions reported will include but not be limited to the following:

- air temperature
- wind speed/direction
- precipitation/moisture at the time of the sampling event, and if known, previous days' precipitation

- ambient odors
- airborne dust

6.0 SAMPLE PRESERVATION, HANDLING AND TRANSPORT

{*For technical guidance, refer to page 96 of the MPCA Ground Water Sampling Guidance Document:, Chapter Four, Section 4.6:* "SAMPLE PRESERVATION, HANDLING AND TRANSPORT"**}**

6.1 Sample Preservation

Samples will be preserved as shown in Table 2. All chemical preservatives, added to containers in the laboratory or field will be produced and controlled within the laboratory's QA/QC program as reflected in the Lab-QAPjP. Field supplies of preservatives and sample containers with pre-dosed preservatives will be discarded and replaced with fresh preservatives no later than 14 days after receipt from the laboratory.

All samples will be thermally preserved in the field immediately after sample collection by placing the samples in an insulated ice chest containing [**ice, Blue Ice**]. The ice chest temperature will be checked **[**by measuring the temperature of the water within the temperature blank container**]** and recorded upon receipt at the laboratory, to verify whether or not samples are kept refrigerated at approximately 4 degrees C.

6.2 Sample Handling And Transport

All ice chests shipped will be accompanied by an SI-FCCR form and contain a complete destination and return address on the outside of the cooler. The samples will be kept at approximately 4 degrees C during transport to laboratories.

Maintain the chain-of-custody according to procedures described in Section 5.2.

Table 1: Sample Containers, Filling Method, Preservation and Holding Times

{note: this is only an example; the protocol developer is responsible for laboratory coordination on these items}

PARAMETER[1]	*BOTTLE*[2] *VOLUME/TYPE*	*FILL METHOD*[3]	*PRESERVATION*[4]	*HOLDING TIME*
MAJOR & MINOR IONS	1L P	No head space	Cool	28 days
NITRATE	250 ml P	Leave head space	H_2SO_4/pH<2 Lab, Cool	28 days
CYANIDE	500 ml P	Leave head space	NaOH/pH>12 Lab, Cool	14 days
TRACE METALS (unfiltered)	500 ml P	Leave head space	HNO_3/pH<2 Lab, Cool	6 months
(mercury)				28 days
TRACE METALS (filtered)	500 ml P	Filter [5 micron] No head space	HNO_3/pH<2 Lab, Cool	6 months
(mercury)				28 days
CHROMIUM VI (unfiltered)	125 ml P	No head space	Cool	24 hours
CHROMIUM VI (filtered)	125 ml P	Filter [5 micron] No head space	Cool	24 hours
MISCELLANEOUS	1 L P	No head space	Cool	
(TDS and TSS)				7 days
(specific conductance)				28 days
(turbidity)				48 hours
VOLATILE ORGANICS	3 x 40 ml P & T	Positive meniscus	HCl/pH<2 Field, Cool	14 days to analysis
purgeable halocarbons purgeable aromatics non-halogenated volatiles				
NON-VOLATILE ORGANICS	2 x 1L AG	No head space	Cool	7 days/extraction 40 days/analysis
base-neutral/acid extractable organics phthalate esters				

PARAMETER[1]	*BOTTLE*[2] *VOLUME/TYPE*	*FILL METHOD*[3]	*PRESERVATION*[4]	*HOLDING TIME*
phenols				

Table 1: Sample Containers, Filling Method, Preservation, and Holding Times (continued)

{note: this is only an example; the protocol developer is responsible for laboratory coordination on these items}

PARAMETER	*BOTTLE[2] VOLUME/TYPE*	*FILL METHOD[3]*	*PRESERVATION[4]*	*HOLDING TIME*
polynuclear aromatic hydrocarbons chlorinated herbicides organochlorinated pesticides & PCBs organophosphorus pesticides acid herbicides carbamate pesticides				
DIOXINS AND DIBENZO FURANS	1L AG	No head space	Cool	7 days/extraction 40 days/analysis
EQUILIBRIUM GEOCHEMISTRY *pH* *alkalinity* [dissolved oxygen[5]] [Eh]	*2x 1 L P*	*Fill from bottom* *(do not filter for pH)* *Filter [5 micron]*	*Cool*	*2 hours*
TOTAL COLIFORM BACTERIA	125 ml P	Leave head space	Cool	6 hours
TOTAL ORGANIC CARBON	1 L G	Leave head space	H_2SO_4/pH<2 Lab, Cool	48 hours
TOTAL PHOSPHORUS	125 ml P	Leave head space	H_2SO_4/pH<2 Lab, Cool	28 days
SULFIDE	250 ml P	Leave head space	$Zn(C_2H_3O_2)2*2H_2O$ & NaOH/pH>9 Lab, Cool	7 days
RADIUM, GROSS ALPHA, GROSS BETA	1 Gallon P	Leave head space	HNO_3/pH<2 Lab	6 months

Table 1: Sample Containers, Filling Method, Preservation, and Holding Times (continued)

{note: this is only an example; the protocol developer is responsible for laboratory coordination on these items}

(1) PARAMETER NAMES/GROUPS

Some of these parameter names {e.g., "trace metals"} actually represent a set of several or many individual analytes. Specific analytes for each parameter/bottle type are listed in Appendix 1. ***{****Some laboratories may request separate containers (with preservatives) for anions and cations.* ***}***

(2) BOTTLE TYPE

L: liters;
ml: milliliters;
P: polyethylene;
P & T: 40 ml purge and trap vial fitted with a Teflon® septum;
G: glass bottle fitted with Teflon®-lined cap
GG: glass bottle fitted with glass stopper
AG: amber glass bottle fitted with Teflon®-lined cap

(3) FILL METHOD

Positive meniscus:	fill container completely with zero head space resulting in a positive meniscus with no air bubbles in container, add acid and cap container quickly;
No head space:	fill container completely; container will not be rinsed; overfilling will be minimized.
Leave head space:	fill container about 90 to 95 % full - do not allow preservative (if present) to be diluted by overfilling container
Fill from bottom:	fill container completely from the bottom of container using tubing; allow several bottle-volumes of water to overflow before sealing bottle
Filter [5 micron]:	filter in-line with positive pressure through a filter with [5] micron pore size.

(4) PRESERVATION

Cool:	place container inside sealed Zip-Lock bag; place in cooler with sufficient ice to quickly bring temperature down to 4 degrees C and hold at approximately 4 degrees C until received by laboratory personnel
HNO3/pH<2:	add a predetermined amount of high-purity HNO3 to sample to bring the sample pH down to 2 or less;
HCl/pH<2:	add a predetermined amount of high-purity HCl to sample to bring the sample pH down to 2 or below;
NaOH/pH>12:	add a predetermined amount of high-purity NaOH to sample to bring the sample pH up to 12 or above; (for Cyanide, use 50% NaOH solution and add ascorbic acid if oxidizing agents are present)
Zn(C2H3O2)2*2H2O:	predetermined amount added by laboratory staff to prevent oxidation of sulfide
Field:	preservative added in the field by field personnel
Lab:	preservative added to container in laboratory before going into the field

(5) DISSOLVED OXYGEN

For Winkler method, if holding time might exceed 2 hours, field staff will make arrangements with the laboratory to prepare a separate 1 L glass stoppered (GG) bottle by adding preservatives in the field immediately after sample collection.

Table 2: Order of Purging and Sampling of Wells

PURGING/SAMPLING SEQUENCE #	WELL NAME	CRITERIA*	ANALYTE SUITE**

{*THIS TABLE MUST BE SAMPLING SITE/EVENT SPECIFIC***}**

{** For the "CRITERIA" column, specify sequence criteria with descriptive terms such as "background well", "previously clean", far from contamination source", "moderately contaminated", "very contaminated", etc.***}**

{** For some sampling projects, the number or type of containers that need to be filled and analyzed may vary from one well to the next. When that is the case, Table 2 should be duplicated and modified to show two or more lists of containers (representing the analytical suites). Each list should be assigned an identifying name or number such as "Table 2, List a", "Table 2, List b", etc. Reference that identifier in the column above titled "ANALYTE SUITE". **}**

Figure 1: Location of Sampling Points

{Create a map showing locations of all sampling points**}**

APPENDIX A: SELECTED ANALYTICAL PARAMETERS, METHOD NUMBERS AND REPORTING LIMITS

{*In order to create this appendix, edit (customize) Appendix B from the main body of the MPCA Ground Water Sampling Guidance Document or insert a table of parameters required by the applicable program. Indicate what analytical method and reporting limit will apply for each parameter. Appendix B was created in a computer spreadsheet and should be available on diskette along with the Appendix A text. While editing an electronic copy of Appendix B, unwanted parameters can quickly be deleted. Selected analytical methods can easily be "selected" by selecting appropriate cells and formatting them as "shaded".****}*** ***{****It is the responsibility of the end user to check the list of analytical parameters, methods and practical quantitation limits to verify that they are appropriate for the particular site and sampling event of interest****.}***

APPENDIX B: EXAMPLE FORMS

Sampling Information Form

Weather Conditions:
Cloud Cover_____
Wind Speed & Direction_____
Temperature:_____
Precipitation:_____

Facility ID#_____
Facility Name_____
MPCA Master ID:_____
Project Name:_____

Station ID#_____
Location:_____
Well Depth (ft. Below TOC):_____
FID/PID reading @ Wellhead:_____
Depth to Water (below TOC):_____

Sample Date:_____
Sample Time:_____
Casing Diameter_____
FID/PID Background Conc.:_____

Purge
Rate:_____gpm

Well Volumes Removed Prior to Sampling_____

Gallons per Lineal Foot 2”ID=0.163, 4”ID=.0661, 6”ID=1.5, 12”ID=5.88

Sampling Method:_____Tap _____Submersible Pump _____Bailer Other (detail)_____

Pump intake or bailer set at _____ft. Below TOC.

Tubing Type:_____, New, Previously Used and Cleaned was used to collect all samples Y N

Flow Cell Used Y N Purging and Stabilization Protocol Followed Y N

Sample Appearance (describe)_____

Field Cleaning of Equipment Performed _____

Describe any deviations from Sampling Protocol_____
_____
_____

Transportation (Thermal Preservation) Type:_____

Comments:_____
_____

Form Completed By:_____ Well Sampled By:_____

Purging and Stabilization Form

Facility ID#:_____ Site Name:_____ Date:_____

Well #:_____ Sampling Personnel:_____ Time:_____

Time	pH	Temp.	Cond.	Dis. O_2	Turb.	Notes

APPENDIX 3: WORKPLAN CHECKLIST FOR NATURAL ATTENUATION

The following is a list of tasks and data needed to demonstrate that natural attenuation is a remedy for chlorinated solvents in groundwater. Items are grouped by those required for an initial screening and those required for a detailed demonstration that natural attenuation is an acceptable remedy for the site.

Tasks that are considered essential for each phase of the evaluation are marked with a shaded box, while those that are optional (but may be necessary at a later stage in the investigation) are preceded by an open box. The collection of data beyond the required minimum may be attractive because of mobilization costs, time factors, or other site specific considerations.

A more detailed discussion of the purpose of these tasks can be found in the *Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater* prepared for the Air Force Center for Environmental Excellence (AFSCEE), Technology Transer Division (15).

A. Screening Tasks

1. Well/Groundwater locations and samples:

- ■ 1 background, upgradient from suspected source area
- ■ 1 background, side-gradient to plume or source area
- ■ 1 in source area
- ■ 2 within area of dissolved portion of contaminant plume
- ■ 1 downgradient of "toe" of plume

Sample number and frequency:

- ■ One round of sampling for each well

2. Geochemical Data:

Field*:

- ■ Oxygen (Field test kit and/or probe)
- ■ Temperature
- ■ Eh (oxidation/reduction potential)
- ■ pH
- ■ Reduced iron (FeII)
- ■ Reduced manganese (MnII)
- □ Carbon dioxide (CO_2)
- □ Hydrogen (From PVC wells only. For a detailed discussion of this method, see references 8 and 15.)
- □ Conductivity

Laboratory:

- ■ Nitrate (NO_3^{-2})
- ■ Sulfate (SO_4^{-2})
- ■ Sulfide (H_2S)
- ■ Methane (CH_4)
- ■ Chloride (Cl^-)
- ■ Total organic carbon
- □ Alkalinity

Contaminant

- ■ Tetrachloroethylene (PCE)
- ■ Trichloroethylene (TCE)
- ■ Trichloroethane (TCA)
- ■ Dichloroethane (DCA)
- ■ Chloroethane
- ■ Dichloroethylene (DCE)
  (*cis*-1,2-dichloroethylene and *trans*-1,2-dichloroethylene)
- ■ Vinyl chloride (VC)
- ■ Benzene, toluene, xylene, ethylbenzene (BTEX)
- ■ Ethene/Ethane
- □ Soil contaminant data, by depth in source area
- □ Soil total organic carbon

(See Table 2.1 in the *Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater* (15) for a compilation of analysis and sample preservation methods)

3. Aquifer data:

- ■ Hydraulic conductivity
- ■ Hydraulic gradient
- ■ Summary of local geologic features (aquitards, aquifer types, etc.)
- □ Vertical data on geochemistry
- □ Risk analysis of downgradient receptors
- □ Regulatory point of compliance

4. Biodegradation potential:

- ■ Assumed conservative biodegradation rate from literature (13, 18).
- □ Laboratory microcosm study (16, 17).
- □ Site specific estimate using employing groundwater tracers (14).
- □ Site specific estimate using regression analysis (6)

5. Calculations:

- ■ Groundwater velocity estimate using hydraulic conductivity and hydraulic gradient data.
- ■ Comparison of electron acceptor and chloride concentrations to background samples.
- □ Site specific biodegradation rates.

6. Screening/Modeling

- ■ "Score" site data on AFSCEE Protocol Table 2.3 (15).
- □ Screening model, using "worst case" and "best case" scenarios of biodegradation rate, dilution, source mass, and groundwater velocity terms (10)**
- □ Fate and transport modeling (provided that the necessary data has been collected to support this level of analysis. (1-5, 11)

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** Field measurements can be made with a combination of probes and commercially available field test kits. Field analytical measurements using these kits must supported with quality control measures. This may include duplicate samples, occasional duplicate laboratory analysis for certain analytes (such as sulfate), standard samples of varying concentrations, field blanks, and rinseate controls. When not addressed in site-specific standard operating procedures, approval from regulatory QA/QC personnel is recommended prior to proceeding with field testing. This should include agreement on the level of data quality that these measurements will represent in subsequent analysis and decisions.*

** *These screening procedures are intended to provide a basis for deciding whether contaminant attenuation is occurring, and whether further sampling and analysis of this remedy is worthwhile. Though not essential, running a screening model is strongly encouraged. This work can minimize the cost and effort at sites where contaminant attenuation is unlikely to be occurring at rates necessary for a natural attenuation remedy.*

B. Verification

1. Wells/Groundwater samples:

(From the screening phase)

- 1 background, upgradient from suspected source area.
- 1 background, side-gradient to plume or source area.
- 1 in source area.
- 2 within area of dissolved portion of contaminant plume.
- 1 downgradient of "toe" of plume.
- Additional monitoring wells based on a Site specific analysis of data needs.
  (This might include: definition of the downgradient extent of the plume; well nests to determine the vertical distribution of contaminants and electron acceptors; estimation of DNAPL extent; contouring of groundwater electron acceptors, etc.)

Sample number and frequency:

For each well:

- a) Four rounds of groundwater samples, approximately 6 months apart,

Or

b) Based on the groundwater velocity term, determine the time needed for groundwater to travel the length of the plume. Sampling should occur at a minimum of four times within this "residence" time of groundwater flow:

{Plume length (ft)]/Groundwater velocity (ft/year)}/ 4 = time between samples

2. Geochemical Data:

Field:

- O_2
- Temperature
- Eh (oxidation/reduction potential)
- pH
- FeII
- MnII
- Hydrogen (from PVC wells only)
- Conductivity
- CO_2

Laboratory:

- NO_3^{-2}
- SO_4^{-2}
- H_2S
- CH_4
- Cl^-
- Total organic carbon
- Estimate of FeIII available in aquifer sediments
- Alkalinity
- Fatty acids

Contaminants‡

- ■ PCE
- ■ TCE
- ■ TCA
- ■ DCA
- ■ Chloroethane
- ■ DCE (*cis*-DCE and *trans*-DCE)
- ■ VC
- ■ BTEX
- ■ Ethene/Ethane

‡ *Subsequent sampling may reveal the presence of contaminants (or their reductive metabolites) not detected in the screening phase. However, this list may be amended to reflect particular site characteristics through consultation with the regulatory staff.*

Other contaminant information:

- ■ Estimate of DNAPL or LNAPL extent
- ■ Estimate of source area boundaries
- ■ Estimate of mass released in the source area based on historical data

Soil:

- ■ Soil contaminant data, by depth in source area
- □ Soil total organic carbon
  (may also be required to evaluate the potential leaching to groundwater)
- ■ Contaminant Kd or Koc values from literature (12)
  (required to calculate retardation constant for contaminants; may also be required to evaluate potential leaching to groundwater)
- ■ Soil density
- ■ Soil/sediment porosity

3. Aquifer data

- ■ Summary and analysis of local geologic features that may include: confining units; aquifer types; drinking water aquifers; analysis of boring logs; hydrogeologic section maps.
- ■ Depth of aquifer
- ■ Lithology
- □ Vertical data on geochemistry and contaminant concentrations†
- ■ Risk analysis of downgradient receptors, including ecological receptors and future exposure points
- ■ Regulatory point of compliance
- ■ Advection and dispersivity assumptions
- ■ Potentiometric water table maps
- ■ Isopleth maps of daughter products
- ■ Isopleth maps of electron acceptors
- ■ Isopleth maps of contaminants
- □ Isopleth maps of above subjects by depth, if warranted
- □ Identification of zones of high transmissivity and preferential flow paths through boring log analysis, cone penetrometer studies, or downhole flowmeter (9)†

† *Strongly recommended.*.

4. **Biodegradation potential:**
   - ■ Field measurement of degradation using tracers.
     Or
   - ■ Using data from three monitoring wells, an analysis using regression analysis (6).
   - □ Laboratory microcosm study.

5. **Calculations:**
   - ■ Retardation coefficient
   - ■ NAPL/water partitioning constants
   - ■ Site specific biodegradation rate for each contaminant
   - ■ Refined estimates of groundwater velocity and direction based on additional data

6. **Modeling and Analysis:**
   - ■ Fate and transport modeling.
   - □ Soil leaching modeling for source area. (This can assist in determining the flux of contaminants to groundwater if needed to evaluate source removal options.)
   - ■ Refined three-dimensional conceptual model for the site (7).
   - ■ Evaluate source removal effect on attenuation processes.
   - ■ Conduct additional sampling and analysis to fill data gaps, if needed.

   - □ Evaluate "active" remedies to augment natural attenuation
   - ■ Compile "weight of evidence" arguments; solicit regulatory approval for a natural attenuation remedy.

C. Long term monitoring plan

1. **Wells/Groundwater samples:**
   - ■ Monitoring wells from verification phase (modified if necessary after modeling and development of refined conceptual model
   - ■ Sentinel well locations, based on analysis and modeling results

   Sample number and frequency:
   - ■ Quarterly during the first year
   - □ Annual sampling after one year if stable results from first year.

   Monitoring wells (in area of plume):
   - ■ Contaminants of concern
   - ■ O_2
   - ■ NO_3^{-2}
   - ■ FeII
   - ■ MnII
   - ■ SO_4^{-2}
   - ■ CH_4
   - ■ Water level
   - ■ NAPL thickness, if appropriate
   - ■ Other analyte of regulatory concern

   Sentinel or compliance monitoring wells (downgradient of plume):
   - ■ Contaminants of concern

2. **Other:**
   - ■ Contingency plans for unexpected plume expansion.

D. Sampling Recommendations.

The following is a compilation of the data needs for a natural attenuation evaluation, the methods of analysis, and recommendations for sample collection procedures. The types of samples and the methods of collection may vary depending on site-specific considerations. Thus, individual work plans for sampling should be approved before proceeding with sampling.

Table 1.

Analyte/parameter	Method[1]	Sampling comments and recommendations
Reduced iron (Fe+2)	Field test kit[2]	--
Reduced manganese (Mn+2)	Field test kit	--
Oxygen	Field test kit O2 Probe[3]	-- Use in flow cell apparatus.
Eh	Eh Probe	Use in flow cell apparatus.
pH	pH Probe Litmus paper	Use in flow cell apparatus.
Conductivity	Conductivity probe	Use in flow cell apparatus.
Temperature	Thermocouple or thermometer	Use in flow cell apparatus.
Carbon dioxide	Field test kit	--
Sulfide	Field test kit	--
Methane	Laboratory analysis[4]	40 ml serum bottle with crimp cap. Preserve sample with 5 drops of 50% H_2SO_4[5]
Dissolved organic carbon	EPA lab method 9060	40 ml serum bottle with crimp cap. Preserve sample with 5 drops of 50% H_2SO_4[6]
Alkalinity	Field test kit	--
	EPA lab method 310	Screw-cap plastic bottle, no preservative necessary
Sulfate	Field test kit	--
	EPA lab method 9035; 9036	Screw-cap plastic bottle, no preservative necessary

Chloride	Field test kit	--
	EPA lab method 9250; 9251	Screw-cap plastic bottle, no preservative necessary
Nitrate	Field test kit	--
	EPA lab method 352	250 ml Screw-cap plastic bottle, preserve with 5 drops H_2SO_4
Hydrogen	Field hydrogen analyzer	Sample collected via dissolved gas flow cell as per reference ( )[7]
BTEX	EPA lab methods 465E; 8015; 8021B	40 ml VOA bottles, preserved with Hcl
Chlorinated VOCs	EPA lab methods 465E; 8121; 8260	40 ml VOA bottles preserved with Hcl

1. Laboratory methods may vary depending on the individual laboratory standard operating procedures. Consult with lab personnel to make sure sample collection is consistent with laboratory analytical standard procedures.

2. a) Several commercially available and reliable test kits are available. Ensure that the range of the test kit analysis is consistent with the expected range to be sampled.
   b) Provision for QA/QC requirements should be included in using field test kits. As for any other sampling protocol, sample blanks, duplicate sample analysis, and duplicate laboratory analysis (when possible) are appropriate in gathering data. Check with quality assurance personnel to verify QA/QC sampling, frequency of duplicates, and other precautions.

3. All probes and electronic instruments must be calibrated as per the manufacturers' instructions on a daily basis prior to making measurements.

4. The laboratory standard operating procedure for methane analysis followed by EPA Kerr labs is enclosed.

5. Preservative is added to reduce the pH so that any methane is not degraded biologically; the vials need to be sealed tightly to eliminate volatilization to the atmosphere

6. Preservative is added to reduce the pH so that organic carbon is not biologically degraded.

7. Can be collected only from PVC wells; cannot be collected using any electrical pump. Use peristaltic, or some form of pneumatic pump.

E. Recommended Field Sampling Procedure.

1. Record pH, temperature, Eh, and conductivity readings using a low flow cell apparatus and appropriate probes until readings are stable.
2. Fill (4) HCl preserved VOA vials for BTEX and solvent analysis.
3. Fill (2) 250 ml plastic screw cap bottles. Add H_2SO_4 to one and label this one as "preserved".
4. Completely fill (2) 40 ml serum crimp vials; add H_2SO_4 and immediately seal with crimp cap.
5. Perform all field test kit analyses.
6. Set up the glass equilibration vessel for hydrogen sampling at a flow rate of approximately 200 ml/min; allow 15 minutes for gas equilibration. After flushing the gas syringe once with the equilibrated gas contained within the vessel, extract 10cc of bubble for analysis on hydrogen analyzer.

Tanks and Emergency Response Section
Minnesota Pollution Control Agency

Soil Sample Collection and Analysis Procedures

Fact Sheet #3.22
July 1996

This fact sheet provides procedures for field screening of petroleum contaminated soil and collection and laboratory analysis of soil samples.

I. FIELD SCREENING PROCEDURE

Minnesota Pollution Control Agency (MPCA) staff recommends the polyethylene bag headspace method described below as the field procedure for characterization of soil contamination. We no longer recommend using glass jars for this procedure because 1) the collapsible nature of bags allows more uniform flow of actual headspace gas into the field instrument resulting in more accurate readings and 2) the soil clumps can be broken up when bags are used.

1. Use photoionization detectors (PIDs) with a 10.2 eV (+/-) or greater lamp source, or flame ionization detectors (FIDs). Perform PID or FID instrument calibration on site and at least daily to yield "total organic vapors" in volume parts per million (ppm) of a benzene equivalent. Follow the manufacturer's instructions for operation, maintenance, and calibration of the instrument. Keep calibration records. MPCA staff reserve the right to request these records.

2. Use a self-sealing quart-size polyethylene freezer bag. Half-fill the bag with the sample to be screened so the volume ratio of soil to air is equal then immediately seal it. Manually break up the soil clumps within the bag. *Note:* Soil collected from a split spoon should be transferred to the bag immediately after opening the split spoon; soil collected from an excavation or soil pile should be collected from freshly exposed surfaces.

3. Allow headspace development for at least 10 minutes. Vigorously shake bags for 15 seconds both at the beginning and end of the headspace development period. Headspace development decreases with temperature. When temperatures are below the operating range of the instrument perform headspace development and analysis in a heated vehicle or building. Record the ambient temperature during headspace screening. *Complete headspace analysis within approximately 20 minutes of sample collection.*

4. Following headspace development introduce the instrument sampling probe through a small opening in the bag to a point about one-half of the headspace depth. Keep the probe free of water droplets and soil particles. (Syringe withdrawal of a headspace sample and injection to an instrument probe or septum-fitted inlet is acceptable, provided the method accuracy is proven by means of a test gas standard.)

5. Record the highest meter response. Maximum response usually occurs within about two seconds. Erratic meter response may occur at high organic vapor concentrations or if moisture is present. Note any erratic headspace data.

APPENDIX 5: POLICY ON EPA METHOD SW-846 5035

The latest approved update to EPA SW-846 contains a new method that changes the way volatile soils samples are taken. Method 5035 contains a number of methods that may be used to sample volatiles in soils depending on required reporting limits needed for a site. A number of laboratories and consultants have requested a formal position from the MN Pollution Control Agency (MPCA) on this method driving this policy letter. The following recommendations are made:

1. The MPCA approves the use of method 5035 and will require that this method be used on all Superfund, solid waste, emergency response, RCRA, and tank sites.

2. The methods used prior to this (jar packing) will be unacceptable for work to take place after 1 September 1998. Current work plans that use the old method should be updated to reflect the new method. (A few sites in the state that are doing comparative soils analysis may want to employ both methods.)

3. Environmental laboratories will be prepared for the transition to 5035 (most are already using the method). The laboratories will include a sheet with their bottles that outlines the exact procedure of sampling to be used (e.g. 5 grams of soils and 5 mls of methanol, etc.).

The method has four procedures used for sampling. Each of the procedures is specific to the data quality objective for the data. The following gives an outline of the different procedures used under 5035.

1. Methanol Preservation - This method is a high level method due to the reporting limits being at 100 ug/kg and greater. The method consists of sampling with a vial that is preweighed and labeled. The vial has a sample of soil added to it using a coring device (usually a cut syringe) to get roughly 10 grams of soil into the vial with minimal disturbance of the soil, and then adding (or already having in the vial) 10 mls of methanol. The sample is then shipped to the laboratory on ice (four degrees Celsius).

2. Sodium Bisulfate - This is a low level method similar to the methanol procedure, but instead of methanol being added, Sodium Bisulfate is used as a biocide (eliminating the dilution and giving a reporting limit of <100ug/kg). The sodium bisulfate is added at a rate of 0.2 grams of preservative to 1 gram of soil. Normally 5 grams of soil and 1 gram of sodium bisulfate are used. Five milliliters of water is added to the vial after the soil and sodium bisulfate are present to form an acid which prevents biodegradation. The vial is then sealed in a cooler with ice, and shipped to the laboratory.

3. High Concentration Samples - This method is used for oily samples or samples of very high concentration. No preservative is used. The sample is taken and put in a container (normally 4 oz. jar) with zero headspace and shipped to the laboratory (on ice).

4. Encore Sampler - The Encore sampler may be used to sample the soil. The Encore is a device that allows for a zero headspace sample to be taken without “jar stuffing”. The Encore is used to transport the sample to the laboratory. The laboratory then can do a direct purge (water and soil) on the sample, add methanol, or sodium bisulfate (thereby giving a <100 ug/kg reporting limit). The current accepted

method 5035 allows a 48 hour hold time on the Encore sample prior to analysis or transfer to a container with preservative in it. It is understood by the State of MN that EPA will be allowing a one week hold time on the Encore within the year. There are a few versions of the Encore that staff should be aware of. The old samplers were made of stainless steel with "o" rings used to seal the system. They were reusable (after being sent back to Enchem for baking and reassembly). The newer samplers are made of Teflon, are disposable, and packaged individually.

In considering which method to use the driving factor will be the reporting limits needed. These must be considered up front to avoid the possibility of resampling. Additionally, multiple samples should be taken (a minimum of three) by any method to allow for reanalysis. If employing the low level method, at least one methanol sample should also be taken too, as dilutions cannot be taken from a sample preserved by the sodium bisulfate. A sample for dry weight must also be taken to allow for this calculation.

At this time, most sites will allow for the use of either the methanol or low level method (or Encore). In reviewing the soil limits for Minnesota, only the leaching limits used on a site specific basis were found to be lower than the limits achievable by the use of methanol extraction (on a clean sample). As newer methods evolve with the introduction of PBMs (performance based methods), one can be certain that there will be other choices in the near future. Therefore, it is recommended that documentation on field sampling allow for these changes with wording to the effect of "use of methanol preservation or sodium bisulfate or equivalent per method 5035".

Any questions or comments on the policy can be directed to Luke Charpentier at (651) 296-8445.

Minnesota Pollution Control Agency
Site Remediation Section
520 Lafayette Road
St. Paul, Minnesota 55155-4194