FIELD QA/QC PROTOCOLS FOR MONITORING AND PREDICTING IMPACTS OF ACID ROCK DRAINAGE

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EXECUTIVE SUMMARY

In recent years there has been a growing emphasis on quality assurance/quality control (QA/QC) protocols for environmental monitoring programs. Although the elements of QA/QC programs for laboratory analyses are reasonably well defined, there has been less emphasis on defining QA/QC protocols for field programs. Therefore, the B.C. Acid Mine Drainage Task Force contracted Norecol Environmental Consultants Ltd. to undertake a review of QA/QC protocols for field studies. The review focuses on components of programs for predicting or detecting acid rock drainage (ARD) and its environmental effects.

A QA/QC program begins with setting a data quality objective, which defines the level of uncertainty that a decision maker is willing to accept in decisions made with environmental data. The quality assurance component of the program is the set of operating principles that, if strictly followed, should produce results which meet the data quality objective. The quality control component of the program is the assessment, through measurement of precision, accuracy, and representivity, of whether the data quality objective has been achieved.

Common elements of QA/QC programs for field studies include selection of the sampling locations, definition of the required number of replicate samples, and choice of the appropriate sampling device to achieve precision, accuracy, and representivity in the data. Other common elements include maintenance of field notes, technician training and evaluation, and standardization of sample collection, preservation and storage protocols.

This document reviews the elements of field QA/QC programs for sampling surface and groundwater, effluents, sediments, soils, tailings and waste rock for chemical parameters related to ARD. It also reviews QA/QC programs for stream flow measurements and biological effects monitoring, including sampling populations of fish, benthic invertebrates, periphyton, zooplankton, and phytoplankton; metals in fish tissues; and the presence of *Thiobacillus*.

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INTRODUCTION

1.1 Background

In recent years, there has been a growing emphasis on quality assurance/quality control (QA/QC) protocols for environmental monitoring programs. This interest is based upon the recognition that large resource expenditures associated with environmental monitoring cannot be justified if data quality is not defined and controlled to meet pre-determined criteria (Sutherland 1990).

The elements of QA/QC programs for laboratory analyses of environmental samples are reasonably well defined, but monitoring programs typically fail to define QA/QC protocols for field collection procedures. Therefore, the British Columbia Acid Mine Drainage Task Force has contracted Norecol Environmental Consultants Ltd. to undertake a review of QA/QC protocols for the field. The review is limited to the components of programs for predicting or detecting acid rock drainage (ARD) and its environmental effects.

Monitoring programs for ARD typically include samples to determine chemistry (pH, acidity, alkalinity, conductivity, sulphates and/or sulphur species, metals, and metalloids) of surface and groundwater, effluents, sediments, soils, tailings, and/or waste rock. Baseline programs and proposed Environmental Effects Monitoring programs may include the measurement of biological parameters such as metal concentrations in fish tissues or population density and species composition of benthic invertebrates, periphyton, zooplankton and phytoplankton. Samples may also be collected for tests specific to the prediction or detection of ARD such as acid-base accounting, measuring tailings pore gases, or detecting the presence of bacteria such as *Thiobacillus*. In addition, stream or effluent flows may be measured to determine the effects of dilution.

This report reviews field protocols for parameters associated with ARD monitoring with emphasis on QA/QC procedures. The report describes the elements of an optimal QA/QC program. The actual program implemented for any study will be determined by the defined data quality requirements and the program budget.

1.2 Definitions

In order to establish a QA/QC program, it is necessary first to define its elements. *Quality assurance* is a set of qualitative operating principles that, if strictly followed, will produce data of known and defensible quality. *Quality control* is the quantitative assessment of the precision and accuracy of the data (APHA 1989).

Precision is the measurement of the repeatability of a set of values. It is estimated by the analysis of replicate samples and can be described by the coefficient of variation (USEPA 1984 cited in Sutherland 1990). In the laboratory, repeated analyses of the same sample (or split sample) determine the precision of the analytical method. In field replicates, however, the variability associated with precision of sampling method is confounded by the spatial or temporal variability of the medium being sampled.

Accuracy is the measurement of how closely a measured value approximates a true value. It encompasses precision and the measurement of systematic error or bias (USEPA 1984 cited in Sutherland 1990). In analytical chemistry, the determination of accuracy involves analyses of standard reference materials containing known amounts of certain metals, for example. There are no comparable standards for field procedures, but methods of assessing biases do exist.

The quality of field data is influenced by a another element, representivity, which could be considered a component of accuracy. *Representivity* is the degree to which the data measure the actual state of the component being sampled (Sutherland 1990). It is a function of spatial and temporal environmental variability.

1.3 Elements of a QA/QC Program for Field Investigations

Although the actual protocols for sampling different media vary, the basic elements of QA/QC programs are similar.

The QA/QC program for a field study begins at the planning phase. It is first necessary to define the objective of the study. The study objective will lead to a *data quality objective (DQO)*, or a definition of the level of uncertainty that a decision maker is willing to accept in decisions made with environmental data (Beak Consultants Ltd. 1991a). The data quality objective is the standard against which data quality is measured. It may determine the budget to be allocated to the project or it may be modified to accommodate the funding available.

Once a data quality objective is established, the study coordinator can determine the field sampling design and other QA/QC measures necessary to meet it. This procedure may involve a pilot study to determine environmental variability and/or to assess sampler biases. It will lead to a definition of the required number of replicate samples, selection of sampling locations, and/or choice of the appropriate sampling device.

Other common elements of a field QA/QC program (Beak Consultants Ltd. 1991a) include:

- maintenance of field notes;
- technician training and evaluation; and
- standardization of sample collection, preservation, and storage protocols.

Regardless of the medium being sampled, field crews should maintain notes in a field log book which records all pertinent information on field activities and sampling efforts. The log book should include (Tetra Tech 1987):

- date and time of starting work;
- names of field supervisor and crew members;
- purpose of sampling;
- locations of sampling sites;
- descriptions of sampling site, including records of any photographs taken;
- field observations;
- field measurements made (including data records);
- details of sampling effort, particularly any deviations from standard operating procedures;
- type(s) and numbers of samples collected;
- sample identification; and
- sample handling, packaging, labelling, and shipment information (including destination).

A protocol manual should be developed to provide for technician training and ensure standardization of sample collection, preservation, and storage procedures. The manual should specify sampling equipment appropriate to the study objectives, proper use of equipment, criteria for locating stations, sample containers, labelling and preservation of samples, and any constraints on sample storage (Beak 1991b). Video recordings are also useful for technician training and standardization of procedures, although they should not be used as a substitute for written protocols (Beak 1991b).

The following sections provide details of sampling different media, including specific records to be maintained, appropriate protocols, and training requirements. They also discuss QC protocols specific to particular media, such as the use of sample blanks for water samples.

FLOW (OR DISCHARGE) MEASUREMENTS

2.1 Introduction

Stream discharges are usually monitored as part of the baseline program for a proposed mine. In this context, they are useful for predicting available dilution which can moderate the impacts of ARD. In addition, flows may be measured in conjunction with water sampling to evaluate existing ARD discharges. They aid in data interpretation as both background receiving water quality and ARD impacts may be flow-related (see, for example, Robertson 1990). The following sections describe QA/QC protocols for the most common flow measurement techniques.

2.2 Instantaneous Flow Measurement by Wading Stream

This section discusses the method for conducting an instantaneous flow measurement at one location on the cross section a stream, assuming that the stream can be waded at the time of the measurement. The measurement of streamflow by wading is the most common method of measuring flow. The need to utilize other methods such as weirs, flumes, dye, cableways or boats would be identified during the study planning phase.

2.2.1 Field Sampling Design and Quality Control Aspects

2.2.1.1 Selection of Measurement Location

The study planning phase will include selection of flow measurement sites, which will be determined, in part by the study objectives. For example, one would normally sample just upstream of a known (or expected) effluent location.

Within the predetermined measurement area (which can be up to 500 m long in some streams) the selected measurement location should be the one which has the best cross-section for measurement. Ideally the stream cross-section at the location to be measured should have the following characteristics:

- all the flow confined to a single well defined channel for at least 3 stream widths upstream of the measurement section;
- no obvious leakage around the measurement section;
- no tributary inflows entering within 3 stream widths upstream of the measurement section;

- a fairly straight section of stream;
- a rectangular or U-shaped cross-section;
- a fairly uniform velocity and depth (and flow) distribution across the stream;
- bed material that is fairly small (not always possible); and
- no vegetation, debris or obstacles that impede the flow.

In most streams the downstream end of a pool or the upstream end of a riffle provides a good measurement section.

If the general measurement location is not defined by other sampling criteria, then selecting the site with the best flow measurement cross section will ensure the most accurate flow measurement.

2.2.1.2 Selection of Measurement Method

As discussed above it is assumed that conditions permit wading the stream. Flow measurements using current meters and wading across the stream offer the possibility of selecting the best available cross-section for the measurement as well as simplicity in conducting the measurement and computing the discharge. Measurement procedures are discussed in detail in a number of publications (Terzi 1981; Buchanan and Somers 1969).

2.2.1.3 Replicate Measurements

In locations where a reasonably good measurement cross-section exists and procedures are carefully followed by experienced technicians the flow measurement should have an accuracy within 5 percent of the true value 95 percent of the time (Terzi 1981). If the data quality objective requires an improved level of accuracy, and conditions permit, multiple simultaneous flow measurements could be conducted in the same reach of stream and the data averaged.

2.2.2 Field Notes

Detailed descriptions of the field information to be noted are presented in Terzi (1981) and Buchanan and Somers (1969). Generally, 20 to 25 measurements should be made across the stream. For each measurement location, field notes should include:

- depth of water;
- location (or distance) relative to the stream banks; and
- number of velocity meter revolutions in a given time.

In addition, information needs to be recorded on: the staff gauge level; the station name and number; the date, time when flow measurement started and finished, and the equipment used, etc.

2.2.3 Technician Training and Evaluation

Ideally, technicians should have some experience around streams and feel comfortable wading in them. Knowledge of flow hydraulics and river processes is also useful. Careful reading of manuals describing measurement of streamflow (Terzi 1981; Buchanan and Somers 1969) along with field training should be sufficient to train most technicians to a reasonable level of competence. During technician training and during the sampling program, simultaneous flow measurements by experienced field technicians can be used to assess the performance of inexperienced technicians.

2.2.4 Conducting Flow Measurements

2.2.4.1 Equipment Preparation

The manuals describing measurement of streamflow include sections on pre-trip and pre-measurement planning and equipment preparation (Terzi 1981; Buchanan and Somers 1969). Generally, check lists are used to ensure that all required equipment is maintained and checked before proceeding into the field.

2.2.4.2 Procedures for Reducing Biases

When carefully followed, the procedures given in manuals describing measurement of streamflow should reduce systematic bias (such as measuring the streamflow as less than the true value because of an improperly maintained flow meter) (Terzi 1981; Buchanan and Somers 1969). Biases caused by improperly maintained or calibrated equipment or poor field techniques may be detected by conducting simultaneous measurements using different sets of equipment.

2.2.4.3 Data Processing and Screening in the Field

If the stream flow (or discharge) is calculated immediately after completing the field measurement, it may be possible to spot random errors and conduct another measurement.

2.2.5 Conclusion

In locations where a reasonably good measurement cross-section exists and procedures are carefully followed by experienced technicians the flow measurement should have an accuracy within 5 percent of the true value 95 percent of the time (Terzi 1981).

2.3 Continuous Stage (Water Level) Measurements and Conversion to Continuous Flow Data

In situations where it is desirable to have a continuous record of streamflow, continuous stage (water level) measurements are made and then converted to streamflow using a stage-discharge relationship.

2.3.1 Field Sampling Design and Quality Control Aspects

2.3.1.1 Selection of Stage and Flow Measurement Locations

The accuracy of streamflow data is highly dependent on the accuracy and stability of the stage-discharge relationship at the measurement location. Ideally, the stream at the measurement location should be very stable, with good locations for monitoring stage and measuring stream flow in close proximity to each other (Buchanan and Somers 1968). These factors should be considered at the study planning stage and appropriate steps (such as choosing a stable measurement section) taken to ensure an adequate level of accuracy.

2.3.1.2 Selection of Frequency of Stage/Discharge Measurements

Ideally for a stable stream about 10 stage/discharge measurements should be made over the entire range of recorded stage levels prior to establishing a stage/discharge curve (Terzi 1981) and calculating the continuous streamflow record for the period. If the stream is unstable, then the number of stage/discharge measurements should be increased. If stage data are collected over only a small range of stages (in a short-term study), then this guideline can be relaxed. Therefore the project objective and schedule along with the stability of the stream should be considered to determine timing and frequency of stage/discharge measurements.

2.3.1.3 Replicate Stage/Discharge Measurements

In establishing a stage/discharge relationship, measurements that are made at the same or at similar stage levels can be used to indicate the accuracy of flow measurements, if the stream is stable. Replicate measurements can also indicate the degree of instability if the stream is unstable.

2.3.2 Procedures for Converting Stage Data to Flow Data

When carefully followed, the procedures given in manuals describing the computation of continuous streamflow records should produce results free of random error (Kennedy 1983).

2.3.3 Technician Training

Ideally, technicians should have been involved in collecting the continuous stage data and the stage/discharge measurements for the subject location. Knowledge of flow hydraulics and graphical techniques is helpful in converting the continuous stage data to continuous streamflow data.

2.3.4 Quality Assurance Procedures

Manuals describing the computation of continuous records of streamflow discuss ways of checking the computations and the adequacy of the stream flow record (Kennedy 1983). In some locations, data can be compared to other regional stations using hydrologic techniques (Kennedy 1983).

There are published criteria for data evaluation. The U.S. Geological Survey in Alaska (U.S. Geological Survey 1990) defines the accuracy of their streamflow records as follows:

- "excellent" if about 95% of the discharge records are within 5% of the true value;
- "good" if about 95% of the discharge records are within 10% of the true value; and
- "fair" if about 95% of the discharge records are within 15% of the true value.

2.3.5 Conclusion

The accuracy of continuous streamflow data calculated from continuous stage data depends primarily on:

- the stability of the stage-discharge relationship;
- the frequency of discharge measurements; and
- the accuracy of observations of stage, measurements of discharge and interpretation of records.

In locations with excellent stage measurement characteristics, excellent flow measurement cross-sections and stable stream channels, it may be possible to obtain a streamflow record of "excellent" accuracy if procedures are carefully followed by experienced technicians. With inexperienced technicians records of only "fair" accuracy are more likely. In situations where ideal flow measurement cross sections and/or stable stream channels do not occur, it may not be possible to achieve a data quality objective of "excellent" or even "good". However, the effect of the technician on data quality can be controlled if the QA/QC program includes adequate technician training and evaluation procedures.

CHEMISTRY OF SURFACE AND GROUNDWATER

3.1 Surface Water

Surface water quality is usually monitored as a component of baseline programs and to assess the impacts of ARD discharges. Study design criteria and protocols for monitoring surface waters were recently documented by Tetra Tech (1987) and Sutherland (1990).

3.1.1 Field Sampling Design and QC Aspects

3.1.1.1 Site Selection

Field sampling design includes selection of sampling sites and determination of sampling frequency or replication. Site selection should always include a spatial control or sampling station which is unlikely to be affected by the mine (or other ARD source being investigated). Normally, this site will be upstream of the ARD source. However, in cases where the ARD source is in headwaters of a stream or discharging into a lake that has no permanent inlet, a control site should be established in an unaffected tributary or reference lake.

If a baseline monitoring program is being planned, a pilot study should be used to determine that the control site is representative of the site of expected impact. In cases where impact monitoring must be conducted in the absence of baseline data (which may be lacking for older or abandoned mines), it will be necessary to make the assumption that the reference site is representative of baseline conditions at the impacted site (Sutherland 1990).

Selection of other sampling sites will depend upon the point(s) of impact or expected impact. Ideally, sampling sites should be downstream of the ARD source being monitored and upstream of all other potential sources of ARD-related parameters (acidity, sulphate, and metals). Where ideal sample siting is not possible, additional sites should be established to assess the contributions of other potential contaminant sources (tributaries and seeps).

Depending upon the objective of the study, at any or all sampling sites it may be necessary to sample a single point that is representative of the cross section of a stream or river. For large rivers, a pilot study may be necessary to determine that the cross section is well mixed and the sampling point is representative.

3.1.1.2 Sample Replication and Sampling Frequency

Replicate samples (usually triplicates) are often collected at one or more sites to assess precision of the sampling method. The use of replicates for this purpose assumes that the variability among replicates is affected by the sampling method or technician. However, the variability among replicates may also be affected by the natural variability at the site. In streams, where the water is constantly moving, variability may be either temporal or spatial. In most cases the variability associated with several replicate samples collected close together or in quick succession at a single point will be low. The ideal pilot study would assess "instantaneous" variability to confirm that this is the case for all sites (Robertson 1990).

Since receiving waters are subject to seasonal variability, the monitoring will need to be frequent enough to ensure that measured water quality is representative of the range of seasonal variability. The data quality objective (which may involve a compromise between the study objective and budget) should define an acceptable coefficient of variability for the annual or seasonal mean. A pilot study can be used to determine the sampling frequency required to meet the data quality objective (Robertson 1990). In practice, because of the costs of chemical analyses and travel to remote sites, both the extent of the pilot study and the eventual data quality objective may be a compromise between the study objective and budget.

3.1.1.3 Assessment of Bias

The study design should incorporate appropriate QC techniques for assessing bias in the data. The major source of bias in water quality studies is sample contamination. The major sources of contamination include:

- contamination by the technician during sample collection or transportation;
- contamination by the sampling device (eg. Niskin bottle), if used;
- contamination during filtration; and
- contamination by the sample bottle.

Unusually high variability among replicates may indicate contamination by the technician during sample collection. This suspicion could be confirmed by having a second technician collect replicate samples at the same time and place and comparing the variability of the two sample sets.

Most of the other major sources of contamination are assessed using sample blanks. Types of blanks include:

- **transportation blanks:** deionized water placed in a sample bottle at the analytical laboratory and carried to and from the field with the sampling bottles and samples;

- **sampler blanks:** deionized water passed through the sampler prior to sampling and after sampling to assess contamination from the sampler itself and from sample carry over; and

- **pre- and post-filtration blanks:** deionized water passed through the filtration equipment prior to sampling and after sampling to assess contamination from the equipment itself and from sample carry over.

Contamination in transportation blanks could indicate either contamination during the transportation process or contamination due to the sample bottle itself. Contamination by the sample bottle is not normally a problem when properly prepared bottles have been supplied by an experienced analytical laboratory. However, if this type of contamination were suspected, it could be assessed with sample bottle blanks which were kept at the analytical laboratory.

A less commonly recognized source of bias is the short-term temporal variability associated with sampling a stream where the water mass passing a fixed sampling point is constantly changing. This type of bias has occasionally been assessed. For example, in a pilot monitoring study of the Columbia River, the Inland Waters Directorate of Environment Canada (IWD) collected 10 samples in quick succession and another 10 samples at one-minute intervals to compare the variability. In this case, the difference between sampling frequencies was significant only for phosphorus and nitrate (Sheehan and Lamb 1987; Sigma Engineering Ltd. 1987).

The temporal bias may be more significant for samples collected across the cross section of a large river. Some IWD personnel collected samples at half-hour intervals at a fixed point on the bank while others sampled the cross section of the Columbia River downstream of Trail, British Columbia. In one data set, visual comparison of the bank and cross section samples collected at corresponding times showed a clear temporal effect. Statistical assessment of the data (using a runs test) showed a temporal bias for most parameters measured in three separate data sets (Sheehan and Lamb 1987, Smith 1987b).

The Columbia River reach studied is downstream of a major effluent source, the Cominco smelter and fertilizer plant at Trail. Much of the temporal variability likely was due to this discharge. Most ARD discharges would not show such pronounced short term variability. Therefore, short term temporal bias is not expected to be a significant factor in most receiving waters monitored for ARD. However, for some ARD investigations, pilot studies to assess temporal bias may be appropriate.

3.1.2 Field Notes

The QA program should include maintenance of adequate field notes. The notes should include a description of each site in adequate detail that the site can be located again, records of field data (such as pH measurements), and descriptions or data on factors that could influence water quality, such as:

- sampling date and time (which may allow water quality to be related to stream flow data or to known events upstream);
- air and water temperature;
- weather conditions (to allow evaluation of the effects of storm events); and
- locations of discharges, seeps, or ephemeral streams; observations about bank slumpage; or other potential sources of environmental contamination.

3.1.3 Technician Training and Evaluation

Technicians should be trained by a person experienced in water sampling. Training should include methods of sample collection, preservation (with emphasis on filtering), and selection of appropriate sampling sites (within general sampling locations, which should be selected by a water quality specialist). Techniques for avoiding sample contamination should be emphasized.

To evaluate technician performance, the supervisor should review field QC results. Frequent contamination in sample blanks would suggest substandard field techniques. Unusual variability among replicates could also indicate a problem. If a problem is suspected, the supervisor should observe the technician in the field and provide additional training, if required.

3.1.4 Sample Collection

The study objectives and water depth will determine the appropriate sampling device. In a shallow (wadable) stream, samples are usually collected directly in the sample bottles. Polyethylene bottles are recommended for most ARD parameters, but samples for mercury should be collected in glass or teflon bottles (IWD 1983). For deeper waters (lakes, large rivers, estuarines) accessed by boat, subsurface samples are usually collected with a remote sampling bottle (Niskin or similar device made of metal-free plastic). Sampling pumps are appropriate if a depth-integrated sample is desired. In addition, automatic sampling devices (IPSCO or similar) are available, if intensive sampling is the objective of the study (e.g. hourly sample collection for 24 hours).

Appropriately cleaned (acid washed) bottles are usually supplied by the analytical laboratory. The bottles should be rinsed again in site water prior to sampling, unless they already contain preservative (as for mercury). Remote samplers such as Niskin bottles should be cleaned between sampling trips by soaking in a weak solution of nitric acid. In the field, they should be rinsed with distilled water between sites and rinsed again with site water prior to sample collection.

A field meter should be used to measure pH, which can change during sample transportation for a variety of reasons, including iron precipitation and biological activity. The meter should be calibrated with standard buffer solutions prior to use and periodically checked for drift.

3.1.5 Sample Preservation, Temporary Storage, and Shipment

Sample preservation has been summarized by IWD (1983). Immediately upon collection, samples should be placed in a cooler (ideally at 4°C) for transportation and storage. Metals samples should be preserved as soon as possible. For most metals, the preservative is 2 mL concentrated HNO_3 per litre of sample. Mercury should be preserved with 1 mL concentrated H_2SO_4 and 1 mL of a 5% $K_2Cr_2O_7$ solution per 100 mL of sample.

Samples for dissolved metals should be filtered using a 0.45-μm acid-rinsed filter prior to preservation. Filtration should occur on the same day the samples are collected, but stream side filtration is not recommended. Rather, filtration should normally be done indoors in as clean (dust-free) an environment as possible.

The following protocol should be followed to minimize the potential for contamination. Filtration equipment should be rinsed with distilled water prior to use and between samples. A small portion of each sample should be passed through the filter and discarded prior to filtration of the remaining sample. Filters should be handled only with plastic forceps. The technicians' hands should never touch the filter or any other part of the equipment that will come in contact with the sample.

Samples should be shipped to the analytical laboratory as soon as possible. Parameters such as acidity and alkalinity should be measured within 24 hours. Holding times under a week should not affect analyses for other parameters.

3.1.6 Conclusions

Sample contamination during sample collection, filtration, and (less likely) transportation is the most common cause of poor quality results for water samples. Careful field techniques, assured by appropriate technician training and monitoring, can minimize this problem.

Study design must address the natural variability of water quality (particularly seasonal or flow-related), if data are to be representative of the true annual or seasonal conditions.

3.2 Effluents

3.2.1 Introduction

Mine effluents that might be sampled as part of an ARD monitoring study include tailings impoundment effluent, effluent from a water treatment facility (eg. lime treatment to neutralize ARD), and interceptor ditches which collect runoff from pit walls, ore or waste rock stockpiles.

Effluent monitoring is a permit requirement for operating mines. The Waste Management permit normally will specify the required sampling location(s) and method(s). However, the objectives of special studies may require different sampling approaches, while abandoned mines may not be bound by permit regulations.

Criteria for effluent sampling have been prepared by Bollans et al. (1989) and Tetra Tech (1987). The following discussion is based on these sources and on the experience of the study team.

3.2.2 Field Sampling Design and QC Aspects

3.2.2.1 Site Selection

Samples should be collected from a representative point in the effluent stream. For tailings impoundments, the discharge pipe or stream, if accessible, is an appropriate location. In some cases, grab samples taken from the impoundment itself may be adequate. Effluent from a water treatment facility should be collected from a zone of turbulent mixing, for example, immediately downstream of a flow disturbance such as a pipe constriction, bend, or flow control device (Bollans et al. 1989). Site selection criteria for ditches are similar to those for streams. If two or more ditches combine, the sampling point should be far enough downstream of the last confluence to ensure that complete mixing has occurred.

3.2.2.2 Sample Replication and Sampling Frequency

Depending upon the study objectives, a pilot study could be used to determine required sample replication. However, composite samples are typically used for effluents which vary considerably in quality or flow rate (Bollans et al. 1989). Continuous monitoring may be use for parameters such as pH and conductivity for which appropriate equipment is readily available.

Sampling frequency is specified in the Waste Management Permit. Composite sampling requires a specified sampling frequency done over a specified period of time. The most common time period is 24 hours, although some Waste Management Permits may specify four-hour composite samples (Bollans et al. 1989). For 24-hour composites, individual grab samples may be collected as infrequently as every three hours or as frequently as four times per hour (Bollans et al. 1989). The more variable the effluent with respect to either quality or flow, the higher the sampling frequency should be.

For special studies, particularly those involving interceptor ditches, seasonal sampling may be appropriate. For these studies, criteria for determining sampling frequency will be similar to those described for surface waters (Section 3.1.1.2).

3.2.2.3 Assessment of Bias

Effluent samples are less subject to bias from contamination than are surface water samples due to their typically high levels of substances such as metals. However, transportation blanks, sampler blanks, and pre-and post filtration blanks can and should be used to assess potential sample contamination. In addition, the use of transportation and post-filtration blanks is important when effluent and receiving water samples are collected, filtered and transported during the same field trip to assess potential contamination of the receiving water samples from the effluent samples.

3.2.3 Field Notes

Field notes should include the general information described in Section 1.3 plus:

- weather conditions (such as heavy rain) that might influence the quality of water in tailings impoundments or runoff in interceptor ditches;

- notations of process upsets or other unusual conditions that might influence the quality of effluent from water treatment plants; and

- other pertinent observations such as seepage entering a ditch or the presence or iron staining in ditches or on tailings.

3.2.4 Technician Training and Evaluation

Technicians who collect effluent samples usually collect surface water samples as well, and training procedures are generally similar (see Section 3.1.3). Technicians will require training in the proper operation of automatic composite samplers, if these devices are used.

3.2.5 Sample Collection

The procedure for collection of grab samples from tailings impoundment outlets or ditches is similar to that described for surface water (Section 3.1.4).

Composite samples can be collected manually or with an automatic sampler. However, pH samples should not be collected with an automatic sampler as this parameter is likely to change during storage (Tetra Tech 1987). If an automatic sampler is used for other parameters it must be constructed from materials that will not introduce contaminants. For ARD-related samples, this means that portions of the sampler which come in contact with the sample must be glass, plastic (polyethylene or polypropylene), or teflon. Stainless steel components are not recommended for collecting metals samples (Bollans et al. 1989).

For composite sampling, the sample size should be adjusted so that the final sample volume is manageable (<10 L) (Bollans et al. 1989). Flow proportional composite samples should be prepared if effluent flow varies by more than ±15% of the daily mean more than 10% of the time (Bollans et al. 1989).

3.2.6 Sample Preservation, Temporary Storage, and Shipment

Sample preservation, storage, and shipment procedures for effluent samples are identical to procedures for surface water samples (Section 3.1.5). However, one special precaution is needed when effluent and surface water samples are collected and preserved at the same time. All surface water samples should be filtered before any effluent samples are filtered to reduce the potential for carry over of contaminants in the filtration equipment.

3.2.7 Conclusions

Many procedures and sources of contamination for effluent samples are similar to those for surface water samples. Specialized devices for effluent sampling include automatic composite samplers. Water quality technicians who also sample effluent will require training in the use of these devices.

3.3 Groundwater, Pore Waters and Seepage

3.3.1 Introduction

Groundwater, pore waters and natural groundwater discharges (seeps) may be monitored for indications of acid generation in the vicinity of waste rock dumps, mine walls and tailings impoundments, and down gradient of all mine facilities, including open pits and underground mines.

Special procedures are required for these waters because they are usually not in equilibrium with the atmosphere and may alter rapidly if oxygenated. General protocols for handling water samples described in Section 3.1 and 3.3 apply to groundwater and pore water.

Canect Environmental Control Technologies (CECT 1989) comprehensively described and evaluated 20 different methods for obtaining groundwater samples in the context of monitoring acid generating tailings piles. The majority of these methods were not developed specifically for ARD monitoring but have general application to subsurface water sampling. Specific procedures for sampling natural groundwater discharges (seeps) have not been described, but several aspects are comparable to other sub-surface water sampling programs.

3.3.2 Field Sampling Design and QC Aspects

3.3.2.1 Site Selection and Sample Size

Selection of monitoring well locations will depend on the hydrogeological conditions at any individual site. Usually sites will be selected up-gradient and down-gradient of a facility to compare background and potentially contaminated groundwater (Steffen Robertson and Kirsten *et al.* 1990). The screen in a monitoring well must be carefully selected (CECT 1989) to avoid monitoring units which are not hydrogeologically connected to the mass of waste rock or tailings.

Sample size is selected based on the requirements of the analytical procedure.

3.3.2.2 QC Aspects

General guidelines for including replicates in a water sampling program should be followed. Deionized water blanks should not only be included at the beginning and end of the sample batch but at several randomly selected positions within the batch. Monitoring of carry-over contamination during field filtration and analysis is essential because metal concentrations may be high in this type of sample and suspended solids levels elevated.

3.3.3 Field Notes

Standard field notes for monitoring groundwater quality include:

- number of well volumes removed during purging;
- general description of the appearance of the water (colour, suspended sediment, precipitates formed upon contact with the atmosphere, odour); and

- field water quality measurements (conductivity, pH, dissolved oxygen, Eh, temperature).

3.3.4 Technician Training and Evaluation

A qualified hydrogeologist should be responsible for selection of locations and installation of monitoring wells. However, technicians can readily be trained to sample groundwater. Technician performance can be evaluated in the manner described in Section 3.1.3.

3.3.5 Sample Collection

3.3.5.1 Selection of Sampling Method and Equipment Preparation

CECT (1989) identified two important criteria for selecting an appropriate sampling method:

- degree of isolation from the atmosphere and other contamination; and

- degree of sample disturbance during collection.

The first criterion is particularly important when monitoring ARD contamination in groundwater. Interaction with the atmosphere could result in oxidation and precipitation of reduced metal species, and changes in pH and conductivity. Decrease in pressure could also cause de-gassing of sulphur compounds and carbon dioxide.

The second criterion should be considered if the water is to be collected from a specific sampling interval. Seepage meters, peristaltic pumps, piston pumps, bladder pumps, syringe samplers, and double valve bailers combine high degree of sample isolation with low sample disturbance (CECT 1989).

All equipment should be carefully washed between sample stations to remove water and sediments carried over from the previous station.

3.3.5.2 Sample Container Selection and Sample Collection

A variety of sample containers are available for collection of the water samples. Guidelines for selection of containers for receiving water samples apply equally to ground water samples. If alteration of the samples by contact with the atmosphere is a concern, gas impermeable bottles with sealed lids should be used, and the headspace minimized during sampling.

Water samples from groundwater wells may be contaminated by a variety of routes during sampling:

- sampling equipment, such as bailers, and tubings must not come into contact with soil or tailings;

- observation wells must be thoroughly purged before sampling to remove contamination introduced during drilling. However, the water level in the well must not be allowed to drop below the level of the screen, since this could create an oxidized zone in the sampling interval;

- sample bottles should be stored away from the well area until required to avoid contamination by dust and mud; and

- slug tests must be conducted after all samples have been collected to avoid contaminating well water with water used in the test. The well should be purged prior to collection of the next sample.

Seepage samples may be contaminated by contact with iron-rich sediments deposited around the seep. Since emergent seepage streams are usually shallow, every effort should be made to avoid disturbing the seepage sediments which are commonly metalliferous.

3.3.6 Sample Processing, Preservation, Temporary Storage and Shipment

Groundwater samples for metal analysis should be filtered and preserved as soon as possible after sampling, as described in Section 3.1.5. Guidelines for preservation, storage and shipment of water samples are given in Section 3.1.5.

3.3.7 Conclusions

Sample alteration by contact with the atmosphere, and contamination from previous sites and soils and tailings in the vicinity of observation wells are the most likely causes of poor quality results in groundwater monitoring. Alteration can be controlled by selecting an appropriate sampling device. Contamination can be minimized by thoroughly cleaning equipment between sites, and keeping equipment and bottles away from surface soils and tailings during sampling.

CHEMISTRY OF SOLID MEDIA

4.1 Sediments

4.1.1 Introduction

Sediments can be collected to monitor accumulation of metals resulting from neutralization, dilution and oxidation of acid rock drainage. Sediments may be collected from a variety of environments which include marine, estuarine, lacustrine and fluvial. Methods for sampling sediments can be divided into two types. The first group of methods was developed to sample sediments that are remote from the sampler (that is, under more than a few centimetres of water). The second group was developed to sample sediments which can be sampled directly (non-remote sediments). The latter group is primarily restricted to fluvial and some estuarine sediments.

The American Society for Testing and Materials recently reviewed 196 reports and papers describing techniques for sampling sediments (ASTM 1991). Their review forms the basis of this section of the report.

4.1.2 Methods for Sampling Remote Sediments (oceanic, estuarine, lacustrine)

Methods for sampling remote sediments include corers, grabs and dredges. Advantages and disadvantages of these samplers are summarized in Table 4.1-1 (ASTM 1991).

4.1.2.1 Field Sampling Design and QC Aspects

Site Sample and Selection Size

Selection of sampling locations should be based on the objective of the study. For acid rock drainage monitoring studies, the survey is usually designed to detect a plume of metal-enriched sediments originating from a source such as a discharge point or stream.

The size of sample collected will be dictated by the analytical method. A sample device should be selected to yield an adequate sample. If the sediment is coarse, a larger sample may be required to statistically represent the coarsest fractions (see Section 4.1.3.1).

TABLE 4.1-1

SUMMARY OF BOTTOM SAMPLING EQUIPMENT[a] (ASTM 1991)

DEVICE	USE	ADVANTAGES	DISADVANTAGES
Fluorocarbon plastic or Glass Tube	Shallow wadeable waters or deep waters if SCUBA available. Soft or semi-consolidated deposits.	Preserves layering and permits historical study of sediment deposition. Rapid-samples immediately ready for laboratory shipment. Minimal risk of contamination.	Small sample size requires repetitive sampling.
Hand Corer with removable Fluorocarbon plastic or glass liners	Same as above except more consolidated sediments can be obtained.	Handles provide for greater ease of substrate penetration. Above advantages.	Careful handling necessary to prevent spillage. Requires removal of liner before repetitive sampling. Slight chance of metal contamination from barrel and core cutter.
Box corer	Same as above	Collection of large sample undisturbed allowing for subsampling.	Hard to handle.
Gravity corers, that is, Phleger Corer	Deep lakes and rivers. Semi-consolidated sediments.	Low risk of sample contamination. Maintains sediment integrity relatively well.	Careful handling necessary to avoid sediment spillage. Small sample, requires repetitive operation, and removal of liners. Time consuming.
Young Grab (fluorocarbon plastic- or kynar-lined modified 0.1 m^2 van Veen)	Lakes and marine areas	Eliminates metal contamination. Reduced bow wake.	Expensive. Requires winch.
Ekman or Box Dredge	Soft to semi-soft sediments. Can be used from boat, bridge, or pier in waters of various depths.	Obtains a larger sample than coring tubes. Can be subsampled through box lid.	Possible incomplete jaw closure and sample loss. Possible shock wave which may disturb the fines. Metal construction may introduce contaminants. Possible loss of "fines" on retrieval.
PONAR Grab Sampler	Deep lakes, rivers, and estuaries. Useful on sand, silt, or clay.	Most universal grab sampler. Adequate on most substrates. Large sample obtained intact, permitting subsampling.	Shock wave from descent may disturb "fines". Possible incomplete closure of jaws results in sample loss. Possible contamination from metal frame construction. Sample must be further prepared for analysis.
BMH-53 Piston Corer	Waters of 4 to 6 ft deep when used with extension rod. Soft to semi-consolidated deposits.	Piston provides for greater sample retention.	Cores must be extruded on site to other containers - metal barrels introduce risk of metal contamination.
Van Veen	Deep lakes, rivers, and estuaries. Useful on sand, slit, or clay.	Adequate on most substrates. Large sample obtained intact, permitting subsampling.	Shock wave from descent may disturb "fines". Possible incomplete closure of jaws results in sample loss. Possible contamination from metal frame construction. Sample must be further prepared for analysis.
BMH-60	Sampling moving waters from a fixed platform.	Streamlined configuration allows sampling where other devices could not achieve proper orientation.	Possible contamination from metal construction. Subsampling difficult. Not effective for sampling fine sediments.
Petersen Grab Sampler	Deep lakes, rivers, and estuaries. Useful on most substrates.	Large sample can penetrate most substrates.	Heavy, may require winch. No cover to permit subsampling. All other disadvantages of Ekman and Ponar.
Shipek Grab Sampler	Used primarily in marine waters and large inland lakes and reservoirs.	Sample bucket may be opened to permit subsampling. Retains fine grained sediments effectively.	Possible contamination from metal construction. Heavy, may require winch.
Orange-Peel Grab Smith-MMcintyre Grab	Deep lakes, rivers, and estuaries. Useful on most substrates.	Designed for sampling hard substrates.	Loss of fines. Heavy - may require winch. Possible metal contamination.
Scoops, Drag Buckets	Various environments depending on depth and substrate.	Inexpensive, easy to handle.	Loss of fines on retrieval through water column.

a Comments represent subjective evaluations.

QC Aspects

Specific quality control programs for these sediments have not been described. Initially, collection of triplicate samples is recommended to evaluate within site variability resulting from sediment heterogeneities such as chemical or sedimentological vertical and lateral zonation. Subsequently, frequency of replication may be reduced if within site variability is adequately defined by the initial sampling phases.

4.1.2.2 Field Notes

The following field notes should be recorded:

- location of sample site;
- sediment type and texture;
- sediment colour;
- presence, type and strength of odours. If hazardous materials are expected then a field gas detector (HNu ionization detector) could be useful to measure hydrocarbons. Protective breathing equipment should be considered for situations where H_2S may be encountered;
- depth of penetration of the core or grab sampler;
- degree of leakage or surface disturbance; and
- presence of any large debris or benthic fauna.

4.1.2.3 Technical Training

Some training is required to operate the various sampling devices, and to correctly handle and sub-sample the sediments. Experience is needed to determine whether a suitable sample has been obtained.

4.1.2.4 Device Selection, Sample Collection and Sample Containers

The type of sampler used should be selected based on the depth of water, type of substrate, and the study objectives. Grab and dredge samplers are generally less efficient than corers but are easier to handle, require fewer operators, and are usually more easily obtained (ASTM 1991). Corers should be used where obtaining a good section of the bed with the surface fine sediments in place is important.

The size of sample is also a consideration when selected a sampling device. Corers often yield smaller samples than other devices, possibly necessitating compositing of samples from several locations to obtain sufficient material for testing.

All sampling equipment (corers, dredges, grabs, scoops, mixing bowls) should be thoroughly cleaned prior to sampling using a procedure consisting of: (1) soap and water wash; (2) distilled water rinse; (3) methanol rinse; (4) methylene chloride rinse; and (5) site water rinse (ASTM 1991). Between sites, the equipment should be thoroughly rinsed with deionized water.

Handling of samples during collection will depend on the intended analysis. If alteration by contact with the atmosphere is not a concern, the sample can be deposited in a mixing bowl and, after thorough mixing, a sub-sample collected using a suitable non-reactive plastic sample scoop (ASTM 1991). If the oxidation state of metals is to be determined, a core sampler must be used. The interior part of the core should be sampled since it is least likely to have been disrupted during sampling and contaminated by contact with the corer surfaces. Fast-freezing of core has also been shown to reduce the rate of oxidation of manganese and iron, but stratigraphy of the core is disrupted (ASTM 1991).

Polytetrafluoroethylene (PTFE) or high density polyethylene containers are generally recommended for sediments to be analyzed for metals since these plastics do not sorb metal species. If volatile components (for example, sulphur compounds) are of interest, air tight PTFE containers, or glass containers with PTFE-lined screw caps should be used.

4.1.2.5 Temporary Storage and Shipment of Samples

Samples should be refrigerated or kept on ice to avoid alteration in transit. Analysis within two weeks is advisable but not always essential (ASTM 1991).

4.1.2.6 Conclusions

Methods for sampling remote sediments are well-developed and their advantages and limitations thoroughly investigated and quantified. Sampling devices should be selected based on the sampling environment and the objectives of the sampling program. Most significant biases potentially arise from disruption of fine bottom sediments by the sampling devices, and alteration of samples upon contact with the atmosphere.

4.1.3 Non-remote Sediments (fluvial)

4.1.3.1 Field Sampling Design and QC Aspects

Field Program Design

Field monitoring stations should be selected based on a model for dispersion of contaminated sediments in the drainage system. Control sites should be included in the sampling design.

Sample Size

The size of field sample required is partly controlled by the detection limits of the proposed analytical scheme. Up to 2 grams of sediment passing a 100-mesh screen are required for some environmental metal analysis methods (EVS Consultants 1990). Mineral resource assessment programs commonly use 0.5 grams passing an 80-mesh screen (Gravel and Matysek 1988). Since fluvial sediments are often very coarse, collection of a few orientation samples is recommended to estimate the minimum field sample size to obtain several grams of sediment passing 80- or 100-mesh screens. If the size distribution of the samples is to be determined, a much larger sample may be required to yield statistically representative weights of the coarse material. Formulae are available to estimate minimum sample requirements for size fraction analysis. For example:

$$M_{min} = 4w/\varepsilon^2$$

where M_{min} is the minimum sample size of any component in a mixture weight of any fraction for a relative error of ε, and w is the overall mean particle mass (Hogg 1988). For instance, approximately, a 17 kg sample will be required for sediment with diameters up to 2 cm for a relative error of 0.05.

QC Aspects

Wide variations of stream sediment metal concentrations within a single site are very common due to local variations in drainage sediment supply, sediment texture and chemical conditions. For larger regional programs (>1000 samples), collection of field duplicates every 20 samples has been found to provide adequate characterization of within site variability. However, for smaller programs, duplicate or triplicate samples should be collected at every site. For a long term monitoring project, duplicates or triplicates should be collected initially to characterize within site variation. Single samples or duplicates should then be adequate for the rest of the program.

4.1.3.2 Field Notes

Detailed field notes are required to assess the quality of fluvial sediment samples because within site variation can be very high. Categorical variables such as sediment colour, estimated texture, and organic content have been shown to significantly affect metal content of fluvial sediments (Matysek *et al.* 1983). A complete list of field observations includes:

- **sediment characteristics:** estimated texture, colour, organic content, reaction to acid, main component rock types;
- **location of sample site:** position in channel, proximity to banks; and
- **stream characteristics:** average channel width, wetted width, gradient, channel features, bank type, bank stability, bank height, confinement, stage, flood signs, and estimated discharge rate (recorded on forms developed by FHIIP (1987)).

Streamwaters at the site should be measured for pH, conductivity, oxidation-reduction potential, and temperature to assist with later interpretation of the analytical data.

4.1.3.3 Technician Training and Evaluation

Sediment sampling technicians should be carefully trained to identify appropriate locations for collecting sediments in streams. Standard forms are available for describing sites and should be used by technicians. Samples and sample locations should be periodically checked by an experienced supervisor to ensure that sample quality is being maintained.

4.1.3.4 Sample Device Selection

Very few samplers have been described for collecting non-remote sediments, primarily because collecting a representative sample is relatively simple. Manual sampling devices are being used with increasing frequency for this type of sediment (ASTM 1991; EVS Consultants 1990). In ARD monitoring, surface sediments are usually of most interest since they were most recently deposited. Sampling using a clean plastic scoop is acceptable (EVS Consultants 1990).

In very high-energy streams, fine sediments are often difficult to locate due to dilution by very coarse material. Matysek and Day (1988) found that sediments trapped in mosses yielded the same geochemistry patterns as fine sediments and were much easier to collect. This medium apparently represents a suitable monitoring alternative to stream sediments in mountainous regions.

The collection tool (scoop, corer) should be thoroughly cleaned before sampling as described in Section 4.1.2.4. Between sites, equipment must be wiped and rinsed with stream water, or if streamwater is unsuitable or unavailable with deionized water, to avoid between site contamination. Sample containers should be selected as described in Section 4.1.2.4.

4.1.3.5 Sample Collection

The sampling location should be selected carefully to avoid biases resulting from local conditions. The following guidelines should be followed:

- the sampling location should not be close to local sediment sources such as bank collapses;
- sediments in fast flowing water should not be sampled since the fines component will be lost during collection;
- the sediment should have a high detrital component and low organic content;
- overbank sediments may be sampled if the sediment is fresh, but should be avoided if soil development has begun;
- anomalous sediment (unusual colour, texture, composition) should be avoided; and
- the fines content of the sediment should be tested in the field before collecting the sample.

In ARD monitoring, the surface sediment is most likely contaminated (EVS Consultants 1990), therefore sampling beyond surface sediments is not recommended unless variations with time are being investigated.

Samples collected from streams should be handled, shipped and stored using the same protocols described in Section 4.1.1.4.

4.1.3.6 Conclusions

Biases affecting non-remote samples are commonly due to the complex conditions at the sampling site. A number of factors are known to affect the metal composition of sediments including organic content, texture, and sediment precipitates. Duplicates and triplicates must be collected to evaluate within site sampling variability. Thorough collection of field notes will also assist with interpreting unusual or variable field results.

4.2 Soils

4.2.1 Introduction

Soils and shallow surficial materials may be sampled either as part of planning of overburden handling at proposed and operating mines, as a monitoring tool to identify emergence of contaminated groundwater from mine waste facilities, or to evaluate the development of soils on waste rock piles and tailings. Soil samples may be analysed for metals, nutrients, and acid-base accounting parameters.

Soil sampling in the context of acid generation assessments has been described in detail by Sobek *et al.* (1978). Appropriate techniques for soil sampling are described in the soil contamination literature (see Barth *et al.* 1989 for a literature review).

A large number of soil sampling devices are available including augers, tubes, drills, hoes, and spades. The relative benefits of these tools will be discussed briefly in the following sections.

4.2.2 Field Sampling Design and QC Aspects

4.2.2.1 Field Program Design

The density of sampling should be chosen in the context of the objective of the survey. Sample spacing should be selected to provide a reasonable probability of locating and defining a positive or negative anomalous area (Sinclair 1975). Where the purpose is to characterize a soil unit, formulae are available to estimate the number of samples required to reduce the variability to a given level (Barth *et al.* 1989).

4.2.2.2 Sample Size

The mass of sample required to reduce field sampling variability to an acceptable level will depend on the size of heterogeneities in the soil (see Section 4.3.3.2) (Gy 1982; Ingamells 1973). If the soil is polylithic and the coarse fragments are large, a larger sample will be required than for a finer soil. The size of sample will also depend on the proposed sample processing method. To eliminate the problem of coarse fragments, the soil may be screened to a finer fraction (Fletcher 1986). However, this could result in failure to recognize minerals associated with coarse components.

4.2.2.3 QC Aspects

Initially, a pilot study, involving collection of duplicates or triplicates at every site, should be conducted to analyse within site variability. An appropriate frequency of replicate samples can be then be selected for the main survey according to the objectives of the project.

4.2.3 Field Notes

Soil composition may vary depending on a wide variety of environmental conditions. Hoffman (1986) recommends recording the following in field notes to assist with identifying biases due to field variability.

- overburden type and origin;
- position of sample site in relation to source;
- drainage of site;
- soil moisture content;
- soil Ph;
- sample texture;
- soil horizons present and sampled;
- lithology and abundance of coarse fragments;
- possibly sources of contamination; and
- soil colour.

Standard forms for general description of soils are provided by Ministry of Environment (1980). These forms provide fields for description of, in addition to the above, horizon boundaries, soil structure, consistency, colour aspect, mottles, roots, pores, porosity, clay films, effervescence, salinity, cementation, organic matter, concretions, nodules, and clasts. This list is complete though probably too extensive for most programs conducted as part of acid generation studies (Sobek *et al.* 1978).

Sobek *et al.* (1978) recommend recording the following variables when describing mine soils:

- **Layers.** Note the presence of layers resulting from deposition of different types of materials.

- **Depth.**

- **Colour of the Matrix.** Described using a Munsell Soil Colour Chart.

- **Mottling.** Describe abundance of mottles, contrast with matrix, size, and colour.

- **Texture.** Estimate using the hand texture technique described by Ministry of Environment (1980).

- **Soil paste pH.** See Section 4.3.2

- **Coarse fragments.** Record abundance of coarse fragments and the percentage of each lithology.

- **Bridging voids.** Note the size and abundance of voids caused by irregular placement of large fragments.

- **Artifacts.** Note the abundance and depth of any objects not normally expected in a soil profile such as mine refuse and undecomposed vegetation.

- **Pockets.** Any pockets of dissimilar material should be described in terms of size, texture, colour, and abundance.

Field notes are best recorded on standard code forms so that data collection is systematic, and the data can later be stored digitally if required (Hoffman 1986).

4.2.4 Technician Training and Evaluation

Soil description and sampling must be conducted by a soil specialist, or a technician under supervision of a specialist. Although the procedures can be learnt fairly quickly, interpretation of subtle physiographic and soil features requires experience. Field descriptions and samples should be checked periodically to maintain quality of samples and field notes.

4.2.5 Equipment Preparation and Selection, Sample Collection, and Container Selection

All equipment must be carefully cleaned prior to starting a new excavation. Soil from the previous station should be carefully removed, especially if metal contamination from mine wastes is expected. Ideally, all metal surfaces in contact with soil should be wiped clean and washed with deionized water to reduce cross-contamination. As a final precaution the equipment can be driven into the soil within a few centimetres of the proposed sampling location.

Augers, tubes, and drills allow for rapid sample collection but may not allow observation of the soil horizons. For this purpose, a spade is preferred, but excavation will take longer. Gullies and artificial cuts may provide appropriate sites for soil sampling if the excavation is relatively fresh. However, older surfaces may be significantly altered when compared to unexposed material. Surface material should be scraped away to expose fresh soil for description and sampling.

When the soil is sampled with an auger or drill, examples of each soil horizon should be set aside as drilling proceeds. If a spade is used, the entire section should be opened and samples collected by cutting small steps into the wall of the excavation, thereby avoiding contamination from the horizons above.

The type of sample container used will depend on the proposed analysis. Collection of samples for analysis of soil bacteria are described in Section 5.4. If any analysis of chemical species or volatiles is proposed, gas and liquid-impermeable containers with sealed lids should be used to reduce alteration of the sample storage. The walls of the container should not allow adsorption. Samples for metal analysis and acid-base accounting can be placed in plastic bags or glass jars. Kraft paper bags are appropriate if moisture content will not be determined and prolonged storage is expected (See Section 4.2.6).

The sample number should be written clearly on the sample container and lid. As a further check, the number should also be written on a paper tag and placed in the sample bag.

4.2.6 Temporary Storage and Shipment of Samples

Soil samples should generally be kept cool in the field to avoid alteration in transit. Refrigeration is not necessary unless bacteriological or volatiles analysis will be conducted. Drying of samples in the field is acceptable only if total (or non-species specific) metals analysis will be conducted. Samples in kraft paper bags should be dried after sampling, especially if saturated, to avoid degradation of the sample bags and cross-contamination during transport. Slow drying under cool dry conditions is recommended to avoid loss of mercury and other elements easily volatalized at higher temperatures.

4.2.7 Conclusions

Biases may arise at several stages in soil sampling. The soil program should be carefully designed to ensure that the objective will be achieved. In the field, complete description of subtle soil features is necessary to assist with interpretation of results. Relatively inexperienced technicians should not describe soils unless their work is frequently checked for consistency by a soil scientist. The type of sample container should be selected according to the parameters to be analyzed, to avoid loss or alteration of components in transit to the laboratory.

4.3 Rocks

4.3.1 Introduction

In the context of ARD monitoring, rock sampling is usually conducted to evaluate the potential of a particular material to generate or consume acid and release metals. The types of testing varies from field evaluation of paste pH to kinetic testing. Sampling may be conducted during mine development to plan facilities and operational methods, or during operation and closure to evaluate the success of waste management procedures and identify areas requiring remediation.

Very little literature on sampling of rock for ARD monitoring is available. The following descriptions are mostly based on the Project Team's experience, and informal discussions with other consultants and government personnel.

4.3.2 Field Paste pH Measurement

4.3.2.1 Introduction

Paste pH is a field test used by soil scientists to assess the readily available acidity or alkalinity of a soil. The test has become popular in acid generation assessment because it provides a rapid, semi-quantitative indication of the pH of leachate likely to be produced by water percolating through a rock mass.

The test involves mixing an equal volume of deionized water with **natural** rock fines. Crushing to produce fines artificially is not appropriate. Soil scientists conduct the test with a 0.01 M solution of $CaCl_2$. In addition to pH, conductivity of the leachate may also be determined.

4.3.2.2 Field Sampling Design and QC Aspects

Rigorous guidelines for designing a survey of paste pH have not been developed. Generally, paste pH determinations should be made for each rock type with different quantities of acid-generating and acid-consuming minerals. Determinations should be repeated with sufficient frequency to ensure that results are reasonably reproducible, i.e., that the investigator is not obtaining acidic and alkaline results for visually similar rock types.

The conductivity of the leachate is useful for interpretation of the results. A conductivity near that of the deionized water indicates that secondary minerals are not abundant and the paste pH is likely to take several minutes to stabilize. The reading obtained will probably reflect the pH of the deionized water.

The pH meter should be calibrated frequently using standard solutions, especially when a wide range of results are being obtained. Re-testing of one sample throughout the day may be useful to check for drift in results.

4.3.2.3 Field Notes, Sample Collection and Analysis

In addition to standard notes for location, each measurement should be accompanied by field notes describing the rock type, mineralogy, weathering appearance, composition of fines (secondary minerals), rock odour, and moisture content.

The greatest potential for biases arises during sample collection and testing. Some causes of biases, in order of decreasing importance are:

- failure to clean the pH probe carefully after each test;
- failure to use the same water:rock volume ratio consistently;
- testing of materials with different particle size;
- variable test temperatures;
- inconsistent pH stabilization times; and
- use of domestic water rather than deionized water to conduct the test.

Generally, these problems can be avoided by standardizing procedures and carrying an adequate supply of deionized water stored under constant temperature conditions.

A technician can be trained to conduct the test in a few minutes. However, a basic geological background is needed to take good field notes.

4.3.2.4 Conclusions

Paste pH is a useful semi-quantitative field assessment technique with relatively few, easily-controlled biases. Field measurements must be supported by notes regarding geology. Conductivity measurements on the paste are also useful to interpret unstable paste pH results, or results near the pH of the deionized water.

4.3.3 Sampling for Acid-Base Accounting

4.3.3.1 Introduction

Acid-base accounting (ABA) is used to quantitatively estimate the relative potential for acid generation or consumption. Minerals of interest are sulphur species (especially sulphides) and acid-consuming minerals (such as carbonates).

Two main factors will affect the value of an acid-base account, namely, (1) the means used to ensure that a representative sample is obtained; and (2) the procedures used to ensure that the make-up of the sample does not alter significantly prior to analysis. There are few options for sampling (grab sampling, chip sampling, drilling), and no information on the relative benefits of different approaches.

4.3.3.2 Field Sampling Design and QC Aspects

Survey Design

Representativeness of the survey is controlled by the number and size of the samples collected. At the initial assessment stage a few samples will be adequate, provided that samples are selected to characterize worse than average scenarios. If a waste rock block model is to be prepared, sampling density must be chosen according to a geostatistical model.

Sample Size

Conventionally, acid-base accounting samples are the same size as the samples sent for determination of metals. However, statistical sampling theory shows that this is not appropriate. Gy (1982) developed a detailed theoretical approach for sampling particulate materials. Extreme sampling errors will result if the sample is large enough to occasionally include a particle of the component of interest, but too small to ensure that the particles are frequent enough to eliminate sub-sampling variability. For example, biases in interpretation could occur in sampling for acid-base accounting if the rock contains large sulphide grains which are very infrequently present in the sample, or only occasionally present. Each sample should be large enough to include enough of the grains to avoid extreme random variability. Gy (1982) and Ingamells (1973) provide several formulae for the appropriate number of samples for given sizes of heterogenous components. An orientation study should be conducted to determine the various sampling constants described by Ingamells (1973).

Quality Control

Collection of field duplicates is rarely described for acid-base accounting projects though it is essential to assess the selection of sample size and local rock heterogeneity. Replicate samples should be collected every 20 samples, or frequently enough to permit an analysis of variance. If enough duplicates (more than 50 pairs) are collected, the true detection limit (including field sampling errors) can be calculated (Thompson and Howarth 1978) for the ABA parameters.

The following procedure is used to estimate the precision curve and calculate detection limits (Fletcher 1986).

- From the duplicate analyses obtain a list of the means and absolute differences;
- Arrange the list in increasing order of concentration means;
- From the first 11 results obtain the mean concentration and median difference for that group;
- Repeat this procedure for each successive group of 11 results, ignoring any remainder less than 11; and
- Calculate the linear regression of the median differences on the means and multiply the intercept and coefficient by 1.048 to obtain σ_o and k, respectively. Precision (P_c) in percent at concentration c is given by:

  $$P_c = (2\sigma_o/c + 2k) \times 100.$$

  Using a suitable value for P_c (for example, 100%), the detection limit can be estimated.

4.3.3.3 Field Notes

To assist with interpretation of acid-base accounting results, the following field notes should be collected:

- overall mineralogy and petrography of the rock, with particular reference to sulphide and carbonate minerals;
- field paste pH of fines of the rock;
- degree of oxidation (presence of surface iron staining, thickness of weathering rinds, extent of weathering along fractures, sulphide or carbonate mineral-shaped voids, presence of secondary weathering products such as gypsum and iron and non-ferrous sulphates, corrosion of nearby metal);
- storage conditions, (dry, underwater, frozen), climate of region and age of samples; and
- overall integrity of individual rock pieces ("rotten" rock, slaking).

Notes should be collected on standard forms to avoid missing observations (Norecol Environmental Consultants 1991).

4.3.3.4 Technician Training

The sampling program should be designed by an experienced specialist in the area of interpretation of acid-base accounting results and the project geologists. Geological technicians can be trained to collect the samples, although the specialist and project geologists should periodically compare samples and field notes, and check that the samples are representative.

4.3.3.5 Sample Collection

The type of rock collected for acid-base accounting should be compared to the material being characterized. For example, weathered outcrop must not be collected if the purpose of the analysis is to estimate the initial acid generation potential of a rock mass (Sobek *et al.* 1978). In outcrops or old exploration excavations, both acid generating and acid consuming minerals have usually been completely leached from near surface rock. Samples should be collected several centimetres into fresh massive outcrop. Fractured outcrop is usually extremely weathered. Similarly, old core should not be sampled unless it has been stored in cool, dry conditions or frozen. Freezing apparently represents the best option for long term storage of rock for ABA (Geddes Resources 1990). Storage of core underwater may be acceptable (City Resources 1988) although some leaching of acid-consuming minerals may take place without comparable removal of acid-generating minerals.

When sampling at existing or abandoned mines, obtaining fresh material is usually difficult or not consistent with the study objectives. However, surface material is usually not representative of the rock mass as a whole. Waste rock on the weathered surface of rock dumps should be avoided by excavating to sufficient depth to reach representative material (see Section 4.3). Sampling of mine walls is also difficult since weathering may be extremely varied and deep (Sobek *et al.* 1978). Observation of weathering features (see Section 4.3.3) will help with selecting appropriate material for sampling.

Samples can be transported in any type of conventional heavy-duty plastic bags or pails. Puncturing of the bag may help to reduce humidity and reduce the rate of alteration of the sample prior to analysis.

4.3.3.6 Temporary Storage and Shipment of Samples

The samples should be submitted to the laboratory within two weeks after collection, to avoid alteration of the sample by weathering. If storage is necessary, the rock samples should not be left in moist plastic bags at room temperature but should be dried and kept cool (preferably frozen at less than -10°C).

4.3.3.7 Conclusions

The acid-base accounting program should be carefully designed to avoid biases resulting from large scale heterogeneities in the rock mass. Small samples collected during ore deposit evaluation may not be suitable for acid-base accounting.

The effect of natural and storage conditions on rock weathering should be considered. Highly weathered outcrop, pit walls, or core may not be appropriate if the rock mass being evaluated is unweathered.

All sampling must be supported by detailed description of the geology, mineralogy and weathering features.

4.3.4 Sampling for Kinetic Testing

4.3.4.1 Quality Assurance

Since larger samples are collected for kinetic testing than for ABA, material is generally more representative of the rock mass as a whole. However, there are similar concerns with the effect of prolonged storage under damp conditions. Weathered rock will take longer to stabilize than fresh rock when tested due to initial leaching of acidic weathering products. However, weathered material may be selected to compress the length of the kinetic test. Rock appearance (weathering features) and storage conditions (length of time, exposure to moisture) should be noted to assist with selecting representative samples and later evaluation of test results.

4.3.4.2 Quality Control

Since kinetic testing is expensive, field duplicates are rarely collected. Nonetheless, replication is essential to monitor the effect of field sampling variability on results from the tests. The number of replicates should be selected and justified on a site specific basis.

MINE WASTES

5.1 Tailings Solids

5.1.1 Introduction

Samples of tailings solids are frequently required as part of assessment and ongoing monitoring of operating and abandoned tailings impoundments. Surface samples are not representative of the tailings mass as a whole, due to oxidation. Therefore, tailings must be excavated. All common methods for obtaining samples of tailings were recently compiled by Canect Environmental Control Technologies (CECT) (1989). This report provides descriptions and a bibliography for each method, and charts for selecting appropriate equipment. A report prepared by Golder Associates and Senes Consultants (1985) for sampling uranium mine tailings also provides some relevant information.

The CECT (1989) report describes 19 types of sampling equipment varying from powered augers and rotary drills to hand-driven corers and shovels.

5.1.2 Field Sampling Design and QC Aspects

5.1.2.1 Sampling Design

Field sampling design must account for the complex nature of tailings impoundments and piles. Tailings are deposited in layers of differing composition and permeability. Tailings deposited proximally to the discharge point are likely to have less slimes than tailings deposited in distal parts of the impoundment. Movement of discharge points also results in complex stratigraphy. Chemical conditions will also vary laterally through unsaturated zones, hard pan layers, and saturated zones. Sufficient sampling points should be selected on a case-by-case basis to adequately characterize stratigraphic and water table variations.

5.1.2.2 Sample Size

Most tailings are composed of fine material, therefore large samples are not required to statistically represent the coarser fractions. If the typical diameter of the tailings is 2 mm, a minimum 20 g sample would be sufficient (see section 4.1.3.1). If fine laminae are not being sampled, a larger sample should be collected to homogenize local small-scale composition variations.

5.1.3 QC Aspects

Replicate samples should be collected at least every 10 samples, or frequently enough to provide data for an analysis of variance.

Control reference materials have been developed for tailings (Smith and Bowman 1990) and should be included in every batch of samples. Unfortunately, these reference materials have negligible neutralization potential and cannot be used for monitoring this parameter.

5.1.3 Field Notes

Detailed logs of bore holes and trenches are required to interpret tailings samples. Field observations should include:

- moisture content of tailings, degree of saturation, and location of the water table if known;
- texture of the tailings;
- colour and cementation (hardpan);
- primary minerals, particularly sulphides and carbonates;
- secondary minerals such as limonite;
- paste pH; and
- odour.

Any free water should at least be field-tested for pH, conductivity, oxidation-reduction potential and temperature.

Observations should be recorded on standard forms to avoid missing information. Standard borehole logs used for geotechnical studies are not adequate because they lack space for recording data relevant to acid generation studies.

5.1.4 Technician Training and Evaluation

Technicians will require special training in sample collection procedures and identification of stratigraphic features. Excavation logs should periodically be checked against the stratigraphy by a specialist with experience in tailings impoundment assessment.

5.1.5 Sample Collection

Since tailings are fine-textured and normally not in equilibrium with the atmosphere, special care must be taken throughout sample collection, storage and shipping to avoid alteration of samples by prolonged contact with the atmosphere.

5.1.5.1 Preparation of Equipment and Sample Containers

All sampling equipment must be thoroughly cleaned prior to sampling and between sampling stations to avoid cross-contamination of samples. Deionized water is usually adequate, although methanol can be used to prevent bacterial cross-contamination (CECT 1989).

Sample containers should be carefully selected based on the objectives of the study and the composition of the tailings. Plastic bags can be used for less reactive (low sulphide) samples. Air will come into contact with the tailings, but effects should be fairly limited. Air- and water-impermeable sealed containers must be used for highly reactive tailings to limit access of oxygen to the samples.

5.1.5.2 Procedures for Reducing Biases during Sampling

The main cause of biases during sampling is contamination of samples by tailings in the same section. This contamination can be avoided (CECT 1989) by:

- collecting sample material from the interior of cores or grab samples rather than material that has come into contact with the borehole or excavation wall;
- using casing in non-cohesive tailings to minimize collapse of walls;
- minimizing use of drilling mud and analyzing the mud to check for contamination;
- removing cuttings from the borehole;
- replacing blunt drill bits; and
- avoiding excessive vibration and jarring during operation.

When samples are collected from trenches or gullies, surface oxidized material should be removed to expose fresh material.

Samples should be immediately deposited in appropriate containers (see Section 5.1.5.1).

5.1.6 Sample Storage and Shipping

Speciation of elements in the tailings can change significantly during storage and shipping. If total concentrations of elements are to be determined, these changes are not significant. Most studies will involve some determination of species, such as carbonates, sulphides, and sulphates. Therefore, alteration of the samples is a major concern. After removal from tailings the following changes may occur.

- oxidation of sulphide minerals in moist conditions will result in conversion of sulphide to sulphate, elemental sulphur, and gaseous forms of sulphur (for example, H_2S, SO_2);
- increases in temperature allow bacterial oxidation of sulphide minerals to accelerate;
- acid release by oxidation of sulphides will consume acid neutralizing minerals, causing loss of CO_2 and leaching of metals from sulphide ore minerals;
- reduced metal species will be oxidized; and
- evaporation could result in precipitation of minerals from pore waters.

To control these processes, samples should be placed in air tight containers with a small headspace. Transportation of samples on ice or refrigerated at 4°C is strongly recommended to prevent the changes described above. Samples should be analyzed promptly.

5.1.7 Conclusions

The quality of tailings samples can be affected at several stages of the sampling process. Cross-contamination of samples must be controlled since tailings commonly have high and variable concentrations of significant parameters. Alteration of samples during shipment and storage is probably the most significant cause of biases. The high surface area of tailings, and the change from anoxic to strongly oxygenated conditions can result in rapid changes in speciation of sulphur, metals, and acid neutralizing minerals.

5.2 Pore Gases in Tailings and Waste Rock Dumps

5.2.1 Introduction

Techniques for sampling pore gases in unsaturated zones of tailings and waste rock piles were recently compiled and reviewed by Canect Environmental Control Technologies (CECT 1989). The five methods described are either under development for acid generation monitoring, or have been adapted from methods developed for monitoring of soil gases or landfills.

Very few studies are available to suggest procedures for collecting high quality samples. Monitoring ports are usually installed after solid sampling. Samples of gas are either analyzed on-site by an autoanalyzer, or stored in syringes for later analysis.

5.2.2 Sample Collection, Field Analysis, Shipping and Storage

The following guidelines were suggested by CECT (1989):

- all tubing and sample containers must be gas impermeable, and the sampling system must be carefully checked for leaks;
- the sample volume should be minimized to avoid drawing oxygen into the pore spaces;
- sampling of unwanted zones should be avoided by sealing zones with bentonite; and
- development of vacuums should be avoided to limit pressure gradients.

If a destructive autoanalyzer is used, the gas should not be re-circulated to the pore-gas sampler.

Samples collected in syringes should be maintained at temperatures close to that of the sampling environment to reduce the effect of temperature-induced pressure changes. Shipping of samples by air cargo should probably be avoided since barometric changes could result in equilibration of the sample with air. Prolonged field storage of samples is not recommended since seals on syringes may decompose.

5.2.3 Conclusions

Pore gas sampling is a sophisticated technique requiring installation and calibration of specialized equipment. Biases will arise if the sampling interval is not adequately sealed from above, which may be difficult when sampling waste rock dumps (Golder Associates 1989). Analysis on-site is preferred since gas samples must be shipped under carefully controlled conditions.

5.3 Sampling Waste Rock Dumps

5.3.1 Introduction

Waste rock dumps at operating and abandoned mines are sampled where a potential for acid generation exists, and the composition and structure of the dump is unknown. Guides describing procedures for sampling waste rock dumps are currently not available. However, sampling of surface materials is not appropriate because they are more weathered than interior rock. In addition, the surface may only be representative of the last batch of waste, if the material was end-dumped.

In British Columbia, waste rock dumps have been sampled as part of acid generation studies at Mt. Washington, Westmin's Buttle Lake mine, and BHP-Utah's Island Copper mine. At Island Copper, the Becker Hammer drilling method was used (M. Li, personal communication). This method was developed for coarse, poorly-consolidated materials. Golder Associates (1989) preferred to use an air rotary rig equipped with a hammer bit at Mt. Washington. A tricone bit was also tried but the bit could not establish an adequate cutting surface due to the loose blocky material and large void spaces within the dump. For both the Becker Hammer and air rotary rig, a casing was installed during drilling to keep the hole open for installation of monitoring wells and improve sample recovery. The following discussion refers to both methods since data are not available to permit a comparison.

5.3.2 Field Sampling Design and Quality Control Aspects

5.3.2.1 Sampling Design

Design of the survey should follow the general guidelines discussed in Section 5.5.1. The following problems associated with sampling of waste rock dumps should be considered when designing the survey:

- waste rock dumps normally are extremely heterogeneous both laterally and vertically;
- the dumps commonly contain non-rock waste, such as discarded rail and ties;
- water conditions are usually variable due to the development of perched water tables and preferential flow paths; and
- the drilling methods yield very poor recovery of core (0 to 20%) due to loss of crushed rock in void spaces.

As much information as possible should be gathered about the composition and structure of the dump prior to drilling by referring to mining records, and possibly by excavating shallow surface pits or conducting geophysical surveys. The local surficial geology should be also be understood to allow recognition of the base of waste rock dump and the beginning of native materials.

5.3.2.2 QC Aspects

The high costs of drilling generally preclude opening of replicate holes to assess local variability. Nonetheless, duplicate holes would be useful to demonstrate consistency of stratigraphy and spatial variation in recovery of core.

5.3.3 Technician Training

All waste rock drilling must be supervised by a specialist experienced in drilling coarse unconsolidated materials.

5.3.4 Sample Collection

Approaches for minimizing bias in rock sampling are discussed in general in Section 4.3. Field notes should include the usual drilling observations such as noting the rate of penetration of the drill (for example, blow counts), recording stratigraphy, and recording changes in moisture content.

Contact of drill returns with water should be minimized to reduce loss of soluble weathering products which may be important in the acid generation assessment.

Since the technology of waste rock dump drilling is still being developed, the work should be conducted by an experienced drilling company and monitored by a technician with experience interpreting the results of drilling unconsolidated materials.

General procedures for storage and shipping of rock samples collected for acid-base accounting are described in Section 4.3.

5.3.5 Conclusions

Drilling of waste rock dumps has been used at several mines in British Columbia and elsewhere but routine procedures have not been described. Possible biases and difficulties with sampling have not been addressed.

5.4 Bacterial Measurements

5.4.1 Introduction

Concentrations of bacteria such as *Thiobacillus ferroxidans* may be determined as part an acid generation monitoring program. These bacteria catalyse the acid generation reaction at low pH (2 to 3) and therefore increasing or decreasing populations are indicative primarily of changing pH. Monitoring of bacteria is particularly important during tests of remediation technologies intended to control bacterial activity.

Soils, rocks or waters can be tested for bacteria, although testing of the latter for *Thiobacillus* has never been reported. Techniques are available for directly culturing bacteria in the field without the need for sample collection and preservation (Redigel undated).

5.4.2 Field Sampling Design and QC Aspects

Sampling design specifically to monitor *Thiobacillus* populations has not been described in the literature. Soil and rock samples collected for analysis of other parameters (acid-base accounting, metals) will generally be suitable for bacterial analysis.

Frequent duplicate sampling (at least 10%) should be included in the program to evaluate local variability in factors which affect the bacteria such as changing soil gas, moisture and pH. These conditions will probably vary over short distances, leading to significantly different micro-environments with significant variations in bacterial populations.

5.4.3 Sample Collection

Samples should be collected in pre-sterilized, water- and gas-impermeable containers with sealed lids to assist in preservation of the soil environment (Silver 1986). Aseptic techniques should be used to avoid cross-contamination of samples (Beak Consultants 1991a). In addition to parameters normally measured for soil and rock samples, conditions which affect bacterial growth such as temperature, nutrient availability, moisture content, and paste pH should be noted.

5.4.4 Temporary Storage and Shipment of Samples

Ideally, storage conditions in the field should be equivalent to the source soil or rock environment (Silver 1986). In practice, the samples should be placed on ice and maintained at a temperature of less than 10°C (Beak Consultants Ltd. 1991a). The samples should not be dried or moistened in the field since changes in these conditions could result in altered growth of the bacteria (Sobek *et al.* 1978).

Bacterial samples should be shipped to a laboratory for analysis within 24 hours (Beak Consultants 1991a).

5.4.5 Conclusions

Information is generally lacking on appropriate procedures for monitoring *Thiobacillus* in soils, rocks and waters. Current methods have been adopted from descriptions for other soil bacteria. Cross-contamination in the field and sample storage conditions are probably significant causes of biases and should be carefully controlled.

BIOTA

6.1 Fish

6.1.1 Populations

Fish populations are usually monitored as a part of biotic community characterization in baseline studies for proposed mines. They may also be sampled in post-operational monitoring programs. However, their value for evaluating impacts of ARD (or other mine-related impacts) is limited due to difficulty in distinguishing between these impacts and the many other factors that cause population fluctuations.

In many cases, fisheries studies include only characterization of species present and a qualitative evaluation of relative numbers. Other data frequently collected include length, weight, sex, condition, age, and stomach contents.

Balkwill and Coombes (1991) compiled a manual for lake sampling, including fish collection methods and field measurements, but they generally did not consider QA/QC aspects. Beak Consultants Ltd. (1991a,b) make brief references to QA/QC methods for fish sampling. The following discussion is based on these references and the experience of Norecol's fisheries biologists.

6.1.1.1 Site Selection

The planning phase of the study should consider site location. Beak Consultants Ltd. (1991b) cite criteria for selecting reference sites for pulp mill environmental effects monitoring. These criteria would also be applicable to ARD monitoring. However, establishing control sites for fish population studies is difficult because of the mobility of fish. It usually is not appropriate to establish control sites upstream of actual or potential ARD discharges. If barriers that isolate upstream and downstream populations are present, then species composition will be different (anadromous species will be absent upstream). In addition, habitat conditions may be very different (less nutrient availability, less wetted area and even intermittent flow in headwaters). Control sites can be established in reference tributaries or lakes, if barriers to movement are present and comparable habitat exists. However, in many cases it may not be possible to establish adequate control sites.

For stream studies, once a general sampling area has been identified, the investigator should conduct a reconnaissance to determine the reaches (homogenous section of a stream) to be sampled. The sampling area should include a sufficient number of similar habitat types (riffle, pool) within a reach to account for within-habitat variability. If the study objective is community characterization, then site selection should ensure that all habitat types are sampled. Sample stratification by habitat type with proportional allocation of effort may be appropriate, if prior knowledge or initial sampling results suggest significant differences in population densities or variability. In some cases, the study objective may focus on populations of a particular species whose preferred habitat would be sampled.

For lake studies, the field crew should attempt to sample in both the shallow areas and the deeper waters as well as different basins, if present. Appropriate shallow areas include creek mouths and shoals, which are often preferred feeding areas for some species (Balkwill and Coombes 1991).

6.1.1.2 Field Notes

For stream sampling, habitat notes should be recorded on the standard Resource Analysis Branch (RAB) reach and point sample cards.

Proper documentation for lake sampling includes netting times, weather and water conditions (including results of field measurements such as pH, dissolved oxygen, Secchi disc depth), mesh type and size, net size, net depth and locations (Balkwill and Coombes 1991).

Field notes should also include data from field measurements (weight, fork length, condition) and identification (by individual fish) of any samples retained for laboratory analysis (scales, stomach contents).

6.1.1.3 Technician Training and Evaluation

Technicians should be carefully trained in the proper use of equipment, including techniques for net-setting and electroshocking. For example, in electroshocking, proper settings can be the difference between catching fish, killing fish, or having no effect. In addition, technicians should all learn and follow standard procedures for weighing fish, measuring fork length, and assessing fish condition.

Since return-for-effort (i.e. catch per unit effect) depends in part on field technique, it provides a method of assessing technician performance. Additional effort checks (Section 6.1.1.4) can be used to evaluate technician performance in relation to pre-determined return-for-effort performance criteria (Beak Consultants Ltd. 1991a).

Substandard performance may indicate a need for additional training. However, some of the causes of poor catch per unit effort may not improve with training. For example, the effectiveness of a beach seining effort depends in part on the technician's strength (which affects the rate at which the net is pulled). Similarly, the effectiveness of electroshocking depends partially on the operator's ability to see fish under water and the time it takes him/her to respond. Deficiencies in these areas can be overcome by practice and training, but differences among individuals will remain.

Results of field measurements can be affected by the technician's training and technique. Occasionally the supervisor should observe the technician's weighing and measuring techniques. Comparison of measurements and condition assessments of the same fish by different investigators may be used to evaluate performance in addition to evaluating data quality. The supervisor should observe whether fish and equipment are relatively dry for each measurement and whether weight scales are shielded from wind.

6.1.1.4 Sample Collection

Equipment Selection

The choice of equipment will depend upon the type of habitat and species being sampled and the weather conditions. For shallow streams the appropriate equipment includes electroshockers and beach seines. Minnow traps (gee-traps or similar) can be used in lakes and stream inlets and outlets, if the study objective requires only qualitative data. As electroshockers and beach seines do not work effectively in water depths >10 m, other equipment is required for lakes and deep rivers. Commonly used sampling methods for deep water include fyke nets, angling, and (where destructive sampling is not a concern) gill nets. Mesh size selection is important, as large mesh sizes may undersample the smaller size classes or species, while small mesh may result in no captures and/or gilling. Weather conditions can affect performance of some samplers; for example, minnow traps are considered ineffective below 4°C due to inactivity of the fish.

Field Collection and Assessment of Data Quality

Fish populations in streams are usually estimated by removal methods, which typically use beach seining or electroshocking to collect the majority of fish in a reach. The accuracy of methods which use a fixed number of passes can be checked by the occasional additional effort (Beak Consultants Ltd. 1991a). For example, the data quality objective may be to retrieve a fixed percentage (95%) of the fish present in a reach or to achieve a specified coefficient of variability for the estimated mean population density. If sampling normally consists of three passes, a fourth pass can be made to determine whether the first three achieved the objective. Alternatively, if a field computer is available, the data can be assessed progressively, with sampling continuing until the data quality objective is achieved (eg. using the formula of Seber and LeCren 1967).

Lake fish populations are generally estimated based on catch per unit effort (relating population size to decreasing return for angling or netting time). Additional effort checks are appropriate for assessing the accuracy of the data collected.

Removal and catch per unit effort methods are highly subject to bias due to the technician's ability or technique. Occasional duplication of effort (having two technicians sample the same site or reach and comparing their results) can be used to assess technician bias. If this QC assessment is used for electroshocking studies, the evaluator should be aware of possible confounding bias. Mesa and Schreck (1989) demonstrated abnormal behaviour of cutthroat trout (including seeking cover) for at least 24 hours after electroshocking. This potentially could affect fish catchability, making the second technician appear less efficient than the first.

Collecting and recording certain field data can aid in identifying the potential for bias. For example, high visibility waters will yield fewer fish per netting effort. Overcast conditions and/or turbid waters generally improve catch success (Balkwill and Coombes 1991). While the investigator cannot control weather and turbidity, understanding the conditions will help with data evaluation. Similarly, returns per unit effort of electroshocking are often lower in low-conductivity water. Field conductivity measurements can suggest that certain populations have been underestimated. Measurement of conductivity during a pilot study will allow for switching to an alternative sampling method, if necessary.

6.1.1.5 Field Processing and Sample Preservation

Field processing includes weighing, measuring fork length, determining sex, assessing condition and collecting samples as necessary for age determinations (scales) and food preference (stomach contents). Training to reduce biases in field processing was discussed in Section 6.1.1.3.

If there is any doubt about the species caught, a representative sample should be retained for identification in the laboratory. In addition, some study objectives may require that a proportion of the fish caught be retained for detailed measurements in the laboratory.

These samples need to be preserved. The preservative of choice is a 10% solution of formalin. Specimens larger than about 15 cm should be slit just below the lateral line to allow the preservative to enter the body cavity. For smaller fish, the preservative should be injected directly into the body cavity using a hypodermic syringe (Balkwill and Coombs 1991). Formalin is also appropriate for preserving fish stomach and their contents.

In some cases, field processing should include photography. For example, if the study objective or taxonomy require an assessment or description of fish colour, the specimens should be photographed as soon as possible after capture (Balkwill and Coombs 1991). Preserved fish rapidly lose their colour.

6.1.1.6 Conclusions

Fish populations are often measured as part of baseline programs, but the use of fish populations in evaluating impacts presents problems. Even with careful study design, it may not be possible to select suitable control sites.

Fish population estimates are subject to a number of biases due to the technician's ability and the effects of environmental and weather conditions on sampler performance. Many of these biases can be reduced or eliminated through careful training and selection of the appropriate sampling method for the local conditions. Other biases can be anticipated and noted when reporting the data.

6.1.2 Tissues

Fish are frequently collected for tissue metals determinations in conjunction with both baseline programs and assessment of suspected ARD impacts. Tissue metal levels are considered reasonably good indicators of available metals in the environment, although some metals (such as copper and zinc) are less useful indicators because they are metabolically regulated (Smith 1987a).

Field QA/QC procedures to reduce sample contamination are frequently described in reports of tissue metals studies (eg. Smith 1987a) and are discussed by Tetra Tech (1987). The following sections are based on these sources and on project team experience and discussions with IWD personnel.

6.1.2.1 Field Sampling Design and QC Aspects

Site Selection

Difficulties in selecting appropriate sampling sites for fish tissue sampling are similar to those described for fish population sampling, except that habitat differences may be less important. Fish motility can cause serious biases. In addition to the difficulty of finding a control population that is isolated from the study population, it may not be possible to ensure that fish collected downstream of a suspected ARD source did not migrate to that site from another area where they were exposed to contaminants.

The ideal study site would have barriers both upstream and downstream of the suspected ARD source. Only resident species would be available for study, but this would not normally be a problem so long as the same species were present both upstream and downstream.

In the absence of an ideal study site, limiting samples to juvenile fish may be an acceptable alternative (City Resources 1988). Juveniles are usually less mobile than adults, and when they do move, it is generally downstream. However, there are limitations to the use of juveniles. They are not appropriate if the study objective is to assess potential effects of fish consumption on human health. In addition, composite samples of juveniles are generally required to provide sufficient tissue for analysis. Compositing results in the loss of information on population variability.

Species Selection

An important study design consideration is the species to be sampled. Since different fish species show different patterns of metals accumulation, a good indicator species for one metal may not be useful for another metal. In some cases, a pilot study may be necessary to determine the appropriate species for monitoring. However, a good general rule of thumb is to include at least one predator and one bottom-feeding species (May and McKinney 1981, Smith 1987).

The study objective will also guide the species selection. If the effect of fish consumption on human health is a concern, commercial or game species should be sampled.

Sample Replication and Sampling Frequency

Ideally, the planning phase of the study should include setting a data quality objective for the standard deviation or coefficient of variability of tissue metals levels, and a pilot study should be conducted to determine the appropriate sample size. In practice, this level of effort is beyond the scope of most baseline programs and is probably appropriate only for long-term monitoring studies.

In the absence of a baseline study, sample size should be in excess of 10 replicate fish (or enough juvenile fish to provide more than 10 composite samples). This recommendation is based on Hakanson (1984) who concluded that 10 samples are too few to provide adequate estimates of metal levels in fish under most circumstances. In addition, Hakanson suggested that the number of samples should increase with increasing contamination.

Most baseline monitoring programs consist of a single collection of fish for tissue metals determinations. However, this approach provides no method for evaluating variability due to seasonal or longer term changes in fish condition, behaviour, or baseline metals availability. There are no guidelines available for determining an appropriate sampling frequency even for long term monitoring studies.

Assessment of Biases

The potential for bias due to sample contamination is likely to be as significant for tissue samples as it is for water samples (Section 3.1.1.3) Tissue monitoring studies typically do not include sample blanks, probably because there is no tissue equivalent of a distilled water blank. Theoretically, reference tissue samples (which are available) or hatchery-reared fish (in which population metal levels could be estimated) could be used as transportation blanks. Using transportation blanks might be appropriate if the study objective were to collect legal samples (G. Masson, BC Research, personal communication).

6.1.2.2 Field Notes

Field notes should include information similar to that recorded for water sampling sites (Section 3.1.2). In addition, they should report observations about fish condition or behavioural abnormalities (if noted).

6.1.2.3 Technician Training and Evaluation

The technician training and evaluation procedures described for fish population sampling (Section 6.1.1.3) should generally be adequate for tissue metals sampling, provided that the field technician is not responsible for dissecting the fish. The training should be supplemented with some instruction on sample handling (Section 6.1.2.4).

6.1.2.4 Sample Collection

Any of the sampling equipment described for fish population estimates (Section 6.1.1.4) is appropriate for collecting fish for tissue metals determinations. The sampling methods should be simplified, because the maximum sampling effort is that required to collect the desired number and species.

6.1.2.5 Sample Preservation, Storage, and Shipping

Avoiding contamination is a major consideration when sampling fish tissue for trace metals. To minimize contamination, the fish should be handled as little as possible. Crew members should wear clean polyethylene gloves when handling the samples (Tetra Tech 1987). Other sources of contamination include sampling gear, engine exhaust, and ice used for cooling. When sampling is done from a boat, the boat should be positioned so that engine exhaust does not fall on deck (Tetra Tech 1987). To avoid contamination from melting ice, Tetra Tech (1987) recommends that samples be wrapped in aluminum foil and placed in water tight bags. They note that the fish skin will protect samples from contamination from the aluminum foil.

Dissection of the fish is the most significant potential source of sample contamination. For this reason, dissections should be done in the laboratory where dust and other potential contaminants can be controlled more effectively (Tetra Tech 1987).

Fish must be weighed and measured, but this also can be done in the laboratory. Some changes in these parameters may occur due to sample preservation (freezing), but the changes should not create significant biases for the intended use of the data.

Because freezing may cause internal organs to rupture, Tetra Tech (1987) recommends that fish be placed on ice but not frozen during transportation if analyses will be performed on "selected tissues". However, much ARD-related sampling will be conducted in remote areas where it may not be possible to ship samples to a laboratory for several days. In this event, freezing will be necessary. Fish should be frozen at -10°C, if possible, as soon as possible after collection. They should remain frozen until and during shipment to the laboratory.

6.1.2.6 Conclusions

Sampling design is important to ensuring high quality (and interpretable) tissue metals data. The major considerations are site selection, species selection, and replication. Sampling frequency is a potentially important factor that is rarely addressed.

Sample contamination is potentially a significant bias but one whose risk can be reduced by minimizing sample handling in the field.

6.2 Benthic Invertebrates

Benthic invertebrate sampling is becoming a component of baseline monitoring programs with increasing frequency. Benthic invertebrates are also being considered as a core component of environmental effects monitoring for metal mines (EVS Consultants 1991).

Beak Consultants Ltd. (1991a) described some QA/QC protocols for field collections of benthic invertebrates. Other aspects of field QA/QC such as biases related to sampling and required sample sizes have been reviewed by Resh (1979), Klemm et al. (1990), and Beak Consultants Ltd. (1991b). The aspects of a QA/QC program for benthic invertebrate sampling are considered in the following sections.

6.2.1 Field Sampling Design and QC Aspects

6.2.1.1 Site Selection

Basic considerations for selecting benthic invertebrate sampling sites in streams are similar to those for selecting water sampling sites. These include selecting at least one control site (MacDonald 1991 recommends two control sites) and having other sampling locations dependent upon the point(s) of impact or expected impact. Ideally, sampling sites should be downstream of the ARD source being monitored and upstream of all other potential sources of impacts (not limited to ARD-related parameters). In addition, sampling sites chosen should encompass comparable habitat(s).

Within each site, the number of habitat types sampled will depend upon the study objectives. Normally for ARD-related studies the objective includes some comparison of population numbers or community structure over space and/or time. This type of comparison can be limited to a single habitat type (eg. riffle) and does not require complete characterization of the invertebrate fauna of the reach. If the study objective requires more extensive characterization, a stratified sampling design that includes representative habitat types is appropriate.

Within lakes or estuaries samples may be located at increasing distances from a known or suspected discharge point (river inflow, effluent pipe, or seep). The sampling design should include at least one control site in a reference lake (if available) or at a location well away from the anticipated zone of impact in a large lake or estuary. For some studies, sample stratification by overlying water depth or tidal level may be appropriate.

6.2.1.2 Sample Replication and Sampling Frequency

Replicate samples are necessary to permit statistical analysis of benthic invertebrate data. Monitoring studies typically rely on a low number of replicates (three or four). Timms (1985) justified the use of four replicates in lake sampling, noting that this number typically captured >95% of the species at a site and that the coefficient of variation was usually <20%. A test with more replicates did not improve these numbers much, while use of three replicates dropped the number of species captured to <90% and increased the coefficient of variation to >30%. Timms' analysis illustrates the use of the additional effort check recommended by Beak Consultants Ltd. (1991a).

For stream benthos (Resh 1979) indicates that larger numbers of replicates may be necessary to achieve a 95% confidence interval of even ±40%. Resh suggests a sequential sampling approach to determine the required number of replicates. He notes that since sample processing (sorting, identification, and enumeration) is usually the cost-limiting factor, a large number of samples can be collected. Sorting should proceed until the pre-determined data quality objective (eg. a relative error ≤20%) has been achieved. Although this approach has merit, apart from cost considerations, the number of samples may have to be limited to avoid impacts from destructive sampling techniques.

A stratified sampling design may be useful for reducing variability and the consequent requirement for larger sample numbers. Stratification based on current, depth, and a combination of physical factors may be possible even within an apparently "uniform" riffle (Resh 1979). Bass (1985) recommends stratified sampling of different substrate types, noting differential distributions of chironomid species among wood debris, leaf debris, and sand microhabitats.

Sampling frequency is not adequately addressed by most benthic studies. Freshwater benthic populations, in particular, show great temporal variability due to the life histories of aquatic insects. High densities of early instar larvae may be present immediately after a hatch, but densities may be much lower within a few weeks due to poor survival. Furthermore, the inability of taxonomists to identify early instar larvae past the family level confounds the analysis of benthic data. Pilot studies to determine adequate sampling frequency usually are not practical or feasible. Alternatively, MacDonald (1991) recommends sampling in spring (before freshet) and late fall.

6.2.2 Field Notes

Field notes should contain a detailed site description. The Resource Analysis Branch point sample card is generally appropriate for this purpose, although it should be supplemented with notations of potential effluent inputs. The record of sampling procedure should be modified for benthos to include number and locations of replicate samples, type of sampler, sampling time, and sampling intensity (for time-limited methods). If stratified sampling is used, then the field notes should contain information on microhabitat characteristics (substrate type, velocity and depth).

6.2.3 Technician Training and Evaluation

Since the effectiveness of many benthic sampling devices is subject to operator biases (Section 6.2.4.1), technicians should be thoroughly trained in the operation of the selected sampler. Additional effort checks (return for number of samples) can be used to assess technician performance (Beak Consultants Ltd. 1991a).

6.2.4 Sample Collection

6.2.4.1 Equipment Selection

A wide variety of benthic sampling devices is available. Different types of samplers are appropriate for sampling from rock/cobble, sand, and mud substrates, and different water depths. These samplers also have different biases. Resh (1979) has reviewed most of the general types and some specific samplers. His summary of their biases and possible bias remedies is presented in Table 6.2-1. Klemm et al. (1990) provide similar tables.

A sampler type not covered in detail by Resh's discussion is the artificial substrate sampler. This type of device reduces between-sample variability by providing a uniform habitat. However, the samples it collects usually represents the invertebrate

TABLE 6.2-1

SELECTED EXAMPLES OF FACTORS THAT AFFECT BENTHIC SAMPLING DEVICES AND MAY RESULT IN SAMPLING BIAS

FACTOR	SAMPLERS AFFECTED	PROBLEMS CREATED	REMEDY
A. FACTORS RELATED TO CHARACTERISTICS OF THE SAMPLERS			
Backwash created in sampler by water not being able to pass through net	Netted and kick samplers	4 to 30% loss of benthos around sides of sampler	Increase the net's surface area and/or decrease size of net opening; use enclosed double netted sampler; alternatively use a hand-operated Ekman grab or cylinder box sampler
Washout of surface organisms upon placement of sampler	Hess sampler	Turbulence scours substrate surface	Use permeable sides
Disruption of substrate surface by shockwave when sampler strikes bottom	Corer and Grab samplers	Loss of small organisms and surface dwellers	Modify Ekman grab by removing screens and incorporating heavier materials in design; alternatively use a pneumatic grab, a box sampler, or a modified corer
Disturbance of biota	Surber sampler and Allan grab	Underestimation of biota due to disruption when sampler is set in place	Modify Allan grab by adding screened openings on top
	Shovel sampler	Loss of motile organisms	
Variable depth of penetration into substrate by sampler	Grabs	Inconsistent volume of sediment sampled; loss due to overfilling or incomplete closure	Leave 5-cm space above substrate; alternatively use a corer whenever possible
	Surber	Failure to consider stream hyporheic zone	Two stage sampling, surface and hyporheic
Variable area sampled	Shovel sampler	Area sampled laterally is variable	
Sampler mesh size too coarse	Netted samplers	Early instars, small and slender organisms missed	Finer mesh, or preferably a double bag sampler
Sampler mesh too fine	Netted samplers	May cause backwash (see above)	Coarser mesh as in double bag samplers
Sampler dimension too large	All Samplers	Increases sorting time; may not detect population aggregations	Smaller samples
	Grab samplers and corers	Inefficient cost/sample ratio	When density > several hundred/m^2 use corer, when < use Ekman grab, alternatively use multiple corer
Sampler dimension too small	All samplers	May not detect aggregations; variability increased due to edge effect	Use nested sampler to determine optimal sampler dimension
Operator inconsistency	All samplers	Systematic error in population estimates	Single operator; or correction factor for each operator

TABLE 6.2-1

SELECTED EXAMPLES OF FACTORS THAT AFFECT BENTHIC SAMPLING DEVICES AND MAY RESULT IN SAMPLING BIAS

FACTOR	SAMPLERS AFFECTED	PROBLEMS CREATED	REMEDY
B. FACTORS RELATED TO CHARACTERISTICS OF THE ENVIRONMENT			
Water depth limitations in lotic environments	Surber and Hess samplers	Surber sampler limited to <30 cm depth	From 0.5 to 4 m use an airlift sampler; 0.4 to 10 m deep, SCUBA and dome suction sampler or modified Hess sampler; or use a modified Allan hand operated grab or bottombasket samplers
Substrate - stony	Grab samplers, corers	Grabs may not close; cylinder sampler cannot penetrate	Substitute airlift or dome suction sampler and artificial substrate as above
Substrate - mixed	Grab samplers, corers	Differential penetration	Specific samplers for different substrate types; stratified sampling
Current too slow	Surber and kick samplers	Organisms do not drift into net	Enclosed sampler such as modified Surber or Hess sampler
Current too fast	Netted samplers	Backwash, resulting in a loss of organisms	Substitute a modified sampler with controlled flow
Current fluctuations	All samplers	Rapid change in flow may scour study area	
Low air temperatures	Netted samplers	Samples freeze in net before organisms are removed	Catch bottle or a zippered net
Sampling in vegetation	All samplers	Loss of organisms during removal; inability to close sampler	Use samplers summarized by Resh (1979) or artificial vegetation
Sampling in open water	Lotic: drift samplers; lentic: all samplers	Lotic: net clogging and changes in current and flow pattern affect estimation of water volume sampled. Lentic: sampling a consistent volume of water; scattering of organisms	Lotic: use Parshall flume drift net or waterwheel drift sampler; Lentic: use column samplers or pull-up trap
Habitat small	All samplers	Sampling destroys habitat	Smaller sampler dimension; artificial substrates
Water chemistry	All samplers	Presence of springs, man-made outfalls, and other conditions may influence microhabitat distribution of biota	Reconnaissance

Source: Resh 1979.

community in only a limited way. Artificial samplers tend to select for colonizing organisms or species which prefer the substrate type the sampler provides.

Sampler selection will depend upon the sampling location (depfh, substrate type, and whether standing or flowing water). Netted samplers (Hess, Surber, Waters and Knapp) are appropriate for stream sampling. Grabs and corers are appropriate for the soft substrates lakes and estuaries, with grabs being designed for sampling deeper waters and corers being confined to shallow water (unless operated by divers).

In addition, sampler selection should include consideration of sampler biases and the study objective. For example, the fact than an artificial substrate sampler fails to collect a representative faunal sample may not be significant if the study objective is to compare changes in some subset of the fauna over time.

6.2.4.2 Field Collection and Assessment of Data Quality

Once a sampling device has been selected, it should be operated correctly. Proper operation can be achieved by adequate technician training (Section 6.2.3).

Data quality can be assessed against an established data quality objective related to variability and/or by extra effort checks, as described in Section 6.2.1.2.

6.2.5 Field Processing and Sample Preservation

Field processing is generally limited to sample screening or sieving to remove debris and reduce the size of samples. In many cases, not even screening will be done, and the samples will simply be preserved as collected and shipped to the laboratory.

Resh (1979) has summarized the biases associated with screening and preservation. The major bias associated with screening is loss of organisms by using too coarse a mesh. A finer mesh may be required for sieving live organisms than for sieving dead organisms (Storey and Pinder 1985). The extent of the bias can be assessed by screening the sample through two or more mesh sizes and comparing the results (a variation on the additional effort check).

Recommendations for sample preservation vary. Klemm et al. (1990) suggest that samples should be preserved in 70% to 80% ethyl alcohol (ethanol), but that soft-bodied organisms (oligochaetes, leeches) should first be placed in 10% formalin for at least 10 minutes to fix the tissue. MacDonald (1991) recommends field preservation in 10% formalin, with possible transfer to alcohol at the laboratory. Formalin, if used, should be buffered to a neutral or slightly alkaline condition with borax (Klemm et al. 1990).

Weight loss bias is associated with preservation in alcohol (Resh 1979). For most ARD study objectives, weight loss may not be a significant consideration. Where weight is important, formalin should be substituted.

A more serious problem associated with preservation is sample deterioration caused by incomplete penetration of the preservative. This is a particular problem with unscreened samples. Remedies include sample screening in the field (Beak Consultants Ltd. 1991b) or use of additional preservatives (MacDonald 1991) and careful shaking to ensure that the preservative is distributed throughout the sample. Buffered formalin should be used for samples that contain significant amounts of sediment or debris because of its superior ability to penetrate the sample.

6.2.6 Conclusions

Careful planning is essential for benthic studies. A clear study objective and data quality objective are necessary to determine sampling design and sampler selection. Even with careful planning the data quality objective which can be achieved may be limited by considerations of cost and potential for impacts from destructive sampling techniques.

6.3 Periphyton

Periphyton (benthic algae) is not included in ARD monitoring programs as frequently as are benthic invertebrates. Nevertheless, changes in algal species composition or biomass (chlorophyll *a*) can be used as an indicator of ARD impacts. In addition, algae are useful for monitoring responses to other mine impacts such as discharges of nitrate related to explosives use.

Sampling protocols for periphyton are less well documented than protocols for invertebrates. The following sections are based largely on experience of the study team and discussions with scientists from IWD.

6.3.1 Field Sampling Design and QC Aspects

6.3.1.1 Site Selection

General site selection criteria for periphyton monitoring are similar to those for benthic invertebrate studies (Section 6.2.1.1). Sampling may be confined to a single habitat type (eg. riffle). Alternatives include stratification by microhabitats or preparation of composite samples from many randomly collected subsamples (Stevenson and Lowe 1986). For most ARD-related study objectives, all samples should be collected from areas having similar current conditions (e.g. the thalweg).

6.3.1.2 Sample Replication and Sampling Frequency

As discussed for benthic invertebrates (Section 6.2.1.2), the required number of replicates should be determined based on a pre-established data quality objective such as collecting 95% of the species. Sequential sampling similar to that described for benthic invertebrates (Section 6.2.1.2) can be used to determine the required sample size. Cost considerations may modify the number of replicates actually used; but the impact to destructive sampling will usually be a lesser concern for periphyton than for invertebrates because most techniques affect only the organisms collected and not the habitat.

Baseline monitoring programs may involve only a single sampling time. This approach fails to identify the effects of algal biomass changes and algal species succession, which may occur seasonally (Sheehan et al. 1980). Ideally, monitoring frequency should be sufficient to provide some understanding of these changes.

6.3.2 Field Notes

Field notes for periphyton studies should be similar to those for benthic invertebrate studies (Section 6.2.2). The extent to which the study area is shaded and any observations of apparent scouring by the current should be noted.

6.3.3 Technician Training and Evaluation

Technicians should be adequately trained in the selected sampling method and preservation techniques. Training may involve operation of a sampling device (such as a Stockner-Armstrong sampler) or proper installation of artificial substrates.

6.3.4 Sample Collection and Assessment of Bias

A limited number of methods are available for sampling periphyton. These fall into two categories, methods of scraping algae from rocks or other substrates and collection on artificial substrates.

Several approaches to collecting a sample from natural substrates are possible, including:

- removing algae from a portion of a rock or other substrate;
- removing all algae from a single rock; and
- removing all algae from all substrates within a defined area (quadrat).

Sheehan et al. (1980) compared the former two approaches. Removing all algae from a portion of a rock yielded consistently higher biomass estimates per unit area than removing all algae from an entire rock. They concluded that the difference resulted from investigator bias: the field personnel were unconsciously selecting sections of the rock having higher algal biomass.

Using an entire rock as a sampling unit removes a potential source of bias. It also is likely to reduce sampling time compared with a quadrat method. However, it may maximize between-replicate variability, because a rock is potentially a habitat unit or patch.

Artificial substrates (plexiglas plates, clay tiles) provide a number of advantages over natural substrate sampling. They are easier to sample and produce reduced variability (and increased precision) because of their uniform texture and history (Stevenson and Lowe 1986). Lamberti and Resh (1985) found that chlorophyll *a*, phaeophytin, and total organic material (ash-free dry weight) on unglazed clay tiles accurately represented those parameters on natural substrates.

To ensure that data from artificial substrates are representative of natural substrates, the microhabitat of the artificial substrate should closely resemble that of the natural substrate. Artificial substrates should be placed as close as possible to the natural substrate to increase the probability that they will be exposed to similar conditions of light, temperature, current and water chemistry (Stevenson and Lowe 1986).

To achieve reliable results with artificial substrates, recommended sampling protocols should be followed carefully. For example, Sheehan et al. (1980) found that when plexiglas plates were left in a stream for six to nine weeks (as opposed to the three weeks recommended by Patrick et al. 1954) algal competition and the development of an invertebrate grazer fauna biased measures of algal production and growth rates. They also reported difficulties with scouring due to the orientation of the tiles relative to the current.

The three-week rule for incubating plexiglas plates apparently does not apply to clay tiles (or does not apply in all situations). Lamberti and Resh (1985) found that a 28-day exposure was required before chlorophyll *a* concentrations on unglazed clay tiles accurately represented natural substrates, while a 63-day exposure was necessary before ash-free dry weight represented natural conditions.

6.3.5 Field Processing and Sample Preservation

Field processing of periphyton includes preserving samples for species identification and enumeration and for chlorophyll *a* analyses. Samples for species identification should be filtered if necessary to reduce water volume, washed into sample bottles, and preserved in acid Lugol's solution. Samples for chlorophyll *a* analyses should be filtered, mixed with a magnesium carbonate slurry, and frozen. They should be maintained frozen and in the dark until and during shipment to the analytical laboratory.

6.3.6 Conclusions

Periphyton samples may be useful for some ARD monitoring programs. They can be collected efficiently on artificial substrates, although some study objectives may require natural substrate sampling.

6.4 Plankton

Plankton are not commonly sampled in conjunction with baseline studies for mines, nor are they recommended as part of the environmental effects monitoring program (EVS Consultants Ltd. 1991). However, they are often sampled to characterize communities or productivities of lakes and estuaries. Acid rain studies have shown some phytoplankton species to be sensitive to acidic inputs, and the same or similar species may be useful indicators of ARD.

Balkwill and Coombes (1991) briefly describe a method of collecting plankton in lakes but do not discuss QA/QC aspects. However, in the literature on marine plankton sampling methods, sampler biases and other factors affecting precision and accuracy have long been recognized (eg. Fleminger and Clutter 1965; Pillar 1984).

6.4.1 Field Sampling Design and QC Aspects

6.4.1.1 Site Selection

For baseline studies, sampling sites should be chosen to characterize the phytoplankton and/or zooplankton communities of the lake or estuary. If the lake of interest contains several basins, sampling might be limited to the basin expected to receive the discharge and a control basin. If the lake is small, a reference lake with similar characteristics should be sampled. A pilot study may be necessary to select a suitable control lake, and in some cases no adequate control may be available. In estuaries, a control point well removed from the expected discharge should be sampled. Tide and current patterns should be considered to ensure that the "control" point will not be impacted when or if a discharge occurs.

For monitoring an existing or suspected ARD source, sampling sites should be located at increasing distances from the discharge site. However, Stevenson and Lowe (1986) note that when sampling phytoplankton that it is difficult and time consuming to determine where the water masses sampled have originated. A control site in a reference lake or well-removed from the zone of expected impact should also be included, if possible.

Within a site, sampling depth is a consideration. Vertical plankton hauls can be used to sample a range of depths simultaneously, while tows may sample a limited range of depths. Plankton traps (such as the Schindler trap) are available to sample discrete depths, if stratification by depth is desired. The type of depth sampling selected will depend on the objective of the study and the data quality objective.

6.4.1.2 Sample Replication and Sampling Frequency

Balkwill and Coombes (1991) state that the "normal procedure" for lake sampling is to take three total vertical hauls per site, allowing plankton to be sampled from all parts of the water column. They do not consider how this approach affects the precision of the data.

The contagious or patchy distribution of plankton and its effect on sampling precision is widely recognized (Fleminger and Clutter 1965, Pillar 1984). Ideally, a pilot study or sequential sampling exercise (Section 6.2.1.2) should be used to determine the number of replicates (and number of sampling points within a lake or estuary) necessary to characterize the plankton based on a pre-established data quality objective for percent of total species or variability (in numbers or biomass). The sequential sampling approach is particularly appropriate for plankton sampling as the cost of sorting and enumeration greatly exceeds that of collecting extra samples. In addition, over sampling plankton is less likely to cause impacts than over sampling benthos. If cost or other considerations dictate a limited number of replicates, the precision of the data can be assessed by the occasional additional effort check.

Plankton distribution varies in time as well as in space, with species succession of both phytoplankton and zooplankton occurring over the course of the growing year. Accurate characterization of lake or estuarine plankton communities requires frequent enough sampling to monitor these natural changes. Complete characterization of seasonal succession is probably not feasible. However, at a minimum, sampling times should correspond to the phytoplankton spring maximum and summer plateau periods, if possible.

6.4.2 Field Notes

Field notes should include the following information:

- locations of the sampling sites (mapped);
- site depth (for vertical hauls);
- number of hauls or tows;
- towing speed (for boat tows);
- weather conditions;
- water clarity (Secchi disc reading);
- dissolved oxygen and temperature profiles; and
- notations of any conditions that might affect sampling performance.

6.4.3 Technician Training and Evaluation

Technicians must be trained in proper handling of plankton nets or samplers. Practising vertical hand hauls is essential to achieve a constant retrieval speed. Instruction on proper towing speeds for boat tows is also necessary. Considerable operator bias is possible, particularly in hand hauling but also in boat towing.

The supervisor should occasionally observe the technicians working in the field. Technician performance might also be evaluated by comparing variability of data achieved by two or more individuals. However, it could be difficult to distinguish between variability due to the patchy distribution of the plankton and variability due to performance of the technicians.

6.4.4 Sample Collection

6.4.4.1 Sampler Selection

The selection of general sampler types will depend upon the sampling location and the study objectives. Hand-held nets are appropriate for small lakes, while boat-towed nets are appropriate for estuaries and some large lakes. Discrete depth samplers (plankton traps) should be used if information on vertical population stratification is required. Phytoplankton is best sampled by collecting water samples and examining all of the algae in the sample as phytoplankton nets are not fine enough to filter the nanoplankton (Stevenson and Lowe 1986).

The biases associated with particular samplers should also be considered in relation to the study objectives. Biases associated with plankton nets include (Pillar 1984):

- mechanical problems with nets such as loss of species through coarse mesh (relative to the size of the organisms being sampled), clogging of fine mesh, and imperfect measurement of the volume of water being filtered by the net; and

- avoidance of the sampler by zooplankton (especially the larger, more active animals).

The effects of avoidance are more pronounced for smaller nets (measured by opening diameter) than for larger nets (Fleminger and Clutter 1965, McGowan and Fraundorf 1966). Even when avoidance is not a problem, samples may be biased by loss of animals through coarse mesh (Pillar 1984). Thus, the size of the organisms being sampled can be an important consideration in selecting both mesh size and net diameter. Size factors are expected to be more important in estuarine sampling than in lake sampling because of the greater size range of marine zooplankton.

6.4.4.2 Field Collection and Assessment of Data Quality

Consistency in towing or hauling practices is often difficult to achieve, but it is essential to obtaining comparable samples. Net speed influences the avoidance ability of zooplankton; in addition, speed may alter "bow wave" effects that can reduce the efficiency of the net (Pillar 1984). In boat tows, differences in speed or towing time will cause variable volumes of water to be filtered, biassing estimates of population density. The extent of these biases will be difficult to assess.

Biases related to operator differences (particularly in hand hauling) can be reduced by ensuring that the same individual undertakes all the sampling for a particular study.

It is easier to assess biases related to mesh size or net aperture. If it is important to the study objective, samples can be collected using different equipment and compared. On a less intensive scale, an additional effort check could employ a net with finer mesh or wider opening.

6.4.5 Field Processing and Sample Preservation

Phytoplankton samples are processed in a manner similar to that described for periphyton samples (Section 6.3.5). Processing of zooplankton samples normally consists of washing the organisms from the nets and preserving them in alcohol or formalin.

6.4.6 Conclusions

Plankton samples are not a common component of ARD monitoring studies, but they can be useful in some instances. Sampling design for these studies must account for both the spatial patchiness and temporal variability of plankton. Collection equipment should be selected with an understanding of the inherent bias or potential bias of the particular sampler. Biases related to the sampler can be assessed with additional effort checks using different equipment. Biases related to operator differences can be controlled through proper training and ensuring (as much as possible) that one individual conducts an entire sampling program.

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