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Project Summary

Soil Sampling Quality Assurance User's Guide

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The inherent inseparability of a cost-effective Soil Sampling Quality Assurance/Quality Control (QA/QC) Plan from the objectives of a soil monitoring program is emphasized. Required precisions and confidence levels for the data cannot be defined until the decisions which will be made on the basis of the data are clearly stated and the consequences of making Type I (false positive) or Type II (false negative) errors are weighed. Statistical considerations are presented with special attention to analyses of variance of soil monitoring data, methods of calculating required numbers of soil samples to achieve desired precisions and confidence levels, possible applications of Kriging, and assignment of control limits to QA/QC data. The value of an exploratory or preliminary study to the cost-effective achievement of both the soil monitoring objectives and the objectives of the Soil Sampling QA/QC Plan is strongly emphasized. The value of developing a hypothetical model to estimate the distribution in space and time of soil pollutants and thus to assist in the design of the monitoring network is discussed. Methods for determination of the number and location of soil sampling sites; sample collection methods and procedures to include frequency of sampling; sample handling to include labeling, preservation, preparation for analysis, and transport; together with QA/QC aspects are presented and discussed. Finally, the importance of systems audits and training to the achievement of soil sampling QA/QC objectives is presented and discussed.

This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Las Vegas, NV, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

An adequate quality assurance/quality control (QA/QC) program requires the identification and quantification of all sources of error associated with each step of a monitoring program so that the resulting data will be of known quality. The components of error, or variance, include those associated with sampling, sample preparation, extraction, analysis, and residual error. In the past, major emphasis has been placed on QA/QC aspects of sample analysis and closely associated operations such as sample preparation and extraction. For monitoring a relatively inhomogeneous medium such as soil, the sampling component of variance will usually significantly exceed the analysis component. Thus, in this case a minimum adequate QA/QC plan must include a section dealing with soil sampling. The purpose of this document is to provide guidance in QA/QC aspects related to soil sampling.

Generally, soil monitoring is undertaken to carry out the provisions and intent of applicable environmental laws with high priority requirements associated with hazardous waste management. The objectives of soil monitoring programs are often to obtain data on the basis of which to answer one or more of the following questions:

- Are the concentrations of specified soil pollutants in a defined study region significantly different from the concentrations in a control region?

- Do the concentrations of specified soil pollutants in a defined study region exceed established threshold action levels?
- At the measured concentrations of specified soil pollutants in a defined study region what is the associated risk of adverse effects to public health, welfare, or the environment?

For each of these applications the QA/QC required to determine precisions and confidence levels for the data cannot be specified without giving careful consideration to the consequences of making an error, for example, in a decision to require, or not to require, cleanup of a contaminated region. It follows in general that to be maximally cost-effective and defensible the QA/QC objectives of a soil monitoring program *cannot be separated from the objectives of the soil monitoring program itself.*

Approximately 20 percent of the total monitoring program sample load should be allocated to QA/QC with about 5 percent of this being dedicated to the analytical effort and 15 percent to the sampling effort. Soil sampling programs must incorporate statistical designs and QA/QC plans to provide quantitative measures of both precision and representativeness.

Control samples are normally as important to a soil monitoring study as are samples taken from the study region. The data from control samples aid in the interpretation of the results from the study region and also help to identify sources and important transport routes for the soil pollutants. Accordingly, the same level of effort and degree of QA/QC checks should go into selecting and sampling a control region as goes into sampling the study region.

Experience has shown that requiring approximately 5 percent of all analytical samples to be duplicate samples will provide adequate QA/QC for determining variance between samples collected at approximately the same site. A precision less than ±20 percent is probably unrealistic for a field soil sampling effort. Table 1 provides recommendations for confidence levels and precisions for soil sampling related to hazardous waste investigations.

All statistical sampling plans are based on frequency distributions with the most common being normal or log normal. Generally, the concentrations of pollutants in soil and transport-related properties of these pollutants are distributed log normally. In addition to obtaining information on the areal distribution of soil pollutants it is necessary to determine the distribution with depth.

Table 1. *Illustrative Confidence Levels and Precisions*

	Confidence Level (Percent)	*Precision (Percent)*
Emergency Cleanup Activities	*90*	*20*
Remedial Response Studies	*95*	*20*
Planned Removal Studies	*≧95*	*≦20*

Both Type I (false positive) and Type II (false negative) errors should be considered in hypothesis testing. Tables are provided for use in determining the required number of samples to achieve defined precision and confidence levels. The location of sampling is important, and a random process should normally be used for selecting specific sampling sites. Stratification of the sampling region may reduce the variance in cases where the variance is considered to be unacceptably large. Compositing of samples is generally not recommended since it allows no estimate of the variance among the samples being composited.

Suggested QA/QC procedures for soil samples include preparation of the following samples, generally on the basis of one QA/QC sample for each 20 samples: field blank, sample bank blank, reagent blank, calibration check standard, spiked extract, spiked sample, total recoverable, laboratory control standard, re-extraction, split extract, triplicate sample, and duplicate sample.

The major technique used to detect bias in a soil sampling effort is the adding of known amounts of standard solutions to some of the samples and comparing the resulting data. It is especially difficult to demonstrate the complete absence of bias.

The confidence interval for soil samples is bounded by the confidence limits (bounds of uncertainty about the average caused by the variability of the experiment). The confidence interval is used in the development of control charts, in identifying outliers, and in determining if a set of samples exceeds some established standard. Generally, the analysis of variance of the data provides the best method for obtaining the information needed for calculating the confidence interval. An approximation of the confidence interval can be obtained by use of the ranges of replicates in a series. The tolerance limits are similar to the confidence limits but are used to identify the interval and limits into which data from the individual samples should fall.

The simplest test of hypotheses is either comparison of two mean values or comparison between the mean and some established standard, or action, value. The Student's t test is generally used for both cases.

Once objectives have been defined for a soil monitoring study, a total study protocol, including an appropriate QA/QC program must be prepared. Usually not enough is known about the sources and transport properties of the soil pollutants to accomplish this in a cost-effective manner without additional study. The suggested approach is to conduct an exploratory study including both a literature and information search followed by selected field measurements based on an assumed dispersion model. The data resulting from this exploratory study serve as the basis for the more definitive total study protocol. If one is dealing with a situation requiring possible emergency action to protect public health, it is necessary to compress the planning and study design into a short time period and proceed to the definitive study without delay. In either case, the objectives of the monitoring study constitute the driving force for all elements of the study design including the QA/QC aspects.

To develop the exploratory study protocol with its associated QA/QC plan one needs to combine into an assumed dispersion model the information obtained prior to any field measurements. On the basis of this model the standard deviation of the mean for soil samples is estimated. Value judgments are used to define required precision and confidence levels (related to acceptable levels of Type I or Type II errors). A control region is selected. The numbers of required samples may then be calculated. Additional samples should be required to provide for the validation of the assumed model. The locations of the sampling sites should be selected by an appropriate combination of judgmental (use of the assumed model), systematic (to allow for the fact that the model may be wrong), and random (to minimize bias) sampling. Sampling and sample handling must be accomplished according to standardized procedures based on principles designed to achieve both data of adequate quality and maximal cost-effectiveness. Particular attention should be given to factors surrounding the disposition of non-soil materials collected with the soil samples.

The requirements for QA/QC for the *exploratory study need not be as stringent* as for the more definitive study in the sense that acceptable precisions and confidence levels may be relaxed somewhat. Allowance should be made, however, for the collection of a modest additional number of QA/QC samples over that specified in the QA/QC plan to verify that the QA/QC study design is adequately achieving its assigned objectives. Also, all normal analytical QA/QC checks should be used.

If the exploratory study is conducted well, it will provide some data for achieving the overall objectives of the total monitoring study; it will provide a check of the feasibility and efficacy of all aspects of the monitoring design including the QA/QC plan; it will serve as a training vehicle for all participants; it will pinpoint where additional measurements need to be made; and it will provide a body of information and data which can be incorporated into the final report for the total monitoring study.

For the more definitive study, the selection of numbers of samples and sampling sites, sample collection procedures, and sample handling methods and procedures follow and build on the principles discussed and results obtained in the exploratory study.

Frequency of sampling is an important aspect of the more definitive study which usually cannot be addressed in the exploratory study because of the relatively short time span over which the exploratory study is conducted. The required frequency of sampling depends on the objectives of the study, the sources of pollution, the pollutants of interest, transport rates, and disappearance rates (physical, chemical, or biological transformations as well as dilution or dispersion). Sampling frequency may be related to changes over time, season, or precipitation. An approach that has been used successfully has been to provide intensive sampling early in the life of the study (e.g., monthly for the first year) and then to decrease the frequency as the levels begin to drop. The important principle is that the sampling should be conducted often enough that changes in the concentrations of soil pollutants important to the achievement of the monitoring objectives are not missed.

The important questions to be answered in the analyses and interpretation of QA/QC data are, "What is the quality of the data?" and "Could the same objectives have been achieved through an improved QA/QC design which may have required fewer resources?" It is *desirable to provide summarized tables of* validated QA/QC data in the final report. This approach allows users to verify the reported results as well as to begin to build a body of QA/QC experimental data in the literature which allow comparisons to be made among studies. Special emphasis should be placed on how overall levels of precision and confidence were derived from the data. If portions of the study results are ambiguous and supportable conclusions cannot be drawn with regard to the reliability of the data, that situation must be clearly stated.

The adequacy of all aspects of the QA/QC plan should be examined in detail with emphasis on defining for future studies an appropriate minimum adequate plan. Some aspects of the QA/QC plan may have been too restrictive, some may not have been restrictive enough. Soil monitoring studies should have checks and balances built into the QA/QC plan which will identify early in the study whether the plan is adequate and, if required; allow for corrective action to be taken before the study continues. This is one of the major advantages of conducting an exploratory study.

There is insufficient knowledge dealing with soil monitoring studies to state with confidence which portions of the QA/QC plan will be generally applicable to all soil monitoring studies and which portions must be varied depending on site-specific factors. As experience is gained it may be possible to provide more adequate guidance on this subject. In the meantime, it is recommended that many important factors of QA/QC plans be considered as site-specific until proven otherwise.

Another important aspect of QA/QC is auditing. The purpose of an audit is to ensure that all aspects of the QA/QC system planned for the project are in place and functioning well. This includes all aspects of field, sample bank and laboratory operations. Whenever a problem is identified, corrective action should be initiated and pursued until corrected. Sample chain-of-custody procedures and raw data are checked as appropriate and results of blind QA/QC samples routinely inserted into the sample load are reviewed. Spot-checks of sampling methods and techniques, sampling and analysis calculations, and data transcription are performed. Checks are made to ascertain that required documentation has been maintained and in an orderly fashion, that each of the recorded items is properly categorized, and cross-checking can be easily accomplished. Checks are made to ensure that data recording conforms to strict document control protocols and the program's QA/QC plan.

It is recommended that an audit of the overall QA/QC plan for sample documentation, collection, preparation, storage, and transfer procedures be performed just before sampling starts. This is to review critically the entire sampling operation to determine the need for any corrective action early in the program.

The project leader of a soil monitoring project is responsible for ascertaining that all members of his project team have adequate training and experience to carry out satisfactorily their assigned missions and functions. This is normally accomplished through a combination of required classroom training, briefings on the specific monitoring project about to be implemented, and field training exercises. Special training programs should be completed by all personnel prior to their involvement in conducting audits.

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The complete report, entitled "Soil Sampling Quality Assurance User's Guide," (Order No. PB 84-198 621; Cost: $13.00, subject to change) will be available only from:

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