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**Environmental Monitoring** Systems Laboratory Las Vegas NV 89193-3478



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## **Project Summary**

## Soil Sampling Quality Assurance User's Guide— Second Edition

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Use of the first edition of the "Soil Sampling Quality Assurance User's Guide" as a text in a series of seminars conducted at various U.S. EPA Regional Offices elicited many constructive comments for improvements from seminar attendees. Many of these suggested improvements have been incorporated in this second edition.

Specifically, the references have been updated, particularly through the incorporation of recent U.S. EPA guidelines documents. More attention has been given to experimental design, specifically to procedures for developing data quality objectives. The statistical coverage has been expanding considerably to include an introduction to applications of geostatistics and a discussion of requirements for the definition of support in conjunction with guidance for soil sampling.

This report is intended to be a living document providing state-of-theart guidance. Accordingly, from time to time revisions will be prepared to maintain harmony with improvements in soil sampling quality assurance methodology. Future revisions will be prepared, and authorship identified, on a chapter-by-chapter basis.

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 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 often 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 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 methods and procedures 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.

In general, the progression of events leading to the development of an adequate Quality Assurance Program Plan (QAPP) follows the outline shown below.

- 1 State study objectives.
- 2. Evaluate impacts of mistakes.
- 3. Define data quality objectives (DQOs).
- 4. Design study to achieve DQOs.
- Design QAPP to confirm achievement of DQOs.

Often it will not be possible to specify in advance what DQOs are possible to achieve. In such cases DQO goals should be set, a QAPP prepared, and a pilot study conducted to determine the achievability of the goals.

Present U.S. EPA guidance for development of DQOs requires that specifications for the following factors must be addressed:

- precision,
- accuracy,
- completeness,
- representativeness, and
- comparability.

A sixth factor of importance to all of the above is the detection limit of the measurement method used. Other important factors which should be considered in specifying DQOs include:

- acceptable probability of a Type I error (judging a clean area to be dirty);
- acceptable probability of a Type II error (judging a dirty area to be clean); and
- desired minimum detectable relative difference between two different geographical areas.

The development of DQOs involves an iterative interaction between management and technical staff. Management identifies the needs and resources available. The technical staff develops guidance for assisting management in making the decisions required to develop the DQOs. The DQO process usually involves a three-stage process as outlined below.

- 1. Identify decision types.
- 2. Identify data uses/needs.
- 3. Design data collection program.

The end result is site-specific guidance for evaluating and interpreting sampling data.

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 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.

In sampling of a continuous medium such as soil, it is necessary to put extra emphasis on the definition of a sampling unit. In addition to having a specified location, each sampling unit of soil has a certain three-dimensional volume, shape, and orientation. These latter three characteristics, when taken together, are

called the **support** of the sample Changes in support not only change the means of distribution, they also change the variances of concentrations and the correlations of concentrations between sampling units.

It is essential that any action level fo soils be defined as a concentration over a particular support and location relative to the ground surface. In this definition of an action level, the support is referred to in this document as the action support. For example, the action support might be defined as the top ten cm of soil over square of 100 m<sup>2</sup>.

The following table provides recommendations, as part of the DQO process for confidence levels, powers, and min mum detectable relative increases ove background for different operations situations.

Both Type I (false positive) and Type (false negative) errors should b considered in hypothesis testing. Table and an equation are provided for use i determining the required number ( samples to achieve defined confidence levels and powers. The location of san pling is also important. Stratification ( the sampling region may reduce th variance in cases where the variance considered to be unacceptably large Compositing of samples is generally no recommended since it allows no estimat of the variance among the samples bein composited. However, some compositir of samples increases the representative ness of samples and may be justified c that basis.

Suggested types of QA/QC sample include various types of blanks, labo atory control standards calibration cher standards, triplicate samples (splits), dul licate samples, various kinds of auc samples, etc. How many samples of eac type would be needed in a specific stuc is a question of considerable importanc. The recommended approach is determine how each type of QA/C sample is to be employed and the

	Confidence Level (1 - a)	Power (1 - β)	Relative Increase ove Backround [100(μ <sub>S</sub> · μβ)/μβ  to be Detectable with a Pri bability (1 - β)
Preliminary Site Investigation	70 - 80%	90 - 95%	10 - 20%
Emergency Cleanup	80 - 90%	90 - 95%	10 - 20%
Planned Removal and Remedial Response	90 - 95%	90 - 95%	10 - 20%

where  $\alpha = \text{probability of a Type I error and}$  $\beta = \text{probability of a Type II error.}$  netermine the number fro that type based n the use. For example, field duplicates are used to estimate the combined variance contribution of several sources of variation. Hence, the number of field duplicates to be obtained in a study should be dictated by how precise one wants that estimate of variance to be.

Geostatistics (or kriging) is an apolication of classical statistical theory to geological measurements that takes into account the spatial continuities of geological variables in estimating the distribution of variables. In many ways, geostatistics is for measurements taken in 2-, 3-, and 4-dimensional space (the three spatial dimensions and the time dimension), what time series is for measurements taken in one-dimensional space (time). However, a principal use of time series is in forecasting; in geostatistics the principal emphasis is on interpolation. Nevertheless, both statistical procedures emphasize modeling the process to get an insight into the system being investigated.

The application of classical statistical procedures to soil measurement data requires that the samples be collected randomly (i.e. not on systematic grids), that the data be independent and identically distributed (with the distribution being a normal distribution), and that the measurement error variance (particularly the between-batch error variance) be a very small part of the total variance of the measurements in a sample survey of a region.

In man soil sampling studies one or all of the following questions will be of primary interest.

- Are there any action supports within the study area that have pollutant concentrations above action level?
- Where are the above-action-level action supports located?
- What is the spatial distribution of pollutant concentration levels among action supports that have pollutant concentrations above action level?

The problem with posing soil sampling methods and objectives in terms of population means is that the mean will depend on the size of the area chosen and the distribution of contamination throughout that area. For example, the mean in a small area may exceed the action level; but if the size of small area is increased by adding a substantial amount of less contaminated soil, the mean in the larger area may not exceed the action limit. Decisions on the need or remedial action should not be based on how one chooses the size of the area

to be sampled, but rather on whether action supports exist that are above designated action limits. A comparison of means is reasonable in comparing pollutant concentrations at a background site with pollutant concentrations of a site down-gradient from a suspected hazardous waste source. Also, cleanup areas may be defined so that the average concentration in those units of soil may be compared with a standard.

It follows from the above discussion that for most applications, geostatistical procedures for designing soil sampling studies and analyzing resultant data are generally preferred over classical statistical procedures.

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 error). A control region is selected. The numbers of required samples may then be calculated. Additional samples should be required to validate 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 data of both adequate quality and maximal cost-effectiveness. Particular attention should be given to factors surrounding the disposition of non-soil material collected with the soil samples.

The requirements for QA/QC for the exploratory study need not be a 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 objective 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 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,

ifrequired, 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 must vary 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 insure that all aspects of the QA/QC system planned for the project are in place and functioning well. This include 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 QAQC 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 docimentation has been maintained and in orderly fashion, that each of the recorde items is properly categorized, and crossichecking can be easily accomplished Checks are made to insure that the da recording conforms to strict documentation of the program QA/QC plan

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

The project leader of a soil monitorii project is responsible for ascertaining the all members of his project team has adequate training and experience to car out satisfactorily their assigned mission and functions. This is normally accordished through a combination of requiriclassroom training, briefings on the specific monitoring project about to implemented, and field training exercises Special training programs should completed by all personnel prior to the involvement in conducting audits.

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The complete report, entitled "Soil Sampling Quality Assurance User's Guide—Second Edition," (Order No. PB 89-189 864/AS; Cost: \$28.95, subject to change) will be available only from:

National Technical Information Service

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