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Project Summary

Direct/Delayed Response Project: Field Operations and Quality Assurance Report for Soil Sampling and Preparation in the Southern Blue Ridge Province of the United States

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The Direct/Delayed Response Project Soil Survey includes the mapping, characterization, sampling, preparation, and analysis of soils in order to assess watershed response to acidic deposition within various regions of the United States. Soil samples collected by sampling crews in the Southern Blue Ridge Province were processed at two preparation laboratories before being sent for analysis at four analytical laboratories. Volumes I and II summarize the procedural and operational compliance with protocols used by the sampling crews and by the preparation laboratories, respectively. Deviations from protocols and difficulties encountered are identified and discussed. Recommendations are made for program improvement.

In general, soil sampling activities during the survey proceeded as planned. A review of the soil data suggests that the integrity of the soil samples was maintained during the preparation activities. In most cases, sampling crews and laboratory personnel adhered to protocols.

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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 two separate volumes of the same title (see Project Report ordering information at back).

Introduction

The U.S. Environmental Protection Agency (EPA), in conjunction with the National Acid Precipitation Assessment Program (NAPAP), has designed and implemented a research program to predict the long-term response of watersheds and surface waters in the United States to acidic deposition. Based on this research, each watershed system studied will be classified according to the time scale in which it will reach an acidic steady state, assuming current levels of acidic deposition. The Direct/Delayed Response Project (DDRP) was designed as the terrestrial complement to the Aquatic Effects Research Program.

After a pilot soil survey was accomplished, a sampling design for the soil survey of the Southern Blue Ridge Province was developed. Representative watersheds were selected for soil and vegetation mapping, and specific sampling sites later were characterized and sampled. Through an interagency agreement, soil scientists from the U.S. Department of Agriculture Soil Conservation Service were assigned the task of mapping and sampling soils in the region.

The services of two preparation laboratories were obtained through interagency agreements to receive and process incoming soil samples and to perform laboratory analyses for certain parameters. Laboratories located at the University of Tennessee in Knoxville and Clemson University in Clemson, South Carolina were selected for these tasks because of the proximity of each laboratory to the sampling sites and analytical experience with soils of the region. The laboratory managers were responsible for supervising the preparation of the soil samples and for ensuring that the integrity of the samples was maintained at the laboratories.

All soil survey participants were required to comply with specified soil sampling and preparation protocols, which are included as appendices in both Volume I and Volume II.

Soil samples processed at the preparation laboratories were obtained from routine sampling sites located in Georgia, Tennessee, South Carolina, and North Carolina. Additional samples were obtained from special interest watersheds in North Carolina and Virginia. Upon receipt of bulk soil samples from the sampling crews, laboratory personnel performed the following analyses on the samples: (1) air-dry moisture determination, (2) determination of the 2- to 5-millimeter and 5- to 20-millimeter rock fragment percentages by weight, (3) qualitative test for inorganic carbon, and (4) clod analysis for determination of bulk density.

Laboratory personnel prepared analytical samples derived from homogenized, air-dry bulk samples. The analytical samples were labeled and were organized according to their parent pedons. Analytical batches were assembled, each containing no more than 42 samples. The samples were randomized within each batch by the laboratory manager. The assembled batches were shipped to various analytical laboratories contracted by EPA for further analyses.

Three types of quality assurance (QA) samples were included in each batch of samples submitted to the analytical laboratory: (1) field duplicates, (2) preparation duplicates, and (3) natural audit samples. Portions of the data from the field duplicates are evaluated in Volumes I and II, and additional data for all QA samples will be evaluated in the forthcoming QA report for the analytical laboratory data.

One soil horizon per sampling crew per day was sampled in duplicate as specified in the protocols. The first sample of the pair is considered the routine sample, and the second sample is referred to as the field duplicate. The field duplicate underwent the same preparation steps as its associated routine sample. This procedure allows an estimate to be made of spatial horizon variability and sampling bias.

One sample per batch was chosen by the laboratory manager to be processed and then split into two subsamples. One of the pair retained the routine sample code and the other was assigned the preparation duplicate designation. Analytical data from the preparation duplicates allows the range of physical and chemical characteristics for splits of the sample material to be determined and allows an estimate to be made of the error attributed to subsampling.

Each batch contained two natural audit sample pairs supplied by EPA, but the samples did not undergo processing at the preparation laboratory. They were submitted as blind samples in order to assess the performance of the analytical laboratories.

Field data received from the sampling crews and raw data from the preparation laboratory analyses were documented in log books and were submitted to EPA for use during data verification.

QA and quality control (QC) measures were applied in order to maintain consistency in soil sampling and preparation protocols and to ensure that the soil sample analyses would yield results of known quality. Field and laboratory personnel received training in the sampling and preparation procedures and analytical methods. QA representatives conducted on-site systems audits of the sampling crews and the preparation laboratories. Weekly communication between the QA staff and the sampling and preparation personnel was established to identify, discuss, and resolve issues.

Volume I of the report presents the results of the soil sampling operation and QA program, and Volume II of th report presents the results of the prep aration laboratory operations and Q program. Recommendations for prograr improvement are made in bot documents.

Procedures

The QA/QC design for the soil sam pling and preparation included trainin personnel in the protocols to be followec establishing a communications network assessing data quality, and accomplish ing on-site systems audits. The data ar evaluated statistically using analytica data from the replicate clods and fron the duplicate samples that were include in each batch of routine samples.

Field and laboratory personne attended a regional pre-sampling work shop in Knoxville, Tennessee durin March 1986. The purpose of the work shop was to review the sampling an preparation protocols and discuss ke activities.

A computer algorithm was used t make random selections of samplin locations within representativ watersheds containing the desired sam pling and vegetation classes. Samplin crews were tasked with collectin approximately 5.5-kilogram bulk sample from selected sampling sites that met th specific sampling class and vegetatior class requirements. A soil horizon nor mally was subdivided for sampling if its thickness was greater than 20 centime ters. A coded field data form was usec by all sampling crews to facilitate data entry into computer files by QA person nel. The sampling effort resulted in a tota of 110 routine pedons sampled.

The sampling crews were responsible for the full characterization of thei assigned pedons to a depth of 200 centimeters, or to bedrock if shallower Samples were taken from representative parts of delineated horizons, were sievec to exclude rock fragments exceeding 20 millimeters in diameter, and were sealec in plastic bags. All samples were tem porarily refrigerated in styrofoam coolers until they could be delivered to colc storage facilities at the preparatior laboratories. Replicate soil clods used to estimate bulk density also were collected.

After retrieval from cold storage, bulk samples were spread out on paper to ai dry until constant weight was achieved After recording the weight of the air-dry bulk sample, the soil peds were crushec to allow passage of the less than 2-

millimeter soil fraction through No. 4 and No. 10 sieves. The rock fragments retained on the No. 4 sieve constituted the 5- to 20-millimeter coarse pebble fraction, and the fragments retained on the No. 10 sieve constituted the 2- to 5-millimeter fine pebble fraction. The fragments were weighed and then packaged for storage.

A Jones-type, ⅜-inch riffle splitter was used to homogenize the less than 2-mm fraction of the soil samples. Each sample was placed through the riffle splitter at least seven times in succession. One-half of each sample was placed into a plastic sample bag for archiving, and the other half was passed through the riffle splitter until a 500-gram subsample was obtained. The subsample was labeled and placed into a batch of samples for delivery to an analytical laboratory.

One gram of air-dry soil was placed in the well of a porcelain spot plate, was saturated with deionized water, and was stirred to release any entrapped air. The sample was observed through a microscope or stereoscope in order to detect any chemical reaction when three drops of 4N HCl were added.

The soil clods collected by the sampling crews were weighed at the laboratory and were dipped in a 1:7 saran:acetone mixture. The dipping procedure was repeated until each clod was assumed to be impervious to water. The clods were dried briefly and were reweighed.

Approximately 800 milliliters of deionized water in a one-liter beaker was degassed by boiling, was allowed to cool to room temperature, and was tared on a balance. Each clod was submerged in the water to determine the weight displacement on the balance. The clods were placed in a drying oven for 48 hours, and, after cooling, were weighed. A two-hour heat treatment in a 400°C muffle furnace allowed the saran to vaporize, and the clods were cooled and reweighed. Each clod was crushed and was passed through a 2-millimeter sieve to determine percent by weight of rock fragments, which was used to adjust the bulk density calculation for rock fragment content.

Results and Discussion

The sampling crews encountered a number of logistical and procedural difficulties that are detailed in Volume I. The presence of thick vegetation made it difficult to locate and gain access to the sampling sites. Helicopters were used to access two watersheds in Great Smoky Mountains National Park. Vegetation class or sampling class modifications were made at six sites, and two sites were removed from the sampling list because the appropriate sampling class could not be located in the watershed. The vegetation class did not always correspond to the class expected at a sampling location. Site selection and sampling protocols generally were followed by all of the sampling crews.

Hand pumps were used to control seepage when sampling wet or saturated soils. Efforts were made to avoid contamination of the samples from sampling equipment, adjacent soil horizons, and agricultural chemcials such as fertilizers or herbicides. Occasional discrepancies were noted in regard to sample labeling, collection of clods, and entry of data on the field data forms.

Each preparation laboratory provided the sampling crews with convenient access to cold storage. A sample receipt log book was kept at each faciity to allow sampling crews to log in the samples. Each laboratory was responsible for checking that all samples delivered by the sampling crews were recorded accurately in the log book. The temperature of the storage facilities was maintained at the contract-specified 4°C.

The laboratory managers were responsible for tracking the distribution of equipment to the sampling crews. The sampling crews usually picked up supplies at the time samples were being delivered to the cold storage facility. The sampling crews were asked to list in the equipment log book all supplies taken. Equipment shortages were reported to EPA during the weekly conference call. Laboratory personnel usually provided the saran dipping solution used for coating clods in the field. After the soil preparation activities were completed, leftover supplies were inventoried and then sent to EPA for storage.

Although there were no deviations from the specified protocols for sample drying, concerns were raised about the length of time necessary to dry the samples. The extended drying period allowed the samples to be exposed to possible airborne contamination for longer periods than was necessary.

There were no deviations from the specified protocols for the air-dry moisture determination, the crushing and sieving operation, the rock fragment determination, the soil homogenization, the test for inorganic carbon, or the bulk density determination.

The preparation laboratories were provided with packaging materials and an express mail charge number for overnight shipment of samples to the designated analytical laboratories.

The sampling crews and the preparation laboratories were provided with log books to use in recording data. Each laboratory manager was instructed to organize log books containing the following labels, information, and analytical data: bulk sample labels, clod labels, sample receipt, equipment, percent moisture, percent rock fragments, bulk density, inorganic carbon, and sample processing. Because a standard format for each log book was not specified, there was variation among the laboratories. As a result, verification of the data took more time than was expected.

Weekly conference calls assisted in keeping the sampling crews and preparation laboratories operating efficiently and consistently by providing a forum that allowed potential difficulties to be discussed and resolved. Issues discussed during the conference calls included site access difficulties, supply shortages, record keeping, and clarification of protocols.

The quality of the sampling effort and the preparation activities are assessed according to the following data quality characteristics: (1) precision, (2) accuracy, (3) representativeness, (4) completeness, and (5) comparability. Both Volume I and Volume II provide details on the evaluation of data quality for various survey activities. As an example, a completely randomized design model was selected for the statistical estimation of precision, using a pooled standard deviation and coefficient of variation to quantify the error due to imprecision.

Conclusions and Recommendations

A series of useful recommendations are made in both volumes, based on information supplied by the sampling crews, preparation laboratory personnel, QA staff, and other survey participants. The recommendations are presented for possible implementation in future surveys, and can be summarized as follows:

- Improved site selection criteria
- Equipment and supply tracking and inventory system
- Procurement of specialized sampling equipment
- Uniformity of staff evaluations and QA systems audits

- Modifications to the computerized field data form
- Standardized forms and log books for record keeping
- Computerized data entry procedures
- Sample drying techniques
- Alternative methods for determining field moisture
- QA/QC measures for bulk density determination
- Alternative method for identifying inorganic carbon
- Documentation of conference calls
- Development of data quality objectives

The Southern Blue Ridge Province soil survey was successful in terms of collecting data of known and documented quality that will be utilized by many end users. The coordination of sampling and preparation activities among the many participants was a large-scale, complex task that was performed as originally conceived with a minimum of unanticipated difficulties and modifications.

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The complete report consists of two volumes, entitled "Direct/Delayed Response Project: Field Operations and Quality Assurance Report for Soil Sampling and Preparation in the Southern Blue Ridge Province of the United States:"

"Volume I. Sampling," (Order No. PB 88-154 257/AS; Cost: $25.95, subject to change)

"Volume II. Preparation," (Order No. PB 88-154 265/AS; Cost: $14.95, subject to change)

The above reports will be available only from: (costs subject to change)

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