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Project Summary

Direct/Delayed Response Project: Field Operations and Quality Assurance Report for Soil Sampling and Preparation in the Northeastern United States

D. S. Coffey, M. L. Papp, J. K. Bartz, R. D. Van Remortel, J. J. Lee, D. A. Lammers, M. G. Johnson, and G. R. Holdren

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 Northeastern region were processed at preparation laboratories before being sent for analysis at 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 soil study complement to the aquatic resources program.

After a pilot soil survey was accomplished, a sampling design for the soil survey of the Northeastern region 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 Soil Conservation Service were assigned the task of mapping and sampling soils in the region.

Four preparation laboratories were contracted through interagency agreements to receive and process the soil samples and to perform laboratory analyses for certain parameters. Laboratories located at the University of Maine, the University of Connecticut, the University of Massachusetts, and Cornell University in New York 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 ensuring that the integrity of the soil samples was maintained after the samples were delivered by the sampling crews to the preparation laboratories.

All soil survey participants were required to comply with specified soil sampling and preparation protocols, which are included as Appendix A in both volumes

Soil samples processed at the preparation laboratories were obtained from routine sampling sites located in Maine, Vermont, New Hampshire, Massachusetts, Rhode Island, Connecticut, New York, and Pennsylvania. Additional samples were obtained from special interest watersheds in new York and Massachusetts. 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 20-mm rock fragment percentage 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 sampling error and horizon variability.

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 samples supplied by EPA, but the samples did not undergo processing at the preparation laboratory. These samples were used to assess the performance of the analytical laboratories.

Field data received from the sampling crews and raw data from the preparation laboratory analyses were doucmented 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 sampling operations and QA program, and Volume II of the report presents the results of the preparation laboratory operations and QA program. Recommendations for program improvement are made in both documents.

Procedures

The QA/QC design for the soil sampling and preparation included training personnel in the protocols to be followed, establishing a communications network, assessing data quality, and accomplishing on-site systems audits. The data are evaluated statistically using analytical data from the replicate clods and from the duplicate samples that were included in each batch of routine samples.

Field and laboratory personnel attended a regional pre-sampling workshop in Orono, Maine during August 1985. The purpose of the workshop was to review the sampling and preparation protocols and discuss key activities.

A computer algorithm was used to make random selections of sampling locations within representative watersheds containing the desired sampling and vegetation classes. Sampling crews were tasked with collecting 5.5-kilogram bulk samples from selected sampling sites that met the specific sampling class and vegetation class requirements. A soil horizon normally was subdivided for sampling if its thickness was greater than 20 cm. A coded field data form was used by all sampling crews to facilitate data entry into computer files by QA personnel. The sampling effort resulted in a total of 306 routine pedons sampled.

The sampling crews were responsible for the full characterization of their assigned pedons to a depth of 150 cm, or to bedrock if shallower. Samples were taken from representative parts of delineated horizons, were sieved to exclude rock fragments exceeding 20 mm in diameter, and were sealed in plastic bags. All samples were kept in styrofoam coolers until they could be delivered to cold storage facilities at the preparation laboratories. Soil clods used to estimate bulk density also were collected.

After retrieval from cold storage, bulk samples were spread out on trays to air dry until constant weight was achieved. After recording the weight of the air-dry bulk sample, the soil peds were crushed to allow passage of the less than 2-mm soil fraction through a No 10 mesh sieve. The rock fragments retained on the sieve constituted the 2- to 20-mm pebble fraction. The fragments were weighed and packaged for storage.

A Jones-type, ⅜-inch riffle splitter was used to homogenize the less than 2-mm fraction of the samples. The soil 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 spiltter until an approximately one-kilogram subsample was obtained. The subsample was labeled and placed into a batch of samples for delivery to an analytical laboratory.

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

The soil clods collected by the sampling crews were weighed at the laboratory and were dipped in a 1.4 or 1:7 saran:acetone mixture The dipping procedure was repeated until each clod was impervious to water. The clods were suspended from a line, were allowed to dry briefly, and were reweighed.

Approximately 800 mL deionized water in a one-liter beaker was de-gassed 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 No. 10, 2-mm mesh sieve to determine percent by weight of rock fragments, which was used to adjust the bulk density for rock fragment content.

Results and Discussion

The sampling crews encountered a number of logistical and procedural difficulties that are detailed in Volume I Sampling was begun under unusually dry conditions, however, mid-way through the sampling, Hurricane Gloria brought excessive rainfall in a short period that impeded access to some locations for several weeks Helicopters were used to obtain access to three watersheds in New York Access was denied by landowners to four watersheds in the region. Inappropriate site conditions also were encountered occasionally. The vegetation class did not always correspond to the class expected at a sampling location. Site selection protocols were followed by all but one of the sampling crews.

Difficulties were encountered when sampling wet or saturated mineral soils and organic soils. Soils with a dense substratum or numerous large rock fragments posed some additional sampling constraints. Efforts were made to avoid contamination of the samples from sampling equipment, adjacent soil horizons, and agricultural chemicals such as fertilizers or herbicides. Occasional discrepancies were noted in regard to the sieving of rock fragments, 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 facility 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 contact-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 responsible for listing in the equipment log book all supplies taken. Equipment shortages were reported to EMSL-LV during the weekly conference call. Laboratory personnel were tasked with mixing the saran dipping solution used for coating clods in the field. After the soil preparation activities were completed, leftover supplies were inventoried, were packed in boxes, and were returned to EMSL-LV for storage.

Although there were no deviations from the specified protocols for sample drying, concerns were raised about the collection of water on the trays and the encrustation of certain organic samples. Both situations encouraged microbial growth that may have altered the composition of the affected samples.

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 incorganic carbon, or the bulk density determination.

The preparation laboratories were provided with packaging materials and a Federal Express charge number for overnight shipment of samples to the designated analytical laboratories. The individual samples were labeled, were placed in canvas bags, and were packed in cardboard boxes for shipment.

The sampling crews and the preparation laboratories were provided with log books to use for 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 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:

- Prior site selection by sampling leaders

- Methods for artificially draining saturated soil pits
- Procurement of specialized sampling equipment
- Uniformity of staff evaluations and QA systems audits
- Standardized forms and log books for record keeping
- Computerized data entry procedures
- Sample drying techniques
- Assessment of air-dry moisture content
- QA/QC measures for bulk density determination
- Documentation of conference calls
- Development of data quality objectives

The Northeastern 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.

D. S. Coffey is with Tetra Tech, Inc., Bellevue, WA 98005; M. L. Papp, J. K. Bartz, and R. D. Van Remortel are with Lockeed Engineering and Management Services Co., Inc., Las Vegas, NV 89119; the EPA authors ***J. J. Lee*** *and* ***D. A. Lammers*** *are with the Environmental Research Laboratory, Corvallis, OR 97333; M. G. Johnson and G. R. Holdren are with Northrop Services, Inc., Corvallis, OR 97333.*

L. J. Blume *is the EPA Project Officer (see below).*

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 Northeastern United States:"

"Volume I. Sampling," (Order No. PB 88-120 597/AS; Cost: $25.95)

"Volume II. Preparation,"(Order No. PB 88-120 605/AS; Cost: $19.95)

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

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5285 Port Royal Road
Springfield, VA 22161
Telephone: 703-487-4650

The EPA Project Officer can be contacted at:

Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
P.O. Box 93478
Las Vegas, NV 89193-3478

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