CHAPTER III
QUALITY ASSURANCE/QUALITY CONTROL FOR BASELINE INFORMATION

The purpose of quality assurance and quality control (QA/QC) procedures is to ensure that data collected for the permit represents actual conditions at the site for the time of sampling. These data need to be accurate and reproducible. Effective QA/QC procedures are essential to ensure the validity of geologic and hydrologic data and ultimately the decisions utilizing geologic and hydrologic data in the coal mine permit process. QA/QC procedures commonly apply to sample collection, sample preservation, control and analysis, as well as the effectiveness of geologic and hydrologic data in monitoring a permitted operation.

Quality control refers to specific procedures used to achieve prescribed standards of performance. Quality assurance is an integrated planning process for assuring the reliability of geologic and hydrologic data so that it can be used independently or with other comparable kinds of data with some definable degree of confidence. Quality assurance components commonly include:

- Outlining the intended use of the data, such as to support permit issuance or revisions, to verify compliance with performance standards, or to verify self-monitoring data.

- Identifying factors that influence the design of the monitoring system such as the homogeneity or lack thereof of the geologic and hydrologic systems being measured or monitored.

- Selecting the parameters to be monitored and the frequency of monitoring.

- Specifying sampling protocols and analytical methods to be used.

- Specifying detection limits where applicable and required precision and accuracy for all types of geologic and hydrologic measurements.

- Identifying quality control procedures to document whether these requirements are being met.

A. Precision, Accuracy and Bias

In scientific measurement, there are three main attributes that describe the quality of the resulting information: precision, bias, and accuracy. The 19th edition of *Standard Methods for the Examination of Water and Waste-Water* (1995) defines them as follows:

- Precision is a measure of the degree of agreement among replicate analysis of a sample, usually expressed as the standard deviation.

- Bias is the consistent deviation of measured values from the true value, caused by systematic errors in procedure.

- Accuracy is a combination of bias and precision of an analytical procedure, which reflects the closeness of a measured value to a true value.

B. Data Quality Objectives

The majority of geologic and hydrologic data will be collected through sampling of the mine permit area. Other data may come from data bases or other sources such as nearby operations. Regardless of the type of information assembled and collected, it is important to determine the objectives of data collection. Surprisingly, sampling objectives are often not clearly defined. As a result, data may be collected that do not need to be collected or data may be collected that do not provide the information necessary to support a particular decision.

Formulation of the appropriate sampling objectives can be achieved in a variety of ways. One model used in environmental applications is the USEPA's *Data Quality Objectives (DQO)* approach (U.S. EPA, 1994). The purpose of DQOs is to (1) clarify the study objective; (2) define the most appropriate data to collect; and (3) specify tolerable limits on decision errors, which will be used as the basis for establishing the quantity and quality of data needed to support the decision. It has been used effectively to establish sampling priorities, manage sampling budgets, and reduce conflict between regulatory and industry groups. The seven basic DQO steps are set forth below:

Step 1: State the problem.
Step 2: Identify the decision.
Step 3: Identify the inputs to the decision.
Step 4: Define the study boundaries.
Step 5: Develop a decision rule.
Step 6: Specify tolerance limits on decision errors.
Step 7: Optimize the design.

Complete discussion of the detailed DQO process is beyond the scope of this document. The above discussion is extracted from a report edited by Hossner (Texas Mine Land Reclamation Monitoring Program Issues, A report of the Soils Working Group October 1998). (Link to: http://www.osmre.gov/pdf/TXMONITORING.pdf)

C. Water Quality Sampling Procedures

Much information has been developed for QA/QC over the years for collection and analysis of water samples. Recently, the USGS released a report dealing with standard methods for sampling ground and surface waters. The purpose is to provide sampling methods that result in accurate data that are reproducible within defined limits of accuracy. Some of the topics covered include: preparations for water sampling, selection of equipment, collecting and processing of water samples and field measurements. The report can be accessed at the following web site. Link to: http://water.usgs.gov/owq/FieldManual

TIPS has software programs to perform cation and anion balance for standard complete water samples. Through programs like HydroChem and AquaChem, one can determine the validity of water quality analyses.

QA/QC discussions and procedures for analysis of water samples are contained in Part 1000 of the Standards Methods for Examination of Water and Wastewater (1995) publication. Topics covered include precision, bias, calibration with standards and analysis of duplicates.

D. Geology and Overburden Sampling Procedures

The following discussion on sampling collection, sample preparation and storage, quality assurance program elements, and related topics was taken from OSM's Overburden Sampling and Analytical QA/QC Requirements. (See Reference in Chapter V.) Other adequate sources of QA/QC procedures exist and may be used.

1. Sampling Methods

The following sampling procedures can be used:

- Core drilling produces a continuous record of the geologic column encountered; it is therefore a preferred method. Drilling may be used to produce continuous cores using air, air-water mist, or water (non-contaminating, low in salts) as the drilling medium. Care must be taken to ensure that mud, water, and lubricants do not contaminate the core.

- Air rotary chip sampling is often used because it is quicker and less costly than core drilling. However, because the chips tend to get mixed as they are blown from the borehole, sampling of discrete intervals becomes more difficult. Chip sampling is often used as a supplement to continuous core drilling.

- Thin-wall tubes, split-barrel samplers, or other drive or press devices can be used to sample unconsolidated materials.

- Each sample should represent a single lithologic unit except where intervening strata are less than 1 foot in thickness. However, single samples should not represent more than 5 feet of the core.
- Coal stringers or lithochromic strata (10YR 3/2 or darker) greater than 6 inches should be sampled separately.
- When core sampling, sampling intervals should be broken if an obvious change in chroma, texture, mineralogy, or weathering intensity is noted, or where an anomalous strata, such as a coal/lithochromic zone, appear
- To avoid contamination of the top 5 feet of each core, one sample from each soil horizon to 5 feet should be obtained separately from each of the core locations. These soil samples should be retrieved from excavated pits by a soil scientist. All analytical parameter analyses, listed in these guidelines, must be completed on each sample. The data from these samples will complement the soil survey and may identify suitable top dressing materials to the 5-foot depth. These procedures are required to assure the acquisition of reliable overburden data to identify materials for root zone reclamation and topsoil substitution are available.

- All analytical methods, parameter limits, and suitability criteria required by the RA must be followed by the industry when characterizing potentially acid- and toxic-forming materials, topsoil substitutes/supplements, and materials proposed for root zone reclamation.

2. Drill Logs

Geologic logs of each drill hole must be recorded and submitted as part of the drilling report in the Permit Application Package (PAP) or required annual reports. Both the cores and any other lithic samples should be kept in case they are needed in the future. Drill logs must be a complete and accurate record of drilling activities and should include the following information:

- Name and qualifications of individual making interval separations and sampling of cores

- Core number, location (northing and easting), collar elevations, depth intervals, lithology and lithological constituents, and Munsell color

- Dates of drilling

- Dates samples sent to laboratory for analyses or storage

- Personnel conducting drilling

- Drilling method used
- Interpretation of water bearing zones
- Identification of lost or non-retrievable cores
- Zones of lost circulation of drill fluids
- Any unusual conditions encountered during drilling activities

3. Sample Collection, Handling and Transport

To meet the requirements of 30 CFR 780.22 (b) (2) and (c) and 30 CFR 777.13(a), the company and laboratory must list, with references, the methods of sample preparation and analyses used to analyze soil, overburden and regraded spoil. The following procedures are suggested for preparation and storage of samples:

- When sampling for monitoring programs, the RA must be notified at least 2 weeks in advance so that it may have the opportunity to observe, and/or participate in, sampling practices and procedures.
- It is important that samples not be exposed to high temperatures. During the warmer months, an insulated container should be provided for storage during sampling and transport. If temperatures exceeding 35º C are possible, the overburden/spoil samples should be kept cold using clean ice or a similar product that will not contaminate the samples.
- Samples will be identified and labeled using a system that will correspond with submitted sample location maps and laboratory analytical results. Samples will be labeled in a numerical sequence, not by site and strata/horizon, when submitted to the laboratory.
- Sufficient sample quantities must be collected to meet the needs for sample splits outlined in the following sections of this document.
- Time between sample collection and preparation should not exceed 30 days.

4. Sample Preparation and Storage

The following are suggested procedures to be followed for preparation and storage of samples:

a. Laboratory Preparation

All analyses must be performed on air-dried samples with data being reported on an oven-dry weight basis (110° C).

b. Drying

Drying must be initiated as soon as possible after the samples arrive at the laboratory. Air dry at a temperature not exceeding 35° C by spreading samples on non-metallic trays to a depth of 1-2 cm. Drying can be excluded for sample analyses that require an "as received basis" for analyses completion. Break up large soil masses so that there are none larger than 1 cm in diameter. Mix the sample daily and re-spread to allow for faster drying. Drying may be accelerated by passing air from a fan over the samples.

c. Core Crushing

Core crushing must be accomplished utilizing equipment that minimizes particle size reduction. Equipment used must also have a minimal chance of chemical contamination of the samples used and be easily cleaned between samples. The entire core sample must be crushed to < 2 mm with constant sample removal.

d. Soil Flailing

Soil samples are to be flailed to <2 mm with constant removal of reduced material. Determination of the coarse fraction (weight basis) is to be made on the basis of the field sample. Soil samples are to be sieved, prior to flailing, with separation of >1 cm coarse fragments.

e. Grinding

Samples for Leco Furnace analysis (total sulfur and organic carbon) must be ground to <0.25 mm.

f. Splitting

The sample should be passed through a mechanical sample splitter and recombined four to five times to insure complete sample mixing. Depending upon how many subsamples are needed, the mixed sample is to be split using a mechanical splitter into several small samples and then recombined to achieve the desired number of sub-samples.

The laboratory must prepare and maintain three splits of the original sample with one sample being retained by the laboratory for analyses, one returned to the company, and one to be provided to the RA when requested.

g. Sample Storage

Samples are to be stored in the laboratory, or elsewhere, at temperatures between 10º and 30º C. After preparation, samples are to be stored in sealed glass containers under controlled storage conditions. Storage conditions used must be outlined in the final reports to the RA.

5. Quality Assurance Program Elements

The following information should be provided to the company and RA for each sample/set of samples:

- Names and qualifications of personnel handling sample at each stage of collection, preparation and analyses
- Name and qualifications of individual making interval separations and sampling of cores
- Dates and conditions of each stage of sample collection, transport, preparation and analyses
- Detailed description of core collection, storage, and preparation procedures
- Laboratory procedures as specified. If procedure modifications are made, the laboratory must describe the modifications in detail and should submit statistical data correlating data with original methodology.

Additional internal laboratory QA/QC procedures on all analytical parameters are to be supplied to the Company and to the RA in final reports.

6. Quality Control Program Elements

The following elements should be considered in the quality control program:

a. Sample Control

A sample is physical evidence collected from a facility or from the environment. An essential part of soil, overburden or spoil sampling programs is to control the evidence gathered. To accomplish this, companies will require that their drillers and contracted laboratory initiate and complete chain-of-custody and document control programs. Copies of program procedures must be submitted to the RA with all soil, overburden, and spoil analytical results.

Time from sample collection to completion of analyses should be as short as possible because minerals begin to break down upon exposure to air.

b. Instrumentation

Detection limits must have been established for each instrument immediately prior to analyses of any field samples. A log of each instrument's detection limits should be kept.

Establish working limits prior to beginning each test. Determine the concentration that is three times the detection limits of the instrument for each analyses in the extraction solution. Prepare calibration standards in graduated amounts.

c. Analytical Controls

Calibration standards must be prepared:

- At an appropriate range approximating that of field samples
- Using the same acids or salts used in the digestion or extraction of the field samples
- As a blank, and at least three calibration standards in graduated amounts for each analytical parameter

Duplicate samples must be prepared and analyzed:

- For samples of a similar matrix type and concentration as the major samples analyzed
- With ten percent duplication for analyses which are routinely accomplished with minimum difficulty in accuracy and precision; i.e., pH, electrical conductivity (EC), etc. These samples should be randomly selected.
- With twenty percent duplication for analyses which are known to be difficult to accomplish with high levels of accuracy and precision; i.e., cation exchange capacity (CEC), exchangeable sodium percentage (ESP), extractable selenium and boron. These samples should be randomly selected.

d. Reports

Control charts must be developed, including:

- Chronological data tables for calibration standards, control samples, duplicate samples, and detection limit results
- Chronological statistical computation of data

Data must be presented using systematic report formats including:

- 0Uniform analytical units
- Uniform significant figures
- Uniform tables for computer reader use (See Data Submittal Format Section)
- Submittal of data in hard copy and in electronic format