ANALYST'S COUCH

Quality Control/Quality Assurance in Geochemical Laboratories

A good part of the success of most mineral exploration programs in North America rests on the data produced by independent laboratories; yet, the mineral laboratory industry is not subject to government certification, or monitoring for quality. Thus, the principle of "buyer beware" must be the rule when dealing with mineral laboratories. The explorationist is forced to put on his "chemist hat" and determine if the sales agent sitting in front of him represents an organization that really can provide fast, quality analytical determinations at a reasonable price. One of the evaluations that should be made of any laboratory is the effectiveness of their Quality Control/Quality Assurance (QC/QA) program.

In modern geochemical analytical laboratories, an elemental determination is usually the culmination of numerous steps and the efforts of many technicians. The quality of the determination depends on the quality of the work carried out at each step. Since the variance introduced at each step is cumulative, the tolerance for error at every individual step must be much more stringent than that for the final product. Explorationist's must take the time to confirm that the laboratory being considered has a QC/QA program effective enough to ensure that the analytical determinations produced are of the required quality for the proposed exploration program.

QC/QA Program Components

Quality is a subjective and a relative term. What is high quality in one situation may be unacceptable in another case, eg., the quality control required for a geochemical Cu determination will be less stringent than that required for an assay Cu determination. The quality required is dictated by the needs of the user and is also subject to the nature of the sample and the analytical technology applied. A good QC/QA program will be able to respond to these situations.

An effective laboratory QC/QA Program will have the following five components; 1) a rigorous, well defined employee training and re-training program; 2) documented methodology protocols; 3) clear, easily accessed, responsibility tracking system; 4) routine use of geostandards, round robin participation and an independent audit program; and 5) well defined and documented analytical data acceptance/data rejection control measures.

Employee Training and Re-training Program

The single most important factor in reducing the occurrence of laboratory error is professional competence. This requirement has become even more critical as the mineral laboratories in North America increasingly adopt computerized automation and expert systems. There is no question that the number of people required to run a mineral laboratory has gone down, but the training and technical ability of the remaining work force has increased.

Documented Methodology Protocols

An important aspect of any laboratory QC/QA program is to ensure control of the methodology applied in the laboratory. All methods must be documented and regularly up-dated to ensure that "gradual method creep" does not occur as each generation of new employees is trained. This is particularly important for sample digestion and calibration protocols. Slight variances in technique can create very different analytical data.

Responsibility Tracking System

In order for any laboratory to ensure "analytical control" an extensive responsibility tracking system is required. Complete work records must be kept, so that the exact sequence of processing can be determined. Technicians must "sign off" their work, thereby assuming responsibility for their actions. It is also very important that someone accept over-all responsibility for the work performed in laboratory sections. In addition, the increasing dependence on computers and "smart systems" has demanded new ways to track work history. Electronic data movement must be documented in order to prevent abuse and gross errors from occurring.

Geostandards, Round Robins and QC Audit Programs

The routine introduction of reference samples and participation in round robin studies is essential in order to ensure that a laboratory is producing analytical determinations that are free of bias. These studies also serve to point out gross errors, e.g. equipment malfunctions, improper standards, etc. The round robin study is the best tool available to effectively evaluate laboratory performance.

A QC Audit program usually involves surprise inspections of laboratory operations by senior personnel, or outside experts. The QC Audit's purpose is to ensure adherence to exact methodology and general good laboratory practice.

Analytical Data Acceptance Control Measures

To guide the acceptance or rejection of analytical data, a monitoring measure should be built into each operational system. This is accomplished by including control samples, weighed replicates, sample preparation replicates, standardized pulps and reagent blanks. The accumulated quality control data is then analyzed statistically and outliers are highlighted. The most common technique for visual monitoring of quality control data is a control chart. Data for all control samples, replicates and sample preparation duplicates are monitored and evaluated against established criteria during the measurement process.

Use of Control Charts

A control chart has time along the horizontal axis and concentration units along the vertical axis. The maximum, minimum and mean observed values of an analytical standard are plotted on the chart for successive time intervals. The central line is the established reference value, X. The lines above and below are the upper and lower warning and control limits, UWL, UCL and LWL, LCL, respectively. As data accumulates, any significant trends plotting above, or below X will become visible. This visual representation of the control of techniques allows the technician to respond to problems that may not have been discernable without the chart.

Data Acceptance/Rejection Criteria

There is no universally recognized criteria for data acceptance/rejection in the fields of geochemical analysis and assaying; however, in the late 1970's, the Geological Survey of Canada (GSC), Standards and Data Services Section, began compiling a guide to control the quality of analytical work performed by outside contractors. The GSC guideline applies a system that monitors and controls short and long term precision, as well as accuracy. Essentially,

Continued on Page 24

Analyst's Couch

Continued from Page 23

reference values for a control sample are established by compiling 20 to 40 results from different analytical runs and calculating the mean and standard deviation. A tolerance limit for the standard is then set at ± 2.5 standard deviations from the mean.

Quality Control data can be of considerable value to the explorationist, yet, not all mineral laboratories will provide these data. Calculated variance data enable the explorationist to determine the overall laboratory variance for any given project. In addition, the variance determined from sample preparation duplicate data can be separated from the total laboratory variance leaving the variance created during the sample preparation process. One now has a tool that can be used to determine the appropriateness of the sample preparation procedure. If duplicate samples are taken in the field, qualitative variance information will be available for the entire project. By simple subtraction, the laboratory variance can be removed from the total project variance leaving the variation introduced during the field sampling stage.

Common Laboratory Errors and Mistakes

The major sources of error in a mineral laboratory can be classed using the following general categories:

A) Incorrect identification of samples.
B) Contamination.
C) Improper, or inappropriate sample preparation.
D) Inaccuracy of sample weights, or volumes.
E) Improper, or inappropriate sample dissolution/treatment.
F) Chemical and physical interferences.
G) Improper, or inappropriate instrumentation/inaccurate measurement.
H) Calculation errors.
I) Incorrect data handling/reporting.

In this list of possible errors and mistakes, all of them except one, chemical and physical interferences, have a "human component." That is why the single most important requirement in any QC/QA program is the ability to effectively train and re-train laboratory staff. The movement of mineral laboratories to automation and expert systems has reduced the number of people required, but it has also increased the requirement for trained, competent people, who can respond to situations and effectively trouble shoot and problem solve.

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