



SCHNEIDER LABORATORIES INCORPORATED

ASPECTS OF LABORATORY QUALITY ASSURANCE

A thorough and complete quality assurance program is composed of many parts, each like a little piece of a puzzle. Together they make a complete quality plan. These parts have been broken down here and described in a short “Quality Assurance 101” seminar-style presentation.

BEFORE SAMPLES ARRIVE

WHAT ARE LABORATORY CERTIFICATIONS?

In the lab, we spend a lot of our time and energy maintaining Laboratory Certification. There are many agencies and governments that have certification programs, but there is a move to consolidate these into a national program. NELAC (National Environmental Laboratory Accreditation Conference) is sponsored by the EPA as a voluntary association of State and federal officials. The purpose of the organization is to foster the generation of environmental laboratory data of known and documented quality in a cost-effective manner through the development of nationally accepted standards for environmental lab accreditation. NELAC encompasses all fields of testing associated with compliance with EPA regulations. NELAC announced its first group of labs in 2001, following about 10 years of planning, and has brought into reality the first effort at a single program for environmental certification.

Many states still do not participate in NELAC and instead maintain their own programs, but NELAC is already very widely recognized. Schneider Laboratories became NELAC-certified in January 2001 with New York as its Accrediting Authority with the first group of laboratories in the nation to achieve this certification. We also maintain a lengthy list of other state and local certifications. We’re proud to have been a leader in our industry in the process of achieving certifications. We hold the first license given in the state of Virginia for PLM and PCM asbestos testing, and were the first laboratory in the nation to achieve certification through the National Lead Laboratory Accreditation Program through AIHA.

WHAT IS THE VALUE OF LABORATORY CERTIFICATIONS?

Our laboratory certification is valuable to our clients because it assures them that we have been evaluated against rigid standards and have met the expectations. These standards include a very inclusive list, including evaluation of staff, training, facilities, equipment, documentation practices, written procedures, and laboratory practice. Laboratory certification programs include system reviews, on-site assessments, and proficiency testing requirements. We participate in proficiency testing from a NIST-approved provider for non-potable water and solid & hazardous waste twice per year. Our participation in certification programs assures our clients that appropriate oversight is done by a qualified agency. It assures us, as the laboratory, that equivalent standards are being applied to all laboratories. It assures data users that good laboratory practice is being observed and that data is traceable and reliable. If you’re interested in knowing more about certification programs or policies, the agencies each have websites and most have policies, checklists, and news releases available for public use.

HOW ARE METHODS ESTABLISHED BY A LABORATORY?

The process of setting up a method is called method validation. When we determine that we want to offer a test, there are certain things that we do to set up that test and validate it before we begin to process unknowns by that method. We begin with a published method, then get reagents, standards, equipment, and supplies specified in the method. We then run initial demonstration of capability testing (or, IDCs), which is a process of running QC samples and evaluating results as percent recovery. NELAC policies outline the procedure, which was initially

published in the Federal Register, so the process for IDCs is standardized and subject to oversight by certifying agencies. In addition to running an IDC for a certain test, each analyst who performs the test repeats the IDC procedure to show individual proficiency. Of course, applicable training is provided for analysts as needed for a new method or procedure. NELAC also requires annual documentation of proficiency per analyst per method.

WHAT ARE METHOD DETECTION LIMITS?

The method detection limit, or MDL, as defined by the EPA, is the minimum concentration of a substance that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from analysis of a sample in a given matrix containing analyte. It is important to remember that the matrix of a client sample is an unknown that can only be approximated in the laboratory. The procedure for determination of an MDL is analysis of 7 replicate samples at a concentration that is 1 to 5 times the estimated MDL. The data from these 7 replicates is statistically treated to arrive at the lab's MDL, by calculating the standard deviation of the 7 replicates, and multiplying the standard deviation by 3.143 which is the Students' t-value at the 99% confidence level for 7 replicates. Another similar measure is called the IDL, or instrument detection limit. The IDL is similar to the MDL with the exception that the IDL is determined on reagent water as opposed to a blank matrix. The IDL is also referred to as the LOD or lower order of detection.

MDL studies are required when the initial method validation is done, and must be repeated with any major change in methodology or instrumentation. Some methods require annual MDL studies; others require annual verification.

HOW ARE REPORTING LIMITS ESTABLISHED?

Once the MDL or IDL is determined, the laboratory uses that information to establish a PQL (practical quantitation limit) or RL (reporting limit) or EQL (estimated quantitation limit). There are other terms for this limit, including LOQ (lowest order of quantitation) and DL (detection limit) and RQL (reportable quantitation limit). Now, we don't use all these terms, but these terms all are used interchangeably to refer to the lowest concentration of an analyte that the laboratory will routinely report. At Schneider Laboratories, we generally define the PQL, as recommended by EPA methods including SW-846, to be 5-10 times the MDL. The purpose of an MDL study is to provide analytical proof that the PQL is valid. Remember that any blank matrix used in the lab to determine a MDL, or for any QC use, is an estimation of a real-world matrix and not the same as testing on an unknown matrix. Real-world matrices tend to be much more complex than a laboratory's "clean" matrix and always add more variability to the testing. This is probably the primary reason for the EPA recommendation for the EQL to be 5 to 10 times the MDL. As you know from your perspective and we know from the lab's, it is very important that a laboratory not report a false positive. This guidance provides an appropriate margin between detection and quantitation limits and generally gives a reporting limit well below the level of interest.

The reporting limit is also greater than or equal to the lowest standard determined. This is based on the concept that all sample results must be bracketed by the lowest and highest standards used in the instrument calibration.

WHAT ABOUT VALUES BETWEEN THE MDL AND PQL?

There is a special convention used for some analyses, by special request, for reporting values that are above the MDL and below the PQL. These values are labeled with a "J-flag" which indicates that the value is estimated. J-flags can indicate an estimated value at either end of the calibration curve, either between the MDL and the low standard (which is at the PQL) or above the highest standard. Our policy for reporting with J-flags is as follows:

First, only mass-spec results are reported with this convention for low-level estimated values (those between the MDL and PQL) since by nature of the technology, mass-spec results are a positive ID.

Second, low-level estimated mass-spec values are reported upon request of the client, but not in our standard reporting mode.

Third, the laboratory will initiate reporting of estimated values without the client's request if laboratory takes action that raises the PQL above the usual level. For example, if a sample cannot be concentrated to the usual 1ml volume, and instead has to be run at a 10ml volume, this is, in effect, a dilution of 10 which subsequently raises the PQL by a factor of 10. If we get a 'hit' below the reporting limit but above the detection limit for this sample, we would report this to the client as an estimated value.

Fourth, there are also situations when the sample is over-range but cannot be diluted, and a laboratory-initiated J-flag style report would be issued in this case.

HOW IS THE CLIENT'S REPORT DEVELOPED?

We have been asked, "Who sets the reporting format, the client or the applicable regulations?" The answer to that is both, actually, because reports are designed to give certain basic information required by our certifying agencies. Reports are also designed to present data clearly and understandably in a format that suits our clients' needs. All reported values are reported with the units of measure as well as the corresponding reporting limit. At Schneider Laboratories we are able to accommodate many requests for customized reports within our LIMS system, and we frequently work with clients on an individual basis to provide a report of a desired format.

WHEN SAMPLES ARRIVE

WHAT HAPPENS TO MY SAMPLE WHEN IT ARRIVES IN THE LAB?

When a sample arrives in the laboratory, more quality assurance measures come into play. First, the sample is evaluated against acceptance criteria applicable to the sample type and tests ordered. This evaluation may include such things as checking temperature, pH, and free chlorine so that documentation can be made about the sample's preservation before arrival at the laboratory. Samples are also evaluated based on whether or not they were packaged and sealed in a manner that will preserve their integrity and prevent cross-contamination with other samples. Any exceptions to normal acceptance conditions are noted in laboratory documents, and the client may be contacted for authorization to continue testing if there is cause to believe that the sample condition can compromise the test results.

WHAT OCCURS AT SAMPLE LOGIN?

The sample is logged into a computer system so that it can be uniquely identified for the rest of its handling in the laboratory. The appropriate method selection is made based on the client's data request and the matrix submitted. Our computer system also provides many other conveniences for sample handling, such as prioritizing samples, providing associated demographic information for the client, providing labels and worksheets, and giving a sample tracking system so that any sample's status can be quickly checked at any time in the process.

WHAT MEASURES GIVE CONFIDENCE TO MY REPORTED RESULT?

As the sample is prepared and tested, many quality control measures accompany the sample through the process. Most QC samples are run at the rate of one per 20 samples, but some individual method requirements may be different.

INSTRUMENT CALIBRATION AND CONTINUING CALIBRATION VERIFICATION

Instruments are calibrated using certified reference standards over a linear range of quantitation that is bracketed by the low and high standards. In other words, results below the low standard or above the high standard are not

reported or are reported with a qualifier such as the J-flag we discussed. Generally, results below the low standard are reported as “less than the detection limit” and results above the high standard are diluted and repeated so that the analysis is within the linear range of calibration. Frequency of calibration is instrument-dependent and is specified in laboratory SOPs. The integrity and stability of instrument calibration is monitored with calibration verification samples, which are calibration standards that are independent of the standards used to calibrate the instrument. These calibration verification samples are either from a different manufacturer or a different lot or prepared independently from the calibration standards and are used to assure validity of the original calibration. They are also repeated at a rate of 1 per 10 samples for most assays and once per 12 hours for GC/MS analyses as a confirmation of calibration stability.

NEGATIVE CONTROLS

Laboratory blanks

Laboratory blanks or media blanks monitor lab reagents and media for analyte contamination. Blanks are always an important part of lab QC, as they provide a “negative control”, which is a basic part of any scientific analysis. Lab blanks are prepared on a known analyte-free or “blank” matrix when available, and other blanks such as trip blanks and equipment blanks are run as submitted by clients. Trip and equipment blanks play an important role in the client’s sampling plan because they provide assurance that positive results are not from sources other than the one being tested.

Trip blanks

The EPA Technical Enforcement Guidance Document for groundwater testing describes the process for collection of trip and equipment blanks. A trip blank is prepared by filling one of each type of sample bottle with Type II Reagent grade water, transporting to the site, handling like a sample, and returning it to the laboratory for analysis. One trip blank per sampling event is recommended.

Equipment blanks

An equipment blank ensures that the sampling device has been effectively cleaned. The device is filled with Type II Reagent grade water or the water is pumped through the device, transferred to the sample collection container, and returned to the laboratory for analysis. A minimum of one equipment blank for each day that ground-water monitoring wells are sampled is recommended.

The EPA Technical Enforcement Guidance Document specifies that if contamination is found in the trip blank or equipment blank, the source of the contamination should be identified and corrective action, including resampling, should be initiated. The guidance document does not approve the practice of blank-corrected sample data.

POSITIVE CONTROLS

Laboratory Control Samples or Laboratory Fortified Blanks

Laboratory control samples or laboratory fortified blanks are used to monitor the accuracy of the sample handling and analysis. Usually an analyte-free or blank material is spiked or fortified with a known amount of a certified

standard. These spiked samples are prepared and processed with client samples, so their role is to provide the basic element of scientific analysis called a “positive control”. These spiked samples are analyzed and evaluated as percent recovery to provide accuracy data, or data that characterizes the method’s ability to get the expected or “right” answer.

Duplicates to Monitor Precision

When these spikes are done in duplicate, additional information can be derived from the analysis, because comparison of the duplicates can be used to evaluate method precision or reproducibility. Duplicates of a client sample give information about the homogeneity of a sample and/or the precision of the analytical method. Spikes onto a client’s sample give information about recovery of the analyte of interest on the client’s matrix, including matrix interference or suppression of the analyte.

Surrogate Spikes

Surrogate spikes are a quality control tool used in organic analyses in which a compound other than the target compound is spiked onto the sample at the onset of its processing. Surrogate compounds are similar to the target analytes in chemical composition and behavior in the analytical process, but are not typically found in environmental samples. Surrogate spikes allow quantitation of the percent recovery of these similar analytes without introducing target analytes into the process. When methods require surrogate spiking, surrogates are spiked onto every sample, not the 1 per 20 frequency that is typical of many other QC protocols.

Evaluation of Quality Control Measures

These quality control samples and calibration verification samples are part of the stream of samples processed side-by-side with samples of unknown content submitted to the laboratory by clients. Acceptable performance of these measures give confidence to the analysis results for the unknown sample. Acceptance criteria for each of these quality assurance measures are established, and every value has specific evaluation criteria. That criteria is established in various ways. For some, acceptance criteria is given by the method, and for others the criteria is given by an accrediting authority. For most quality control samples, acceptance criteria is established by statistical evaluation of data using the mean plus/minus 3 standard deviations to establish control limits. In the event of a quality control failure, the probable cause is determined, and samples may be repeated when it is determined that the cause of the failure no longer exists.

More quality assurance measures are done after sample processing is completed. Blank, spike, and calibration data is summarized into tables and control charts for evaluation. The data is used to calculate control limits, and also to evaluate shifts and trends in the data.

Data Review

Individual report data is reviewed and validated in the final stages of the laboratory process. Quality control and associated raw data are reviewed at the processing stage by the analyst. Data is reviewed for transcription errors by the data entry staff. Most calculations are done by computer software. The final review stage includes review of the client’s test request and the analysis and quality control data. This final report review is done by the department manager or his or her designee. The amount of detail and data included in the final report package can be customized to meet client needs.

CONCLUSION

The quality assurance program at Schneider Laboratories, Inc. is a robust, mature one that has been evaluated against many sets of standards. We are confident that you will be pleased with the outstanding quality of testing we provide. We look forward to being of service to you for your laboratory testing needs.