A Practical Approach to Differentiate and Comprehend the QC Components of a Commercial Analytical Testing Laboratory

Commercial laboratories providing sample data to clients may provide the quality control information associated with the samples analyzed. In most cases, the quality control information provided or requested by the client is the reference material data that is run with the submitted samples and blank and duplicate data from the analysis. This QC information is not always defined or differentiated and may not completely cover all stages of the process; initial sample preparation to the final data. This can result in misleading and misunderstood information.

In a commercial laboratory it is not practical in both time and cost to provide estimations of the measurement uncertainties at each stage of the process. However, it is necessary for the laboratory to understand, identify and monitor the components of measurement uncertainty so QC information available to the client is useful. There are suitable and practical ways for commercial laboratories to provide the client with sufficient quality control information so that the variances can be differentiated within reason and therefore evaluated to meet the needs of the client.

In best practice situations, the reference materials used are certified with an established degree of confidence and associated uncertainties. In house reference materials are also deemed suitable if there is demonstrated path to a certified source. In both cases, certain criteria must be met. Reference materials must be homogenous and stable for a specified time period, the analyte in question must be at a comparable concentration range and preferably with similar mineralogy. A good reference material provider will produce a certificate of analysis and report that includes the number of participants, the within laboratory standard deviation and the between laboratory standard deviation to ensure that method dependency is evaluated. The laboratory itself can suitably calculate the measurement uncertainty of reference materials when the material is in sufficient quantity to generate statistical data over a reasonably long time period. Involving different analysts, different environmental conditions, different instruments and/or equipment provides a degree of rigour that enables confidence in the data to be generated. Certified reference materials (CRMs) are prepared and purchased commercially. These are suitably homogenous and ready for use. These materials are introduced during weighing at the digestion or fusion stage and can only provide a partial estimation of the overall measurement uncertainty associated with the entire process. If used appropriately i.e. conditions of homogeneity, stability, concentration, mineralogy and method dependency are met, they aid in the determination of the accuracy associated with the digestion or fusion stage through to the finish technique. CRMs fail to provide a correlation to the sample preparation stage, which is commonly the largest source of error involved during the entire procedure, unless we consider the sampling error involved prior to arrival at the laboratory.

Duplicate data can provide a good estimation of the precision of the method but is dependent upon the stage of duplicate insertion and the number of duplicate pairs. Reporting duplicate data from the weighing stage during the digestion process provides a partial estimation of the precision of the method, but not the entire precision of the process. For best practice, the duplicates should be taken at both the crushing-splitting and weighing-digestion stage. The difference in the variances obtained from a minimum of twenty pairs of duplicate data can provide the overall precision of the process, the

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significance of the differences can also be assessed to differentiate the possible errors involved including sample heterogeneity.

The measurement uncertainty associated with the instrument or finish stage of a method is best differentiated with the use of certified calibration materials, secondary source control checks, duplicate insertion and in some cases spike recovery. To minimize the error associated with the instrument and the reference materials used to establish the instrument response, it is important to store the materials appropriately to ensure stability and to matrix match the materials as close as possible to that of the samples analyzed. In best practices, the calibration materials are checked against secondary source control materials purchased separately. This ensures the materials used to calibrate the instrument are suitably stable and correctly made, as these often involve serial dilutions from a master source. The degree of diligence involved in the calibration can contribute to the error involved and must be evaluated. The finish technique must have demonstrated linearity and the samples analyzed must fall within the linear range or be diluted accordingly. Duplicates inserted at this stage differentiate the precision that can be associated with the instrument response.

The laboratory can best demonstrate and differentiate all of these quality control measures by the use of control charts. Most LIMS systems have the ability to chart data automatically to provide a quick visual aid when data evaluation is performed. Control charts are used to monitor performance and provide crucial audit trails. Almost all QC information, and therefore errors associated with the entire process, can be charted; this includes reference materials, calibration and control check materials, blanks, duplicates at different stages in the process, verification checks for balances, ovens, furnaces, dispensers and autopipets, spike recoveries and screen tests. Evaluating these errors and combining the significant errors, generates the overall measurement uncertainty, a useful and robust statistical value.

There are many types of control charts available for use, all which have the ability to demonstrate and monitor the accuracy and precision of a method. The Shewhart Chart [1] is a reliable resource for demonstrating the accuracy or bias associated with the reference materials involved in the process. This chart involves drawing a horizontal line at the mean value and at locations two standard deviations (the warning level) and three standard deviations (the control limits) above and below the mean. After generating a minimum of twenty data points, the chart can easily demonstrate the accuracy and bias of the

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method. There are specific rules for Shewhart charts [1] that ensure statistical control of the measurement process.

The range control chart [2] is another reliable source for demonstrating the precision of a method. Using a minimum of twenty sets of duplicates, the mean is calculated from the absolute value of the differences between the duplicate pairs (the mean of the ranges). The mean value (R) is plotted along with lines at 0.845R, 2.51R and 3.27R. Statistical control is monitored and deemed acceptable when 50%, 95% and 99% of the ranges lie below the 0.845R, 2.51R and 3.27R lines, respectively. For best use, the concentration ranges of the duplicate data should be grouped according to orders of magnitude and charted individually.

A practical approach to providing the client with meaningful QC information without producing multiple control charts and error values is to report the combined estimation of the measurement uncertainty. This is calculated from the data generated by the use of reference materials preparation duplicates and digestion duplicates, over a reasonable time period. All associated errors involved in the entire process are accounted for and easily differentiated by the laboratory's use of control charts as described above. Reporting the reference material and duplicate data, both preparation and digestion duplicates, also allow the clients to calculate the precision and accuracy and differentiate the variances of their own test data.

References

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