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1.0 SCOPE

The AIHA LAP, LLC (AIHA LAP) Policy on the Evaluation of Measurement Uncertainty documents the requirements for accredited laboratories to maintain accreditation to ISO/IEC 17025 with regard to evaluating and reporting measurement uncertainty. This guidance document provides additional information intended to assist laboratories with efficiently and effectively implementing the AIHA LAP Policy. AIHA LAP wishes to thank and acknowledge the Canadian Association for Laboratory Accreditation (CALA) for its permission to incorporate elements of CALA P19 –*Policy on the Estimation of Uncertainty of Measurement in Environmental Testing* in preparing the initial version of this guidance document.

2.0 REFERENCES

The following documents provide the basis and assist with application of the principles stated in this guidance document.

- **General requirements for the competence of testing and calibration laboratories,** ISO/IEC 17025:2017
- **AIHA LAP Policy Appendix G on the Evaluation of Measurement Uncertainty;** www.aihaaccreditedlabs.org
- **Policy on the Estimation of Uncertainty of Measurement in Environmental Testing,** CALA P19; www.cala.ca
- **Guidance on the Implementation of the CALA Measurement Uncertainty Policy,** CALA P19-02; www.cala.ca
- **ILAC G17:01/2021: ILAC Guidelines for Measurement Uncertainty in Testing;** https://ilac.org/publications-and-resources/ilac-guidance-series/
- **ILAC P14:09/2020 ILAC Policy for Measurement Uncertainty in Calibration**; https://ilac.org/publications-and-resources/ilac-policy-series/
- **Quantifying Uncertainty in Analytical Measurement**, 3rd Edition, 2012, Eurachem/CITAC, http://www.eurachem.org

3.0 TERMS AND DEFINITIONS

Refer to the AIHA LAP Policy on the Evaluation of Measurement Uncertainty for additional terms and definitions. Pertinent terms (and associated sources) used in this document are presented below:

bias (measurement bias) (VIM 2.18 JCGM 200:2012): estimate of a **systematic measurement error**
NOTE: Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value.

combined standard uncertainty (combined standard measurement uncertainty) (VIM 2.31 JCGM 200:2012): **standard measurement uncertainty** that is obtained using the individual **standard measurement uncertainties** associated with the **input quantities in a measurement model**

coverage factor (VIM 2.38 JCGM 200:2012): number larger than one by which a **combined standard measurement uncertainty** is multiplied to obtain an **expanded measurement uncertainty**
NOTE: A coverage factor, *k*, is typically in the range of 2 to 3.

empirical test method: a test method intended to measure a property that is dependent on the test method used to measure it. Different methods for the same test parameter may return different results that may not be related. In many cases, one method cannot be verified using another test method.

expanded uncertainty (Expanded measurement uncertainty) (VIM 2.35 JCGM 200:2012): product of a **combined standard measurement uncertainty** and a factor larger than the number one
NOTE 1: The factor depends upon the type of probability distribution of the **output quantity in a measurement model** and on the selected **coverage probability.**
NOTE 2: The term "factor" in this definition refers to a **coverage factor.**
NOTE 3: Expanded measurement uncertainty is termed "overall uncertainty" in paragraph 5 of Recommendation INC-1 (1980) (see the GUM) and simply "uncertainty" in IEC documents.

Coverage probability (VIM 2.37 JCGM 200:2012): probability that the set **of true quantity values** of a **measurand** is contained within a specified **coverage interval**
NOTE 1: This definition pertains to the Uncertainty approach as present in the GUM
NOTE 2: The coverage probability is also termed "level of confidence" in the GUM

measurand (VIM 2.3 JCGM 200:2012): **quantity** intended to be measured
NOTE 1: The specification of a measurand requires knowledge of the **kind of quantity,** description of the state of the phenomenon, body, or substance carrying the quantity, including any relevant component, and the chemical entities involved.
NOTE 4: In chemistry, "analyte", or the name of a substance or compound, are terms sometimes used for 'measurand'. This usage is erroneous because these terms do not refer to quantities.

Precision (measurement precision) (VIM 2.15 JCGM 200:2012): closeness of agreement between **indications** or **measured quantity values** obtained by replicate **measurements** on the same or similar objects under specified conditions.
NOTE 1: Measurement precision is usually expressed numerically by measures of imprecision, such as standard deviation, variance, or coefficient of variation under the specified conditions of measurement.
NOTE 2: The 'specified conditions' can be, for example, **repeatability conditions of measurement, intermediate precision conditions of measurement,** or **reproducibility conditions of measurement** (see ISO 5725-3:1994).
NOTE 3: Measurement precision is used to define **measurement repeatability, intermediate measurement precision,** and **measurement reproducibility.**
NOTE 4: Sometimes "measurement precision" is erroneously used to mean **measurement accuracy.**
NOTE 5 (ISO3534-1): Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is reflected by a larger standard deviation.

rational test method a test method intended to measure a property that is defined independent of any test method. There is an objective "true" value to that property and a method can be verified using other test methods. It is recognized that, although there is an objective "true" value, it may be very difficult to measure that value.

repeatability (measurement repeatability) (VIM 2.21JCGM 200:2012): **measurement precision** under a set of **repeatability conditions of measurement**
NOTE: Repeatability conditions include: the same measurement procedure; the same observer; the same measuring instrument used under the same conditions; the same location; repetition over a short period of time.

Reproducibility (measurement reproducibility) (VIM 2.25 JCGM 200:2012) **measurement precision** under **reproducibility conditions of measurement**
NOTE: Reproducibility conditions of **measurement** include different locations, operators, **measuring systems,** and replicate measurements on the same or similar objects

standard uncertainty (standard measurement uncertainty) (VIM 2.30 JCGM 200:2012): **measurement uncertainty** expressed as a standard deviation

type A evaluation of measurement uncertainty (VIM 2.28 JCGM 200:2012): evaluation of a component of **measurement uncertainty** by a statistical analysis of **measured quantity values** obtained under defined measurement conditions
NOTE: For various types of measurement conditions, see **repeatability condition of measurement, intermediate precision condition of measurement,** and **reproducibility condition of measurement.**

type B evaluation of measurement uncertainty (VIM 2.29 JCGM 200:2012): evaluation of a component of **measurement uncertainty** determined by means other than a **Type A evaluation of measurement uncertainty**
EXAMPLES Evaluation based on information:
- associated with authoritative published **quantity values,**
- associated with the quantity value of a **certified reference material,**
- obtained from a **calibration** certificate,
- about drift
- obtained from the **accuracy class** of a verified **measuring instrument,**
- obtained from limits deduced through personal experience.

4.0 BACKGROUND

The guidance provided in this document is intended to be helpful suggestions. It is important to note that it is not possible to provide guidance and examples for every testing activity in every laboratory. It is the responsibility of each laboratory to investigate this subject and choose to implement any or none of the methods described in this document, as long as the requirements of the AIHA LAP Policy on the Evaluation of Measurement Uncertainty are met. Links to several references and tools are provided for the convenience of the laboratories. It is also the responsibility of each laboratory to evaluate the effectiveness of the method it chooses to implement and take responsibility for the consequences of the decisions taken as a result of the method chosen.

As a general metrological principle, known systematic errors that contribute to method bias should be eliminated, or corrections should be made to account for them. However, there are many cases where biases cannot be eliminated and where correction factors cannot be reliably applied, because the biases are inconsistent and poorly characterized. If bias is corrected for in a manner that eliminates its impact on test results, then it will not contribute to measurement uncertainty. On the other hand, if a systematic bias exists and is not corrected for, then it should be reported separately (see section 5.3.2.6).

5.0 GUIDANCE ON IMPLEMENTATION

There are three approaches that can be taken in estimating the measurement uncertainty associated with testing.

1) One approach is that stipulated in Note 1 to Clause 7.6.3 of ISO/IEC 17025 that states*: "In those cases where a well-recognized test method specifies limits to the values of the major sources of measurement uncertainty and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions."* This approach is applicable to test methods such as NIOSH Method 7400 where the method is followed exactly as written and test results are reported in compliance with the method. This approach does not apply to a test method that is based on (i.e., a modified version of) a reference method.

2) A second approach (termed the Type A Method) uses existing data from routine laboratory quality control samples such as, certified reference materials, laboratory control samples, duplicates, or data from method validation studies and proficiency testing (PT) programs.

3) A third approach (termed the Type B Method) involves the evaluation and compilation of individual uncertainties for each contributing measurement.

The AIHA LAP Policy on the Evaluation of Measurement Uncertainty accepts any of the three approaches described. The first approach can only be used provided the test method is followed exactly as written and achieves bias and precision estimates similar to those stated in the reference method. In all other cases the laboratory must choose between using the Type A or Type B approach.

5.1 *Choosing Type A or Type B*

The Type A Method is the approach used by most specifier agencies when requiring evaluations of the measurement uncertainty for analytical laboratories. It is also typically the first choice of laboratories because it is the most efficient and cost-effective approach.

The Type A Method allows laboratories to statistically analyze their existing quality control (QC) sample results, method validation/verification data, and/or proficiency testing results to provide a reasonable evaluation of measurement uncertainty. This assumption can be made as long as the following criteria have been met:

- Where applicable, the analyses have been carried out over an extended period of time and involve a representative number of combinations of operators and equipment, to provide reproducibility conditions.
- The measurement procedure is stable and has remained in statistical control during the period of the analysis.
- All variables and processes that contribute significantly to the uncertainty of the test have been captured by the selected QC data. Any factors not captured must be considered separately.

Other advantages of using the Type A Method

- The Type A approach is consistent with requirements found in the reference documents cited in Note 3 ISO/IEC 17025 Section 7.6.3.
- It is accepted as a valid approach in the international community. For example, the EURACHEM CITAC Guide *Quantifying Uncertainty in Analytical Measurements* states: “Where a factor has been representatively varied during the course of a precision experiment … that factor needs no additional study.”
- Virtually all of the data required is already present in laboratory quality control data.

- Little time is required to evaluate the uncertainty for individual methods using the laboratory historical data. Most applicable components of the laboratory measuring system are addressed.

- The resulting evaluation is robust, internationally accepted, and defendable to customers and specifiers.

The Type B Method, on the other hand, looks individually at the contribution to uncertainty of each measurement made during the sample handling and testing activities. It involves the cause-and- effect-based metrological estimation of specific uncertainties from each identified source. This is similar to the approach used by calibration laboratories. The variability of each measurement is quantified either by using measurement uncertainty information from calibration certificates, from specifications, or by performing studies that will quantify the variability from isolated measurements. An advantage of this approach is that it is an in-depth examination of each critical measurement step in the test method. A disadvantage is that it ignores variability information provided by QC samples.

In other words, the Type A Method uses data the laboratory has already collected to provide long term variability information on the test method processes. The Type B Method on the other hand involves looking at each measurement step in a test method and determining a way to measure the variability attributed to that step.

Although Type A is the most commonly used method because of ease of use and cost effectiveness, each laboratory is responsible for deciding on the method that best meets its needs.

5.2 *General considerations*

When using either method, documented training is required to enable laboratory staff to do the necessary work and perform the needed calculations.

Identifying contributors to uncertainty and evaluating an initial measurement uncertainty is considered to be part of method validation or verification. It is recognized this uncertainty will not include long term variability. Therefore, evaluations must be repeated using long term data when available.

Contributors to measurement uncertainty may be common to several test methods. Once contributors have been identified for a test method, the same contributors can be used for the evaluation of uncertainty for other similar test methods. Although the following provides a step-by-step approach, any reasonable and valid approach to evaluating measurement uncertainty is accepted, as long as the requirements of AIHA LAP Policy on the Evaluation of Measurement Uncertainty are satisfied.

5.3 *Sequential Steps*

5.3.1 Define the measurand – Applies to both Type A and Type B Methods

It is recognized that the measurand is sometimes defined by the test method used for the analysis, such as in the case of empirical methods, and the test result may not be directly traceable to SI units. Alternatively, for rational test methods, the "true" value of a sample may not be easily measured.

It is therefore important to begin the exercise by defining what is being measured and understanding any limitations imposed by the test method, specifically, document the parameters being measured, in what matrix, and by what test method.

For example: Total micrograms of lead on filters by HNO_3 digestion and ICP-AES analysis (Laboratory SOP-123).

5.3.2 Using the Type A Method

The following general steps apply:

5.3.2.1 Identify the measurand as in 5.3.1 above.

5.3.2.2 Using the test method SOP and the final result-calculation equations, identify and list all factors that may contribute to uncertainty (sources of uncertainty). Consider the following categories or processes as listed by CALA and ILAC Guide 17:

- Sampling or sub-sampling – in-house sub-sampling typically applies only to bulk samples tested in AIHA LAP laboratories. Effects due to variation between sub samples and potential for bias in the sub-sampling procedure (e.g., due to sample size, processing techniques, etc.) may be significant contributors to uncertainty. Note that field sampling is most often outside the responsibilities of the laboratory. **In such cases, the laboratory should clearly state that any evaluations of uncertainty reported with samples relate only to analytical uncertainty.** Sub-sampling within the laboratory does not apply where the entire sample is consumed during sample preparation or analysis.

- Transportation, storage and handling of samples – for those processes under the control of the laboratory and where test samples are stored prior to analysis, consider the impact of the variability of environmental conditions (e.g., temperature, time, contamination in storage area) on the test results. Note that when transportation of samples prior to their submission to the laboratory is outside of the control of the laboratory, variability attributed to transportation is not considered part of analytical uncertainty. As well, when samples are stored according to well established references, the impact of storage on measurement uncertainly can be considered to be minimized and need not be included in uncertainty evaluations.

- Preparation of samples - all steps of the sample procedure prior to analysis. This can include variations in drying, grinding, filtering, weighings, dispensing of materials, extractant backgrounds, etc.

- Environmental and measurement conditions - those conditions that can impact some test results (e.g., gravimetry, microbiology) when they vary (e.g., temperature or humidity of the balance room, seasonal changes in microbiological background of micro labs, etc.).

- The personnel carrying out the tests - different analysts impact test results over time. Each individual brings their own training, experience and technique to critical steps in the test method. This is especially important with subjective tests such as microscopy and organism identification.

- Variations in the test procedure - for example, different recoveries for different batches of media, impurities in reagent lots, the effect of different extraction, digestion or incubation times and temperatures, percentages of microscopic samples read, etc.

- The measuring instruments - variations in baseline drift, day to day calibration differences, carry over effects, interferences specific to the test method, microscope magnification used, etc.

- Calibration standards or reference materials - uncertainty related to reference materials or due to preparation differences, etc. Uncertainty estimates may come from certificates of analysis or estimations based on provider claims.

- Methods of generating test results - uncertainty due to data interpretation (e.g., peak integration, baseline manipulation, etc.), blank corrections, differences in how the software was used, other data manipulations, etc.

- Corrections for systematic effects - if test results are corrected for bias, include the uncertainty of the correction.

As you proceed through the test method, ask if variability from each process will impact the test result. If the answer is yes, then that step or process is a contributor to uncertainty for the test method and should be added to your list of contributors.

For qualitative test methods, completing these steps to document the understanding of the variability of the test method and using this information to minimize variability, where possible, fulfils the policy requirements and needs no further work.

5.3.2.3 Identify the QC Data to be Utilized

QC data to be used are sources of repeated measurements from which standard deviations (SDs) or relative standard deviations (RSDs) can be calculated. Since the laboratory typically varies one or more of the above contributors to uncertainty during the collection of the repeat data, the SD or RSD calculated will include uncertainty attributed to the varied source(s).

The AIHA LAP Policy on the Evaluation of Measurement Uncertainty indicates the use of the following types of QC samples. These are listed in descending order of preference.

5.3.2.3.1 Laboratory Control Samples (LCS), or Matrix Spikes (MS) from long term data collected from routine sample runs or from method validation/verification studies for new test methods. These are examples of quality control sample data that have gone through every sample preparation and analysis step, and will provide feedback on the preparation of samples, environmental conditions, personnel, test procedure, instrumentation, calibration standards, and software processes. These data may be part of routine quality control and/or those used in method validation/verification.

Laboratory control samples may include matrix matched certified reference materials and in-house reference materials (e.g., media spikes and reference slides for environmental microbiology direct examination air). The LCS data are used for routine runs for control charting applications and are a source of long-term uncertainty data. Components that contribute to the uncertainty (e.g., analysts, calibration sets, calibration solution sources, environmental conditions, instrument drift, and many more) vary during repeated insertion of these materials. When the laboratory uses chemical, environmental, or microbiological test methods based on published reference methods (e.g., NIOSH, OSHA, EPA, AOAC, ASTM), and when the CRM or LCS has been through all sample preparation and analysis steps, the CRM or LCS precision data can typically be used as an estimate of combined standard uncertainty.

Bias can also be estimated using the average recovery of the certified reference materials or in-house reference materials. When bulk samples are tested and such samples are subject to laboratory sub-sampling, the CRM or LCS does not reflect all sample preparation steps and the contribution of laboratory sub-sampling must also be consider using laboratory sample duplicate data (see Section 5.3.2.3.2 below).

In microbiology test methods, variability is most often attributed to growth characteristics and recovery from media (culturable analyses), variability between analysts (organism identification and counting), and sample handling steps. Similar to chemical analysis laboratories, microbiology laboratories incorporate QC samples that provide information on these steps; however, there are few quantitative reference materials for microbiology tests. As indicated above, reference slides prepared by the laboratory and required for the direct examination air program can be treated in the same manner as laboratory control samples to evaluate uncertainty. For other test methods, intra-analyst and inter-analyst comparisons are routinely conducted.

Method validation replicate data provide a source of data to establish precision estimates at different analyte concentration levels. The results from runs at low concentrations for the calculation of detection and/or reporting limits can also be used to assess uncertainty at low analyte concentration ranges. The validation data can also serve as a source of information on the uncertainty contributed by other sources (such as analyst, instrument, temperature, time, etc.), depending on how the validation work was planned and executed to include such variables. This is especially the case if ruggedness studies were incorporated as an integral part of the validation program to assess the effect of varying parameters likely to be significant sources of uncertainty. A thorough discussion of the use of method validation data in the estimation of uncertainty is *VAM Project 3.2.1 Development and Harmonization of Measurement Uncertainty Principles; Part (d): Protocol for uncertainty evaluation from validation data,* by V.J. Barwick and S.L.R. Ellison, January 2000, Version 5.1. This can be downloaded as a .pdf file from the VAM web site.

Matrix spike recovery data from spiked customer samples yield information similar to laboratory control samples. With the exception of the Environmental Lead Laboratory Accreditation Program (ELLAP) paint and soil matrices, matrix spikes are rarely performed in AIHA LAP laboratories. The standard deviation of the matrix spike recovery reflects the uncertainty contributions from the same sources as certified reference materials and LCS, but also includes the effects of the customers' sample matrices. Customer matrix spike data should be used with caution since these data reflect variability outside of the laboratory's control and typically will overestimate analytical uncertainty.

5.3.2.3.2 Duplicate data (when sub-sampling occurs in the lab) from long term routine sample runs or from method validation/verification studies for new test methods. Duplicate samples inserted at the earliest step of sample handing will provide feedback on the variations in sub- sampling and sample handing processes. These may be part of routine quality control or produced during method validation/verification.

Duplicate samples are a valuable source of uncertainty data – known as **repeatability** SD_{dupl} - that reflects the variability due to differences between analytical portions (non-homogeneity) and other sub-sampling factors that can vary between replicates (e.g., weighing, volumetric manipulations, short term instrument drift, and percent of sample mount examined during microscopic analyses).

NOTE: If the duplicates are measured in the same analytical chemistry run, as is usually the case for most IHLAP test methods, any uncertainty associated with the duplicate measurements is not accounted for. The standard deviation of the within batch LCS and LCS duplicate (required as part of routine batch QC for most IHLAP test methods) should not be used as duplicate data for estimating uncertainty. Uncertainty is captured by the standard deviation of the LCS data over time (multiple preparation and analytical batches) as stated in section 5.3.2.3.1.

For microscopy and environmental microbiology test methods, intra- analyst and inter-analyst comparisons are routinely conducted. The use of inter-analyst rather than intra-analyst duplicates reflect more sources of uncertainty associated with the process and analysts and provide a better evaluation of uncertainty.

When utilizing duplicate data, more than 20 duplicate pairs should be used of samples at a similar concentration. The $SD_{dupl} = \sqrt{(\sum R^2/2N)}$ where R is the difference between duplicate pairs and N is the number of duplicate pairs. This should be calculated for low, medium and high concentration ranges, as necessary, to reflect the concentration dependence of the SD. Alternatively, the RSD can be calculated (at low, medium and high concentration ranges, as needed) as $RSD_{dupl} = \sqrt{\{\sum[(a_i - b_i)/-x_i]^2/2N\}}$ where $(a_i - b_i)/-x_i$ is the relative difference between duplicates for sample "i" and N is the number of samples for which duplicates have been run. The RSD can also be determined for each duplicate pair and the RSDs can be "pooled" to give an overall RSD. The RSD value makes allowances for the concentration dependence of the SD for concentrations between those at which the calculation was made.

Environmental microbiology data may often be statistically analyzed in this manner; however, transforming data by taking the square root or the log of the data prior to statistical analysis may also be appropriate. Consult the **"Statistical Analysis of EMLAP QC- Guidance Document"** for additional guidance on this subject as well as the "pooling" of RSD values.

5.3.2.3.3 Method blank data. When taken through the entire test method process, method blanks are an important source of variability information for test methods where the absolute value of the blank is typically greater than the reporting limit. The values for these method blanks will represent variability within the laboratory's control, such as the grade or purity of solvents or contamination or will represent sampling media variability or instrumental variability (such as carry-over, baseline variation). In the first case, laboratories are encouraged to identify the source and reduce the impact. However, when method blank values impact test results, they represent contributors to measurement uncertainty and must be considered.

5.3.2.3.4 Proficiency Testing (PT) Sample Data. Inter-laboratory bias and standard deviations derived from proficiency testing programs can be used if no laboratory specific quality control data are available. These include components of uncertainty that include both intra- and inter- laboratory sources. Estimates of uncertainty derived from proficiency testing (inter-laboratory **reproducibility** SD (SD_R) data) are larger than the intra-laboratory **repeatability** SD (SD_r)) of a laboratory whose methods are in statistical control. In the absence of any other source of repeated data, reproducibility from proficiency testing and other round robin studies can be used as an evaluation of measurement uncertainty provided the laboratory can demonstrate that their bias is within certain bounds, consistent with the collaborative study estimate of between-laboratory SD. It is, however, very likely to be an overestimate of what the intra-laboratory uncertainty

actually is (by a factor of 2 according to conventional wisdom). Some proficiency testing includes the component of bias depending on the study design.

5.3.2.4 *Review the contributions*

Verify that all significant sources are covered by the QC samples. When possible, minimize counting the contribution of a source of uncertainty more than once in the calculation of the combined standard uncertainty. In unusual cases there may be processes for which the QC data will not provide complete variability information. For example, if duplicate samples are split after sample digestion or extraction, they will not provide sub-sampling information. In these cases, information can be obtained from studies performed during method validation or verification studies, or the information can be obtained from a similar method, or a separate study may be needed. If separate studies are needed, consider changing the QC practices to include as much variability information as possible.

5.3.2.5 *Compile the data and perform the calculations*

Compile the data for the chosen QC samples and calculate the standard deviations (SD) or relative standard deviations (RSD) for each QC type and any other individual components, where needed. The contribution from reference materials is most often difficult to quantify. If the reference material is certified, the certificate will contain measurement uncertainty information, or at least values for purity or variability. Many reference materials are not certified or are not certified by an accredited reference material provider, so the certificates are often lacking. In these cases, an attempt should be made to evaluate the contribution to the overall uncertainty of the test method and determine its significance.

Those sources that have an SD of less than 1/3 the largest SD can be eliminated from the subsequent calculations since their contribution to the combined uncertainty will be negligible.

This process assumes that the test has been operating within statistical control for the timeframe of the data set. There are outlier tests available to test the data set against this assumption. Examples are the Grubbs' Test and Dixon's Test. If such a test is used, the results should be interpreted with care. Data should not be rejected unless there is a sound reason for believing they represent unique and unusual occurrences rather than normal test method performance. If in doubt, err on the side of caution by using all of the data.

5.3.2.5.1 Calculate the combined uncertainty

SDs cannot be manipulated to calculate the combined uncertainty. Instead, SDs are converted into variances by squaring them. The combined uncertainty is calculated by adding the squares and taking the square root.

$$SDc = \sqrt{[SD_1^2 + SD_2^2 + ...+SD_n^2]}$$

It may be beneficial to use RSD instead of SD as it allows for the concentration dependence of SD. For example, if SD is calculated for LCS at a high concentration, applying the SD to low level data may not be appropriate. However, if the SD is divided by the mean of the data set, it becomes more relative to other concentrations.

5.3.2.5.2 Calculate the expanded uncertainty

Apply the appropriate coverage factor 'k'. Calculate the expanded uncertainty by multiplying the combined standard uncertainty by the appropriate coverage factor (k) to give an expanded uncertainty with the desired confidence level. The factor k is the confidence interval Student distribution t-factor for n-1 degrees of freedom. For a confidence level of 95%, k is approximately 2 for a data set of 30 points or more, for normally distributed data sets.

Expanded measurement uncertainty = k x SDc

5.3.2.6 Reporting test results with the expanded measurement uncertainty

When the reporting of uncertainty is required, the test result and the expanded measurement uncertainty are reported in the same units. The test result and expanded measurement uncertainty should both be rounded in a similar manner (i.e., with the same number of significant figures). Include a description of the coverage factor used in a manner such as the following example:

Total benzene concentration of 88 ug/sample $\pm$ 11 ug/sample at the 95% confidence level (k=2).

Where bias is present, report it along with the uncertainty as a probable bias in a manner such as the following example:

Total lead concentration of 78 ug/sample $\pm$ 12 ug/filter at the 95% confidence level (k=2). This method has an average recovery of 94%, or at this level, a probable bias of -5 ug/filter.

Alternate forms of reporting uncertainty and bias are acceptable as long as required information is clearly presented.

5.3.3 Using the Type B Method

The Type B Method involves the contribution of each individual uncertainty source.

- Identify the measurand as in 5.3.1 above.

- Using the test method document and final test results calculations, list or otherwise document each individual measurement such as mass, volume, temperature, instrument calibration, and any factors that would provide other sources that contribute to the variability of test results, such as the impact by different analysts.

- Tabulate the individual standard uncertainties for each contributor. This information will be available from calibration certificates in the case of calibrated equipment and reference standards, and certificates of analysis of reference materials. Where these uncertainties have been expanded, be sure to divide them by the expansion factor. Where the uncertainties are not readily available, studies will be needed to isolate the uncertainties from that individual step.

- Once data are compiled for each source of uncertainty, determine the applicable distribution and convert the uncertainties to SDs. For example, for normal distributions attributed to reference weights, balances and thermometry, divide the expanded uncertainty by 2. For uniform distributions attributed to readability and corrections divide by the square root of 3. Refer to the Eurachem document and GUM for further information regarding distributions and estimating Type B uncertainty.

- Calculate the combined standard uncertainty and report in the same manner as in the Type A methods as above.

6.0 ADDITIONAL SOURCES OF INFORMATION AND EXAMPLES

In addition to the references listed at the beginning of this document, the following references provide alternate acceptable approaches and additional guidance. Laboratories are encouraged to consider many sources of information to ensure their approach meets the needs of their customers.

AIHA LAP has posted several examples of the Type A approach to evaluating measurement uncertainty on its website at https://www.aihaaccreditedlabs.org/policies/additional-resources

- Example Chemistry Measurement Uncertainty Calculations, https://www.aihaaccreditedlabs.org/policies/additional-resources
- Example Microbiology Measurement Uncertainty Calculations https://www.aihaaccreditedlabs.org/policies/additional-resources
- A Discussion on the Uncertainty Associated with the Bacterial Colony Count Obtained by Membrane

Filtration, Canadian Council of Independent Laboratories.

- **Evaluation of measurement data – Guide to the expression of uncertainty in measurement (GUM),** JCGM 100:2008 https://www.bipm.org/en/publications/guides/gum.html
- **Protocol for Estimating Measurement Uncertainty Using QC Data (Type A),** Canadian Council of Independent Laboratories (CCIL)
- **Quality Assurance of Chemical Measurements,** Taylor, J.K., 1987, Boca Raton, FL: Lewis Publishers Inc.
- **Standard Operating Procedure- Estimation of Analytical Measurement Uncertainty,** Navy Laboratory Quality and Accreditation Office, Navy Shipyard Laboratory Standard Operating Procedure, SOP:06, Revision 2, June 2003 (Uncertainty SOP.doc)
- **Statistical Analysis of EMLAP QC- Guidance Document**, AIHA LAP, https://www.aihaaccreditedlabs.org/policies
- **Uncertainty Calculator**, William Ingersoll, NAVSEA, Charleston, S.C, 2003 (Uncertainty- Calc.xls)
- **Uncertainty of Quantitative Determinations Derived by Cultivation of Microorganisms**, Niemela, S.I., 2002, Centre for Metrology and Accreditation: Helsinki, Finland. p. 75