Measurement of Uncertainty; Calibration Lab
Procedure
1.0 **PURPOSE AND SCOPE**

1.1 **Purpose**
1.1.1 The purpose of this procedure is to define methods for determining and handling Measurement of Uncertainty for Calibration Lab at our Company.

1.2 **Scope**
1.2.1 This procedure is applicable to all programs at ATS.

2.0 **APPLICABLE DOCUMENTS**

The following documents are applicable as specified here in:

- **Industrial/Commercial/Government Documents**
  - ISO 9001: Quality Management System - Requirements

- **Document(s)**
  - ATS-HRP-1001: Training and Certification
  - ATS-QAP-1004: Quality Records
  - ATS-QAP-1008: Internal Audits

- **Form(s)**
  - None

2.1 **Definitions**
2.1.1 **Uncertainty** - is defined by ISO (International Vocabulary of Basic and General Terms in Metrology) as the parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurement.

3.0 **RESPONSIBILITIES**

3.1 **General**
3.1.1 **Quality Assurance** - is responsible for maintaining and performing the activities defined in this procedure.
3.1.2 Laboratory Operations - are responsible for ensuring that the requirements specified in this procedure are being followed.

3.1.3 All Other Functions - are responsible for ensuring that the requirements specified in this procedure are being followed.

4.0 REQUIREMENTS

4.1 General
4.1.1 Quality Assurance and Laboratory management shall be responsible for calculating and determining Measurement of Uncertainty for Calibration Lab as per ISO 17025, General requirements for the Competence of Testing and Calibration Laboratories.

4.1.1.1 The summation of deviation/error from the following three are used to determine the Measurement of Uncertainty:
   1) Uncertainty 1 = Test Process Error
   2) Uncertainty 2 = Test Equipment Error
   3) Uncertainty 3 = Calibration Standard Error (i.e. the error included on the Calibration standard certificate used to calibrate the Test equipment used to conduct lab tests)

4.1.1.2 The ultimate use of such uncertainty estimates is to:
   • Be able to state just how good a test result is.
   • Allow the client or end user of data to properly interpret data in the report.

4.1.2 There are many process factors that influence the correctness and reliability of the tests performed by Laboratory Operations. These factors include contributions from:
   • human factors
   • accommodation and environmental conditions
   • test methods and method validation
   • equipment
   • measurement traceability
   • sampling
   • the handling of test items

4.1.2.1 The extent to which the factors contribute to the total uncertainty of measurement differs considerably between types of tests. The Laboratory Operations and Quality Assurance does take account of these factors in developing test methods and procedures, in the training and qualification of personnel, and in the selection of the equipment it uses.
4.1.3 In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases the ATS laboratory does at least attempt to identify all the components of uncertainty and make a reasonable estimation, and do ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation is based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

4.1.3.1 When estimating the uncertainty of measurement, all uncertainty components which is of importance in the given situation are taken into account using appropriate methods of analysis.

4.2 Approaches to Uncertainty -
4.2.1 There are two (2) contributors to measurement uncertainty (refer to ISO Guide to the Expression of Uncertainty in Measurement):
   • Type A contributors are those that may be determined statistically (random contributors).
   • Type B contributors are those that must be determined by non-statistical methods (see below).

4.2.2 Type A contributors - may be estimated by estimating the overall uncertainty in the entire analytical process by measuring the dispersion of values obtained from well-chosen standards or samples. It is not necessary to break down the Type A contributors any further, i.e., how much is digestion error, weighing error, instrument error, etc., although such a breakdown may be useful in improving analytical precision.

4.2.2.1 If the Type A determination has been all-inclusive, the only common Type B contributor will be the uncertainty of the reference standards and reference materials, which can never be picked up statistically. There is also an interesting, although small, Type B contribution from the uncertainty of atomic weights, but this is small enough to be ignored (in most cases). Generally, in Chemistry, Type B errors are usually insignificant and may be ignored. The Lab should, however, ensure that this is the case before doing so, as in some instances this will not be the case (e.g., references with large uncertainties).

4.2.2.2 Once the Type A and Type B errors have been determined, they may be combined using the technique of root-mean-square sum:

$$\mu_0 = \sqrt{\sum \mu_i^2}$$
4.2.2.3 If only Type A errors are significant, then the test’s random variability (however determined) serves as the measure of uncertainty. Since accredited Labs must do Statistical Quality Control (SQC) for all tests wherever possible (ISO 17025:2005, 5.9), the data generated from this effort also serves the measure of uncertainty; a separate effort is not required.

4.3 Overall Process Estimation
4.3.1 If Type B contributors are indeed negligible, quality control data provide an estimate of the laboratory component (as opposed to the sampling component) of overall uncertainty. For example:

4.3.1.1 Reference Standards: The analyses of reference standards (Standard Reference Material, SRM, or Certified Reference Material, CRM), usually calculated as %yield (100*result/reference content) produce an estimate of bias (average %yield-100), and also precision (standard deviation of %yield). These standards often give the only estimate of bias. However, precision calculated from reference standards is likely to under-estimate (be more precise than) the dispersion of data of real-world samples, due to the extensive homogenization these materials have undergone. To properly estimate bias and precision using a reference standard, a proper number of QC data points (typically 20-30) should be considered.

4.3.2 Duplicates: A better estimate of day-to-day precision can be determined by comparison of duplicate samples (two samples taken from and representative of the same population) or duplicate analyses (the analysis of the variable of interest on two sub-samples of the same sample). The two values are usually compared by their relative percent difference, RPD: (A-B)/(average of A and B). The average RPD for many duplicate pairs can be used as a surrogate for the expected precision component of uncertainty of a sample. Often the RPD will vary with the amount of analyte, lessening as the amount of analyte increases. If such variation is >100% over the analytical range, then estimating an average uncertainty might be misleading, and, therefore, any reported uncertainties should take analyte load into account. To properly estimate precision using duplicate analyses, a proper number of QC data points (typically 20-30) should be considered.

4.3.3 Published Methods: For certain analyses, published methods, having published, well-characterized uncertainties, are available. For example, NIOSH 7400, a commonly used method for PCM fiber counting, has formulae for calculating 95% confidence limits. Use of these published uncertainties
may be used in lieu of laboratory-derived uncertainties provided the laboratory is following the published method without modification.

4.3.4 Sampling Uncertainty: Uncertainty due to sampling should be added to yield a total uncertainty for an analysis if the laboratory performs the actual sampling process. If the laboratory does not perform sampling as part of the analysis, then estimating uncertainty due to sampling errors should not be included in the overall analytical uncertainty. There is no simple way to do this, short of taking duplicate field samples.

4.3.5 Analyst Uncertainty vs. Overall Lab Uncertainty: For most laboratory quality assurance programs, QC data used to assess both analyst and laboratory precision and bias are maintained. For some methods (e.g., NIOSH 7400), it is a requirement to maintain separate QC databases for each analyst. However, for the purposes of reporting and of satisfying AIHA-LAP, LLC policies on measurement uncertainty, bias and precision data for all analysts in the laboratory shall be utilized to estimate the overall laboratory uncertainty of measurement.

4.3.6 Reporting: Where necessary for the interpretation of the test results, the estimate of uncertainty shall be reported to the client (ISO 17025:2005, 5.10.3.1c). When using laboratory QC data to estimate measurement uncertainty, uncertainty should be reported as bias and precision. Bias may be determined using LCS % yield data. For example, an analysis having an average LCS yield equal to 107% has a bias of +7%. Precision may be determined using relative percent difference (RPD) as determined from duplicate analyses or from % yield of LCS measurements. For example, precision, represented by the 95% confidence interval for duplicates (or the range in which the result should fall 95% of the time) can be estimated to be ±2 times the RPD. The LCS 95% confidence interval is equal to ±2 times the standard deviation of the % yield. When using the mathematical approach to the step-by-step estimation of measurement uncertainty, a single, overall ± value should be reported.

4.4 Taking A Type B Approach

4.4.1 Some procedures are best handled by step-by-step uncertainty calculations. For example, a nuisance dust method involves the subtraction of a filter weight from a (sample+filter) weight; the result is divided by an air sample volume. In this case, there are no “dust on filter” standards to be run to determine precision and bias. As a substitute, precision could be estimated for each of the weighing steps (perhaps obtained from the dispersion of repetitive measurements of a blank filter); another precision could be estimated for the pump volume (if the laboratory is performing the
actual sampling process), and the overall precision would then be calculated using the root-mean-square sum. A step-by-step uncertainty calculation would also be useful for analytical procedures that are used so infrequently that there is insufficient data to determine uncertainty by other means.

4.5 Calculating Measurement of Uncertainty

4.5.1 The following method is used by ATS to calculate Measurement of Uncertainty.

4.5.2 Uncertainty Calculator

4.5.2.1 ATS Laboratory Operations management or Quality Assurance uses “Uncertainty Calculator 3.2” software to determine the level of uncertainty (see www.eCalibration.com). The Uncertainty Calculator 3.2 (UnCal3.2), a Windows 2000 application, is a "FREEWARE" software program developed by Christopher Grachanen of Hewlett-Packard.

4.5.2.2 UnCal3.2 addresses uncertainties for commonly made measurements in a simple, straightforward manner; congruent with the basic guidelines contained within measurement uncertainty publications such as ISO "Guide to the Expression of Uncertainty in Measurement", 1993, NIST Technical Note 1297, etc.

4.5.3 The Measurement of Uncertainty data shall be traceability to the item (i.e. P/N, process, etc.) where uncertainty is being calculated and shall be approved and dated by Laboratory Operations management or Quality Assurance. See Figure 1.1 – Example of Measurement of Uncertainty for an example on how to calculate and document Measurement of Uncertainty information.

4.6 Measurement of Uncertainty Records Retention and Use

4.6.1 Laboratory Operations management and/or Quality Assurance maintain records of Measurement of Uncertainty as required by the Customer order. In addition, where deemed necessary by Laboratory Operations management and/or Quality Assurance, Measurement of Uncertainty documentation is approved and dated by the person creating and approving the information. Measurement of Uncertainty documentation is considered Quality Records and shall be identified and controlled per ATS-QAP-1004, Quality Records.

4.7 Training

4.7.1 Quality Assurance and Laboratory management personnel shall be trained in accordance with this procedure per ATS-HRP-1001, Training and Certification.
5.0 QUALITY ASSURANCE

5.1 Audits
5.1.1 QA shall audit this process as scheduled per ATS-QAP-1004, Internal Audits.
Figure 1 – Example of Measurement of Uncertainty

Uncertainty Calculator 3.2” software