ASCLD/LAB-*International*

ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty – ANNEX A

Details on the NIST 8 Step Process

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ASCLD/LAB customers should use this document in conjunction with AL-PD-3051 and AL-PD-3055 for conformance with ASCLD/LAB Measurement Uncertainty policy requirements.

NOTE AL-PD-3008 *Estimating Uncertainty of Measurement Policy* and AL-PD-3033 *Updated Approach to Uncertainty of Measurement Requirements are* withdrawn, effective August 5, 2011, and should no longer be used for conformance with ASCLD/LAB measurement uncertainty requirements.

Document History / AL-PD-3056

Date	Version	Description of Activity or Revision	Approved By	Effective Date
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Purpose

To provide guidance to laboratories that must achieve compliance with the ASCLD/LAB Policy on Measurement Uncertainty[1] as they prepare for and maintain ASCLD/LAB-*International* accreditation.

Scope

This guidance document will cover:

- Details on the NIST 8 Step Process for the estimation of measurement uncertainty
    - This framework can be applied to those tests and calibrations where by policy ASCLD/LAB-*International* has required that the uncertainty be estimated (as demonstrated by the examples provided). The process can also be applied for other tests where statute or customer request requires a laboratory to estimate the uncertainty of a test result.

This guidance document is intended to be used by:

- Testing and Calibration laboratories either currently accredited or seeking accreditation by the ASCLD/LAB-*International* programs,
- Technical assessors for the ASCLD/LAB-*International* programs, and
- Users of ASCLD/LAB-*International* accredited laboratory services.

For ease of use:

❖ Denotes a definition

➢ Denotes a requirement from the cited accreditation requirement document. Accreditation documents include: ISO/IEC 17025:2005,[2] ASCLD/LAB-*International* Supplemental Requirements for both Testing[3] and Calibration[4] programs, ASCLD/LAB Policy on Measurement Uncertainty[1] and ASCLD/LAB Board Interpretations[5]

✓ Denotes a mathematical formula

Introducing NIST's Eight-Step Process for Estimating Uncertainty

The steps in the process are:

- Step 1 • *Specify the Measurement Process*
- Step 2 • *Identify uncertainty sources*
- Step 3 • *Quantify uncertainty sources*
- Step 4 • *Convert factors to standard uncertainties*
- Step 5 • *Calculate combined standard uncertainty*
- Step 6 • *Expand the uncertainty by coverage factor (k)*
- Step 7 • *Evaluate the expanded uncertainty*
- Step 8 • *Report the uncertainty*

The remainder of this document will go through the NIST 8 Step process explaining each step.

Discipline specific examples of estimating the uncertainty of a test or calibration are provided in Annexes B-E. For ease in future document control maintenance, each annex will be issued as a separate document.

Annex B - Testing - Drug Chemistry – Currently under development
Annex C - Testing - Firearms - Currently under development
Annex D - Testing - Toxicology - Currently under development
Annex E - Calibration - Toxicology - Breath Alcohol - Currently under development

1. *Specify the measurement process*

State the measurand - what is being measured and how. If possible, do this both as a written expression and as a mathematical expression showing the measurement result and the parameters that it depends on.

Being specific is important. Performing the same measurement with different measuring devices or parameters may result in a different estimation of uncertainty.

Expect to come back to Step 1 a number of times throughout the 8 Step process. As you identify uncertainty sources, it may mean revising both your written and mathematical expression. The final description of the process may in fact be a combination of a number of measurement processes. It is easy to see how a mathematical model can become complex and might never be fully delineated.

2. *Identify uncertainty sources*

- The goal of this step is to comply with ISO/IEC 17025:2005, Clause 5.4.6.3 which states "when estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account …"

ISO/IEC 17025:2005, Note 1 of section 5.4.6.2 provides additional guidance when it states: "The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

- the requirements of the test method;
- the requirement of the customer;
- the existence of narrow limits on which decisions on conformity to a specification are based."

In the foreword to the second edition of the EURACHEM/CITAC Guide: Quantifying Uncertainty in Analytical Measurement,[6] the author's advice is that "the evaluation of uncertainty requires the analyst [however named] to look closely at all the possible sources of uncertainty. However, although a detailed study of this kind may require a considerable effort, it is essential that the effort expended should not be disproportionate. In practice, a preliminary study will quickly identify the most significant sources of uncertainty … and the value obtained for the combined uncertainty is almost entirely controlled by the major contributions."

In general, if a mathematical expression has been determined for Step 1, then all parameters in the expression will have one or more components that contribute to the uncertainty.

The *Guide to the expression of uncertainty in measurement* (GUM)[7] advises that it may be useful to consider a test or calibration method as a number of discrete processes and to estimate the uncertainty for each process. This is especially useful if the process is performed in many different test methods.

The key is to realize that you will probably not identify "all" uncertainty components, but you are required to take into account all contributions which are of importance. "Importance" may not be able to be determined until later in the process; therefore, in Step 2, more components may need to be considered and evaluated.

Is there any guidance on where to start to identify uncertainty components?

Yes. Many references on measurement uncertainty also provide a list of possible sources that they recommend be considered. Three references are provided below to allow you to see both the similarities and the differences. The laboratory personnel most familiar with the test or calibration method are generally those who can most easily and completely identify the potential contributions to uncertainty.

The GUM[7] in section 3.3.2 includes:

a. Incomplete definition of the measurand;
b. Imperfect realization of the definition of the measurand;
c. Non-representative sampling – the sample measured may not represent the defined measurand;
d. Inadequate knowledge of the effects of environmental conditions on the measurement or imperfect measurement of environmental conditions;
e. Personal bias in reading analog instruments;
f. Finite instrument resolution or discrimination threshold;

g. Inexact values of measurement standards and reference materials;
h. Inexact values of constants and other parameters obtained from external sources and use in the data-reduction algorithm;
i. Approximations and assumptions incorporated in the measurement method and procedure; and
j. Variations in repeated observations of the measurand under apparently identical conditions. (may be impacted by a-i)

ILAC[8] lists the following Factors Contributing to Uncertainty of Measurement:

Consideration should be given to the different factors which may contribute to the overall uncertainty of a measurement (not all are relevant in all cases). Some examples:

1. definition of the measurand
2. sampling
3. transportation, storage and handling of samples
4. preparation of samples
5. environmental and measurement conditions
6. the personnel carrying out the tests
7. variations in the test procedure
8. the measuring instruments
9. calibration standards or reference materials
10. software and/or, in general, methods associated with the measurement
11. uncertainty arising from correction of the measurement results for systematic effects

A Beginner's Guide to Uncertainty of Measurement[9] lists:

- The measuring instrument
- The item being measured
- The measurement process
- 'Imported' uncertainties
- Operator skill
- Sampling issues
- The environment

Is there a required format for the list of components to uncertainty?

No. You can use any mechanism that works for your laboratory as long as the records are reviewable and maintained. The mechanism can be as simple as a list, a "fishbone" diagram, a "budget" or another choice of the laboratory. The ASCLD/LAB Annexes provide different examples.

An Ishikawa diagram, also referred to as a fishbone diagram or a cause-and-effect diagram is a causal diagram that identifies potential factors causing an overall effect.

Two examples and a blank fishbone diagram are available for your use on the ASCLD/LAB website at www.ascld-lab.org (Choose "Forms" from the main menu). The downloadable diagram can be edited to include more or less "bones" and to reflect the contributions to uncertainty in your process. The "bones" can be typical basic categories in all methods, steps in a specific test or calibration method, or parameters in the mathematical equation stated in Step 1.

A spreadsheet program may be used for the entire 8 Step process. An EXCEL file is available for your use on the ASCLD/LAB website at www.ascld-lab.org (Choose "Forms" from the main menu). The components can be entered into the "budget" directly or after they have been gathered using any other format. Use of the sample EXCEL spreadsheet is not required for accreditation. Components to uncertainty may be gathered using any other format.

Sample of EXCEL spreadsheet:

Item	Uncertainty Component
1	
2	
3	
4	

- Per ISO/IEC 17025:2005, Clause 5.4.7.1, each laboratory will need to ensure that functions of the EXCEL spreadsheet work properly after downloading from the ASCLD/LAB website. Records of this performance verification will need to be maintained and be available for review.

3. <u>Quantify uncertainty sources</u>

In Step 3, depending on the test or calibration method, there may be a blending of the approaches to estimating measurement uncertainty as described in the *ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty - Overview*.[10]

Review the list of uncertainty sources created in Step 2. Which of the components listed, can be quantified by existing data that can be evaluated statistically?

By existing data, do you mean "quality control" data, "method validation" data, or data from "measurement assurance programs"?

Yes. For a new method, the laboratory will have method validation precision data that can be used to quantify uncertainty contributions. Importantly, this is true only if the method validation was structured appropriately to be representative of samples that will be tested or items that will be calibrated. As a part of method validation, it will be necessary to investigate if precision is constant over the range of the method (e.g. extraction efficiency over the expected concentration range, detector response over concentrations, etc.). Method validation should address the impact of the sample matrix, or matrices, on method performance. Once completed, method validation has potentially included the evaluation of a number of significant factors that could contribute to a method's uncertainty.

Once a method has been validated, ISO/IEC 17025:2005 in Clause 5.9.1 states "the laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken…"[2] Quality control data has the potential to provide much of the needed information for estimating uncertainty. Quality control is one aspect of Measurement Assurance.

- Measurement Assurance - Practices put in place to monitor a testing or calibration process and/or the reference standards or reference materials used in a process.

The goal of Measurement Assurance is to mimic the test or calibration method. Depending on how well the quality control sample(s) or the measurement assurance sample(s) or check standard(s) mimic the actual test or calibration process will determine how many of the uncertainty components listed in Step 2 will be covered by this precision data.

The Annexes to this document give examples of measurement assurance processes that mimic the test or calibration process.

The GUM has classified uncertainty components that can be evaluated statistically through a series of observations as *Type A.*

- *Type A* evaluation (of uncertainty) - a method of evaluation of uncertainty by the statistical analysis of a series of observations.[7]

What type of statistical evaluation is performed?

The statistic that is calculated is the standard deviation of the numerical results from the series of measurements gathered through method validation data or ongoing quality control or measurement assurance programs. Standard deviation is a mathematical expression of the variability (dispersion) in this repeatability[11] or reproducibility[12] data – an expression of the average distance from the mean. The mean is the sum of the measurement values divided by the number of measurements. The mean is the best available estimate of the value after a series of independent observations.

Arithmetic Mean:

✓ $$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

The distribution, the spread of a set of data, can take many forms. If an infinite number of measurements were taken and available to calculate the mean, then the mean would approach the 'true value (symbolized as μ)' and the standard deviation would be σ. In practice, the mean and spread are calculated based on a smaller number of measurements and the mean is symbolized as $\bar{x}$ and the standard deviation as *s*.

Standard Deviation (Population):

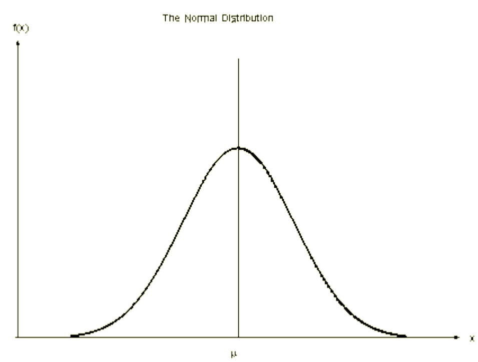
✓ $$\sigma = \sqrt{\frac{\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}{n}}$$

Standard Deviation (Sample):

✓ $$s = \sqrt{\frac{\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}{n-1}}$$

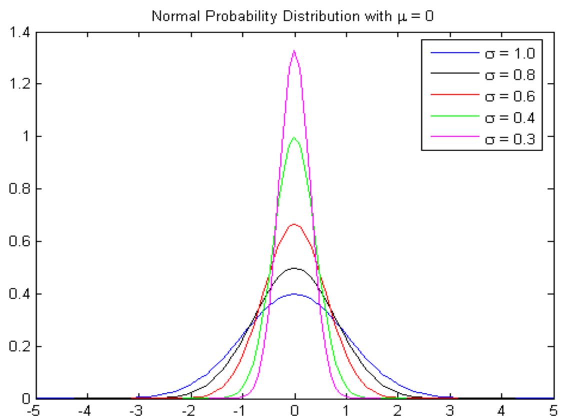
The standard deviation is the variability due to random effects from various sources that affect the measurement result. If the variation is due to random effects, with a large enough number of measurements the data will approximate a normal distribution. In a normal distribution (Figure 1), the majority of the measurement results center around the mean and the shape of the curve is symmetrical about the mean. It is less likely to observe data in the margins of the distribution.

Normal Distribution (Gaussian distribution)
Figure 1



Source: http://www.itl.nist.gov/div898/handbook/pmc/section5/pmc51.htm

Figure 2 shows five populations of data with the same mean but different standard deviations. Greater variability (the width of the bell shaped curve gets broader) translates to a larger standard deviation.

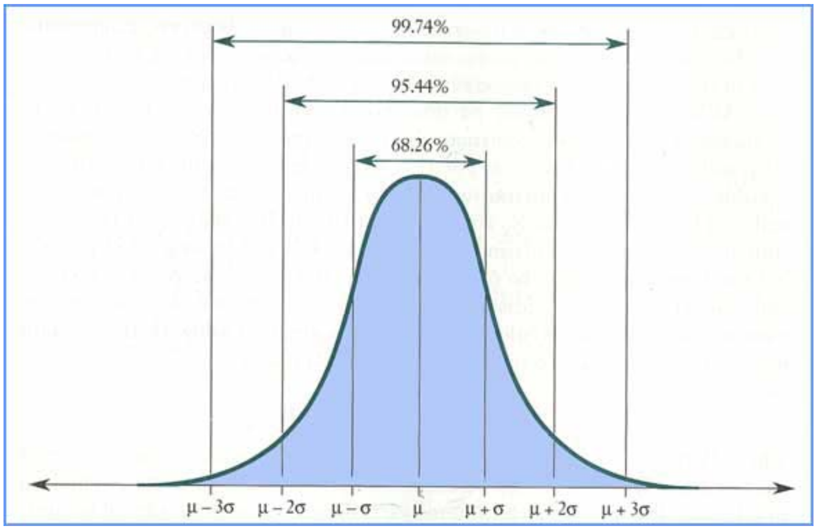


Normal Distribution (Gaussian distribution)

Figure 2: Five populations with the same mean but different standard deviations

If your data follows a normal distribution, then the probability that the value of your next measurement result of the same measurand, under similar operating conditions, would be within one standard deviation of the mean is approximately 68%. The probability that the value would be within 2 standard deviations is approximately 95% and the probability that the value would be within 3 standard deviations of the mean is approximately 99 %.(Figure 3)

Normal Distribution (Gaussian distribution)
Figure 3: Figure 1 graph with probabilities added



After identifying contributions to uncertainty for which the laboratory has experimental data that can be statistically evaluated (*Type A*), there are still components to uncertainty identified in Step 2 that have not been quantified or evaluated. What does the laboratory do with these other components?

Even when quality assurance or measurement assurance processes closely mimic the test or calibration method, there will be uncertainty attributed from other sources. These may include, but are not limited to, reference materials, reference standards or, at times, an environmental condition that is not covered by the *Type A* data.

How do I evaluate these types of uncertainty components?

The evaluation of the remaining uncertainty components is performed by a different classification of evaluation. The GUM has classified this type of evaluation as *Type B.*

- *Type B* evaluation (of uncertainty) - a method of evaluation of uncertainty by means other than the statistical analysis of a series of observations.

If the laboratory determines that an uncertainty component cannot be evaluated statistically, or that a statistical evaluation would be impractical, or that a statistical evaluation may be unnecessary, this demonstrates that the uncertainty can be categorized as a *Type B* uncertainty.

The GUM[7] states in Section 4.3.1, the pool of information to evaluate *Type B* components may include:

- experience with or general knowledge of the behavior and properties of relevant materials and instruments;
- manufacturer's specifications;
- data provided in calibration and other certificates;
- uncertainties assigned to reference data taken from handbooks.

The most common *Type B* uncertainty component is the data provided in calibration and other certificates.

All that is available is a manufacturer's specification. How is the standard deviation estimated from a specification?

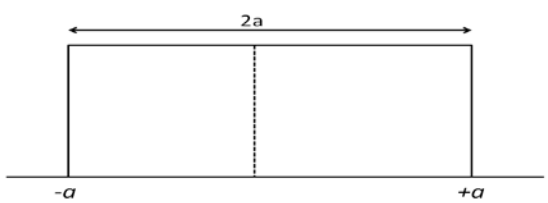
There are situations when it may only be possible to estimate the upper and lower limits of the variability in an uncertainty component. The amount of knowledge that is available is limited compared to the knowledge that is available for the normal distribution.

If:

- you know the limits
- you know that the probability that a value lies outside these limit boundaries is zero
- one value is just as likely as another between the limits (equal probability)

Then, a Rectangular Distribution is used to describe the probability.

Figure 4 - Rectangular Distribution (Uniform Distribution)



Upper limit = +*a*
Lower limit = -*a*
Possible range of values = *(-a)-(+a)*

For a rectangular distribution, the standard uncertainty is calculated by:

✓ standard uncertainty = $a/\sqrt{3} = 0.5774a$

If a measurement near the mean is more likely than one at the limit, is a rectangular distribution still used?

In this scenario, more knowledge is available.

If:

- you know that it is more realistic to expect values near the center than at the two limits
- you know that extreme values are less likely

Then, a Triangular Distribution is used to describe this probability

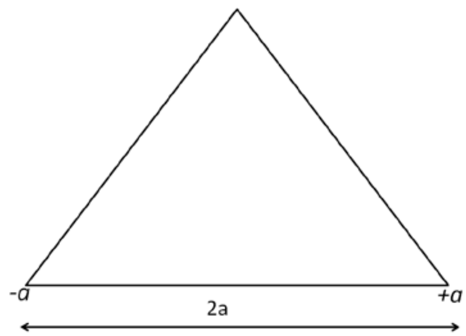


Figure 5 - Triangular Distribution

For a triangular distribution, the standard uncertainty is calculated by:

✓ standard uncertainty = $a/\sqrt{6} = 0.4082\ a$

If I don't know which distribution is appropriate for evaluating an uncertainty component, what is the most conservative way to evaluate a *Type B* uncertainty component?

When in doubt, use the Rectangular Distribution as it is the most conservative.

Are there other types of Distributions to describe the spread of a set of data?

Yes, but due to their infrequent application, they will not be covered here. It is the laboratory's responsibility to consult references or with a statistician if a different distribution is used as part of the evaluation.

Is a *Type A* evaluation method better or more accurate than a *Type B* evaluation method?

No. The two "types" of evaluation are for convenience only. Both are quantified by variances or standard deviations and can be equally reliable.[7]

Is there a relationship between random effects and systematic effects on uncertainty and *Type A* and *Type B*?

No. *Type A and Type B* are methods of evaluating the data. Per the GUM,[7] contributions to uncertainty are not classified as either random or systematic.

- Random effects – every time a measurement is taken under nominally the same conditions, random effects from various sources influence the result.[13]

  More measurements = better estimate
  = potentially less variability but not eliminated

- Systematic effects – an effect on a measurement result of a quantity that may not have been included in the original specification of the measurand but nevertheless influences the result.[13]

  More measurements = no additional information
  = introduces a bias or offset

  The source may be identified by using a different procedure or participating in inter-laboratory proficiency testing. If identified, it can be corrected.

4. Convert factors to standard uncertainties

A standard uncertainty is equivalent to one standard deviation.

It is imperative to know how the *Type B* uncertainty component has been expanded and reported so that the proper divisor is used to convert to a standard uncertainty which is equivalent to one standard deviation.

If, for example, a calibration certificate or a reference material certificate indicates approximately 95% confidence (k=2) in a reported measurement uncertainty, then that value must be divided by a factor of 2 to arrive at the standard uncertainty.

At this point in the process, it may be beneficial to review, and if necessary, re-evaluate uncertainty contributions with an unacceptably large standard uncertainty.

With uncertainty contributions coming from many different sources it seems likely that the units will not all be the same. Must the units on uncertainty components be the same?

Yes. In order to be combined, the next step in this 8 step process, all standard uncertainties must be expressed in the same units. It is most common to express standard uncertainties in the units of the measurement being made, but standard uncertainty may also be expressed as a percentage variation for each component.

5. Calculate combined standard uncertainty

Individual standard uncertainties quantified by *Type A* or *Type B* are now combined to calculate the combined standard uncertainty. The combined standard uncertainty is an estimated standard deviation and characterizes the dispersion of the values that could reasonably be attributed to the measurement result.

The combined standard uncertainty, denoted by $u_c(y)$, is the positive square root of the variance of all components combined. This formula is commonly called the Root Sum of the Squares or RSS.

- ✓ $$u_c(y) = \sqrt{\sum (c_i u_i)^2}$$
- ✓ $$u_c(y) = \sqrt{u_s^2 + s_p^2 + u_o^2 + u_1^2 + u_2^2 + ... + u_i^2}$$

This is the RSS formula when the measurement result is the sum of a series of components. The formula also assumes that the uncertainty components are independent or uncorrelated. If in fact the components are correlated, a large change in one component could cause a large change in another component. Performing the calculation as if the components are independent will result in an overestimation of the combined uncertainty value. Determining the degree of correlation and the correlation coefficient to be used in the RSS equation can be difficult. If a laboratory wants to pursue a combined uncertainty for correlated contributions, they should seek further guidance from published references or a statistician.

If the mathematical expression of the measurement from Step 1 identifies a different relationship such as subtraction, multiplication, division, squaring or square root, then the RSS formula must also reflect this and becomes more complicated.

6. Expand the uncertainty by coverage factor (k)

The combined standard uncertainty, calculated in Step 5, is an estimated standard deviation and characterizes the dispersion of the values that could reasonably be attributed to the measurement result.

Assuming a normal distribution, a standard uncertainty is equivalent to one standard deviation. Based on the normal distribution, if you were to perform the test or calibration again there is a 68% probability that the result would be your current result +/- this standard uncertainty.

Represented by the equation:

- ✓ y +/- u_c(y)

Graphically: Refer to Figure 3 and the area under the curve within 1 standard deviation of the mean.

Is probability the same as confidence interval, coverage interval, or coverage probability? Which is the better term to use?

The terms do not have the same meaning and should not be interchanged.

The terms that can be used and be correct for all estimations of uncertainty are "interval" and "coverage probability" or "level of confidence."

From the GUM[7] Section 6.2.2: The terms confidence interval and confidence level have specific definitions in statistics and are only applicable to the interval when certain conditions are met, including that all components of uncertainty that contribute to $u_c(y)$ be obtained from *Type A* evaluations. There may be scenarios in forensic science where after going through this 8 Step process and evaluating both *Type A and Type B* uncertainty components, where all contributions of importance to an estimation of uncertainty are *Type A,* making the use of the terms confidence interval and confidence level correct.

The ASCLD/LAB Policy on Measurement Uncertainty[1] requires that an expanded uncertainty with a coverage probability of not less than approximately 95% be reported. What does this mean?

A level of confidence of 68% of a standard uncertainty is not acceptable in forensic science.

Assuming a normal distribution, a laboratory can increase the interval covered and therefore, the level of confidence by multiplying the combined standard uncertainty by the coverage factor *(k)*. The value chosen for *k* is determined by the laboratory and the customer. Common values for *k* are 2 and 3.

k	Level of Confidence
2	95.45%
3	99.73%

Often these are generalized to say that a coverage factor of 2 is approximately 95% level of confidence and a coverage factor of 3 is approximately a 99% level of confidence.

Once a combined standard uncertainty is multiplied by a coverage factor *(k)*, it is now an expanded uncertainty and denoted by *U*

- ✓ Expanded uncertainty $U = k{*}u_c(y)$

Are you limited to using a coverage factor of 2 or 3?

No. A laboratory is responsible for making the decision on the coverage factor that will be used based on the use of the test or calibration result and the needs of their customer but the resulting coverage probability must not be less than approximately 95%. Other values for *k* assuming a normal distribution (an infinite number of observations for the *Type A* uncertainty component) can be found in Table 1.

Table 1: The *t*-distribution and degrees of freedom[7]

Value of $t_p(v)$ from the *t*-distribution for degrees of freedom *v* that defines an interval $-t_p(v)$ to $+t_p(v)$ that encompasses the fraction *p* of the distribution

Degrees of freedom *v*	Fraction *p* in percent					
	68.27[a)]	90	95	95.45 [a)]	99	99.73 [a)]
1	1.84	6.31	12.71	13.97	63.66	235.80
2	1.32	2.92	4.30	4.53	9.92	19.21
3	1.20	2.35	3.18	3.31	5.84	9.22
4	1.14	2.13	2.78	2.87	4.60	6.62
5	1.11	2.02	2.57	2.65	4.03	5.51
6	1.09	1.94	2.45	2.52	3.71	4.90
7	1.08	1.89	2.36	2.43	3.50	4.53
8	1.07	1.86	2.31	2.37	3.36	4.28
9	1.06	1.83	2.26	2.32	3.25	4.09
10	1.05	1.81	2.23	2.23	3.17	3.96
11	1.05	1.80	2.20	2.25	3.11	3.85
12	1.04	1.78	2.18	2.23	3.05	3.76
13	1.04	1.77	2.16	2.21	3.01	3.69
14	1.04	1.76	2.14	2.20	2.98	3.64
15	1.03	1.75	2.13	2.18	2.95	3.59
16	1.03	1.75	2.12	2.17	2.92	3.54
17	1.03	1.74	2.11	2.16	2.90	3.51
18	1.03	1.73	2.10	2.15	2.88	3.48
19	1.03	1.73	2.09	2.14	2.86	3.45
20	1.03	1.72	2.09	2.13	2.85	3.42
25	1.02	1.71	2.06	2.11	2.79	3.33
30	1.02	1.70	2.04	2.09	2.75	3.27
35	1.02	1.70	2.03	2.07	2.72	3.23
40	1.01	1.68	2.02	2.06	2.70	3.20
45	1.01	1.68	2.01	2.06	2.69	3.18
50	1.01	1.68	2.01	2.05	2.68	3.16
100	1.005	1.660	1.984	2.025	2.626	3.077
∞	1.000	1.645	1.960	2.000	2.576	3.000

a) For a quantity *z* described as a normal distribution with expectation μ_z and standard deviation □, the interval μ_z+/□ encompasses *p* = 68.27 percent, 95.45 percent and 99.73 percent of the distribution for *k* = 1, 2 and 3, respectively.

So far, the values of *k* have been based on a normal distribution assuming an infinite number of observations/measurements for the *Type A* component in the estimated uncertainty. If a laboratory is using method validation data, has limited or no historical data to use for this uncertainty component, is this still a valid approach for the laboratory to take?

The assumptions made are modified when a limited number of measurements are used for the *Type A* repeatability data or reproducibility data. The laboratory does need to take into account that they have less information about how the test or calibration method performs but the laboratory does know that the limited data available does indicate a normal distribution.

With limited data, the values used for k are found in Table 1 above. With limited data, instead of using the value of k found in the last line of the table with infinite observations, the laboratory must use the number of observations available minus one.

✓ Degrees of freedom = The number of observations minus 1
= n - 1

Example: A laboratory has determined in Step 3 that precision data obtained using a traceable reference material during method validation adequately mimics a portion of the test method and by calculating the standard deviation can be used to quantify a number of uncertainty components identified in Step 2.

The laboratory has 10 values to use in the calculation, not an infinite number.

Using the t-distribution or Student's T-table, Table 1, the value for a 95% level of confidence with 9 degrees of freedom is 2.26 compared to 1.96 with an infinite number of degrees of freedom.

U in this example = 2.26*$u_c(y)$

Are there other scenarios when using the values of 2 and 3 to approximate a 95% and 99% level of confidence should be reviewed?

If the distribution of the dominant component does not approximate a normal distribution, the expanded uncertainty will be conservative but it might be unrealistically large.[12]

Care must be taken if the measurement results lie over a range of values – some components within the uncertainty budget may have been assumed to be constant over the range, while others can be shown to be proportional to the measured value. In such circumstances it may be necessary to determine the uncertainty at the expected upper end and lower end of the range, and then interpolate the uncertainty value for a particular case. In complex situations, it may be necessary for the range of expected values to be divided into smaller ranges and the applicable uncertainty determined separately for each range.[12]

7. *Evaluate the expanded uncertainty*

Step 7 is the time to critically evaluate the estimation of uncertainty and to determine if it "makes sense" and is "reasonable."

- The laboratory may identify calculation errors in this Step.
- The laboratory must decide if the uncertainty results are appropriate for the test or calibration method. If the laboratory placed limits on the acceptable expanded uncertainty, does the calculated value meet the stated requirement?
- Does the expanded uncertainty meet the customer's needs?

If the laboratory determines that the expanded uncertainty is not acceptable, what is the next step?

If the laboratory determines that the expanded uncertainty is not acceptable, areas of method improvement (e.g., improved reference standard, improved measuring instrument, etc.) can be identified and evaluated for the impact that a change would have on the estimation of uncertainty using the information available from Steps 3 and 4.

Once changes to a test or calibration method have been validated, the appropriate edits to Steps 1-6 can be made and the estimation of uncertainty reevaluated.

What is the relationship between Proficiency Test Results and a laboratory's estimation of uncertainty for a given test or calibration method?

If the principles of traceability, and therefore the estimation of uncertainty, have been incorporated in the development of the proficiency test, then proficiency test results are used to support and demonstrate that your estimation of uncertainty is complete. Stating this concept another way, rigorous proficiency testing is one tool to evaluate your estimation of uncertainty for appropriateness.

8. *Report the uncertainty*

Now it is time to report your best estimate of the test or calibration result and the expanded uncertainty at your chosen level of confidence.

Specific reporting requirements are found in the ASCLD/LAB Policy on Measurement Uncertainty[1] and are repeated below for convenience. These requirements are based on ISO/IEC 17205:2005[2], the GUM[7] and ILAC Policy[14]

- The numerical value of the expanded uncertainty shall be reported to, at most, two significant digits.
    - The laboratory shall establish a procedure for the process of rounding reported uncertainty.
    - The usual rules for rounding of numbers must be used, subject to the guidance on rounding provided in Section 7 of the GUM, which recognizes the appropriateness of rounding uncertainties upwards rather than to the nearest digit, or subject to the guidance on rounding provided in ISO 80000-1:2009[15] or as stipulated by statute.
        - In some cases it may be necessary to retain additional digits to avoid round-off errors in subsequent calculations.

- The estimated measurement uncertainty, communicated as an expanded uncertainty, including the coverage factor and the coverage probability, must be in the test or calibration report or in an attachment to the report that is communicated to the customer.

  For certain testing applications, the laboratory may have an agreement with the appropriate legal or judicial customer(s) that the estimated uncertainty is not required in a test report unless the measurement result (considering the expanded uncertainty) falls within a certain range around a legal specification. Such an arrangement is acceptable to ASCLD/LAB as long as the agreement with the appropriate customer(s) is in writing; is readily available for review in the laboratory; and is scientifically/mathematically reasonable. The existence of such a written agreement to limit the testing laboratory's reporting of measurement uncertainty does not excuse the laboratory from estimating the measurement uncertainty of the test method.

- This measurement result shall include the measured quantity value, y, along with the associated expanded uncertainty, U, and this measurement result shall be reported as $y \pm U$ and be consistent with the units of y.

- The measurement uncertainty that the expanded uncertainty or the coverage interval are derived from shall always be a non-negative parameter characterizing the dispersion of the quantity/values being attributed to the measurand, and based on the information used.

- In some cases, a coverage interval may not be centered at the measured value y. The laboratory shall consult with a statistician to ascertain whether such asymmetric assessment is needed.

Is it correct to round numbers with each calculation or wait until the expanded uncertainty is calculated?

Rounding of values should be carried out only at the end of the calculation, to avoid rounding errors.

Is it acceptable to report the test or calibration and the associated expanded uncertainty in relationship to a statute limit?

A laboratory can include this additional information in the report but it must be additional information and not in place of that which is required by the Policy[1] and re-stated above in Step 8. If a tolerance or specification is used, the uncertainty must be taken into account with the measurement result.

NOTES

1 American Society of Crime Laboratory Directors / Laboratory Accreditation Board (ASCLD/LAB): ***ASCLD/LAB Policy on Measurement Uncertainty*** (AL-PD-3051) (Garner, North Carolina: ASCLD/LAB, 2011). Available at http://www.ascld-lab.org.

The effective date of AL-PD-3051 is July 1, 2012.

Additional information about ASCLD/LAB is available at www.ascld-lab.org.

2 International Organization for Standardization (ISO), *ISO/IEC 17025:2005 General requirements for the competence of testing and calibration laboratories* (Geneva, Switzerland: ISO, 2005). Available for purchase at http://www.iso.org/iso/iso_catalogue.htm or from other authorized distributors.

Additional information about ISO is available at http://www.iso.org/iso/home.html.

3 ASCLD/LAB, ***ASCLD/LAB-International Supplemental Requirements for the Accreditation of Forensic Science Testing Laboratories*** (AL-PD-3040) (Garner, North Carolina, 2011 Edition).

Acquisition and use of that document requires a User License Agreement with ASCLD/LAB and an ISO/IEC 17025 Certification Statement. Acquisition forms are available at http://www.ascld-lab.org/forms/forms_intl.html or by contacting ASCLD/LAB. Contact information for ASCLD/LAB is provided on page 2 of this document.

4 ASCLD/LAB, ***ASCLD/LAB-International Supplemental Requirements for the Accreditation of Breath Alcohol Calibration Laboratories***, 2007 Edition (AL-PD-3026) (Garner, North Carolina, 2007 Edition).

Acquisition and use of that document requires a User License Agreement with ASCLD/LAB and an ISO/IEC 17025 Certification Statement. Acquisition forms are available at http://www.ascld-lab.org/forms/form_intl_calibration.html or by contacting ASCLD/LAB. Contact information for ASCLD/LAB provided on page 2 of this document.

[5] ASCLD/LAB, ***ASCLD/LAB-International Program Applications, Guidance and Board Interpretations [for] Forensic Science Testing Laboratories*** (Web only access, available at http://www.ascld-lab.org/interpretations/applicationsintl_2011.html) and ***ASCLD/LAB-International Program Applications, Guidance and Board Interpretations [for] Breath Alcohol Calibration Laboratories*** (Web only access, available at http://www.ascld-lab.org/interpretations/applicationsbreath.html).

[6] A Focus for Analytical Chemistry in Europe (EURACHEM) and the Co-operation on International Traceability in Analytical Chemistry (CITAC), ***EURACHEM/CITAC Guide: Quantifying Uncertainty in Analytical Measurement***, *second edition*. *QUAM:2000 P1* (Prague: EURACHEM Secretariat, 2000). Available for download at http://www.eurachem.org/index.php/publications/guides/trc.

Additional information about EURACHEM is available at http://www.eurachem.org/index.php/euintro.
Additional information about CITAC is available at http://www.citac.cc/.

[7] Joint Committee for Guides in Metrology (JCGM), *Evaluation of measurement data – Guide to the expression of uncertainty in measurement* (GUM) (GUM 1995 with minor corrections). (Sèvres, France: International Bureau of Weights and Measures [BIPM]-JCGM 100], September 2008). Available at http://www.bipm.org/en/publications/guides/gum.html.

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[8] International Laboratory Accreditation Cooperation (ILAC), ***ILAC-G17:2002*** *Introducing the Concept of Uncertainty of Measurement in Testing in Association with the Application of the Standard ISO/IEC 17025* (ILAC: Rhodes, Australia, 2002). Available at
http://www.ilac.org/documents/ILAC_G17-2002_intro_the_concept_of_uncert_meas_with_17025.pdf

Additional information about ILAC is available at http://www.ilac.org.

[9] National Physical Laboratory (NPL), ***NPL No 11: Measurement Good Practice Guide – A Beginner's Guide to Uncertainty of Measurement***, *Issue 2* (Teddington/Middlesex, United Kingdom: NPL, 2001). Available for download at http://www.npl.co.uk/publications/uncertainty-guide/.

The National Physical Laboratory (NPL) is the UK's National Measurement Institute (NMI). Additional information about the NPL is available at http://www.npl.co.uk.

[10] ASCLD/LAB, ***ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty - Overview*** (AL-PD-3055) (Garner, North Carolina: ASCLD/LAB, 2011). Available at http://www.ascld-lab.org.

The effective date of AL-PD-3055 is July 1, 2012.

[11] VIM Definition - Repeatability: *"repeatability conditions of measurement – condition of measurement, out of a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time."*

All "VIM" definitions are available in:

Joint Committee for Guides in Metrology (JCGM), *International vocabulary of metrology – Basic and general concepts and associated terms* (VIM), 3rd ed. (Sèvres, France: International Bureau of Weights and Measures [BIPM]-JCGM 200, 2008). Available for download at http://www.bipm.org/en/publications/guides/vim.html.

Even though the electronic version of the 3rd edition of the VIM is available free of charge on the BIPM's website, copyright of that document is shared jointly by the JCGM member organizations (BIPM, IEC, IFCC, ILAC, ISO, IUPAC, and OIML).

12 VIM Definition - Reproducibility: *"reproducibility condition of measurement – condition of measurement, out of a set of conditions that includes different locations, operators, measuring systems, and replicate measurements on the same or similar objects."*

13 NPL, *NPL No 36 Measurement Good Practice Guide – Estimating Uncertainties in Testing* (Teddington/Middlesex, United Kingdom: NPL, 2003). Available for download at http://publications.npl.co.uk/.

14 ILAC, *ILAC-P14:12/2010 ILAC Policy for Uncertainty in Calibration* (ILAC: Rhodes, Australia, 2010). Available at http://www.ilac.org/documents/ILAC_P14_12_2010.pdf.

15 ISO, *ISO 80000: 2009 Quantities and Units; Part 1: General* (Geneva, Switzerland: ISO, 2009). Available for purchase at http://www.iso.org/iso/home.html or from other authorized distributors.