Analytical Measurement Uncertainty

APHL Quality Management System (QMS) Competency Guidelines

This course will help staff recognize what measurement uncertainty is and its importance in a laboratory committed to ensuring the consistent quality of the tests performed, the products created, the data generated, and the results reported.

Objectives:

1. Participant will understand what the term "measurement uncertainty" means
2. Participant will appreciate the need for measurement uncertainty when performing analysis and reporting results
3. Participant will review some basic statistics.
4. Participant will comprehend terms and calculations used in determining measurement uncertainty

Measurement uncertainty is the doubt that exists about the result of any measurement.

Public health laboratory competency guidelines:
General (GEN) domain

GEN 1.02. Mathematical and statistical concepts and practices
Applies fundamental mathematical and statistical concepts and practices in work area
Instructs others in fundamental mathematical and statistical concepts and practices
Ensures appropriate utilization of mathematical and statistical concepts and practices
Oversees the policies,* processes,* and procedures* regarding the use of mathematical and statistical concepts and practices

Chemistry (CHM) domain
CHM 1.03. Laboratory statistics
Explains statistical concepts and

To express uncertainty you need to know:

- "How big is the margin (possible range)?"

  Example: working in the linear portion of the calibration curve vs. working close to the detection limits.

- "How bad is the doubt?" (Do I trust this value?)

  Once you know the possible range, you can decide if you trust the value.

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practices
Collects data for statistical analyses
Evaluates summarized data for statistical analyses
Oversees the development and use of statistical programs

Introduction to Analytical Measurement of Uncertainty Dictionary of Terms

Accuracy - The ability of an instrument or testing process to repeat readings close to the true value of an analyte

Analyte - The substance which is being measured

Bias - Systematic error manifested as a consistent unidirectional deviation from the true value

Confidence Interval - Size of the range around a measurement that conveys the precision of the measurement

Confidence Level - The likelihood (expressed as a percentage) that the results of a test are real and repeatable, and not just random

Coverage Factor (k) - Number of standard deviations within which the confidence interval lies. Thus, in a confidence interval of two standard deviations, k=2 and the confidence level for a normal distribution is 95%.

Deviation - Unidirectional measure of the difference between an individual value and the mean for that set of values

Error - The difference between the measured value and the true value of the measurand

Expanded Uncertainty - A measure of uncertainty that defines an interval about the measurement result within which the value of the measurand can be confidently asserted to lie

Laboratory Control Sample (LCS) - Quality control specimen containing a known amount of analyte

Margin - The possible range of results

Mean - The average of a set of values

Measurand -The particular quantity of the substance being measured, including the unit of measure and the matrix, e.g., “mg of lead in a cubic meter of air”

Measurement - The comparison of an unknown quantity with a standard unit of measure

Measurement Uncertainty - The doubt that exists about the result of any measurement, expressed as a quantifiable number or range of numbers, based on what is known about the measurement system

Offset - The amount by which a measurement is affected due to some other factor or effect

Percent Recovery - The quantity of analyte recovered divided by the starting quantity of analyte, times 100 (This can also be expressed as a decimal quantity.)

Precision - The ability of an instrument to repeat the same, or nearly the same, readings for a given analyte (These readings are not necessarily accurate.)

Random Error - Unpredictable error caused by precision limitations of instruments or by operator interpretations (Random error is always present in a measurement.)

Range - The difference between the smallest and largest value in a set

Reference Method - A sampling and/or measurement method that has been officially specified by an organization as meeting its data quality requirements

SOP - Standard Operating Procedure- An administratively established protocol that is to be carried out in a particular operation, function, activity or situation

Spike - Solution prepared as a control by adding a known quantity of standard to a known volume of pre-analyzed sample

Square - The result obtained when any number is multiplied by itself

Square Root - The square root of a number is a number which multiplied by itself, gives you the original number

Standard Deviation - A mathematical formula used to calculate the average distance that values in a set differ from the set's mean. Standard deviation indicates the spread of the values

Standard Uncertainty - Uncertainty expressed as a standard deviation

Statistical Predictability - The ability to indicate the range of accuracy in advance, based on observation, experience or scientific reason and stated in quantifiable terms

Statistics - Branch of mathematics dealing with the collection, analysis, interpretation, and presentation of masses of numerical data

Systematic Error - A consistent deviation in the results of sampling and/or analytical processes from the expected or known value (such error is caused by human and methodological bias)

Target Value - Value sought as the result when analyzing spiked, laboratory control, or other samples containing known amounts of the analyte

Tolerance - The allowable deviation from a standard

True Value - Actual amount of the analyte in a sample

Uncertainty Factor - Value used to compensate for a deficiency in knowledge concerning the accuracy of test result

Uncertainty - Quantification of the doubt about a measurement

Calculating Standard Deviation

Find the sum of all the values in the set.
Find the average by dividing the sum by the number of values in the set.
Subtract the mean from each value in the set to find the deviation of each value from the mean.
This may be a positive or negative number.
Square each deviation.
Find the sum of the squares of the deviations.
Divide the sum of the squares by one less than the number of values in the set.
Find the square root of this number.
The result is the value of one standard deviation for this set of numbers.

Determining Measurement Uncertainty Using Combined and Expanded Uncertainty

1. Determine Percent Recovery for the Procedure
   a. Run at least ten tests for the analyte on a laboratory control sample (LCS) and LCS duplicate.

(LCS contains a known amount of the analyte; i.e., the true value)

   b. Divide each result by the true value and multiply by 100 to find the ***percent recovery*** for that result.
   c. Add the percent recoveries for the 20 values and divide by 20 to find the average, or mean, of these values. This is the ***mean percent recovery***.
   d. Calculate the ***standard deviation*** of the percent recovery values for the twenty laboratory control samples. If one standard deviation is, for example, 1.56%, then two standard deviations = 3.12%.
   e. If 95% of your results are within two standard deviations of the mean, the ***confidence level (k)*** is expressed as *k* = 2.
   f. Divide the SD percentage (3.12% in this case) by 100 to determine the ***Uncertainty Factor***. (In this example, the Uncertainty Factor for the test is 0.0312.

Calculate Uncertainty for the Test Result

   a. Multiply the value of the test result by the Uncertainty Factor.
      - Example: The test result for lead in water is 1.10 µg/dL
        The Uncertainty Factor is 0.0312
        The analytical uncertainty for this result is 1.10 x 0.0312 = 0.0343
   b. Express the result as "1.10 µg of lead per dL of water, with an ***analytical uncertainty*** of plus or minus 0.0343 µg/dL at the 95% confidence level, where *k*=2."

Determine Bias for the Procedure

   a. Subtract the ***mean percent recovery*** (from step 1c above) from 100. This is the ***bias.***

        - If the mean percent recovery is greater than 100, the bias will be positive;
        - If the mean percent recovery is less than 100, the bias will be negative. (Example: The bias is positive, 1.54 %.)

    b. Convert the bias to a decimal by dividing by 100. (Ex. 1.54%= 0.0154)
    c. Multiply the sample result (1.10 µg/dL in this example) by the bias expressed as a decimal (0.0154).
    d. Calculate the probable bias for this result by multiplying the result by the bias expressed as a decimal. 1.10 x 0.0154 = 0.0169 µg/dL. The probable bias for this result is 0.0169 µg/dL.

Reporting the Test Result with Measurement Uncertainty and Probable Bias

1.10 µg of lead per dL of water, with an analytical uncertainty of ± 0.0343 µg/dL at the 95% confidence level (where *k* = 2) with a probable bias of 0.0169 µg/dL.

Determining Measurement Uncertainty Using Combined and Expanded Uncertainty When a Test Sample Cannot be Spiked or Run in Duplicate

1. Determine the Mean Percent Recovery for the Procedure
    a. Run duplicate tests for the analyte on a laboratory control sample (LCS). We will use ten runs in this example. Check your SOP for the appropriate number (LCS contains a known amount of the analyte; i.e., the true value).
    b. Divide each of the ten test results by the true value and multiply by 100 to find the percent recovery for that result.
    c. Add the percent recoveries for the 10 values and divide by 10 to find the average, or mean, of these values. This is the mean percent recovery.

Calculate the Uncertainty Factor for the Procedure

    d. Calculate the standard deviation of the percent recovery values for the ten laboratory control samples. If one standard deviation is, for example, 1.56%, then two standard deviations = 3.12%. If 95% of your results are within two standard deviations of the mean, the confidence level (k) is expressed as k = 2.
    e. In step 2.a., the value of 2 standard deviations is a percentage (3.12%). Change the percentage to a decimal by dividing by 100. This is the Uncertainty Factor, where k = 2. (In this example, the Uncertainty Factor for the test is 0.0312.)

Calculate the Analytical Uncertainty for a Test Result

    f. Multiply the value of the test result by the Uncertainty Factor (from step 2.b. above)
        i. Example: The test result for lead in water is 1.10 µg/dL
        ii. The Uncertainty Factor is 0.0312