10.0 Uncertainty of Measurement for Volatiles Analysis

10.1 BACKGROUND

Any measurement, no matter how carefully obtained, should not be considered as the true value for the measurement. Whenever any quantitative measurement is performed, the value obtained is only an approximation of the true value.[1] According to JCGM 200:2008, the International vocabulary of metrology – Basic and general concepts and associated terms (VIM),[3] measurement uncertainty is defined as "*A non-negative parameter associated with the result of a measurement/quantity value (number and measurement unit used together to express the magnitude of a quantity) that characterizes the dispersion of quantity values that could reasonably be attributed to the measurand (quantity intended to be measured).*" ISO/IEC 17025:2005 clause 5.4.6.2 requires that we make a reasonable estimation of uncertainty that 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.[2] Clause 5.4.6.2, NOTE 1 goes on to state that the degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as the existence of narrow limits on which decisions on conformity to a specification is based.[2] Paragraph 5.10.3.1 states that when applicable, the test report should include a statement on the estimated uncertainty of measurement.[2] For our purposes, it is applicable due to the uncertainty affecting the application of the test results which are compliant to a specification limit. In the analysis of forensic specimens, we do not know the true value for the specimen; hence this information is not the error associated with the analysis. Rather, it is a range of values likely to be encountered during the measurement process.[7] This information is crucial to the legal system because it impacts if and how an individual will be charged with an offense such as DUI.[4,5]

10.2 SCOPE

This analytical method will be applied to analytical methods which report quantitative results. This approach to uncertainty uses the standard deviation of matrix matched controls and other known sources of uncertainty. A 99% confidence interval will be created by three standard deviations of data collected during the process. To properly represent the uncertainty, this data will be expressed as the Uncertainty Of Measurement on the analysis report. Authentication of ethanol containing blood controls is described in Volatiles Analysis Analytical Method 2.0

10.3 EQUIPMENT

Reference analytical methods listed under section 10.6.

10.4 REAGENTS

Reference analytical methods listed under section 10.6.

10.5 QUALITY ASSURANCE MATERIAL

Reference analytical methods listed under section 10.6.

10.6 REPORTING OF QUANTITATIVE ETHANOL RESULTS

10.6.1 Analytical Methods

1.0 Analysis of Volatiles by GC-HS

10.6.2 Determination of Confidence Interval

10.6.2.1 Blood control values obtained during the process are used to establish the UM based on the standard deviation of data as well as incorporating other known sources of uncertainty into the uncertainty budget.

10.6.2.2 Three standard deviations will be calculated for a 99% confidence interval.

10.6.2.3 The mean value as determined by the above analytical method will be reported along with a ± UM.

10.7 MONITORING AND UPDATING THE UNCERTAINTY OF MEASUREMENT

10.6.1 Monitoring

10.7.1.1 The UM for the analysis process will be monitored per the AM 1.0 through the use of certified reference materials. The reference materials shall be run with each batch of samples being analyzed and entered into a spreadsheet.

10.7.1.2 The results of the reference standards shall be reviewed annually. The review will consist of the Discipline Leader checking the results for each lab and issuing a memo to summarize the results of the reference standard analysis.

Note: The memo shall consist of the following summaries at a minimum: Overall system standard deviation, overall system standard error, each regional laboratories overall standard deviation, and a quarterly breakdown of the standard deviation and standard error for each lab to identify trends.

10.7.2 Updating the UM for the system

10.7.2.1 Should a new GC/HS instrument be put into service within the laboratory, the measurement process for the affected laboratory shall be repeated using an available lot of blood control QC samples in the same prescribed manner as the original determination.

10.7.2.2 Should a new analyst be approved to perform volatile substance analysis, the measurement process will be performed by that analyst using an available lot of blood control QC samples in the same prescribed manner as the original determination.

10.7.2.3 Every three years, the process with be reproduced using a different lot of blood QC samples throughout the entire system. Each analyst that is approved and performing volatile substance analysis on blood and other fluids shall produce data used for the determination of the UM for the system. This process shall be substantially the same as the previous determinations and analyses.

10.7.2.4 When the UM is updated, reports that are in progress shall report the UM numbers in accordance with the version that is in effect during the ANALYSIS date found in the case notes, and not with the report issue date.

10.8 REFERENCES AND RECOMMENDED READING

10.8.1 Huber, L., Validation and Qualification in Analytical Laboratories, pp. 146 - 150, Interpharm/CRC, 19910.

10.8.2 International Organization of Standardization (ISO) / International Electrochemical Commission (IEC), *General requirements for the competence of testing and calibration laboratories*, 2005. (ISO/IEC 17025:2005)

10.8.3 Joint Committee for Guides in Metrology (JCGM), *International Vocabulary of Basic and General Terms in Metrology (VIM)*, 2008. (JCGM 200: 2008)

10.8.4 Idaho Code §18-8004. Persons under the influence of alcohol, drugs or any other intoxicating substances.

10.8.5 Idaho Code §18-8004C. Excessive Alcohol Concentration – Penalties.

10.8.6 ISO/IEC 17025:2005: Section 5.4.6: Estimation of Uncertainty of Measurement Workshop, Presented by J.P. Bono and E.A. Mishalanie, AAFS 61st Annual Meeting, Denver, Colorado, 20010.

10.8.7 Mason, F., Uncertain About Uncertainty, Quality Digest, Inside Metrology Column, 06-12-2008.

Revision History

10.0 Uncertainty of Measurement for Volatiles Analysis

Revision #	Issue Date	Revisions
0	09-07-2009	Original issue Analytical Methods 4.1 and 4.2 addressed for quantitative ethanol results.
0	1-20-2011	Initial version as 10.0, split from toxicology discipline analytical methods. Formerly Toxicology AM 5.1.13.
1	4-23-2012	Changes made to reflect correct references to other AM's.
2	4-15-2013	Changes made to sections 10.1, 10.2, 10.6.2.1, 10.6.2.2, 10.6.2.3.
3	1/16/2014	Changes were made to section 10.7 and section 10.8 was added. Section 10.7 became section 10.8 and section 10.7 is new text with this revision.