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Standard for Breath Alcohol Measuring Instrument Calibration

410 North 21st Street, Colorado Springs, CO 80904, www.aafs.org/academy-standards-board.

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410 North 21st Street
Colorado Springs, CO 80904

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DRAFT

Foreword

The field of Toxicology includes breath alcohol testing. Breath alcohol testing is widely used to determine the alcohol (ethanol) content of an individual. Breath Alcohol Programs vary widely in their requirements (statutory, regulatory, programmatic), resources, and oversight/administration. Historically, the National Safety Council (Alcohol, Drugs and Impairment Division, previously known as Committee on Alcohol and Other Drugs) has outlined initial minimum guidelines for various components of breath alcohol testing. This document provides a model for Breath Alcohol Programs to follow in developing and validating a calibration method. Additional ~~program~~Breath Alcohol Program components will be included in other documents.

This document was prepared and finalized as a standard by the Toxicology Consensus Body of the ASB. The draft was developed by the Toxicology Subcommittee of the Organization of Scientific Area Committees for Forensic Science to provide minimum standards of practice for the calibration method development, the validation of such a method and the calibration of breath alcohol instruments used for forensic purposes. By following ~~these standards~~this standard, a Breath Alcohol Program will be able to objectively show that a breath alcohol instrument is capable of successfully performing at its intended level of accuracy and precision using the validated calibration method. This standard cannot replace the knowledge, skills, and abilities acquired through education, training, and experience.

The American Academy of Forensic Sciences established the Academy Standards Board (ASB) in 2015 with a vision of safeguarding Justice, Integrity and Fairness through Consensus Based American National Standards. To that end, the ASB develops consensus based forensic standards within a framework accredited by the American National Standards Institute (ANSI), and provides training to support those standards. ASB values integrity, scientific rigor, openness, due process, collaboration, excellence, diversity and inclusion. ASB is dedicated to developing and making freely accessible the highest quality documentary forensic science consensus Standards, Guidelines, Best Practice Recommendations, and Technical Reports in a wide range of forensic science disciplines as a service to forensic practitioners and the legal system.

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Keywords: *breath alcohol, calibration, methodology, validation.*

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Standard for Breath Alcohol Measuring Instrument Calibration

1 Scope

This standard is applicable to the calibration of breath alcohol measuring instruments for evidentiary purposes. These minimum requirements are included for (1) the development and validation of calibration methods on these instruments; (2) evaluation of performance following adjustments and calibrations; and (3) monitoring the validity of the calibrations performed. This standard is not intended to cover preliminary (non-evidentiary) testing, ignition interlock, or federally-regulated testing.

2 Normative References

For dated references, only the cited edition applies. For undated references, the latest edition including amendments applies.

ANSI/ASB Standard 017, *Standard Practices for Measurement Traceability in Forensic Toxicology*.[a]

3 Terms and Definitions

For purposes of this document, the following terms and definitions apply.

3.1
accuracy
Closeness of agreement between a test result or measurement result and the true value. 1) In practice, the accepted reference value is substituted for the true value. 2) The term “accuracy,” when applied to a set of test or measurement results, involves a combination of random components and a common systematic error or bias component. 3) Accuracy refers to a combination of trueness and precision.[b]

3.2
adjustment
A set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of the quantity to be measured.[c]

~~3.1~~3.3
bias
An estimate of systematic measurement error, calculated as the difference between the mean of several measurements under identical conditions~~,~~ to a known “true” value. It is often reported as a percent difference.

---

[a] Available from: https://www.aafs.org/asb-standard/standard-practices-measurement-traceability-forensic-toxicology

[b] ISO 3534-2:2006

[c] Joint Committee for Guides in Metrology (JCGM), *International vocabulary of metrology - Basic and general concepts and associated terms (VIM)* (Sèvres, France: International Bureau of Weights and Measures [BIPM]-JCGM 200) available from: https://www.bipm.org/en/publications/guides.

~~3.2~~3.4

Breath Alcohol Program

An organizational structure including policies, procedures, responsibilities, and resources necessary for implementing core breath alcohol activities. ~~The Breath Alcohol Program includes~~Activities include, but may not be limited to, requirements or specifications for reference material, training of operators, maintenance and calibration of instrumentation, the evidential breath alcohol test sequence, and record retention.

~~3.3~~3.5

calibration

Operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication.~~1~~[d]

~~3.4~~3.6

calibrator

~~A reference~~Measurement standard ~~or reference material of known concentration~~ used ~~to standardize or calibrate an instrument or laboratory procedure.~~in calibration.[e]

~~3.5~~3.7

carryover

The appearance of unintended analyte signal in samples after the analysis of a positive sample.

~~3.6~~3.8

computer system

A system containing one or more components and elements such as computers (hardware), associated software, and data (e.g., software, firmware, hardware, configuration files).[f]

~~3.7~~3.9

data

A quantitative or qualitative representation that is observed, measured, collected, or gathered that characterizes some static or dynamic attribute of the physical world or the use of it by individuals or groups of people and that is suitable for communication, interpretation, or processing by humans or machines.[g]

~~3.8~~3.10

Lower Limit of Quantitation

LLOQ

An estimate of the lowest concentration of an analyte in a sample that can be reliably measured with acceptable bias and precision.

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[d] Joint Committee for Guides in Metrology (JCGM), *International vocabulary of metrology - Basic and general concepts and associated terms (VIM)* (Sèvres, France: International Bureau of Weights and Measures [BIPM]-JCGM 200) available from: https://www.bipm.org/en/publications/guides.

[e] ISO Guide 30:2015

[f] ISO/IEC 25024:2015(en) *Systems and software engineering — Systems and software Quality Requirements and Evaluation (SQuaRE) — Measurement of data quality* available from: www.webstore.ansi.org.

[g] ASTM E867-06 (2020) *Standard Terminology Relating to Vehicle Pavement Systems* available from: www.webstore.ansi.org.

~~3.9~~3.11

masking

Automated function where results above or below a specified threshold are reported with a defined result. For example, the instrument may report “ethanol below administrative threshold” or “0.000 g/210 L ethanol” for a response below 0.005 g/210 L. Another example may be reporting “Results greater than 0.400 g/210 L” or “Over Range” for a response above 0.400 g/210 L.

~~3.10~~3.12

measured quantity value

A quantity value representing a measurement result.[h]

~~3.11~~3.13

measurement assurance

The process of monitoring the validity of the calibrations performed.

~~3.12~~3.14

nominal quantity value

A rounded or approximate value of a characterizing quantity of a measuring instrument or measuring system that provides guidance for its appropriate use.~~[5]~~[i]

~~3.13~~3.15

precision

The measure of the closeness of agreement between a series of measurements obtained from multiple samplings of the same homogenous sample. It is expressed numerically as the coefficient of variation (% CV).

~~3.14~~3.16

reference material

A material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process.[j]

~~3.15~~3.17

reporting range

~~A range of concentrations that can be reliably measured by an analytical procedure.~~

A range of concentrations that can be reliably measured by an analytical procedure that will be reported per the Program’s specifications (e.g., what appears on a Subject Test Result certificate).

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[h] Joint Committee for Guides in Metrology (JCGM), *International vocabulary of metrology - Basic and general concepts and associated terms (VIM)* (Sèvres, France: International Bureau of Weights and Measures [BIPM]-JCGM 200) available at https://www.bipm.org/en/publications/guides.

[i] Joint Committee for Guides in Metrology (JCGM), *International vocabulary of metrology - Basic and general concepts and associated terms (VIM)* (Sèvres, France: International Bureau of Weights and Measures [BIPM]-JCGM 200) available from: https://www.bipm.org/en/publications/guides.

[j] Joint Committee for Guides in Metrology (JCGM), *International vocabulary of metrology - Basic and general concepts and associated terms (VIM)* (Sèvres, France: International Bureau of Weights and Measures [BIPM]-JCGM 200) available at https://www.bipm.org/en/publications/guides.

3.18

stability

An analyte's resistance to chemical change in a matrix under specific conditions for given time intervals.

~~3.16~~3.19

Upper Limit of Quantitation

ULOQ

The highest concentration of an analyte in a sample that can be reliably measured with acceptable bias and precision.

4 Development of a Calibration Method

4.1 The calibration method shall be a defined procedure with specified components and pre-defined acceptance criteria. Breath Alcohol Programs (hereafter called Program) providing calibration services for evidentiary breath alcohol instruments are often subject to legal, programmatic, legal precedent, and/or accreditation requirements. Consequently, the Program may need to perform various experiments to develop and optimize a method that meets Program requirements and the requirements of this standard.

4.2 Prior to performing calibration method validation experiments, all elements of the calibration method shall be determined and defined~~.~~ (including bias and precision, see Section 5 for elements of the calibration method). The Program may choose to revise computer system parameters during the method development phase to optimize components such as method, instrumentation, or user interface.

4.3 Accuracy (bias and precision) is integral to calibration methods. The needs of the end user should be balanced against instrument capabilities. Bias and precision results obtained during method optimization should be used to determine the method's acceptance criteria.

4.4 The largest calculated within-run and between-run % CV for each concentration shall be used to determine precision acceptability.

4.5 During method development, the LLOQ and ULOQ shall be determined. ~~The range of ethanol concentrations of interest (e.g., statutory concentrations, administrative concentrations) shall be considered when determining the appropriate proposed limits.~~Ethanol concentrations of particular interest (e.g., those that trigger legal consequences) shall be considered in determining these values. LLOQ and ULOQ concentrations may be administratively determined (e.g., choose the concentrations based upon readily available reference material) but shall be supported with experimental data (i.e., data from your instrument showing acceptable bias and precision at the administratively set limits of quantitation). After the method development stage, LLOQ and ULOQ effectively become the lowest and highest concentration calibrators (see also 5.1.d).2). The following is one of multiple paths that can be used to experimentally determine these values.

~~**4.5.1** The following is one of multiple paths that can be used to determine these values.~~

~~4.5.2~~4.5.1 Determine ULOQ using reference material with established traceability.

a) Analyze ~~a minimum of~~ three ethanol concentrations around the proposed upper limit.

b) Each concentration should be analyzed three consecutive times (replicates).

c) The concentrations should bracket the proposed upper limit (e.g., 0.~~38~~380, 0.400, 0.420 for a proposed 0.400 ULOQ).

~~d)~~ The ~~highest data point where acceptable~~ series of concentrations should meet bias and precision criteria ~~are met is the experimentally determined ULOQ.~~

~~e)~~d) ~~If the series of 3 concentrations does not meet the bias and precision~~(section 5 provides minimum acceptance criteria~~, a series of 3~~ for the calibration method). A Program can analyze consecutively lower concentrations ~~shall be repeated~~ until acceptable bias and precision is achieved.

e) If the series of concentrations meet bias and precision criteria, analyzing consecutively higher concentrations can be performed to experimentally determine the ULOQ. Alternatively, an administrative ULOQ can be chosen from acceptable concentrations and referenced in the validation plan.

~~4.5.3~~4.5.2 Determine LLOQ using reference material with established traceability.

a) Analyze ~~a minimum of~~ three ethanol concentrations around the proposed lower limit.

b) Each concentration should be analyzed three consecutive times (replicates).

c) The concentrations should bracket the proposed lower limit (e.g., 0.015, 0.020, 0.025 for a proposed 0.020 LLOQ).

~~d)~~ The ~~lowest data point where acceptable~~ series of concentrations should meet bias and precision criteria ~~are met is the experimentally determined LLOQ.~~

~~e)~~d) ~~If the series of 3 concentrations does not meet the bias and precision~~(section 5 provides minimum acceptance criteria~~, a series of 3~~ for the calibration method). A Program can analyze consecutively higher concentrations ~~shall be repeated~~ until acceptable bias and precision is achieved.

e) If ~~masking is to be utilized during testing, this function shall be removed during performance~~the series of ~~the calibration method. The specific~~ concentrations ~~at which point masking occurs~~meet bias and precision criteria, analyzing consecutively lower concentrations can be performed to experimentally determine the LLOQ. Alternatively, an administrative LLOQ can be chosen from acceptable concentrations and referenced in the validation plan.

4.6 Linearity is integral to a calibration method and shall be ~~determined~~evaluated during ~~the~~ method development ~~and optimization phase~~. The statistical method to calculate the 'curve' used to provide results is typically preset by the manufacturer and not editable by the Program. It can however be impacted by the choice of concentration(s) used to adjust the instrument. The following process shall be used to determine the linearity.

4.6.1 ~~The~~Adjust the instrument using the number and concentration levels documented chosen to produce a linear response. Performing calibration doesn't alter the calibration model (curve), it is the adjustment process that directly impacts the linearity.

4.6.2 Following the adjustment method, a minimum of five non-zero concentrations spanning the reporting range shall be analyzed on the instrument. The reference material shall have established traceability.

4.6.3 There shall be a minimum of ten (10) replicates for each concentration.

4.6.4 If compressed gas is utilized, the results shall be normalized to standard atmospheric pressure prior to continuing with calculations.

4.6.5 The results for all concentrations (n > 50) shall be used to plot a graph using a software program. For example using a graphing calculator or Microsoft Excel to capture the data points {concentration (x), results (y)}.

4.6.6 From the graph, determine the best fit line (e.g., trendline function using Microsoft Excel), calculate the slope, and intercept.

4.6.7 Determine the correlation (fit for the line) by calculating the Coefficient of Determination (r2).

4.6.8 Calculate the bias for each concentration.

4.6.9 The measurement technology (detector) shall be considered linear if all bias results are < 0.005 g/210L or 5% (whichever is greater) and the r2 value is > 0.990.

4.6.10 Should the results not indicate linearity, steps shall be taken to perform calibration within a linear range (e.g., reduce the reporting range, alter the adjustment calibrator(s)) or ensure sufficient calibrator concentrations to ensure bias and precision criteria is met across the reporting range.

4.6.11 The adjustment method shall be documented and approved. It may be approved in conjunction with approving the calibration method.

4.7 Programs should consider the impact of masking. While masking is likely needed during subject testing (i.e, not reporting outside the ULOQ and LLOQ), masking outside the ULOQ and LLOQ could result in unsuccessful calibrations due to the instrument's nonquantitative results (e.g., Sample Over Range).

~~4.7~~4.8 Programs should consider usage, storage, and transportation requirements for reference material (section 6.3.5 provides additional information that would be used during method validation). Requirements may need to be modified to eliminate limitations. In cases when it is not possible to modify, limitations shall be documented in the calibration method.

~~4.8~~4.9 The final adjustment and calibration ~~method~~methods, however determined, shall be validated prior to use on instruments for evidential purposes. Annex A provides an example of a method development and optimization plan with example results.

5 Elements of a Calibration Method

The calibration method shall include, but may not be limited to, the following:

a) method name;

b) instrument make and model;

NOTE This document does not address instrument specifications.

c) computer system ~~parameters~~:

  1) analysis and the subsequent information obtained (e.g., diagnostics, response curves), calculated (e.g., results), retained, and reported is controlled by a computer system,

  2) components of the computer system ~~parameters~~ which shall be uniquely identified and versioned if applicable;

d) reference material:

  1) matrix:

    i) aqueous (wet), and/or

    ii) compressed (dry) gas;

  2) concentrations;

  ~~3) the reporting range is dependent upon the calibrator concentrations;~~

    — The lowest reported concentration shall be equal to or greater than the lowest non-zero calibrator utilized in the calibration method.

    — The highest reported concentration shall be equal to or lower than the highest calibrator utilized in the calibration method.

  ~~4)~~3) a minimum of ~~6~~four non-zero calibrators if measurement technology is linear or six non-zero concentrations shall be used as calibrators~~;~~ if non-linear (see section 4.6);

  ~~5)~~4) the concentrations shall span the calibration range;

  ~~6)~~5) number of replicates per concentration shall be a minimum of 5.

e) limits of quantitation (See 4.~~4~~5);

f) reporting range;

  — The calibration method shall define the reporting range. The calibration method shall ensure acceptable results across the entire ~~reporting~~calibration range. The reporting range may be administratively set but shall be within the validated ~~reporting~~calibration range. Legally mandated ethanol concentrations should guide the decision regarding the reporting range.

g) calibration sequence;

— The calibration sequence is comprised of the number of replicates, number of concentrations, and the order of operations performed during the calibration method. Programs may use an automated process for their calibration sequence.

h) ~~traceability;~~the method shall require the use of traceable reference material;

— Traceability shall be established according to ANSI/ASB Standard 017, *Standard Practices for Measurement Traceability in Forensic Toxicology*.

i) process to evaluate the calibration method's measurement uncertainty;

~~j) —~~ acceptance criteria~~;~~

j) ~~Criteria~~ shall be defined for a successful calibration~~. The~~;

k) acceptance criteria shall include a maximum acceptable bias of ±5% or 0.005 g/210 L, (whichever is greater) at each concentration;

l) precision acceptance criteria (% CV) shall not exceed ± 10% at each concentration;

~~k)~~m) the method shall also specify steps to be taken when the calibration ~~does~~results do not meet the ~~parameters~~acceptance criteria for a successful calibration.

6 Validation of a Calibration Method

6.1 When to Validate the Calibration Method

6.1.1 Calibration methods shall be validated when it is necessary to verify a method's performance parameters are acceptable for use.

Common examples requiring validation include the following:

a) existing calibration method that does not ~~currently~~ meet the current requirements outlined in this document;

b) modifications of an established calibration method to improve performance or extend its use beyond that for which it was originally validated (e.g., expanded reporting range);

c) new calibration method;

d) to demonstrate equivalent uniformity between an established method/instrument and a new method/instrument;

~~e) and modification to the computer system parameter(s). Not all modifications impact the calibration method. The Program shall perform acceptance testing on revised system parameter(s) to determine impact (if any) on the calibration method.~~

6.1.2 The parameters to be evaluated for validation of calibration methods depend upon the circumstances in which the method is to be used. Likewise, it is recognized that after validation has occurred, methods may be revised. The extent and frequency of revalidation of previously validated methods depend upon the nature of the intended changes or Program policy. See Section 7 for further information on revalidation of methods.

6.1.3 A Program using a method that was validated prior to the promulgation of this document shall demonstrate and document that the method meets the Program's needs (i.e., fit-for-purpose) under this standard. The method is likely to have sufficient historical calibration data that can be used to address a number of the required validation parameters. In the absence of sufficient data to fulfill these minimum standards, appropriate studies shall be conducted to ensure conformance with this document.

6.2 Establishing a Validation Plan

6.2.1 The Program is responsible for ensuring the calibration method is satisfactorily validated. Annex B provides examples of a calibration method validation plan.

6.2.2 A validation plan shall be in place prior to starting any validation experiments.

NOTE The validation plan is typically separate from a ~~Programs~~Program's standard operating procedure (SOP) for method validation. It provides direction for the specific experiments that will be performed and acceptance criteria for each parameter.

6.2.3 The plan shall specify that the ~~method~~adjustment and calibration methods (including computer system parameters) shall remain the same throughout validation.

6.2.4 The plan shall include the parameters specified in Section 4.

6.2.5 The plan shall specify acceptance criteria for each parameter necessary to approve the calibration method.

6.2.6 The plan shall ~~document~~specify the ~~validation and method requirements~~range of environmental conditions that ~~may allow it to~~will be ~~fit-for-purpose.~~validated. For example, a Program may require the calibration method to ~~appropriately perform at~~be performed and then validated between a specific temperature range of −5°C to 40°C (~~in a~~ non-controlled environment).

6.2.7 The plan shall specify the number of instruments to be used for validation experiments. A minimum of ~~1~~one instrument shall have all validation experiments performed in totality.

NOTE Although a minimum of ~~1~~one instrument is specified for method validation, it is expected that all instruments would undergo performance verification ~~and calibration~~ prior to evidential use~~.~~ (that topic is outside the scope of this document).

6.2.8 The validation shall be conducted using the same calibration conditions and parameters as specified in the final method.

6.2.9 The validation plan shall require successful completion of all validation experiments on the same ~~method~~adjustment and calibration methods prior to approval.

6.2.10 Programs ~~should~~shall consider uncertainty ~~estimation~~evaluation in developing the validation plan.

6.3 Validation Parameters

6.3.1 General

All validation experiments outlined below shall be conducted in similar environments and conditions to those in which a calibration may take place. Validation experiments shall be conducted on different days. Programs should use different analysts when practicable. The instrument shall be adjusted following the method determined during method development (see 4.6.11). The instrument shall be successfully calibrated (i.e., meet all calibration method acceptance criteria) prior to performing validation experiments.

6.3.2 Bias and Precision

6.3.2.1 General

Accuracy (bias and precision) shall be calculated over multiple days to ensure variability has been evaluated. Accuracy shall be measured using reference material with established traceability that is different than that used for the calibration. A minimum of three concentrations (low, medium, high) shall be evaluated over six ~~different ~~days~~, with~~. For each concentration, there shall be a minimum of three replicates. A minimum of ~~each concentration~~one run (accuracy experiment) shall be conducted each day. A Program may conduct more than one run per day; the days do not need to be consecutive; and the Program may choose to conduct testing over more than 6 days.

Low concentrations shall be no more than approximately 3 times the lowest calibrator (LLOQ) utilized in the calibration method and high concentrations (ULOQ) shall be ~~no less than~~within approximately 80% of the highest calibrator utilized in the calibration method. Medium concentrations shall be near the midpoint of the low and high concentrations.

When using compressed gas reference material, the results shall be normalized for standard atmospheric pressure before bias and precision evaluation. See Annex C for example calculations.

6.3.2.2 Bias Determination and Acceptance

The bias shall be calculated for each concentration. See Annex D for example validation data. The Program may utilize the nominal quantity value or measured quantity value of the reference material to calculate the bias, however, it shall be specified in the validation plan. See Annex C for guidance related to nominal quantity and measured quantity values.

For calculating bias utilizing the nominal quantity value, use the following formula:

$$\boldsymbol{Bias}\ (\%)\ \boldsymbol{at\ Concentration_x} = \left[\frac{\text{Grand Mean of Calculated Concentration}_\text{x} - \text{Nominal Concentration}_\text{x}}{\text{Nominal Concentration}_\text{x}}\right] \times 100 \qquad (1)$$

For calculating bias utilizing the measured quantity value, use the following formula:

$$\boldsymbol{Bias}\ (\%)\ \boldsymbol{at\ Concentration_x} = \left[\frac{\text{Grand Mean of Calculated Concentration}_\text{x} - \text{Measured Concentration}_\text{x}}{\text{Measured Concentration}_\text{x}}\right] \times 100 \qquad (2)$$

The maximum acceptable bias is ±5% or 0.005 g/210 L, (whichever is greater) at each concentration.

6.3.2.3 Precision

Precision is expressed as the coefficient of variation (% CV). The mean and standard deviation (std dev) of the response is calculated for each concentration to determine the % CV.

$$\% \, CV = \frac{std \; dev}{mean \; response} \times 100 \tag{3}$$

The % CV shall not exceed ± 10%. The largest calculated within-run and between-run % CV for each concentration shall not exceed ± 10%.

6.3.2.4 Within-Run Precision Calculations

Within-run ~~precisions are~~precision shall be calculated for each concentration separately for each of the ~~six~~ runs. Within-run precision ~~may~~shall be calculated using the data from each run's triplicate analyses at each concentration as:

$$Within - Run \, \% \, CV = \frac{std \; dev \; of \; a \; single \; run \; of \; samples}{mean \; calculated \; value \; of \; a \; single \; run \; of \; samples} \times 100 \tag{4}$$

6.3.2.5 Between-Run Precision Calculations

Between-run precision ~~is~~shall be calculated for each concentration over the ~~six~~ runs (minimum n=18/concentration). This ~~may~~shall be done by using the combined data from all replicates of each concentration as:

$$Between - Run \, \% \, CV = \frac{std \; dev \; of \; all \; observations \; for \; each \; concentration}{grand \; mean \; for \; each \; concentration} \times 100 \tag{5}$$

6.3.3 Limits of Quantitation

~~6.3.3.1~~ The ULOQ and LLOQ shall be experimentally determined ~~within~~or administratively set during the method development process.

~~Programs may administratively set a ULOQ and LLOQ; however~~ Per clause 6.3.1, the ~~Program shall demonstrate that~~instrument is calibrated prior to conducting validation experiments. A successful calibration ensures the ULOQ (lowest calibrator) and LLOQ ~~levels chosen,~~(highest calibrator) meet ~~acceptable~~the specified bias and precision criteria.

6.3.4 Carryover

Carryover shall be evaluated as part of method validation. To evaluate carryover as part of method validation, ethanol negative sample(s) (e.g., human breath, compressed gas, and/or aqueous solution) shall be analyzed ~~immediately after~~following the highest concentration of the reporting range.

The highest calibrator concentration at which no ethanol carryover is observed (above the method's ~~LLOD~~LLOQ) in the ethanol negative sample is determined to be the concentration at which the method is free from carryover. Determining the concentration at which no carryover exists shall be confirmed by repeating the determination twice (i.e., a total of 3 repeated tests).

The calibration method (or computer parameters) should be modified to remove any carryover. In cases when it is not possible to eliminate the carryover, the Program shall address in writing how carryover shall be managed.

6.3.5 Reference Material

6.3.5.1 Reference material used to calibrate the instrument(s) may be subject to variables including storage, repeated usage, and transportation conditions and handling. The performance of reference material shall be evaluated with respect to the calibration method.

6.3.5.2 Experiments shall be designed and conducted to address situations typically encountered with reference material used to calibrate a breath alcohol instrument. Annex E and Annex F provide examples of validation plans related to reference material used in the calibration method. Characteristics that shall be evaluated include~~.~~ the following.

a) Reference material shall conform to requirements published in ANSI/ASB Standard 017, *Standard Practices for Measurement Traceability in Forensic Toxicology*.

b) Shelf life of reference material if it may be used past the stated date of best use (different terms may be used by manufacturers such as recommended retest date, reanalysis date, and expiration date).

c) Stability of reference material over time and/or usage as permitted in the calibration method. Examples of possible calibration method or Program choices.

   1) The calibration method allows for repeated use of a single aqueous simulator solution over X calendar days or X replicates.

   2) The Program allows for a reference material to be repeatedly opened to produce simulator solution which can impact the concentration of ethanol.

d) Stability of reference material when used, stored, and/or transported outside of manufacturer's recommended conditions (see Annex E for an example validation plan).

e) Capability of compressed gas cylinder at lower pressure when used in the calibration method (e.g., valve limitations, compressed gas cylinder performance). See Annex F for an example validation plan.

6.3.5.3 Any limitations related to reference material identified during the validation experiments shall be addressed.

6.3.6 Environmental Conditions

6.3.6.1 The performance of the calibration method shall be assessed under similar environmental conditions ~~that are~~ typically encountered in the laboratory and/or field (as applicable). If environmental conditions exist that potentially cause an effect on the instrument operation and calibration method, then those conditions shall be evaluated. The Program shall define these conditions in the validation plan. The Program shall calibrate the instrument(s) under the defined conditions and then assess the applicable validation parameters. Annex G provides examples for validation plans for environmental conditions. Environmental conditions may include, but are not limited to:

a) atmospheric pressure;

b) humidity;

c) radiofrequency interference (RFI);

d) temperature.

6.3.6.2 Alternatively, parameters may be addressed through other means (e.g., quality assurance practices, published references) and documented within the validation plan.

7 Revalidation of Previously Validated Methods

7.1 Modifications to a validated method require evaluation to confirm that the changes do not have an adverse effect on the method's performance. The decision regarding which performance characteristics require additional validation is based on logical consideration of the specific parameters likely to be affected by the change(s). These changes may include, but are not limited to the following:

a) analytical changes to the computer system;

b) expanded reporting range;

c) instrumentation (e.g., different model); and

d) location of calibration method performance (e.g., initiating field calibrations).

NOTE For example, an analytical change in the computer system may affect linearity, precision, or bias. Consideration should be given to conducting parallel studies using a previously validated method and the modified method to evaluate the effects of the change(s). The goal is to demonstrate any impact the change(s) have on the performance of the previously validated procedure.

7.2 New models/manufacturers of instrumentation shall require a complete validation study.

7.3 Laboratories using methods that were validated prior to the promulgation of this document shall demonstrate and document that those methods are fit-for-purpose under this standard. These methods are likely to have sufficient historical calibration data that can be used to address a number of the required validation parameters. In the absence of sufficient data to fulfill these minimum standards, appropriate studies shall be conducted to ensure conformance with this document.

8 Validation Documentation Requirements

8.1 Record keeping is an essential part of a ~~Programs~~Program's operating procedures and is a key component of method validation. The following validation records shall be retained, organized, and available for review:

a) name of the adjustment and calibration ~~method~~methods;

b) validation plan;

c) original observations, data and calculations shall be recorded at the time they are made; observations, data and calculations shall include at a minimum:

  1) date, identity of personnel and breath alcohol instrument(s),

  2) source of reference material (e.g., lot number, manufacturer, dates), and

  3) description of all the parameters evaluated, results and calculations~~. If~~; if any of the parameters were not evaluated, then the reason shall be documented and justified;

d) references;

e) conclusion/summary;

f) date of method approval~~;~~(s);

g) and the name and title of person(s) approving the adjustment and calibration method(s) for use.

8.2 Records shall be retained ~~according to the Program's record retention policy or~~ for a minimum of 10 years after the calibration method is no longer used.

9 Adjustment

9.1 An evidential breath alcohol instrument may be adjusted for various reasons (e.g., out of tolerance results, mandated occurrences, preventive reasons). The process to perform the adjustment shall be documented in an approved method.

9.2 Adjustment shall be conducted using reference material ~~with established traceability~~ that is different than that used for the calibration~~.~~ (e.g., different lot, matrix, vendor). If an adjustment is performed without the use of reference material (e.g., auto-cal, internal standard), the Breath Alcohol Program shall still ensure that the requirements in this section are met.

9.3 Instrument performance shall be evaluated before an adjustment. Instrument performance shall be evaluated using reference material with established traceability. Reference material used for the calibration may also be used for the performance evaluation.

9.4 To evaluate instrument performance, a minimum of three concentrations (low, medium, high) shall be evaluated. Low concentrations shall be no more than approximately 3 times the lowest calibrator utilized in the calibration method and high concentrations shall be ~~no less than~~within approximately 80% of the highest calibrator utilized in the calibration method. Medium concentrations shall be near the midpoint of the low and high concentrations. Alternatively, the Program may perform their calibration method prior to adjustment to evaluate instrument performance.

9.5 When using compressed gas reference material, the results shall be normalized for standard atmospheric pressure before bias evaluation.

9.6 If the performance evaluation results do not meet the acceptable bias criteria specified in the calibration method, prior instrument results should be evaluated.

9.7 The results of adjustment(s) shall be documented.

9.8 An adjustment shall be followed by a calibration before the instrument is used for evidential Breath Alcohol testing.

9.9 There are limited scenarios where the performance evaluation is not possible. For example:

a) ~~The~~the instrument is unable to run tests until repairs are performed; or

b) ~~The~~the instrument software is preventing continued testing without first performing an adjustment.

9.10 Instances where a performance evaluation is not performed shall be documented and justified.

10 Performance

10.1 When to Calibrate

The calibration method shall have a specified interval not to exceed 12 months from the date of calibration~~.~~ (analytical work performed). Instruments may be calibrated more frequently. Additionally, instruments used for evidential purposes shall be calibrated under the following circumstances:

a) after a change to the computer system ~~parameter~~ that impacts the analytical results;

b) after any system component that impacts an analytical result is replaced or repaired;

c) after an adjustment (see Section 9);

d) when acceptance criteria are not successfully met (e.g., failed calibration); and

e) prior to being used the first time for evidential testing.

10.2 Measurement Assurance

Programs shall have a scheduled process for monitoring the validity of their calibration activities. Techniques to evaluate calibration results may include, but are not limited to, the following: participation in an inter-laboratory proficiency program; routine calibration checks on evidential instruments; and calibration verification using reference material after a calibration has been performed.

10.3 Unacceptable Calibration Results

The Program shall define the action(s) to be taken when the calibration results do not meet the defined acceptance parameters. This response may be a subsequent attempt at calibration, troubleshooting, and/or repair.

10.4 Documentation Requirements

Programs shall retain all records related to calibration, adjustment, and instrument maintenance according to the ~~Programs~~Program's retention schedule.

11 Elements of a Calibration Certificate

A calibration certificate (however named) shall be created for calibrations that meet the acceptance criteria.

Certificates shall be written clearly and shall include at a minimum the following:

a) a description of the calibration instrument (e.g., instrument make/model);

b) an unambiguous identification of the calibration instrument (e.g., serial number);

c) date of calibration;

d) date of issue of the Certificate;

e) the calibration interval (e.g., "The calibration of this instrument is valid for 12 months from the date of calibration");

f) calibration results, with units of measurement and the associated uncertainty of measurement;

g) the name and address of the Program;

h) the name and address where the calibration was performed (if different than the Program's address);

i) the name of the calibration method (e.g., title of standard operating procedure);

j) the name of the calibration certificate author (e.g., the individual taking responsibility for the calibration certificate); ~~and~~

k) ~~if certificates are multi-page,~~ all pages shall contain the page number; and

~~k)~~l) a clear identification of the ~~final page count~~end of the certificate shall be present (e.g., "1 of 3"; "End of Certificate" footer on the last page).

12 Amended Certificates

When modifications to the original calibration certificate are necessary, an amended certificate shall clearly indicate the amendment. If a new certificate is issued, the certificate shall reference the original certificate.

Annex A

(informative)

Example of a Method Development Plan with Selected Results[k]

A.1 Method Development and Optimization Plan

A.1.1 Objective

Develop and optimize a calibration method for Instrument ABC that provides the:

a) required minimum bias and precision;

b) necessary analytical capabilities;

c) determination of the lower and upper limits of quantitation;

d) evaluation of linearity; and

~~d)~~e) determination of the reporting range.

A.1.2 Development Protocol

Instrument –	Instrument ABC equipped with Anytown P.D. computer system (software/firmware) version 05.3
Equipment –	Company XYZ simulators (temperature metrologically traceable to SI units) External barometer (metrologically traceable to SI units by an accredited ISO/IEC 17025 calibration laboratory)
Standards –	For calibration: compressed gas (EtOH in N_2) (metrologically traceable to SI units) For verification of the calibration method during method development: aqueous reference material (EtOH in H_2O) (metrologically traceable to SI units)
Location(s) –	Anytown P.D. Crime Laboratory 1234 Main Street, Anytown, USA
Maximum bias/precision –	Bias = 5% or 0.005g/210L (whichever is greater) Precision = The % CV shall not exceed +/-10%

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[k] This is an example of a mock Method Development/Optimization Plan and subsequent results, for illustrative purposes only. Actual *concentrations of interest and instrument capability may vary.*

Concentrations of interest –	0.02 g/210 L of breath = underage DUI 0.04 g/210 L of breath = mass transit operators 0.08 g/210 L of breath = rebuttable presumption of DUI 0.15 g/210 L of breath = enhanced penalty for DUI 0.20 g/210 L of breath = enhanced penalty for DUI
Records –	The names and dates of those involved with executing this plan will be recorded with the resultant data.
Analytical process –	The following process will be repeated, as necessary, to achieve the stated objective.

a) Determine the Lower Limit of Quantitation (LLOQ).

A minimum of three samples of decreasing ethanol concentrations shall be analyzed three consecutive times (replicates). The lowest statutorily mandated ethanol level will be considered when determining the appropriate ethanol concentrations. The lowest concentration that is capable of achieving acceptable bias and precision criteria is considered the estimated LLOQ.

b) Determine the Upper Limit of Quantitation (ULOQ).

A minimum of three samples of increasing ethanol concentrations shall be analyzed three consecutive times (replicates). The highest statutorily mandated ethanol level will be considered when determining the appropriate ethanol concentrations. The highest concentration that is capable of achieving acceptable bias and precision criteria is considered the estimated ULOQ.

c) Determine the reporting range.

The reporting range will be determined using data developed from Step a) and Step b) above. The concentrations of interest shall also be considered. Once the reporting range has been determined, no quantitative values will be reported above the upper limit of quantitation. A final computer system version will be programmed for reporting; this final software will go through the method validation testing process.

d) Develop appropriate calibration method(s).

Evaluate linearity of instrument. Specify the instrument parameters, concentrations, acceptance parameters, and number of replicates used for each calibration method used during the method development phase.

e) Evaluate the data obtained from Step d) above to determine if further optimization is desired.

f) At the conclusion of Steps a) through e), identify the appropriate calibration method that will advance to the validation stage.

g) The ~~names~~initials, dates, instrument parameters, and final data will be retained until the method validation is successfully concluded.

A.2 Method Development Results and Summary

A.2.1 Determination of the Lower Limit of Quantitation (LLOQ)

The instrument was adjusted following the approved Adjustment Method. Three (3) different metrologically traceable aqueous standards of decreasing concentration were evaluated to determine the LLOQ. It was determined by experiment and manufacturer's literature that the instrument has a masking function at 0.005 g/210L. The LLOQ was administratively set at 0.020 g/210L. The instrument will utilize a 0.02 g/210L calibrator as the lowest calibrator and lowest reported value for subject test results. Table A.1 provides the data from this experiment.

Table A.1—Determination of LLOQ

Date/initials:	**8/1/2014**	**8/1/2014**	**8/1/2014**
	TNW	**TNW**	**TNW**
Instrument SN	90299	90299	90299
Simulator SN	XN1425	XN1480	XN1454
Sim Solution Lot#	140728A	140301A	140301B
Sim Solution Exp.	7/28/2015	3/1/2015	7/28/2015
Nominal quantity conc (g/210 L)	**0.015**	**0.02**	**0.025**
Replicate #1	0.014	0.02	0.026
Replicate #2	0.015	0.02	0.026
Replicate #3	0.014	0.019	0.025
Mean:	0.014	0.020	0.026
Bias (difference in g/210 L)	-0.001	-0.0003	0.001
std dev	0.00058	0.00058	0.00058
% CV:	4.0	2.9	2.2
Acceptable bias and precision	Yes	Yes	Yes
Final ~~determined~~selected LLOQ:	0.020 g/210 L		

A.2.2 Determination of the Upper Limit of Quantitation (ULOQ)

Four (4) different metrologically traceable aqueous standards of increasing concentration were evaluated to determine the ULOQ. The ULOQ was determined to be at 0.400 g/210 L. Table A.2 provides the data from this experiment.

Table A.2—Determination of ULOQ

Date/initials:	**8/2/2014**	**8/2/2014**	**8/2/2014**	**8/2/2014**
	TNW	**TNW**	**TNW**	**TNW**
Instrument SN	90299	90299	90299	90299
Simulator SN	XN1480	XN1425	XN1454	XN1430
Sim Solution Lot#	140301C	140728C	140301D	140728D
Sim Solution Exp.	3/1/2015	7/28/2015	3/1/2015	7/28/2015
Target conc (g/210L)	**0.380**	**0.400**	**0.420**	**0.~~430~~425**
Replicate #1	0.375	0.401	0.41	Sample Over Range
Replicate #2	0.370	0.392	Sample Over Range	Sample Over Range
Replicate #3	0.368	0.387	N/A	Sample Over Range
Mean:	0.371	0.393	N/A	N/A
Minimum acceptable ~~bias~~low result (-5% from target)	0.361	0.380	0.399	0.404
Maximum acceptable ~~bias~~high result (+5% from target)	0.399	0.420	0.441	0.446
Bias (%)	-2.37	-1.67	N/A	N/A
std dev	0.0036	0.0071	N/A	N/A
% CV:	1.0	1.8	N/A	N/A
Acceptable bias and precision	Yes	Yes	**No**	**No**
Final determined ULOQ:	0.400 g/210 L			

A.2.3 Evaluation of Linearity

Following the adjustment method, five non-zero aqueous concentrations were each run 10 times. Concentrations were chosen across the ULOQ and LLOQ range (0.020 – 0.400 g/210L ethanol). Table A.3 provides the data and calculations for Bias and Coefficient of Determination. Figure A.1 is the graph of the best fit line for the data located in Table A.3.

Table A.3—Linearity Evaluation

-	Linearity Evaluation				
Instrument SN	90320				
CRM matrix	Compressed Gas (results normalized)				
CRM Lot#	AL141202	AL141204	AL141208	AL141220	AL141030
Nominal quantity conc. (g/210 L)	**0.020**	**0.040**	**0.080**	**0.200**	**0.400**
Replicate #1	0.018	0.040	0.080	0.201	0.390
Replicate #2	0.019	0.040	0.080	0.201	0.390
Replicate #3	0.020	0.039	0.079	0.201	0.389
Replicate #4	0.019	0.039	0.079	0.201	0.389
Replicate #5	0.018	0.039	0.079	0.202	0.382
Replicate #6	0.019	0.040	0.079	0.202	0.382
Replicate #7	0.020	0.039	0.079	0.202	0.378
Replicate #8	0.019	0.039	0.078	0.203	0.378
Replicate #9	0.020	0.038	0.078	0.203	0.388
Replicate #10	0.019	0.038	0.078	0.201	0.382
Mean:	0.019	0.039	0.079	0.202	0.385
Bias (%)	N/A	N/A	N/A	0.8	-3.8
Bias (g/ 210 L)	-0.0009	-0.0009	-0.001	N/A	N/A
Correlation (r2)	0.999				
Acceptable bias and precision	Yes	Yes	Yes	Yes	Yes

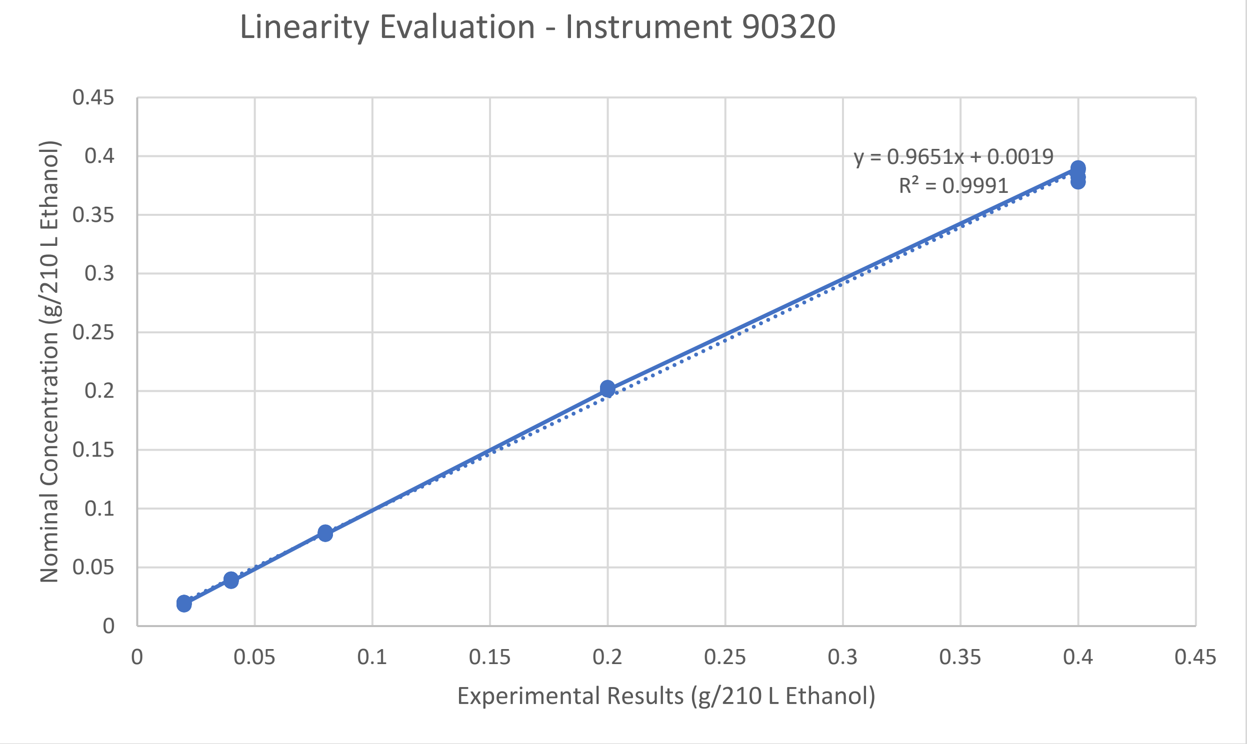

Figure A.1—Best Fit Line for Linearity Evaluation

A.3 Method Development

Table A.~~3~~4 summarizes the instrument parameters, concentrations, acceptance parameters, number of replicates and results.

a) Example using Nominal Quantity Value to calculate Bias Determination

NOTE: Formula #1 is located in Section 6.3.2.2:

$$\boldsymbol{Bias}\ (\%)\ \boldsymbol{at\ Concentration_x} = \left[\frac{\text{Grand Mean of Calculated Concentration}_x - \text{Nominal Concentration}_x}{\text{Nominal Concentration}_x}\right] \times 100 \qquad (A.1)$$

Bias (%) at Concentration$_{80}$ = ((79.45 mg/dL – 80.00 mg/dL) / 80.00 mg/dL) * 100)
Bias (%) at Concentration$_{80}$ = -0.69%

b) Example using Measured Quantity Value to calculate Bias Determination

NOTE: Formula #2 located in Section 6.3.2.2:

$$\boldsymbol{Bias}\ (\%)\ \boldsymbol{at\ Concentration_x} = \left[\frac{\text{Grand Mean of Calculated Concentration}_x - \text{Measured Concentration}_x}{\text{Measured Concentration}_x}\right] \times 100 \qquad (A.2)$$

Bias (%) at Concentration$_{80}$ = ((79.45 mg/dL – 78.53 mg/dL) / 78.53 mg/dL) * 100)
Bias (%) at Concentration$_{80}$ = 1.17%

~~for~~Table A.4—Summary of Calibration & Measurement Assurance Results Using Method A

-	Calibration Method A Data						Measurement Assurance
Instrument SN	90320						90320
CRM matrix	compressed gas (results normalized)						aqueous
CRM Lot#	AL141202	AL141204	AL141208	AL141015	AL141220	AL141030	140728D
Nominal quantity conc. (g/210 L)	**0.02**	**0.04**	**0.08**	**0.15**	**0.20**	**0.40**	**0.10**
Replicate #1	0.020	0.039	0.079	0.148	0.201	0.389	0.099
Replicate #2	0.020	0.039	0.079	0.149	0.202	0.378	0.099
Replicate #3	0.018	0.039	0.079	0.150	0.202	0.382	0.098
Replicate #4	0.018	0.040	0.080	0.149	0.201	0.390	0.097
Replicate #5	0.020	0.040	0.080	0.150	0.203	0.388	0.100
Mean:	0.0192	0.0394	0.0794	0.1492	0.2018	0.3854	0.0986
Bias (%)	N/A	N/A	N/A	-0.5	0.9	-3.7	-1.4
Bias (g/210 L)	-0.0008	-0.0006	-0.0006	N/A	N/A	N/A	N/A
std dev	0.001	0.0005	0.0005	0.0008	0.0008	0.005	0.001
% CV	5.7	1.4	0.7	0.6	0.4	1.3	1.2
Acceptable bias and precision	Yes	Yes	Yes	Yes	Yes	Yes	Yes

A.3.1 Assessment and Comparison of Method A and Method B

No further optimization is necessary as Calibration Method A meets the requirements.

A.3.2 Determination of the Reporting Range

The LLOQ, ULOQ and concentrations of interest were considered in determining the reporting range (for subject testing). The final reported result has 2 significant figures with units = g/210 L of breath. See Table A.5 for additional information.

Table A.5—Determination of the Reporting Range

Concentrations of Interest	Concentration (g/210 L)
LLOQ	0.02
Lowest conc. of interest	0.02
Lowest (subject) reporting limit	0.02
ULOQ	0.40
Highest conc. of interest	0.20
Highest (subject) reporting limit	0.40
Resulting (subject) reporting range	0.02-0.40

For results <0.02 g/210 L of breath, the instrument will report the actual result when in calibration mode. For subject testing, these results will be reported as 0.00 g/210 L of breath.

For results >0.40 g/210 L of breath, the instrument will report the actual result when in calibration mode. For subject testing, these results will be reported as "Seek Medical Attention".

A.3.3 Summary of Method Development

In August 2015, Anytown PD performed method development for the ABC evidential breath alcohol instrument. The quantitation range was found to be 0.02-0.40 g/ 210L of breath. The (subject) reporting range was established to be 0.02-0.40 g/210L of breath for Method A. Method validation experiments will be performed using Calibration Method A to assess the suitability of the method for evidential calibration purposes.

~~Table A.3—Summary of Calibration Results Using Method A~~

~~**METHOD A**~~	~~**Calibration**~~	~~**Verification**~~					
~~Instrument SN~~	~~90320~~	~~90320~~	~~90320~~	~~90320~~	~~90320~~	~~90320~~	
~~CRM matrix~~	~~compressed gas (results normalized)~~	~~compressed gas (results normalized)~~	~~compressed gas (results normalized)~~	~~compressed gas (results normalized)~~	~~compressed gas (results normalized)~~	~~compressed gas (results normalized~~	
~~**Nominal quantity conc. (g/210 L)**~~	~~**0.02**~~	~~**0.04**~~	~~**0.08**~~	~~**0.15**~~	~~**0.2**~~	~~**0.4**~~	
~~Replicate #1~~	~~0.02~~	~~0.039~~	~~0.079~~	~~0.148~~	~~0.201~~	~~0.389~~	
~~Replicate #2~~	~~0.02~~	~~0.039~~	~~0.079~~	~~0.149~~	~~0.202~~	~~0.378~~	
~~Replicate #3~~	~~0.018~~	~~0.039~~	~~0.079~~	~~0.15~~	~~0.202~~	~~0.382~~	
~~Replicate #4~~	~~0.018~~	~~0.04~~	~~0.08~~	~~0.149~~	~~0.201~~	~~0.39~~	
~~Replicate #5~~	~~0.02~~	~~0.04~~	~~0.08~~	~~0.15~~	~~0.203~~	~~0.388~~	
~~Mean:~~	~~0.0192~~	~~0.0394~~	~~0.0794~~	~~0.1492~~	~~0.2018~~	~~0.3854~~	~~0.0986~~
~~(± 5% or 0.005) acceptable bias range~~	~~0.015-0.025~~	~~0.035-0.045~~	~~0.075-0.085~~	~~0.142-0.157~~	~~0.190-0.210~~	~~0.380-0.440~~	
~~Bias (%)~~	~~N/A~~	~~N/A~~	~~N/A~~	~~-0.5~~	~~0.9~~	~~-3.7~~	~~-1.4~~
~~Bias (g/ 210 L)~~	~~-0.0008~~	~~-0.0006~~	~~-0.0006~~	~~N/A~~	~~N/A~~	~~N/A~~	~~N/A~~
~~std dev~~	~~0.0011~~	~~0.0005~~	~~0.0005~~	~~0.0008~~	~~0.0008~~	~~0.0052~~	
~~% CV~~	~~5.7~~	~~1.4~~	~~0.7~~	~~0.6~~	~~0.4~~	~~1.3~~	
~~Acceptable bias and precision~~	~~Yes~~	~~Yes~~	~~Yes~~	~~Yes~~	~~Yes~~	~~Yes~~	

Annex B
(informative)

Example of a Method Validation Plan[12]

B.1 Introduction

ACME Breath Alcohol Program Validation Study

During **Method Development** it was determined that the LLOQ for instrument "Model-123" was 0.01 g/210L, the ULOQ was 0.40 g/210L~~, **and** the resulting~~. The administratively determined Reporting Range ~~was~~is 0.02 to 0.40 g/210L. Linearity testing determined the instrument response to be linear, and therefore 4 non-zero calibrators will be used for calibration.

Because this calibration method will serve as a quantitative procedure for evidential use, the method validation parameters will be assessed for the desired requirements as listed in Table B.1. The assessments shall be performed by multiple analysts and in multiple locations, including field locations. The name of the analyst and the location shall be recorded each time.

B.2 Equipment, Method and Materials

Instrument –	"Model-123" evidential Breath Alcohol measuring instrument, infrared technology calibrated with Method A1216
Adjustment Method ~~–~~ -	Evidential Instrument Adjustment (version 003)
Calibration Method –	Method Validation Procedure for Breath Alcohol Instruments (MVP_001), approved 1/1/2015
	Gas Concentrations: • 0.02 g/210L (statutory limit, LLOQ, & lowest calibrator • 0.08 g/210L (statutory limit) • 0.15 g/210L (statutory limit) • 0.40 g/210L (ULOQ & highest calibrator)
Simulator –	Model "XYZ" simulators from Company-A, with certified thermometers from Company-B
Reference material –	Aqueous, certified ethanol-water solutions from Company-C, multiple concentrations
Reference material –	Gas, certified compressed gas-ethanol from Company-D, multiple concentrations
Water –	Laboratory grade deionized water, made in-house

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[12] This is an example of a mock Method Validation Plan, for illustrative purposes only.

The lot numbers of all reference materials and other reagents shall be recorded as well as the serial numbers of all equipment.

This approved computer program has the capability of toggling masking on and off when the analytical mode is in use.

The instrument is to be calibrated prior to conducting the validation experiments. Calibration is to occur in the analytical mode with no masking.

Validation experiments will be conducted in the analytical mode with masking in place. The approved ACME adjustment and calibration method will be used (i.e., no deviations permitted).

Table B.1—Validation Plan for Instrument "Model-123" (Method A1216)

Parameter (ANSI/ASB 055 document reference)	Pre-Determined Acceptance Criteria	Validation Parameters
Bias (see 6.3.2)	Shall not exceed ± 5%, or ± 0.005 g/210L, whichever is greater	Validation experiment will include 10 replicates/aqueous concentration, 6 separate ~~runs~~days, 5 different instruments at the following concentrations: 0.~~02~~06 g/210L (~~i.e. statutory limit &~~ 3 times the LLOQ) ~~10L (statutory limit)~~ 0.15 g/210L (~~statutory limit)~~ ~~0.30 g/210L (~~mid-range concentration) 0.~~40~~320 g/210L (80% of the ULOQ) Separately evaluate the bias for each instrument.
Precision (see 6.3.2)	Not to exceed +/-10 % CV	

Carryover (see 6.3.4)	~~Carryover at ULOQ does not exceed 0.01 g/210L~~Carryover at ULOQ does not exceed 0.01 g/210L. The top calibrator (0.400 g/210L) will be tested with three replicates. Immediately following the three replicates, a subject test will be run (negative ethanol subject) to evaluate the presence of carryover. This experiment is to be repeated two times (a total of three data sets).
NOTE Several of the validation requirement parameters listed in this table can be assessed simultaneously~~; for example, the~~. The data used for bias assessment ~~can also~~will be used to assess precision	

Annex C

(informative)

Calculations (Background and Examples)

Accurate calculations rely on scientific knowledge as well as mathematical skills. This informative annex provides examples for selected calculations. It does not provide the scientific knowledge necessary to ensure the correct formulae are used.

C.1 Bias and Precision

C.1.1 Nominal vs. Measured Quantity Value

Certified Reference Material (CRM) possess a Nominal Quantity Value. This may also be referred to as a 'stated' or 'certified concentration' (e.g., Ethanol-80 has a certified concentration of 80.00 +/- 0.31 mg/dL). In essence, the reference material producer has prepared the reference material to achieve a target concentration (i.e., the target concentration is 80.00 mg/dL in the previous example).

Certified Reference Material should be supplied with a Certificate of Analysis meeting the requirements of ISO 17034. Table C.1 provides an example of a Certificate of Analysis entry with a Nominal Quantity Value of an aqueous ethanol reference material.

Table C.1 – Example of Aqueous Nominal Quantity Value on Certificate of Analysis

Component	Solution Chromatographic Purity	Certified Concentration
Ethanol	> 99.9%	80.00 ± 0.31 mg/ dL

The result(s) of the reference material producer's testing process are Measured Quantity Value(s). Measured Quantity Values are frequently included on a Certificate of Analysis (see Table C.2).

Table C.2 – Example of Aqueous Measured Quantity Value on Certificate of Analysis

Solution Standard	Lot Number	Results compared to NIST SRM Lot 1234 (mg/ dL)	Homogeneity (ampoule to ampoule consistency) %RSD
New Lot	ABC123	78.53	1.02%
Prior Lot	ABC124	78.99	1.08%
Acceptance Criteria		± 2%	± 2%

C.1.2 Bias Determination Calculations

Section 6.3.2.2 *Bias Determination and Acceptance* in this document provides two separate bias determination formulae.

To demonstrate the calculations, hypothetical values of a single calibrator are provided below:

Nominal Quantity Value:	80.00 mg/dL Ethanol
Measured Quantity Value:	78.53 mg/dL Ethanol

Grand Mean of validation results: 79.45 mg/dL Ethanol

C.1.3 Within-Run Precision Determination Calculations

a) Table C.3 provides replicate data from a single run.

Table C.3 – Example Data from Calibration Run

Low Calibrator	Concentration (g/210 L)
Rep 1	0.050
Rep 2	0.051
Rep 3	0.048
Mean	0.~~050~~0496
Standard Deviation	0.~~002~~00152

b) ~~Using the~~The data from Table C.3 and the Within-Run Precision formula (#4 located in Section 6.3.2.3) was used to determine the precision of this calibrator. A software program was utilized which did not limit significant figures. Significant figures are taken into consideration during the final step. The calculation is as follows:

$$Within - Run\ CV(\%) = \frac{std\ dev\ of\ a\ single\ run\ of\ samples}{mean\ calculated\ value\ of\ a\ single\ run\ of\ samples} \times 100 \qquad (C.1)$$

$$Within - Run\ \%\ CV = \cancel{\frac{0.002}{0.050}}\ \cancel{\times}\ \frac{0.00152}{0.0496} \times 100$$

$$Within - Run\ \%\ CV = 3.1\%$$

C.1.4 Between-Run Precision Determination Calculations

a) Table C.4 provides validation data for a single concentration (6 separate runs with 3 replicates per concentration).

Table C.4 – Example Data from Validation Experiment

Low Concentration	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6
Rep 1	0.050	0.050	0.051	0.049	0.049	0.048
Rep 2	0.049	0.050	0.050	0.048	0.049	0.048
Rep 3	0.049	0.051	0.050	0.049	0.050	0.048
Grand Mean	0.049					
Std Dev	0.001					

b) Using the data from Table C.4 and the Between-Run Precision formula (#5 located in Section 6.3.2.3):

$$Between - Run\ \%\ CV = \frac{std\ dev\ of\ all\ observations\ for\ each\ concentration}{grand\ mean\ for\ each\ concentration} \times 100 \qquad (C.2)$$

$$Between - Run\ \%\ CV = \frac{0.001}{0.049} \times 100$$

$$Within - Run\ \%\ CV = 2.0\%$$

C.2 Converting Aqueous Concentrations to Vapor Concentrations

Breath alcohol measuring instruments that use aqueous reference material measure ethanol concentrations in the vapor phase (headspace). The nominal and measured quantity values published for aqueous reference material arise from the aqueous phase. To accurately use aqueous concentrations in breath alcohol calibrations, a mathematical conversion takes place.

The conversion requires a partition ratio. There are several peer reviewed, published studies outlining aqueous ethanol partition ratios. This informative annex uses the ratio published by A.W. Jones in *Determination of liquid/air partition coefficients for dilute solutions of ethanol in water, whole blood, and plasma*. The water to air partition ratio ($K_{w/a}$) for an aqueous ethanol solution was determined to be 2587 at 34°c. Using this ratio, the following formula can be used to determine the relationship between an aqueous concentration and vapor concentration:

$$\text{Concentration}_w = [\text{Concentration}_a \times 2587] \div 210 \qquad \text{(C.3)}$$

where:

Concentration_w = concentration in the aqueous (water) phase (mg/100 mL)

Concentration_a = concentration in the vapor (air) phase (g/210 L)

2587 = conversion factor [2587 water: 1 air @ 34° ~~c~~C]

210 = conversion factor [g/210 L to mg/100 mL]

Scenario - A Program wants to produce reference material with a targeted 0.080 g/210 L vapor concentration. What aqueous concentration should be prepared? Using the formula above, the calculation becomes:

$$\text{Concentration}_w = [\text{Concentration}_a \text{ x } 2587] \div 210$$

$$\text{Concentration}_w = [0.080 \text{ x } 2587] \div 210$$

$$\text{Concentration}_w = 0.99 \text{ mg/100 mL}$$

The Program would prepare an aqueous ethanol solution with a target concentration of 0.99 mg/100 mL to achieve a target vapor concentration of 0.080 g/210 L.

C.3 Normalizing a Compressed Gas Result to Standard Atmospheric Pressure

The concentration of compressed gas reference material is impacted by atmospheric pressure. To compare results, they are normalized to standard atmospheric pressure. The following calculation may be used to normalize a result:

$$\text{Normalized Result (g/ 210 L)} = [760/\text{Pressure}] \times \text{Result} \qquad \text{(C.3)}$$

where:

Normalized Result (g/210 L) = instrument result normalized to standard atmospheric pressure

Result = the actual measurement result obtained from the instrument (g/210 L)

760 = conversion factor [1 atm = 760 mmHg]

Pressure = barometric pressure at the time of the result (mmHg)

Scenario - An instrument's barometric pressure at 9am MDT was 712.5 mmHg. At 0902 MDT a calibration verification test was performed, with a result of 0.075 g/210 L. What is the normalized result?

$$\text{Normalized Result (g/ 210 L)} = [760/\ \text{Pressure}] \times \text{Result}$$

$$\text{Normalized Result (g/ 210 L)} = [760/\ 712.5] \times 0.075$$

$$\text{Normalized Result (g/ 210 L)} = 0.080$$

The normalized result may now be used to evaluate bias.

C.4 Converting Compressed Gas Results (g/210 L to parts per million)

Compressed gas reference material is frequently available with the nominal quantity value (concentration) expressed as both a breath alcohol value (e.g., grams per 210 liters of ethanol vapor) and parts per million (ppm)). The two values are typically present on the Certificate and the cylinder label of CRM. The following formula converts an unknown breath alcohol result (g/210 L to ppm):

$$\text{Result}_{\text{ppm}} = (\text{Result}_{\text{BrAC}} \times \text{CRM}_{\text{ppm}}) \div \text{CRM}_{\text{BrAC}} \qquad \text{(C.4)}$$

where:

$\text{Result}_{\text{ppm}}$ = converted result (ppm)

$\text{Result}_{\text{BrAC}}$ = instrument (g/210 L)

CRM_{ppm} = certified nominal quality value (ppm)

CRM_{BrAC} = certified nominal quality value (g/210 L)

Scenario - A compressed gas proficiency test (PT) cylinder provides a result of 0.150 g/ 210 L ethanol. The label on one of their compressed gas calibrators provides the following information [0.100 g/210 L: 260 ppm]. The proficiency test results are to be recorded in ppm per the proficiency test ~~providers~~provider (PTP) instructions. What is the converted result?

$$\text{Result}_{\text{ppm}} = (\text{Result}_{\text{BrAC}} \times \text{CRM}_{\text{ppm}}) \div \text{CRM}_{\text{BrAC}}$$

$$\text{Result}_{\text{ppm}} = (0.150 \times 260) \div 0.100$$

$$\text{Result}_{\text{ppm}} = 390$$

C.5 Corrected and Uncorrected CRM

When compressed gas reference material is run on an electrochemical detector (fuel cell), a small but consistent reduction in expected results occurs. This is addressed by increasing compressed gas concentrations in a systemic manner referred to as the wet/dry offset. This offset may be accomplished by automatically converting results in the software prior to reporting or by increasing the concentration of ethanol in the calibrators. The instrument manufacturer should be consulted to determine the specific wet-dry offset approach utilized.

In instances where CRM with higher concentrations are utilized as calibrators, cylinders may be labelled as Corrected. For example, a manufacturer may specify their instrument has a wet dry offset of 4.5%. A Program may purchase Corrected CRM to calibrate their instruments. The ethanol concentration (in ppm) is increased by 4.5% in the Corrected CRM. The reference material producer (RMP) may label their compressed gas cylinders as Uncorrected vs. Corrected and the ppm will correspond accordingly. Using the above example of a 4.5% wet dry offset, a 0.100 g/210 L nominal quantity value CRM will be labelled as Uncorrected (260 ppm) or Corrected (272 ppm).

C.6 Example of Calculations Used in a Proficiency Test

Scenario - A Program calibrates their instrument using Corrected compressed gas reference material ([0.100 g/210 L:272 ppm]) with a wet-dry offset of 4.5%. The PTP provides four compressed gas cylinders and specifies a ratio of [0.100 g/210 L:260 ppm] ethanol for the proficiency test cylinders. The proficiency test results are to be recorded in g/210 L per the PTP instructions. What steps will the Program take to provide results to the PTP?

Step 1- following calibration, each of the unknowns is run

Table C.5 Proficiency Test Data

	Concentration (g/210 L)			
Replicate	**PT cylinder 1**	**PT cylinder 2**	**PT cylinder 3**	**PT cylinder 4**
Rep 1	0.042	0.284	0.118	0.069
Rep 2	0.042	0.279	0.120	0.073
Rep 3	0.041	0.280	0.119	0.070
Mean	0.042	0.281	0.119	0.071
Standard Deviation	0.001	0.003	0.001	0.002

Step 2 -for each unknown, calculate the normalized result following Section C.3

Table C.6—Proficiency Test Data Normalized

	PT cylinder 1	PT cylinder 2	PT cylinder 3	PT cylinder 4
Mean	0.042	0.281	0.119	0.071
Standard Deviation	0.001	0.003	0.001	0.002
Barometric Pressure (mmHg)	764.80	764.80	764.80	764.80
Normalized results (g/210 L)	0.041	0.279	0.118	0.070

Step 3- convert the normalized results to ppm following Section C.4

Table C.7—Conversion of Breath Alcohol Results (g/210 L to ppm)

	PT cylinder 1	PT cylinder 2	PT cylinder 3	PT cylinder 4
Normalized results (g/210 L)	0.041	0.279	0.118	0.070
Normalized results (ppm)	112.62	759.52	321.65	191.01

Step 4- convert the result (ppm) generated from a calibration that utilizes Corrected cylinders to an Uncorrected result (g/210 L)

$$\text{Uncorrected Result (g/210 L)} = (\text{PTP}_{g/210\text{ L}} \div \text{PTP}_{\text{ppm}}) \times \text{Corrected Result}_{\text{ppm}} \quad \text{(C.4)}$$

where:

Uncorrected Result (g/210 L) = the breath alcohol result to be reported to the PTP

$\text{PTP}_{g/210\text{ L}}$ = part of the conversion factor supplied by the Proficiency Test Provider (0.100g/210 L)

PTP_{ppm} = part of the conversion factor supplied by the Proficiency Test Provider (260ppm)

$\text{Corrected Result}_{\text{ppm}}$ = calculated normalized result from Program's run (112.62, 759.52, 321.65, 191.01 respectively)

Table C.8—Conversion of Corrected Results to Uncorrected Results

	PT cylinder 1	PT cylinder 2	PT cylinder 3	PT cylinder 4
Corrected results (ppm)	112.62	759.52	321.65	191.01
Uncorrected results (g/210 L)	0.043	0.292	0.124	0.073

Step 5 – the Uncorrected results (g/210 L) will be reported to the PTP

Annex D
(informative)

Example of Validation Results: Accuracy

Table D.1 provides an example of mock validation results related to bias and precision. This is for illustrative purposes only; results for a ~~successful~~ mock adjustment and calibration are not provided. For this example the computer system would have been updated to force a report of “Sample greater than 0.250 g/210 L Ethanol” for any result above the top calibrator.

Table D.1—Summary of Validation Results: Accuracy

Record Title:	2021 Accuracy Validation Data - Sunset PD
Method Name:	~~Sunset PD~~ Breath Alcohol Adjustment/Calibration Method
Ethanol Adjustment CRM:	0.10 g/210 L
Ethanol Calibrator Range:	0.~~0150 –~~015 - 0.~~350~~250 g/210 L
Maximum concentration for low validation reference material:	0.045 g/210 L
Minimum concentration for high validation reference material:	0.~~280~~200 g/210 L
Validation concentrations for precision + bias:	• 0.020 g/210 L • 0.150 g/210 L • 0.~~300~~200 g/210 L

Low Nominal Quantity Value		0.020 g/210 L	Results			Within-Run Statistics			
Date	Initials	Location	Rep 1	Rep 2	Rep 3	Mean	Standard Deviation	Bias (g/210 L)	% CV
2/10/~~2021~~21	MG	HQ	0.019	0.02	0.02	0.020	0.001	0.000	2.9
2/11/~~2021~~21	SZ	Barrack 5	0.019	0.018	0.018	0.018	0.001	-0.002	3.1
2/12/~~2021~~21	DR	Barrack 2	0.02	0.021	0.021	0.021	0.001	0.001	2.8
2/16/~~2021~~21	CF	Barrack 8	0.018	0.02	0.02	0.019	0.001	-0.001	6.0
2/18/~~2021~~21	BS	Barrack 3	0.017	0.018	0.018	0.018	0.001	-0.002	3.3
2/22/~~2021~~21	MK	HQ	0.018	0.019	0.021	0.019	0.002	-0.001	7.9

Between-Run Statistics	Grand Mean	0.019
	Bias (g/210 L)	0.001
	Standard Deviation	0.001
	% CV	6.5

Middle Nominal Quantity Value		0.150 g/210 L	Results			Within-Run Statistics			
Date	Initials	Location	Rep 1	Rep 2	Rep 3	Mean	Standard Deviation	Bias (%)	% CV
2/10/~~2021~~21	MG	HQ	0.148	0.149	0.15	0.149	0.001	-0.7	0.7
2/11/~~2021~~21	SZ	Barrack 5	0.145	0.147	0.148	0.147	0.002	-2.2	1.0
2/12/~~2021~~21	DR	Barrack 2	0.15	0.149	0.151	0.150	0.001	0.0	0.7
2/16/~~2021~~21	CF	Barrack 8	0.147	0.149	0.15	0.149	0.002	-0.9	1.0
2/18/~~2021~~21	BS	Barrack 3	0.146	0.148	0.151	0.148	0.003	-1.1	1.7
2/22/~~2021~~21	MK	HQ	0.152	0.151	0.153	0.152	0.001	1.3	0.7

Between-Run Statistics		
	Grand Mean	0.15
	Bias (%)	-0.59
	Standard Deviation	0.002
	% CV	1.4

High Nominal Quantity Value		0.~~300~~200 g/210 L	Results			Within-Run Statistics			
Date	Initials	Location	Rep 1	Rep 2	Rep 3	Mean	Standard Deviation	Bias (%)	% CV
2/10/~~2021~~21	MG	HQ	0.~~305~~205	0.~~309~~209	0.~~307~~207	0.~~307~~207	0.002	~~2.3333~~0.0023	1.0~~.7~~
2/11/~~2021~~21	SZ	Barrack 5	0.~~302~~202	0.~~304~~204	0.~~303~~203	0.~~303~~203	0.001	~~1.0000~~0.0010	0.~~3~~5
2/12/~~2021~~21	DR	Barrack 2	0.~~297~~197	0.~~299~~199	0.~~3~~200	0.~~299~~199	0.002	-0.~~4444~~0004	0.~~5~~8
2/16/~~2021~~21	CF	Barrack 8	0.~~301~~201	0.~~302~~202	0.~~301~~201	0.~~301~~201	0.001	0.~~4444~~0004	0.~~2~~3
2/18/~~2021~~21	BS	Barrack 3	0.~~298~~198	0.~~299~~199	0.~~298~~198	0.~~298~~198	0.001	-0.~~5556~~0006	0.~~2~~3
2/22/~~2021~~21	MK	HQ	0.~~3~~200	0.~~302~~202	0.~~304~~204	0.~~302~~202	0.002	0.~~6667~~0007	1.0~~.7~~

Between-Run Statistics		
	Grand Mean	0.~~30~~20
	Bias (%)	0.~~57~~86
	Standard Deviation	0.003
	% CV	1.~~1~~6

Annex E

(informative)

Example of Validation Results: Reference Material Part 1[13]

E.1 Freeze/Thaw Validation Plan

E.1.1 Validation Plan:

To evaluate the effect of freezing and thawing on aqueous calibration material, the Program will examine the effect on 0.080 g/210L reference material. A total of twenty bottles of reference material from the same lot will be used in this experiment. Five bottles (e.g., bottles 1 through 5) that have never been frozen will be analyzed initially five times (replicates). The remaining bottles (e.g., bottles 6 through 20) will then be placed in a freezer at –10°C for a minimum of forty-eight hours. After they are allowed to thaw unassisted at room temperature for forty-eight hours, five bottles (e.g., bottles 6 through 10) will be analyzed five times (replicates). The remaining bottles will undergo a second (e.g., bottles 11 through 15) and third (e.g., bottles 16 through 20) freeze/thaw cycle respectively~~,~~ and be analyzed in the same fashion described previously.

This experiment will be performed on an "ABC-123" instrument with "XYZ" simulators connected to the instrument's calibration port. All reference material shall be of the same lot for adequate comparison.

E.1.2 Acceptance Criteria:

The reference material ~~are~~is considered stable if the combined mean of the analysis of the frozen/thawed samples meets the bias (0.005 g/210 L) and precision (± 10 % CV) acceptance criteria.

E.2 Freeze/Thaw Validation Results and Summary

The validation experiment took place from 02/01/2016 to 02/27/2016 using 0.080 g/210L reference material lot 3456-8. The raw data and all documentation generated from this experiment have been retained in the validation file for this method. A software program was used to perform calculations. The mean of each analysis, as well as the combined mean for each event are recorded in ~~the~~ Table E.1~~.~~ (Note: Consideration of significant figures was applied to reported Bias and Precision results).

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[13] This is an example of additional mock Validation Parameters and subsequent results, for illustrative purposes only.

Table E.1—Freeze/Thaw Experiment Data

	Initial Analysis	**After One Cycle**	**After Two Cycles**	**After Three Cycles**
	Bottles 1-5	Bottles 6-10	Bottles 11-15	Bottles 16-20
Replicate Mean	0.0813	0.0809	0.0805	0.0798
Replicate Mean	0.0807	0.0805	0.0800	0.0795
Replicate Mean	0.0817	0.0815	0.0811	0.0804
Replicate Mean	0.0809	0.0806	0.0802	0.0797
Replicate Mean	0.0811	0.0809	0.0806	0.0801
Within-run Mean:	0.~~0811~~081	0.~~0809~~081	0.~~0805~~080	0.~~0799~~080
Within-run SD:	0.~~00038~~0003	0.~~00039~~0003	0.~~00042~~0004	0.~~00035~~0003
Bias (%)	~~0.2~~1.4	~~-0~~1.1	~~-~~0.6	-~~1.4~~0.13
% CV	0.~~47~~4	0.~~48~~4	0.~~52~~5	0.~~44~~4

The impact of freeze/thaw cycles was demonstrated to be at an acceptable level, with the greatest change in bias being a ~~loss~~gain of 1.4%, which meets the requirement to be less than 5%.~~ However, the lab noted an increasing negative bias with each freeze/thaw cycle; therefore, a decision was made to discard any reference material that goes through more than three freeze/thaw cycles.~~

Annex F
(informative)

Example of Additional Validation Parameters and Results: Reference Material – Part 2[14]

F.1 Minimum Allowable Pressure of Compressed Gas Reference Standards

F.1.1 General

The purpose of this validation is to ensure that compressed gas reference standards used for calibration of evidential Breath Alcohol instruments continue to provide acceptable results at minimum pressure (i.e., running low to empty). Anytown, USA calibrates 2 models of evidential instruments (Desktop A and Handheld B). The software used in Anytown, USA's evidential instruments will provide a "compressed gas tank empty" test result when the pressure reaches 50 psi (pounds per square inch) and will not allow a subject test or calibration to continue.

F.1.2 Validation Plan

Two instruments, installed with software version XXXXX will be used to assess Certified Reference Material (CRM) acceptability.

a) Test a minimum of three different compressed gas CRM of different known values with a pressure close to 50psi; e.g., 60psi.

b) Record the initial pressure in psi for each CRM.

  1) Perform a test to determine if the result is within acceptable parameters (0.005 or +/-5%).

  2) Record the pressure after this test.

c) Continue to perform tests of each CRM until:

  1) The instrument's minimum pressure level of acceptance is reached (tank empty message appears), or

  2) An insufficient sample warning is given, or

  3) A result outside of the acceptable parameters is produced.

d) Perform a test on each CRM on different days and with different analysts, if possible.

e) Record the route of delivery for each test.

  1) Breath port.

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[14] This is an example of additional mock Validation Parameters and subsequent results, for illustrative purposes only.

2) Reference material inlet.

Evaluate the results to determine the acceptable lower pressure levels. Acceptability shall be concluded if CRM results are within the acceptable parameters at all concentrations and all pressure readings unless an instrument message alerts the user of “compressed gas tank empty”.

F.1.3 Validation Results

Table F.1—Summary of Minimum Allowable Pressure

Date/initials:	**2/11/16** **ESZ**	**2/12/16** **RPM**	**3/7/16** **RPM**	**3/8/16** **ESZ**	**3/9/16** **ESZ**	**3/10/16** **RPM**
Instrument Model	Desktop A	Handheld B	Desktop A	Handheld B	Desktop A	Handheld B
Instrument SN	1234	9876	1236	9874	1238	9872
Delivery Route	Inlet	Breath port	Inlet	Breath port	Inlet	Breath port
Dry Gas Std. Lot/Tank #	OP416120/5	OP416121/3	OP335123/8	OP335126/7	OP435127/16	OP435128/12
Dry Gas Std. Exp.	06/10/2016	6/10/2016	12/19/2016	12/19/2016	12/19/2016	12/19/2016
Nominal quantity conc (g/210L)	**0.198**	**0.200**	**0.040**	**0.040**	**0.081**	**0.081**
Replicate #1	0.199	0.203	0.041	0.040	0.080	0.081
Replicate #1 psi	56	54	56	56	54	52
Replicate #2	0.199	0.204	0.041	0.040	0.080	0.081
Replicate #2 psi	51	54	54	55	54	52
Replicate #3	0.199	0.203	0.041	0.040	0.080	0.081
Replicate #3 psi	51	51	51	53	51	51
Replicate #4	0.199	0.198	0.041	0.040	0.080	0.081
Replicate #4 psi	51	46	51	46	51	46
Replicate #5	“Tank Empty”	*Sample Timeout	“Tank Empty”	*Sample Timeout	“Tank Empty”	*Sample Timeout
Replicate #5 psi	46	31	46	41	46	41
Acceptable Results (Y/N)	Yes	Yes	Yes	Yes	Yes	Yes

F.1.4 Validation Summary

A total of 3 Desktop A and 3 Handheld B instruments installed with Anytown, USA software were evaluated at low pressure levels with CRM. The results presented in F.1.3 (Table F.1 – Summary of Minimum Allowable pressure) indicate that all results at lower pressure were either within the acceptable parameters or generated an instrument message. This demonstrates that no negative effects are to be expected while calibrating the instrument using compressed gas cylinders at lower pressure. The instruments will either produce valid results or stop calibration activities. The results objectively support that the Certified Reference Material used to calibrate is acceptable at low pressure levels.

Annex G

(informative)

Example of a Validation Summary for Environmental Impact[15]

Scope and Purpose~~:~~ for the validation experiment:

The performance of the calibration method (SOP 4.3) was assessed under similar environmental conditions that are typically encountered during calibration. To assess the performance of the calibration method, Program personnel shall calibrate the instrument(s) under these expected conditions and evaluate the resultant data to ensure the method's acceptability.~~The~~

The following information captures the environmental conditions that are typically encountered. Town Everywhere Sheriff's Department calibrates evidential instruments in the laboratory and field (external facilities and roadside). The Town Everywhere S.D. experiences great fluctuation in temperatures and humidity throughout the year. Additionally, the instruments located in various facilities across Town Everywhere are subject to different barometric pressures due to differing altitudes. ~~To assess the performance of the calibration method, Program personnel shall calibrate the instrument(s) under these expected conditions and evaluate the resultant data to ensure the method's acceptability.~~

Example experimental design (evaluation plan)

Temperature:

The lowest and highest laboratory temperatures over three months were recorded with a SI-traceable reference thermometer. An instrument was calibrated using SOP 4.3 during the temperature highs and lows. Post-calibration, the following parameters were assessed: bias, precision, and carryover. Results met the defined criteria for acceptance. Data are included in the main summary for the SOP 4.3 calibration method.

Annual temperature data from the National Weather Service- Everywhere Office was obtained to determine reasonable high and low temperature expectations for roadside conditions. The average high temperature was 110°F (43°C) and the average low temperature was −50°F (−45°C). An administrative decision was made to limit field calibration to the temperature range of 32°F to 100°F (0°C to 38°C). An instrument was calibrated three times each using SOP 4.3 at 32°F (0°C) and at 100°F (38°C). Post-calibration, the following parameters were assessed: bias, precision, and carryover. Results met the defined criteria for acceptance. Data are included in the main summary for the SOP 4.3 calibration method.

Barometric Pressure:

Altitude ranges from Town Everywhere were obtained. The altitude ranged from sea level (~10 meters) to 5,000 feet (~457 meters). An instrument was calibrated at the highest and lowest point in Town Everywhere. Post-calibration, the following parameters were assessed: bias, precision, and

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[15] This is an example of a portion of a mock Validation Summary for illustrative purposes only.

carryover. Results met the defined criteria for acceptance. Data are included in the main summary for the SOP 4.3 calibration method.

RFI:

The manufacturer provided independent testing to internationally accepted electromagnetic compatibility (EMC) standards. In addition to this independent testing, Everywhere Sheriff's Office personnel activated their emergency communication devices (radios) during and after the SOP 4.3 calibration method was being performed. Personnel were in close proximity to the instruments during these experiments. Results met the defined criteria for acceptance. A passing result is one which either flags an RFI error *or* provides a valid result whose value is not altered by more than the acceptable bias from the expected result. Data are included in the main summary for the SOP 4.3 calibration method.

Annex H
(informative)

Bibliography

This is not meant to be an all-inclusive list as other publications on this subject may exist. At the time this standard was drafted, these were the publications available for reference. Additionally, any mention of a particular software tool or vendor as part of this bibliography is purely incidental, and any inclusion does not imply endorsement.

For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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