Procedure for Measurement Assurance

1.0 Purpose - This procedure specifies the required elements for measurement assurance in the Drug Chemistry Sections of the State Crime Laboratory.

2.0 Scope – This procedure applies to Drug Chemistry at the Raleigh, Triad, and Western locations of the State Crime Laboratory.

3.0 Definitions

- Coverage factor – numerical factor used as a multiplier of the combined uncertainty in order to obtain an expanded uncertainty.
- Coverage probability (level of confidence) – probability that the set of true quantity values of a measurand is contained within a specified coverage interval.
- Measurement – a process of experimentally obtaining one or more quantity values, typically of physical, chemical, or biological nature. Implies comparison of quantities.
- Metrology – the science of measurement.
- Measurand – the (unknown) quantity subject to measurement.
- Reference standard – measurement standard designated for the calibration of other measurement standards (reference standards or equipment)
- Reference material – material sufficiently homogeneous and stable, with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.
- Measurement Traceability – an unbroken chain of comparisons (using acceptable and documented methods) to national or international standards (SI) with each comparison having stated uncertainties.
- Uncertainty of measurement – a parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.

4.0 Equipment, Materials and Reagents

4.1 Equipment

- Balances (top loading, analytical, and bulk)

4.2 Materials and Reagents

- ANSI/ASTM E617 Class 1 Reference Standard Weights
- OIML R111 Class F Reference Standard Weights or better (may be used for Bulk balances only)
- Weigh paper, boats, or other appropriate vessels

5.0 Procedure

5.1 Standards and Control

5.1.1 Class 1 Reference Standard Weights shall be used for QC checks and to determine the Uncertainty of Measurement for section balances. Class F Reference Standard Weights, or better, shall be used for bulk balances only.

5.1.2 The static weighing process shall be used for weighing events in the drug chemistry section, to include casework as well as monthly QC checks. See the Technical Procedure for Balances – Drug Chemistry.

5.2 Measurement Traceability

5.2.1 The traceability for the measurement process is established through the calibration of the balances and reference standard weights, which have been determined to have a significant effect on the test result and/or the total uncertainty.

5.2.2 See the current Drug Chemistry Section Annual Report for Uncertainty of Measurement for details.

5.3 Balance Measurement Assurance

5.3.1 All common use balances (analytical and bulk) as well as all individual top loading balances currently being used for case analysis shall be included in the data collection.

5.3.2 Each Forensic Scientist has access to a two decimal place electronic top loading balance that is used for most casework.

5.3.3 The section utilizes several four decimal place analytical balances.

5.3.4 The section utilizes either two or three decimal place bulk balances.

5.3.5 A single uncertainty of measurement value will be calculated for similar style balances with the same readability.

5.3.6 The process to determine the Uncertainty of Measurement for balances shall be conducted on a yearly basis according to the procedure outlined below.

5.3.6.1 Refer to **5.3.12** for the process of evaluating the uncertainty assessment for the inclusion of new balances or balances not included in the annual data collection study for use with casework.

5.3.7 In order to determine uncertainty for balances, several factors must be taken into consideration. The factors include but are not limited to:

5.3.7.1 The uncertainty of the measuring instrument (expressed as C1) shall be obtained from the statement of uncertainty from the approved vendor's current Calibration Report.

5.3.7.1.1 If the expanded uncertainty was reported on the certificate provided by the vendor, divide the expanded uncertainty value by the applied coverage factor, k.

5.3.7.2 The uncertainty of the item being measured (expressed as C2) shall be obtained from the approved vendor's current Calibration Report for the Class 1 Reference Standard Weights or Class F Reference Standard Weights (or better, bulk balances only).

5.3.7.2.1 If the expanded uncertainty was reported on the certificate provided by the vendor, divide the expanded uncertainty value by the applied coverage factor, k.

5.3.7.3 The uncertainty of human/environmental influences (expressed as C3) shall be obtained from the monthly QC check data performed in accordance with the Technical Procedure for Balances – Drug Chemistry, which is performed by the Forensic Scientists (or designees) in the Drug Chemistry Sections of the North Carolina State Crime Laboratory.

5.3.8 Calculations

5.3.8.1 Data collection and data analysis may be done in an Excel spreadsheet ("Determination of Uncertainty") due to the volume of data collected.

5.3.8.2 After completion of the data collection, the uncertainty of human/environmental influences (C3) shall be determined for each tested weight for all balances. The following equation shall be used to determine C3:

$$C3 = \frac{s}{\sqrt{n}}$$

Where s = standard deviation
Where n = number of measurements

5.3.8.3 Combined standard uncertainty: (u_c)

5.3.8.3.1 In order to accurately reflect the total uncertainty from all of the contributing factors, the following equation shall be used to determine the combined standard uncertainty (u_c):

$$u_c = \sqrt{[(C1)^2+(C2)^2+(C3)^2]}$$

Where: C1 = highest uncertainty of measuring device
C2 = highest uncertainty of items being measured
C3= highest uncertainty of human/environmental influences

5.3.8.4 Total Uncertainty: (u_{total})

5.3.8.4.1 Due to the static weighing process, the calculation of total uncertainty for the net mass is:

$$u_{total} = \sqrt{[2-2r_1]}*u_c$$

Where r_1 is the correlation coefficient between the weighing associated with the tare and the weighing of the material. Because this is a static weighing process, two separate weighing events are considered. Conservatively, r_1 will be assigned the value of -1, therefore:

$$u_{total} = 2*u_c$$

5.3.8.5 Expanded Uncertainties at an approximate 99.7% Confidence Level (U)

5.3.8.5.1 In order to determine the expanded uncertainty (U), the total combined standard uncertainty (u_{total}) shall be multiplied by a coverage factor (k), in order to state the uncertainty at an approximate 99.7% level of confidence.

$$U=k*u_{total}$$

Where k = coverage factor for an approximate 99.7% confidence level
u_{total} = the combined uncertainty for each type of measurement

5.3.9 The expanded uncertainties for each type of measurement on each type of balance (top loading/analytical/bulk) included in the uncertainty study shall be evaluated.

5.3.9.1 If the final expanded uncertainty (U) is greater than the readability of the balance, the value shall be rounded up and shall have the same number of digits as the balance is capable of reading.

5.3.9.2 If the final expanded uncertainty (U) is less than the readability of the balance, the readability of the balance shall be used as the final expanded uncertainty.

5.3.10 Reporting of Final Expanded Uncertainty for the Weighing Process

5.3.10.1 The Expanded Uncertainty for each type of balance (see above) shall be used to calculate the Final Expanded Uncertainty for the weighing process. This process is repeated when multiple units are weighed for a combined net weight. The following equation shall be used:

$U_{final} = \sqrt{(U_{balance}^2 \text{ x } N)}$ which can be simplified to

$U_{final} = \sqrt{N} \text{ x } U_{balance}$

Where:

U_{final} = Final expanded uncertainty for the weighing process
$U_{balance}$ = Expanded Uncertainty of the Balance
N = Number of weighing events
Approximate 99.7% Confidence Level using the appropriate coverage factor, k

5.3.10.2 The expanded uncertainty for the weighing process for the type of balance used shall be reported only with reported net weights, to include the coverage probability. The calculations shall be recorded in the case notes.

5.3.11 Documentation of Uncertainty Determination

5.3.11.1 The expanded uncertainty shall be updated annually, with all calculations verified by a second Forensic Scientist.

5.3.11.2 This shall be documented in the Drug Chemistry Section Annual Report for Uncertainty of Measurement, which shall include traceability maps.

5.3.12 Additional Assessments Utilizing C1

5.3.12.1 Newly obtained balances or balances which are not subjected to a complete year (twelve months) of QC checks (such as those out of service for a period of time, or balances not assigned) shall be QC checked according to Technical Procedure for Balances – Drug Chemistry. The balance shall then be evaluated to determine the impact, if any, on the current expanded uncertainty.

5.3.12.1.1 The evaluation shall include replacing the C1 value for the newly acquired balance into the associated balance budget worksheet, and observing the impact on the expanded uncertainty.

5.3.12.1.1.1 If there is found to be no impact, the balance shall be placed into service and shall assume the current expanded uncertainty for the balance type.

5.3.12.1.1.2 If there is found to be a significant impact, the balance shall be recalibrated by an external vendor, and then reassessed.

5.3.12.1.2 The balance budget(s) used for balances fitting **5.3.12.1** shall be added to the Resource Manager in Forensic Advantage and are not required to be added to the Drug Chemistry Section Annual Report for Uncertainty of Measurement.

5.4 Secondary Reference Standard Weight Measurement Assurance

5.4.1 The Balances Coordinator, or designee, shall record the weights of the Primary and Secondary Reference Standard Weights annually. Identifiers for the weights/weight sets and balances used shall also be documented.

5.4.2 The balances used shall be checked with the primary standard reference weights according to the monthly QC Check criteria listed in the Technical Procedure for Balances – Drug Chemistry before the secondary weights are checked.

5.4.3 A successful recheck will require the weights recorded for the secondary reference standard weights agree with expected values within the expanded uncertainty measurement as stated on the annual balance calibration certificate of the balance used.

5.4.4 Values recorded during the rechecks shall be verified by the Drug Chemistry Section Technical Leader, or designee.

6.0 Limitations – N/A

7.0 Safety – N/A

8.0 References

ASCLD/LAB Level 100A Traceability presentation. Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB.

ASCLD/LAB Level 100B Measurement Assurance presentation. Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB.

ASCLD/LAB Level 100C Measurement Uncertainty Concepts presentation. Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB.

ASCLD/LAB Level 200 Measurement Confidence for the Forensic Laboratory: Measurement Uncertainty in Drug Chemistry presentation. Copyright 2011; Heusser Neweigh, LLC & ASCLD/LAB

Clark, J.P. and Shull, A.H. *Evaluation of Methods for Estimating the Uncertainty of Electronic Balance Measurements*. Westinghouse Savannah River Company, 2002.

EURACHEM/CITAC Guide CG 4: *Quantifying Uncertainty in Analytical Measurement*, Third Edition 2012.

JCGM 100:2008 Evaluation of measurement data - Guide to the Expression of Uncertainty in Measurement, First Edition September 2008.

LeBeau, Marc. "Introduction to Measurement Uncertainty." RTI International. 2009.

Measurement Uncertainty for Weight Determinations in Seized Drug Analysis Supplemental Document SD-3 Revision 2. Copyright 2011; SWGDRUG.

Taylor, B.N, and Kuyatt, C.E. NIST Technical Note 1297 Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, September 1994 Edition.

Virginia Department of Forensic Sciences. Controlled Substances Procedure Manual. Document 221-D100 Revision 7, February 6, 2012.

9.0 Records

- Drug Chemistry Section Annual Report for Uncertainty of Measurement (includes traceability maps)
- Calibration Reports for section balances
- Reference Standard Weight Calibration Certificates
- Balance Uncertainty Budgets
- Drug Chemistry Measurement Assurance Cause and Effect Diagram
- Drug Chemistry Measurement Cause and Effect Appendix

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
12/16/2020	12	Definitions – Added coverage factor, coverage probability, and uncertainty of measurement 4.1 – Clarified types of balances 4.2 – Clarified types of weights and added weighing vessels 5.1.2 – Addition of static weighing process 5.2 – Added section for measurement traceability 5.3.1, 5.3.2, 5.3.3, 5.3.4, 5.3.5 – Added clarification for measurement assurance study 5.3.6.1 – Added line reference for evaluation of new balances 5.3.7.1.1, 5.3.7.2.1 – Added division of coverage factor k to calculations 5.3.7.3– Reworded process to include uncertainty of human/environmental influences 5.3.8.3 – Clarified calculations for combined standard uncertainty (u_c) and added that the highest C1, C2, and C3 shall be used 5.3.8.4 – Updated u to u_{total} 5.3.8.5 – Updated calculation of expanded uncertainty (U) 5.3.9 – Added instructions for rounding and number of digits 5.3.10 – Clarified references to confidence level/coverage factor 5.3.11 – Added requirements for documentation and verification of calculations 5.3.12 – Added section for evaluation of newly obtained balances 5.4 – Added designee and verification of recheck values Records – Added Balance Uncertainty Budgets, Cause and Effect Diagram, and Cause and Effect Appendix