1.0 Purpose - This procedure specifies the required elements for measurement assurance of weight determinations.

2.0 Scope – This procedure applies to electronic balances used in the Drug Chemistry and Blood Alcohol Concentration (BAC) sections of the Pitt County Sheriff's Office Forensic Services Unit.

3.0 Definitions

- **Approved Vendor –** Supplier of a product or service that meets ISO/IEC 17025:2017 – Forensic Science Testing and Calibration Laboratories Accreditation Requirements
- **Calibration –** Checking or adjusting (by comparison with a standard) the accuracy of a measuring instrument. Calibrations are performed by approved vendors for all balances in the Pitt County Sheriff's Office Forensic Services Unit.
- **Calibration Verification (QC check) –** Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
- **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.
- **Measurement** – A process of experimentally obtaining one or more quantity values, typically of physical, chemical, or biological nature. Implies comparison of quantities.
- **Measurement Traceability** – an unbroken chain of comparisons (using acceptable and documented methods) to national or international standards (SI) with each comparison having stated uncertainties.
- **Reference standard** – measurement standard designated for the calibration of other measurement standards (reference standards or equipment)
- **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

Mettler Electronic analytical balance - XS204 (BAC only)
Mettler Electronic analytical balance - XS204DR
Mettler Electronic table top balance - XS6002-S

Mettler Electronic table top balance - XSR6002-S
Fisher Electronic table top balance - Accu-2202
Ohaus Electronic bulk scales - B300BX Base with T31P Indicator

4.2 Materials and Reagents

Weighing paper and boats
Plain paper, cardboard boxes, plastic bags, flasks or other appropriate weighing vessels
Class 2 primary reference standard weights
Class F primary reference standard weights (bulk scales only)

5.0 Procedure

5.1 Measurement Traceability –

5.1.1 The traceability for this measurement process is established through the calibration of the balances and the primary reference standard weights used to confirm the continued calibration status of the balances. (For details see the current Drug Chemistry Section Traceability Map for Balances and Weights, stored in DM in the "Balance Study" folder for the current year.)

5.1.2 The calibration of the balances and primary reference standard weights was determined to have a significant effect on the accuracy and validity of the test result. Both types of calibration are completed annually by approved vendors that meet the requirements specified by the accrediting body.

5.2 Measurement Assurance –

5.2.1 Intermediate checks are needed to maintain confidence in the calibration status of the balances during the interval between external calibrations. These checks are carried out according to a defined schedule and procedure using calibrated mass reference standards (refer to the Drug Chemistry Section Technical Procedure for Balances).

5.2.2 Data is available from a measurement assurance program that evaluates the variation in the measurement process using reference standard weights. All Forensic Scientists participate. All balances are used as are all methods of weighing allowed by the test method procedure.

5.3 The Drug Chemistry Technical Leader shall determine an estimation of the uncertainty of measurement (UofM) for weight determinations in the Drug Chemistry Section according to the *NIST.IR.6969 Selected Laboratory and Measurement Practices and Procedures to Support Basic Mass Calibrations. SOP 29 Standard Operating Procedure for the Assignment of Uncertainty*. The balances and scales used for a reported test result must be evaluated in the estimation of the UofM for weight determinations.

5.4 Individual sources of uncertainty that are not significant contributors may be excluded.

5.5 When established, the estimation of the UofM shall be performed annually, at a minimum, or when a change in measurement conditions occurs that may have a significant effect on the UofM. (See the Measurement Cause and Effect Diagram in DM)

5.6 Laboratory environmental conditions shall be monitored and any additional effect on UofM shall be evaluated upon collection of data. See the Drug Chemistry Technical Procedure for Balances for details on the collection of data (yearly balance study and monthly QC checks.)

5.7 The Drug Chemistry Section Technical Leader shall maintain records of the estimation of the uncertainty of measurement in the Drug Chemistry Section. The BAC Technical Leader shall maintain records of the estimation of the uncertainty of measurement in the BAC Section. The records shall be stored in DM.

5.8 **Step 1: Specify the Measurement Process**

5.8.1 Weight determinations are performed by multiple Forensic Scientists, using electronic balances according to the Drug Chemistry Technical Procedure for Balances.

5.8.1.1 Each Forensic Scientist has a two decimal place electronic balance at their workstation that is used for most cases. *Mettler* XS6002-S *Mettler* XSR6002-S, and *Fisher* Accu-2202.

5.8.1.2 A four decimal place analytical balance (*Mettler* XS204DR) and a set of bulk scales (*Ohaus* B300BX Base with T31P Indicator) are shared by the Drug Chemistry Section.

5.8.1.3 A four digit electronic balance (*Mettler* XS204) is used only by the Blood Alcohol Concentration (BAC) Section. (No weights are reported for casework purposes.)

5.8.2 The procedure includes the use of gross and net weights and the use of weighing vessels. The yearly balance study and the monthly QC checks incorporate weighing vessels selected to mimic the weighing vessels utilized by the Forensic Scientists.

5.8.3 The procedure describes static weighing events for the Drug Chemistry Section, where the Forensic Scientist(s) remove the tared weighing vessel from the balance, add the measurand to the weighing vessel, and return it to the balance. The BAC Section uses dynamic weighing for casework and monthly QC checks.

5.8.3.1 The static weighing process involves two weighing events (Drug Chemistry). The dynamic weighing process involves one weighing event (BAC).

5.8.3.2 The measurement assurance yearly ten day balance study and the monthly QC checks incorporate static types (Drug Chemistry) and dynamic types (BAC) of weighing events, including tare vessels, to mimic casework.

5.8.4 The measurement of the weight is not made directly. Weight is determined through a functional relationship based on the amount of force on the balance. The functional relationship can be expressed by the mathematical equation:

$$y = (mx + b) \pm U$$

Where,

y = the balance reading (or indication)
m = sensitivity of the weighing device
x = the applied load
b = zero offset, (or bias)
U = assigned measurement uncertainty (expanded uncertainty)

Ideally, b = 0 if the balance was properly zeroed, and m = 1 because the balance indicates one mass unit for each mass unit applied.

5.9 Step 2: Identify uncertainty components

5.9.1 Balance / Scale

5.9.1.1 Display resolution – impact of rounding at zero and at load on the value displayed
5.9.1.2 Balance calibration uncertainty
5.9.1.3 Balance linearity
5.9.1.4 Balance bias

5.9.2 Staff

5.9.2.1 Multiple Forensic Scientists
5.9.2.2 Training
5.9.2.3 Experience

5.9.3 Test method

5.9.3.1 Static process involving two weighing events (Drug Chemistry)
5.9.3.2 Dynamic process involving one weighing event (BAC)
5.9.3.3 The buoyant effect of weighing the measurand in air
5.9.3.4 Differences in centering of measurand on the balance
5.9.3.5 Weighing vessel

5.9.4 Facility

5.9.4.1 Temperature variation of laboratory and difference from the temperature during calibration
5.9.4.2 Drafts
5.9.4.3 Vibration
5.9.4.4 Humidity
5.9.4.5 Static electricity

5.10 Step 3: Quantify uncertainty components

5.10.1 Data is available from an ongoing balance study that evaluates the variation in the measurement process using reference standard weights that reasonably mimic the weight determination process. All balances are used, as are the tare vessels and method of weighing allowed by the test method procedure. All Forensic Scientists participate. The data spans the work day including all days of the work week and normal laboratory environmental conditions. (See the Technical Procedure for Balances – Yearly Balance Study)

5.10.2 Data is available from ongoing monthly quality control checks using calibrated primary reference standard weights to maintain confidence in the calibration status of the balance during the interval between external calibrations. Tare vessels are used to mimic casework. (See the Technical Procedure for Balances - Monthly QC checks).

5.10.3 The uncertainty components and how they will be evaluated:

Uncertainty Component	Method of Evaluation
Measuring Equipment – Balance/Scale	
• Display resolution – impact of rounding at zero and at load on the value displayed	Type A Evaluation – covered in Balance Study (statistical method)
• Balance calibration uncertainty	Type B Evaluation - covered in balance calibration (scientific judgement)
• Balance linearity	Type B Evaluation
• Balance bias	Type B Evaluation
Staff	
• Multiple Forensic Scientists	Type A Evaluation
• Training	Type A Evaluation
• Experience	Type A Evaluation
Test Method	
• Static process involving two weighing events (Drug Chemistry)	Type A Evaluation
• Dynamic process involving one weighing event (BAC)	Type A Evaluation
• The buoyant effect of weighing the measurand in air	Type B Evaluation
• Differences in centering of measurand on the balance	Type A Evaluation
• Weighing vessel	Type A Evaluation
Facility	
• Temperature variation	Type A Evaluation
• Air flow	Type A Evaluation
• Vibration	Type A Evaluation
• Humidity	Type A Evaluation
• Static Electricity	Type A Evaluation

5.10.4 Type A Evaluation of uncertainty components:

5.10.4.1 Balance study and monthly QC checks

5.10.4.1.1 Measurements are collected monthly, and for ten business days during the balance study, which is conducted once per calendar year. The process has been evaluated and the balance accuracy verified through primary reference standard weight checks. To more closely represent the process, only the balance study measurements and monthly QC check data from the last year will be used for all balances. A period of one year encompasses all seasonal variations in environmental factors.

5.10.4.1.2 XS204, XS204DR, XS6002S, XSR6002S, and ACCU 2202, B300BX. The statistic that will be calculated for all occurrences for each weight on each balance is the standard deviation.

5.10.4.1.3 Standard Deviation (sample)

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

5.10.5 Type B Evaluation of uncertainty components:

5.10.5.1 Balance calibration uncertainty

5.10.5.1.1 XS204, XS204DR, XS6002S, XSR6002S, and ACCU 2202 and B300BX

- Review the calibration certificate from the approved vendor external calibration laboratory and identify the greatest calibration expanded uncertainty at a confidence level of at least 95% (k=2).

5.10.5.2 Balance linearity:

5.10.5.2.1 XS204, XS204DR, XS6002S, XSR6002S, and ACCU 2202 and B300BX

- Balance linearity is determined during the balance calibration and is factored into the uncertainty provided by the approved vendor performing the calibration.

5.10.5.3 Balance bias:

5.10.5.3.1 Ongoing quality control checks of balance to maintain confidence in calibration status using calibrated primary reference mass standards provides an ongoing evaluation of bias.

5.10.5.4 The buoyant effect of weighing the measurand in air:

5.10.5.4.1 Refer to SWGDRUG, **Supplemental Document SD-3 for Part IVC – Quality Assurance / Uncertainty Measurement Uncertainty for Weight Determination in Seized Drug Analysis.** (SWGDRUG 2011-07-07). "Buoyancy is difficult to account for in seized drug cases because the density of the material being weighed must be known. However, for material that has a lower density than the steel calibration weights (8.0 g/cm^3) the bias imparted is always negative and the weight displayed by the balance will be less than the true weight of the material. Ignoring buoyancy contributes a small systematic error that represents no more than 0.1 % bias to the weight." Therefore, the buoyant effect of weighing the measurand in air is not a significant contributor and will not be included.

5.11 Step 4: Convert quantities to standard uncertainties (*u*)

5.11.1 Type A Evaluation of uncertainty components:

5.11.1.1 Balance study data

5.11.1.1.1 XS204 and XS204DR

- The measurement unit is the gram.
- Reproducibility data from the balance study is expressed as one standard deviation.

5.11.1.1.2 XS6002S, XSR6002S and ACCU-2202

- The measurement unit is the gram.
- Reproducibility data from the balance study is expressed as one standard deviation using the highest value from the balances.

5.11.1.1.3 B300BX

- The measurement unit is the kilogram.
- Reproducibility data from the balance study is expressed as one standard deviation.

5.11.2 Type B Evaluation of uncertainty components:

5.11.2.1 Balance calibration uncertainty:

5.11.2.1.1 XS204, XS204DR, XS6002S, XSR6002S, ACCU-2202, and B300BX

- The uncertainty on the calibration certificate will be divided by the coverage factor utilized by the approved vendor performing the calibration, to arrive at a standard uncertainty.

5.11.2.2 Balance linearity:

5.11.2.2.1 XS204, XS204DR, XS6002S, XSR6002S, and ACCU-2202, B300BX

- Balance linearity is determined during the balance calibration and is factored into the uncertainty provided by the approved vendor performing the calibration.

5.11.2.3 Quantifying the uncertainty components:

5.11.2.3.1 The greatest calibration expanded uncertainty of the mass reference standards was determined from the calibration certificates. The range of measurements for each balance is considered in determining this value.

5.11.2.4 Convert quantities to standard uncertainties

5.11.2.4.1 The calibration certificates and/or approved vendor documentation indicate that the expanded uncertainty assumes a normal distribution using the coverage factor utilized by the approved vendor performing the calibration. The uncertainty on the calibration certificate will be divided by the utilized coverage factor to arrive at a standard uncertainty for each of the balances.

5.12 Step 5: Calculate the combined standard uncertainty (u_c)

5.12.1 The combined standard uncertainty equals the square root of the sum of each standard uncertainty squared.

5.12.2 XS204 (BAC), XS204DR, XS6002S, XSR6002S, ACCU-2202, and B300BX

$u_c = \sqrt{u_{calibration\,unc}^2 + u_{CalRefStd}^2 + s_{process}^2}$ or for Excel purposes:

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

Where C1 = uncertainty of measuring equipment
C2 = uncertainty of weights being measured
C3 = uncertainty of human/environmental influences

5.12.3 The impact to the calculated combined standard uncertainty can only be seen when additional digits are shown beyond two significant digits.

5.13 Step 6: Expand the combined standard uncertainty (u_c) by the coverage factor (k) to calculate the Expanded combined standard uncertainty ($U_{balance}$):

5.13.1 The expanded combined standard uncertainty is the product of a combined standard measurement uncertainty and a coverage factor larger than the number one.

5.13.2 XS204 (BAC), XS204DR, XS6002S, XSR6002S, ACCU-2202 and B300BX

$$U_{balance} = u_c \times k$$

Where k = a coverage factor for a 99.73% confidence level
u_c = the combined uncertainty for each weight on each balance

5.13.2.1 The data from the measurement process is assumed to follow a normal distribution. To expand the uncertainty to a 99.73% coverage probability the coverage factor k will be calculated for the given measurements and degrees of freedom.

5.14 Step 7: Evaluate the expanded uncertainty ($U_{balance}$)

5.14.1 The uncertainty calculations shall be reviewed for accuracy. The expanded uncertainties should be less than the minimum sample loads in order for the expanded uncertainties to be determined to be acceptable.

5.14.2 The highest value for the expanded uncertainty for the bench top balances shall be used for reporting the expanded uncertainty for all bench top balances.

5.15 Step 8: Report the expanded uncertainty (U_{final})

5.15.1 The expanded uncertainty is reported for net weights of analyzed material. When a gross weight or a weight associated with unanalyzed material is reported the expanded uncertainty shall not be included on the report.

5.15.2 When weights are added to produce a total weight, the uncertainty is propagated. For this type of addition, the combined standard uncertainty is found by squaring the uncertainties, adding them all together, (or multiplying by the number of weighings), and then taking the square root of the total.

$U_{final} = \sqrt{(U_{balance})^2 \text{ x N}}$ 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 weighings
99.7 % Confidence Level using a coverage factor (k) and normal distribution

5.15.3 The reported expanded uncertainty will be rounded up and contain at most two significant digits. The reported expanded uncertainty will be reported to the same level of significance as the reported weight.

5.15.4 The laboratory report shall identify the measured quantity value, *y*, along with the associated U_{final}. The result shall be reported as $y \pm U_{final}$, with the units of U_{final} consistent with the units of *y*. The coverage probability shall be included.

5.15.5 Examples:

Table top balances:
Net weight of material – XX.XX (+/- 0.0X) grams (confidence level 99.7%)

Analytical balance *XS204DR:
Net weight of material – XX.XXXX (+/- 0.000X) grams (confidence level 99.7%)
*Yearly uncertainty calculations shall be conducted for only the lower range of the dual range balance XS204DR. The upper range shall not be used for reported weights.

Bulk scales:
Net weight of material – XX.XXX (+/- 0.XXX) kilograms (confidence level 99.7%)

6.0 References

SWGDRUG Supplemental Document SD-3 for Part IVC – Quality Assurance / Uncertainty Measurement Uncertainty for Weight Determination in Seized Drug Analysis. (SWGDRUG 2011-07-07).

NIST.IR.6919 Recommended Guide for Determining and Reporting Uncertainties for Balances and Scales. (January 2002 Ed) https://www.nist.gov/sites/default/files/documents/2017/04/28/NISTIR6919.pdf

NIST.IR.6969 Selected Laboratory and Measurement Practices and Procedures to Support Basic Mass Calibrations. SOP 29 Standard Operating Procedure for the Assignment of Uncertainty (pages 166-177). (2019 Ed) https://doi.org/10.6028/NIST.IR 6969-2019.

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Operator manuals for each balance model.

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

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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.
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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.

7.0 Records

- Measurement Cause and Effect Diagram in Document Management DM
- Traceability Map for Balances and Weights in DM
- Certificates of calibration for balances in DM
- Certificates of calibration for primary reference standard weights in DM
- Monthly QC Check data in DM
- Uncertainty of measurement budgets (Balance Study Data) in DM
- Measurement Assurance Yearly Report in DM

Revision History		
Current Version	**Effective Date**	**Summary of Changes**
1	2017/11/14	Original Document.
2	2018/01/03	**5.2.9** – Added "type of" and "for each discipline" for reported value.
3	2018/04/01	**Header** – Added "Drug Chemistry" Technical Leader Entire document – Updated "Illicit Drugs" to "Drug Chemistry" "section" instead of discipline **5.2.5 & 5.2.6** – Added "During the ten day data collection"
4	2018/10/22	**Header –** Updated to match other section documents **Equipment** – Added new balance that is currently on order. **5.2.2 –** Changed factors to components of significance. **5.2.2.3 and 5.2.9** – Updated "Illicit Drugs" to "Drug Chemistry" "section" instead of discipline **5.2.2.3.1.6** – Added Operator competence and experience. **5.2.8.4.2** – Added rules for rounding of final expanded uncertainty. **5.2.10.2** - Added coverage probability to reporting requirements, and requirement for same number of decimal places as the balance used. Reporting examples updated. **Records** – Updated references to DM and added Traceability Map for Balances and Weights, Measurement Assurance Cause and Effect Diagram, and Uncertainty Study Data
5	2019/11/19	**ARCHIVED all of Version 4** Started a new document based on *NIST.IR.6969 Selected Laboratory and Measurement Practices and Procedures to Support Basic Mass Calibrations. SOP 29 Standard Operating Procedure for the Assignment of Uncertainty (pages 166-177). (2019 Ed)*
6	2019/11/21	**5.14.2** Added statement reference using the highest expanded uncertainty of the bench top balances to report expanded uncertainty for all bench top balances.