	Instruments Pitt County Sheriff's Office Forensics Services Unit Issued by the Drug Chemistry Technical Leader	Effective Date: 2018/10/22	Ver.: 4
Technical Procedure for Measurement Assurance			

- **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 sections of the Pitt County Sheriff's Office Forensic Services Unit.

#### 3.0 Definitions

- 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.
- **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 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)

# 4.0 Equipment, Materials and Reagents

## 4.1 Equipment

Mettler Electronic analytical balance (XS204DR, XS204)

Mettler Electronic table top balance (XS6002-S)

*Mettler Electronic table top balance (XSR6002-S)* 

Fisher Electronic table top balance (Accu-2202)

Ohaus Electronic bulk balance (300BX Base with T31P Indicator)

# 4.2 Materials and Reagents

Class 2 Reference Standard Weights

Class F Reference Standard Weights (may be used for Bulk balance only)

#### 5.0 Procedure

#### 5.1 Standards and Control

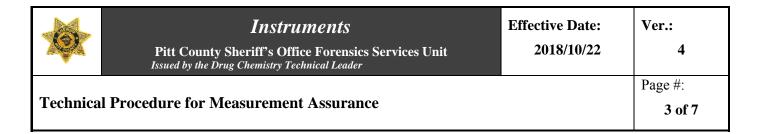
- **5.1.1** Class 2 reference standard weights shall be used for Monthly QC checks and to determine the Uncertainty of Measurement for section balances. Class F Reference Standard Weights shall be used for the bulk balance only.
- **5.1.2** Secondary reference standard weights may be used for the Daily QC checks.

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- **5.1.2.1** The chemist shall record the weights of the primary and secondary reference standard weights annually after the balance and primary weights have been calibrated by an approved vendor. Identifiers for the weights and balances used shall also be documented on data collection form.
- 5.1.2.2 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.

## **5.2** Section Balances

- 5.2.1 The process to determine the Uncertainty of Measurement for balances shall be conducted on a yearly basis for ten business days according to the procedure outlined below.
- **5.2.2** In order to determine uncertainty for balances, several factors must be taken into consideration. The components of significance include:
  - 5.2.2.1 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. **NOTE:** If the expanded uncertainty was reported on the certificate provided by the vendor, divide the expanded uncertainty value by the coverage factor K.
  - **5.2.2.2** The uncertainty of the item being measured (expressed as C2) shall be obtained from the approved vendor's current Calibration Report for the Class 2 Reference Standard Weights or Class F Reference Standard Weights (bulk balance only).
  - 5.2.2.3 The uncertainty of human/environmental influences (expressed as C3) shall be obtained from the data collection performed by the Chemists in the Drug Chemistry and Blood Alcohol sections of the Pitt County Sheriff's Office Forensic Services Unit on an annual basis.
    - **5.2.2.3.1** These factors include, but are not limited to:
      - 5.2.2.3.1.1 Position and leveling of the balance
        5.2.2.3.1.2 Position of weight on the balance pan
        5.2.2.3.1.3 Draft
        5.2.2.3.1.4 Ambient temperature changes



# **5.2.2.3.1.6** Operator competence and experience

- **5.2.3** All balances being used for case analysis shall be included in the data collection.
- 5.2.4 If a Chemist is out of the office for a partial day or partial week during the data collection period, a substitute Chemist shall collect data on that individual's balance.
- 5.2.5 During the ten day data collection the monthly QC check shall be performed at the start of each work day in accordance with the Technical Procedure for Balances, but shall be documented with the replicate weight determinations.
- 5.2.6 During the ten day data collection in addition to the monthly QC check, each morning and afternoon three replicate weight determinations shall be obtained for two reference standard weights. The Chemist performing the determination shall record these values on a data collection sheet along with the identifier for each weight used.
- **5.2.7** The standard deviation of all occurrences for each weight on each balance shall be used.

#### 5.2.8 Calculations

- 5.2.8.1 Data collection and data analysis may be done in an Excel spreadsheet ("Determination of Uncertainty") due to the volume of data collected.
- **5.2.8.2** After completion of the data collection, the uncertainty of human/environmental influences (C3) shall be determined. The following equation shall be used to determine C3:

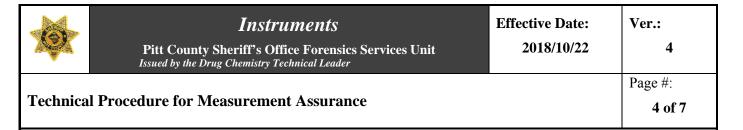
C3 = 
$$\frac{s}{\sqrt{n}}$$
 Where s = standard deviation  
Where n = number of measurements

- **5.2.8.3** Combined Uncertainties (CU)
  - **5.2.8.3.1** In order to accurately reflect the total uncertainty from all of the contributing factors, the following equation shall be used to determine the combined uncertainty (u):

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

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

- **5.2.8.4** Expanded Uncertainties at 99.7 % Confidence Level (EU)
  - **5.2.8.4.1** In order to determine the expanded uncertainty (EU), the combined uncertainty (CU) shall be multiplied by a coverage



factor (k) of 3, which states the uncertainty at a 99.7 % level of confidence.

EU = k \* CU

Where

k = a coverage factor of 3 for a 99.7 % confidence level
 CU = the combined uncertainty for each type of measurement

- **5.2.8.4.2** If the final expanded uncertainty is greater than the readability of the balance, the final value shall be rounded up. If this number is less than the readability of the balance, then the readability of the balance shall be used as the final expanded uncertainty.
- 5.2.9 The expanded uncertainties for each type of measurement on each balance included in the uncertainty study shall be evaluated. The highest value for each type of balance in each section shall be used as the reported value. These values shall be updated annually and used as directed in the technical procedure for each type of measurement.
  - **5.2.9.1** Calculations shall be verified by a second Chemist and documented on the yearly summary chart for each type of uncertainty.
- **5.2.10** Reporting of Final Expanded Uncertainty for the Weighing Process
  - 5.2.10.1 The Expanded Uncertainty for each 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:

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

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

Where:

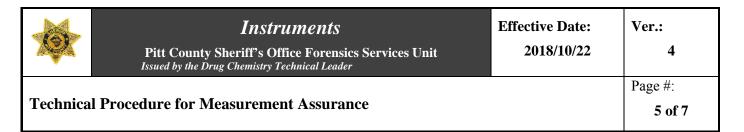
U<sub>final</sub> = Final expanded uncertainty for the weighing process

 $U_{\text{balance}} = \text{Expanded Uncertainty of the Balance}$ 

N = Number of weighings

99.7 % Confidence Level using k=3 coverage factor and normal distribution

5.2.10.2 The expanded uncertainty for the weighing process for the balance used shall be reported with the results of reported net weights and include the coverage probability. In addition, the expanded uncertainty shall be reported



to the same number of decimal places as the readability of the balance used. The calculations shall be recorded in the case notes.

# **5.2.10.2.1** Top loading balances:

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

## **5.2.10.2.2** Analytical balances

Lower range:

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. The upper range shall not be used for reported weights.

# **5.2.10.2.3** Bulk balance:

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

- **5.2.11.2.3.1** A notice shall be posted near the bulk balance stating the range for which the balance has been calibrated and certified for use, as needed.
- 5.2.10.2.4 Gross weights shall not require a reported uncertainty and shall be truncated to the 0.1 place (table top balances) or whole number (bulk balance).
- **6.0** Limitations N/A
- **7.0 Safety** Make sure balance is plugged in and is not near a source of water.

# 8.0 References

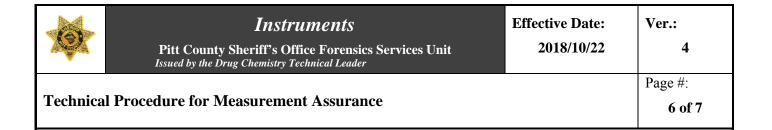
Operator manuals for each balance model.

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

- Yearly Report for Balances Measurement Assurance in Document Management (DM)
- Certificates of calibration for balances in DM
- Certificates of calibration for reference standard weights in DM
- Annual secondary reference weight recheck report in DM
- Measurement Cause and Effect Diagram in DM
- Traceability Map for Balances and Weights in DM
- Uncertainty Study Data in DM



# Instruments

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**Technical Procedure for Measurement Assurance** 

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REVISION HISTORY					
CURRENT VERSION	EFFECTIVE DATE	SUMMARY OF CHANGES			
1	2017/11/14	Original Document.			
2	2018/01/03	<b>5.2.9</b> – 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			