

## 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

- 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.

### 4.0 Equipment, Materials and Reagents

#### 4.1 Equipment

- Balances (individual, analytical, and bulk)

#### 4.2 Materials and Reagents

- Class 1 Reference Standard Weights
- Class F Reference Standard Weights (may be used for Bulk balances only)

### 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 shall be used for bulk balances only.

#### 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. These factors include but are not limited to:

- 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.
- 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 1 Reference Standard Weights or Class F Reference Standard Weights (bulk balances only).
- 5.2.2.3** The uncertainty of human/environmental influences (expressed as C3) shall be obtained from the data collection performed by the Forensic Scientists in the Drug Chemistry Sections of the North Carolina State Crime Laboratory on an annual basis.
- 5.2.2.3.1** These factors include, but are not limited to:
- Position and leveling of the balance
  - Position of weight on the balance pan
  - Draft
  - Ambient temperature changes
  - Vibration
- 5.2.3** 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.
- 5.2.4** A rotation list of Forensic Scientists shall ensure that multiple users contribute to the data collection of common use balances.
- 5.2.5** If a Forensic Scientist is out of the office for a partial day or partial week during the data collection period, a substitute Forensic Scientist shall collect data on that individual's balance.
- 5.2.6** The monthly QC check shall be performed at the start of each work day in accordance with the [Drug Chemistry Technical Procedure for Balances](#), but shall be documented with the replicate weight determinations.
- 5.2.7** In addition to the monthly QC check, each morning and afternoon three replicate weight determinations shall be obtained for two reference standard weights. The Forensic Scientist performing the determination shall record these values on a data collection sheet along with the identifier for each weight used.
- 5.2.7.1** The specific weights used for each type of balance will depend on certified reference weights at the respective laboratories.
- 5.2.8** The standard deviation of all occurrences for each weight on each balance shall be used.

## 5.2.9 Calculations

**5.2.9.1** Data collection and data analysis may be done in an Excel spreadsheet (“Determination of Uncertainty”) due to the volume of data collected.

**5.2.9.2** 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:

$$C3 = \frac{s}{\sqrt{n}} \quad \begin{array}{l} \text{Where } s = \text{standard deviation} \\ \text{Where } n = \text{number of measurements} \end{array}$$

**5.2.9.3** Combined Uncertainties (u)

**5.2.9.3.1** 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):

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

Where

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

**5.2.9.4** Expanded Uncertainties at 99.7 % Confidence Level (U)

**5.2.9.4.1** In order to determine the expanded uncertainty (U), the combined uncertainty (u) shall be multiplied by a coverage factor (k) of 3, which states the uncertainty at a 99.7 % level of confidence.

$$U=ku$$

Where

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

**5.2.10** 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. The highest value for each type of balance (top loading/analytical/bulk) shall be used as the section value. These values shall be updated annually and used as directed in the Drug Chemistry technical procedure for each type of measurement.

**5.2.10.1** Calculations shall be verified by a second Forensic Scientist and documented on the yearly summary chart for each type of uncertainty.

**5.2.11** Reporting of Final Expanded Uncertainty for the Weighing Process

- 5.2.11.1** 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:

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

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

Where:

$U_{\text{final}}$  = Final expanded uncertainty for the weighing process

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

N = Number of weighings

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

- 5.2.11.2** The expanded uncertainty for the weighing process for the type of balance used shall be reported with the results of reported net weights. The calculations shall be recorded in the case notes.

- 5.2.11.2.1** Top loading (individual) balances (lower range):  
Net weight of material – XX.XX (+/- 0.0X) grams

When the upper range of a top loading balance is triggered, the readout drops to the one tenths place. Yearly uncertainty calculations are based on the lower range; therefore, any evidence which triggers the upper range shall be weighed on a bulk balance.

- 5.2.11.2.2** Analytical balances:  
Net weight of material – XX.XXXX (+/- 0.000X) grams

- 5.2.11.2.3** Bulk balances:  
(Raleigh/Western Regional Laboratories)  
Net weight of material – XX.XX (+/- 0.0X) kilograms  
OR  
(Triad Laboratory)  
Net weight of material – XX.XXX (+/- 0.XXX) kilograms

- 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.

- 5.2.11.2.4** Gross weights shall not require a reported uncertainty and shall be truncated to the 0.1 place (table top balances) or whole number (bulk balances).

- 5.2.12** Annual Secondary Reference Standard Weight Checks

- 5.2.12.1** The Balances Coordinator shall record the weights of the Primary and Secondary Reference Standard Weights annually. Identifiers for the weights and balances used shall also be documented.
- 5.2.12.2** 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](#) before the secondary weights are checked.
- 5.2.12.3** 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.

**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**

- Determination of Uncertainty - Yearly Report for Balances
- Calibration Reports for section balances
- Traceability Maps
- Reference Standard Weight Calibration Certificates

**10.0 Attachments – N/A**

<b>Revision History</b>		
Effective Date	Version Number	Reason
09/17/2012	1	Original Document
5/10/2013	2	<p><b>Original 5.1-</b> Moved first statement to apply to Section Balances only. References to traceability maps removed, since this data covered by calibration certificates</p> <p><b>Original 5.2, 5.3 – N/A</b> sections removed</p> <p><b>5.2 –</b> Section renamed and edited to cover Section Balances only</p> <p><b>5.2.2.1 –</b> Removed normalization calculation that is not needed</p> <p><b>5.2.2.2 –</b> Added reference to Class A Reference Standard Weights</p> <p><b>5.2.2.3.1 –</b> Section moved from below</p> <p><b>5.2.3 – 5.2.9 –</b> Sections moved from below; Weights used will depend on availability at laboratories</p> <p><b>5.2.9.1 –</b> Added name of Excel spreadsheet used to calculate data</p> <p><b>5.2.11 –</b> Added clarification on types of balances</p> <p><b>5.2.11.1 –</b> Corrected reference line number</p> <p><b>5.3, 5.4 -</b> Revised to “Currently being established”</p> <p><b>5.5 –</b> Added explanation for Methamphetamine Quantitations via HPLC</p> <p><b>Original 5.7 –</b> Removed section for GHB Quantitations</p> <p><b>5.5.7.1 -</b> Added process evaluated annually</p> <p><b>References –</b> Added EURACHEM/CITAC... Taylor..., and JCGM...references</p> <p><b>Records –</b> Removed Measurement Assurance Cause &amp; Effect Diagram, added Uncertainty Budget for the HPLC Quantitation of Methamphetamine and HPLC Control Charts</p>

06/14/2013	3	<p>Removed Technical in title  <b>5.2.10</b> - Removed last sentence  <b>5.3</b> - Inserted explanation of measurement uncertainty as it relates to Alcohol/Acetone Concentration Determinations  <b>References</b> - Added Garriott, Gullberg, Jones, Kristiansen, LeBeau, Sklerov, and Tilson  <b>Records</b> – Added Uncertainty Budget for the Quantitation of Ethanol, Methanol, Isopropanol, and Acetone using Headspace Gas Chromatography and Toxicology Control Charts</p>
10/16/2013	4	<p><b>5.2.11.2.1</b> – Reworded to remove use of upper range on table top balances  <b>5.2.11.2.3</b> – Clarified nonuse of upper ranges on bulk balances  <b>5.2.11.2.4</b> – Deleted unneeded reference to range of balance  <b>5.2.12</b> – Added section to clarify successful annual Secondary Reference Weight check; added issuing authority to header</p>
12/18/2013	5	<p><b>4.2, 5.1.1, 5.2.2.2</b> – Typo corrected for Class 1 Weights  <b>5.3.2</b> – Reworded statement reference calibration curve  <b>5.3.3</b> – Reworded statement reference validity of calibration curve</p>
03/14/2014	6	<p><b>4.1</b> – Removed HPLC from Equipment list  <b>5.1.3</b> – Removed reference to HPLC Standards and Controls  <b>5.4</b> – Amended section to include discussion of measurement uncertainty as it relates to Cannabinoid Concentration Determinations.  Records - Added Uncertainty Budget for the Quantitation of Cannabinoids in blood using Liquid-Liquid Extraction and LC-MS/MS  <b>5.5</b> – Removed section for HPLC Quantitations via HPLC  <b>Records</b> – Removed Uncertainty Budget for HPLC and HPLC control charts</p>
08/29/2014	7	<p><b>5.2.11.1</b> – replaced incorrect line reference with “(see above)”  <b>5.3.3</b> – 6 % changed to 5 %  <b>Records</b> – added Traceability Maps</p>
02/27/2015	8	<p><b>5.2.11.2.3</b> – Adjusted two and three digit options.</p>
04/06/2016	9	<p>Removed all portions of document that pertain to Toxicology Analysis due to division of Drug Chemistry Toxicology into separate sections within the laboratory system.  <b>4.1</b> – Removed Toxicology equipment  <b>4.2</b> – Removed Toxicology materials and reagents  <b>5.1.2, 5.3, 5.4</b> – Removed Toxicology related sections  <b>References</b> – Removed Toxicology related  <b>Records</b> – Removed Toxicology related  <b>5.2.1</b> – Removed “consecutive” in case a day gets accidentally skipped during the yearly balance study.</p>

---

07/01/2016	10	<b>5.2.12.1</b> – Changed secondary reference weight check from month of September to annually.
11/14/2016	11	<b>Header</b> – updated issuing authority <b>4.2, 5.1.1</b> and <b>5.2.2.2</b> – Added Class F Reference Standard Weights (bulk balances only) <b>5.2.9.1</b> - Reworded <b>5.2.11.2.4</b> –Clarified balances for gross weights <b>4.1, 5.2.1, 5.2.12.1</b> – Removed “section”