Technical 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

- Section balances (individual, analytical, and bulk)
- Liquid Handling System
- Headspace GC
- Toxicology GC-MS
- HPLC

4.2 Materials and Reagents

- Class A Reference Standard Weights
- Primary Reference materials
- Volumetric flasks
- Class A Pipettes
- Glassware

5.0 Procedure

5.1 Standards and Control

5.1.1 The process to determine the Uncertainty of Measurement shall be conducted on a yearly basis according to the procedure outlined herein.

5.1.2 Class A Reference Standard weights shall be used for QC checks and to determine the Uncertainty of Measurement for section balances.

5.1.3 See Drug Chemistry Balances Traceability Map for references back to NIST standards.

5.1.4 Primary reference standards, primary reference materials, Class A pipettes and Class A volumetric flasks shall be used for Section Blood Alcohol Quantitations, Cannabinoid Quantitations, and GHB Quantitations.

5.1.5 See Toxicology *Blood Alcohol Traceability Map* for references back to NIST standards.

5.1.6 See *Cannabinoid Traceability Map* for references back to NIST standards.

5.1.7 See *GHB Traceability Map* for references back to NIST standards.

5.1.8 Primary reference materials, Class A pipettes, Class A volumetric flasks and graduated cylinders shall be used for Section Methamphetamine Quantitations via HPLC.

5.1.9 See *Methamphetamine Traceablility Map* for references back to NIST standards.

5.2 Calibrations – N/A

5.3 Sampling – N/A

5.4 Application of Procedure on Evidence

5.4.1 In order to accurately determine any uncertainty, several factors must be taken into consideration. These factors include but are not limited to:

5.4.1.1 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.

5.4.1.2 The uncertainty of the item being measured (expressed as C2) shall be obtained from the approved vendor's current Calibration Report.

5.4.1.3 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.

5.5 Calculations

5.5.1 Data collection and data manipulation may be done in an Excel spreadsheet due to the volume of data collected.

5.5.2 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

5.5.3 Combined Uncertainties (u)

5.5.3.1 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

5.5.4 Expanded Uncertainties at 99.7 % Confidence Level (U)

5.5.4.1 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

5.6 Uncertainty of Measurement

5.6.1 The expanded uncertainties for each type of measurement on each type of equipment included in the uncertainty study shall be evaluated. The highest value for each type of equipment 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. See Section records for current values.

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

5.6.2 Calibrations – N/A

5.7 Section Balances

5.7.1 (C1) Uncertainty of the measuring device - Current calibration certificates for section balances shall be evaluated to determine the greatest uncertainty for each balance.

- Divide the reported uncertainty by the maximum weight for the range and multiply by 100 to normalize the value**.**
- Choose the highest value of uncertainty between the ranges, and utilize this number for (C1) calculations.
- Once the range with the highest uncertainty value has been determined, divide the expanded uncertainty value by the coverage factor k if expanded uncertainty was reported on the certificate issued by the vendor.

5.7.2 (C2) Uncertainty of the item being measured - ("Red/clear" and "Wood" designate the two sets of reference standard weights stored in a red and clear plastic box and a wooden box respectively.)

5.7.3 (C3) The uncertainty of human/environmental influences - (see Drug Chemistry Cause and Effect Diagram for a detailed list of possible contributing factors.)

5.7.3.1 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

5.7.3.2 Data Collection for (C3) shall be repeated for ten consecutive work days.

5.7.4 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.

5.7.5 A rotation list of Forensic Scientists shall ensure that multiple users contribute to the data collection of common use balances.

5.7.6 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.

5.7.7 The monthly QC check shall be performed at the start of each work day in accordance with the Drug Chemistry Technical Procedure for Balances.

5.7.8 Each morning, 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.

- Top loading balances shall use 1 g and 1000 g weights
- Bulk balances shall use 5 kg and 10 kg weights
- Analytical balances shall use 0.1 g and 5 g weights

5.7.9 Each afternoon, three replicate weight determinations shall be obtained and recorded for the two reference standard weights that were used in the morning data collections.

5.7.10 The standard deviation of all occurrences for each weight on each balance shall be used.

5.7.11 Reporting of Final Expanded Uncertainty for the Weighing Process

5.7.11.1 The Expanded Uncertainty for each type of balance (see **5.5.4.1**) 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:

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

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

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 k=3 coverage factor and

normal distribution

5.7.11.2 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.

5.7.11.2.1 Top loading (individual) balances:
Net weight of material – XX.XX (+/- 0.0X) grams
Or for upper range:
Net weight of material – XX.X (+/- 0.0X) grams

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

5.7.11.2.3 Bulk balances:
Net weight of material – XX.XX (+/- 0.0X) kilograms
Or for upper range:
Net weight of material – XX.X (+/- 0.0X) kilograms

5.7.11.2.4 Gross weights shall not require a reported uncertainty and shall be truncated to the 0.1 place or whole number, depending on the range of the balance.

5.8 Blood Alcohol Concentration Determinations

5.8.1 **(**C1) To be determined

5.8.2 (C2) To be determined

5.8.3 (C3) To be determined

5.8.3.1 Data Collection

5.9 Cannabinoid Quantitations

5.9.1 (C1) To be determined

5.9.2 (C2) To be determined

5.9.3 (C3) To be determined

5.9.3.1 Data Collection – to be determined

5.10 GHB Quantitations

5.10.1 (C1) To be determined

5.10.2 (C2) To be determined

5.10.3 (C3) To be determined

5.10.3.1 Data Collection – to be determined

5.11 Methamphetamine Quantitations via HPLC

5.11.1 (C1) To be determined

5.11.2 (C2) To be determined

5.11.3 (C3) To be determined

5.11.3.1 Data Collection – to be determined

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.

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

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

9.0 Records

- Measurement Assurance Cause & Effect Diagram
- Determination of Uncertainty - Yearly Report for Balances
- Calibration Reports for section balances
- Calibration Reports for section Class A pipettes
- Reference Standard Weight Calibration Certificates

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original Document