Chapter 5: Technical Procedure for Uncertainty of Measurement

1. Purpose / Scope - This procedure is utilized to estimate the uncertainty of measurement for test methods for which a numerical value is reported on a Laboratory Report in the Drug Chemistry Unit of the Wake County Bureau of Forensic Services Laboratory.

2. Definitions
2.1. 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.
2.2. Coverage probability (Level of confidence) - probability that the set of true quantity values of a measurand is contained within a specified coverage interval.
2.3. Coverage factor - numerical factor used as a multiplier of the combined uncertainty in order to obtain an expanded uncertainty.

3. Overview

3.1. The Drug Chemistry Technical Leader shall determine an estimation of the Uncertainty of Measurement (UOM) for each test method for which a numerical value is reported on a laboratory report.

3.2. The estimation of the UOM shall be performed annually, at a minimum, or when a change in measurement conditions occurs that may have a significant effect on the UOM.

3.3. Measurement traceability shall be established through the calibration of the balances and mass reference standards.

3.4. Measurement assurance shall be established through intermediate weight checks on the balances between intervals of external calibration as well as the Process Measurement Assurance (Balance Study).

3.5. Each test method requiring UOM shall be evaluated for contributions from sources of uncertainty, u. The contributions shall be evaluated using Type A methods (by a statistical analysis of measured values obtained under defined measurement conditions such as repeatability and / or reproducibility, including measurement assurance data) and Type B methods (by other means of analysis of components from such things as instrument readability, calibration certificate reported uncertainty, etc.)

3.6. Evaluate the identified sources of uncertainty and combine them to obtain the combined uncertainty of measurement (CU), using the formula

$CU = \sqrt{(u_1^2 + u_2^2 + u_3^2 \ldots .)}$
where
CU = combined uncertainty
u_1, u_2, etc. = individual identified sources of uncertainty

3.7. The combined uncertainty of measurement is an estimation of the uncertainty of measurement, UOM. Individual sources of uncertainty that are not significant contributors may be excluded.

3.8. The expanded uncertainty (EU), shall be calculated to provide a minimum 95.45% coverage probability (or approximately 95%) by multiplying the CU by the appropriate coverage factor, k.

3.9. The reported EU shall contain at most two significant digits and be reported to the same level of significance as the measurement result. The reported EU shall be rounded up.

3.10. The EU shall be reported for each test method where a numerical value is reported on a laboratory report. When numerical results are added to produce a combined result the respective EU's shall also be added.

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

Examples:

Net weight: 4.00 grams ± 0.08 gram at a coverage probability of 99.5%.
Net weight: 0.01875 gram ± 0.00058 gram at a coverage probability of 99.5%.
Net weight: 10.27 pounds ± 0.07 pound at a coverage probability of 99.5%.

3.12. The Drug Chemistry Unit Technical Leader shall maintain records of the estimation of the uncertainty of measurement in the Drug Chemistry Unit.

4. Procedure

4.1. Each test method requiring UOM shall be evaluated using the NIST 8-Step Process for Estimating and Reporting Measurement Uncertainty.

4.1.1. Step 1: Specify the Measurement Processes

4.1.1.1. Weight determinations are made using The Drug Chemistry Unit Technical Procedure for Balances, and balances, Model XP205, Model XP6002S, and XPE6002S, and AND HW100KA1.

4.1.1.2. 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:

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

y = the measurement result
m = slope or sensitivity of the balance linearity
x = the balance reading or indication
b = bias
U = expanded uncertainty

4.1.2. Step 2: Identify Uncertainty Components

4.1.2.1. Balance / Scale

- Display resolution – impact of rounding at zero and at load on the value displayed
- Balance calibration uncertainty
- Balance linearity
- Balance bias
- Temperature coefficient of sensitivity

4.1.2.2. Staff

- Multiple Chemists
- Training
- Experience

4.1.2.3. Test Method

- Dynamic, single weighing event or a static process involving two weighing events
- Differences in centering of measurand on the balance

4.1.2.4. Facility

- Temperature variation of laboratory and difference from the temperature during calibration
- Drafts
- Location of balance
- Vibration
- Humidity
- Static electricity

4.1.3. Step 3: Quantify Uncertainty Components

4.1.3.1. 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 process reproducibility data
• Balance calibration uncertainty	Type B Evaluation - calibration certificate
• Balance linearity	
○ HW100KA1, XP205, XP6002S, XPE6002S	Type B Evaluation - covered in balance Calibration
• Balance bias	Ongoing quality control checks of balance to maintain confidence in calibration status using calibrated mass reference standards provides an ongoing evaluation of bias.
• Temperature coefficient of sensitivity	
○ XP205, XP6002S, XPE6002S	Type A Evaluation - covered in process reproducibility data
○ HW100KA1	Type B Evaluation - using temperature coefficient of 6×10^{-6} / °C
Staff	
• Multiple Chemists	Type A Evaluation - covered in process reproducibility data
• Training	Type A Evaluation - covered in process reproducibility data

• Experience	Type A Evaluation - covered in process reproducibility data
Test Method	
• Dynamic, single weighing event or a static process involving two weighing events	Type A Evaluation - covered in process reproducibility data
• Differences in centering of measurand on the balance	Type A Evaluation - covered in process reproducibility data
• Facility	
Location	The location of the equipment has not been altered since the external calibration - covered in the balance calibration uncertainty.
• Temperature variation	Type A Evaluation - covered in process reproducibility data
• Air flow	Type A Evaluation - covered in process reproducibility data
• Vibration	Type A Evaluation - covered in process reproducibility data
• Humidity	Type A Evaluation - covered in process reproducibility data
• Static Electricity	Type A Evaluation - covered in process reproducibility data

4.1.3.2. Type A Evaluation of uncertainty components:

4.1.3.2.1. Measurement Process Reproducibility data (Balance Study)

4.1.3.2.1.1. To more closely represent the process, only the balance study measurements from the last year will be used for all balances. A period of one year encompasses all seasonal variations in environmental factors.

4.1.3.2.1.2. XP205, XP6002S, XPE6002S

- The statistic that will be calculated is the standard deviation
- Standard Deviation (sample):

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

- XP205, XP6002S, XPE6002S use the greatest standard deviation.
- Temperature coefficient of sensitivity: Balance "Adjust.int" function is performed each day the balance is in use followed by a calibration status check using calibrated mass reference standards.

4.1.3.2.1.3. AND HW100KA1
- Use the greatest standard deviation.

4.1.3.3. Type B Evaluation of uncertainty components:

4.1.3.3.1. Balance calibration uncertainty:

4.1.3.3.1.1. XP205:
Review the calibration certificate from the accredited external calibration laboratory and identify the greatest calibration expanded uncertainty at a confidence level of at least 95 % (k=2).

4.1.3.3.1.2. XP6002S and XPE6002S:
Review the calibration certificates from the accredited external calibration laboratory and identify the greatest calibration expanded uncertainty at a confidence level of at least 95 % (k=2).

4.1.3.3.1.3. AND HW100KA1:
Review the calibration certificate from the accredited external calibration laboratory and identify the greatest calibration expanded uncertainty at a confidence level of at least 95 % (k=2).

4.1.3.3.2. Balance linearity:

4.1.3.3.2.1. XP205, XP6002S/XPE6002S, and AND HW100KA1:
Balance linearity is determined during the balance calibration and is factored into the uncertainty provided by the vendor performing the calibration.

4.1.3.3.3. Temperature coefficient of sensitivity:

4.1.3.3.3.1. AND HW100KA1
A value for the temperature coefficient is not provided by the manufacturer. A temperature coefficient of 6 x 10^{-6} / °C * i will be used, where i is the scale indication. (SD-3)

The temperature in the room is measured with each measurement assurance check standard measurement. If a value of ± 5 °C and the upper limit of the scale are used, the largest impact is: 5 °C X 0.000006 / °C X 220.46 lb = 0.0066138 lb

4.1.3.4. Step 4: Convert Quantities to Standard Uncertainties

4.1.3.4.1. Type A evaluation of uncertainty components:

4.1.3.4.1.1. Measurement Process Reproducibility data (Balance Study)

4.1.3.4.1.1.1. XP205
- The measurement unit is the gram.
- Reproducibility data from the balance study is expressed as one standard deviation.

4.1.3.4.1.1.2. XP6002S and XPE6002S
- 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 in this group.

4.1.3.4.1.1.3. AND HW100KA1
- The measurement unit is the pound.
- Reproducibility data from the balance study is expressed as one standard deviation.

4.1.3.4.1.2. Type B evaluation of uncertainty components:
4.1.3.4.1.2.1. Balance calibration uncertainty:

4.1.3.4.1.2.1.1. XP205, XP6002S, XPE6002S, AND HW100KA1:

- The uncertainty on the calibration certificate will be divided by the coverage factor, 2, to arrive at a standard uncertainty.

4.1.3.4.1.2.2. Balance linearity:

4.1.3.4.1.2.2.1. XP205, XP6002S, XPE6002S, AND HW100KA1:

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

4.1.3.4.1.2.3. Temperature coefficient of sensitivity:

4.1.3.4.1.2.3.1. AND HW100KA1

- This component is evaluated as a rectangular distribution:

$$\text{Standard uncertainty} = \frac{largest\ impact}{\sqrt{3}}$$

- Standard uncertainty = 0.0066138 lb / √3 = 0.0038185 lb

4.1.3.4.1.3. The calibration certificates and/or vendor documentation indicate that the expanded uncertainty assumes a normal distribution, a coverage factor of k = 2 and a coverage probability of approximately 95%. The uncertainty on the calibration certificate will be divided by the coverage factor, 2, to arrive at a standard uncertainty.

4.1.3.5. Step 5: Calculate the Combined Standard Uncertainty:

4.1.3.5.1. XP205, XP6002S/XPE6002S

$$u_c(y) = \sqrt{s_{process}^2 + u_{calibration\,unc}^2 + u_{CalRefStd}^2}$$

4.1.3.5.2. AND HW100KA1

$$u_c(y) = \sqrt{s^2_{process} + u^2_{calibration\,unc} + u^2_{temp} + u^2_{CalRefStd}}$$

4.1.3.6. Step 6: Expand the Combined Standard Uncertainties by the Coverage Factor, k

4.1.3.6.1. XP205, XP6002S/XPE6002S, AND HW100KA1

- The data from the measurement process is assumed to follow a normal distribution. To expand the uncertainty to a 99.5 % coverage probability the coverage factor k will be calculated for the given measurements and degrees of freedom.

4.1.3.7. Step 7: Evaluate the Expanded Uncertainty:

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

4.1.3.8. Step 8: Report the Uncertainty:

4.1.3.8.1. The expanded uncertainty is reported for net weights of analyzed material. When a gross weight is reported, the expanded uncertainty need not be included on the report.

4.1.3.8.2. When weights are added to produce a total weight, the respective expanded uncertainties are also added. This is a conservative approach that likely results in an overestimation of the uncertainty.

4.1.3.8.3. 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. Records

5.1. Uncertainty of Measurement Budgets

6. References

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6.9. *Evaluation of measurement data - Guide to the expression of uncertainty in measurement, JCGM 100:2008 GUM 1995 with minor corrections***,** First edition September 2008**,** JCGM 2008**,** Working Group 1 of the Joint Committee for Guides in Metrology (JCGM/WG 1)

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6.15. *Guide for the Use of the International System of Units (SI).* NIST Special Publication 811, 2008 Ed., (March 2008; 2nd printing November 2008), p. 53.

Document Revision History		
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