Charlotte-Mecklenburg Police Department

Crime Lab - Chemistry Section:

Controlled Substance Analysis Standard Operating Procedures

Table of Contents

Appendices:

1 General Evidence Procedures

1.1 Introduction

This section is intended to provide guidance when accepting, storing, and returning evidence.

1.2 Evidence Acceptance

1.2.1 A completed Lab Request must be received before analysis can proceed on a case.

1.2.2 Sharp objects such as syringes are not routinely accepted for analysis but may be accepted if no other suitable item is available for testing. They should be properly secured using a sharps container or other appropriate method.

1.2.3 Evidence items with no visible or weighable quantity of substance will not be tested when there are other evidence items with weighable amounts available for testing.

1.2.4 Cases that contain large amounts of wet evidence, fresh plant material, or are found to be moldy may be accepted at the analyst's discretion. Such items should be dried prior to submission.

1.2.5 Inappropriately packaged evidence which cannot be immediately corrected will be returned to Property Control (follow QM 5.8.3.1). The submitting investigator must be contacted by either the analyst or Property Control so that any issues can be corrected. Once the issue has been fixed the evidence can be requested by the analyst and work on the case can be resumed.

1.3 Evidence Handling

1.3.1 The chain of custody is recorded and maintained electronically in PLIMS by scanning the unique barcode label attached to the evidence (QM 5.8.1).

1.3.2 The person receiving the evidence is responsible for ensuring that the Complaint number in the PLIMS system matches the evidence labels affixed to the item and that the evidence is properly sealed and marked. (QM 5.8.1.3, QM 5.8.2)

1.3.3 The analyst will mark the evidence/package (at a minimum) to show date received, analyst initials, and laboratory number.

1.3.4 The evidence must be maintained in a secure location both before and after analysis.

1.3.5 Repackaged evidence will be initialed, and a new barcode label will be added on the exterior packaging. The original packaging will be included with the repackaged evidence. The analyst may select the “Repackaged” box on the item worksheet to signify the evidence was placed back in its original packaging or repackaged in another suitable container (QM 5.8.1.3).

1.3.6 When possible, the outer packaging will be opened in a fashion that retains the integrity of previously attached seals. If this is not possible the evidence should be repackaged and the original package/seals should be placed into the new package/container.

1.3.7 Outer packaging/containers will be closed and secured through the use of tape or heat seal. The outer packaging seals will contain the initials and code number of the person sealing the package, along with the date the item was sealed.

1.3.8 Every effort must be made during sampling and analysis to conserve material for additional testing if necessary.

1.3.9 To prevent the cross-contamination of evidence only one case should be opened at a time. If a case is started and not yet completed, and it becomes necessary to proceed with another case, evidence from the incomplete case must be repackaged in a manner that prevents cross-contamination. Also, work surfaces and tools will be cleaned or replaced as needed.

1.3.10 Discrepancies found in evidence weight, count or item description will be documented in the case notes and verified by the Section Supervisor or another analyst. In the case of major discrepancies, the section supervisor and the lab director will be notified. The submitting officer and their supervisor will be notified and the evidence will be returned to Property Control for correction.

2 Analysis

2.1 Introduction

Identifications will be made using techniques accepted in the field of forensic science. The scope of the testing and note taking will be sufficient such that in the absence of the analyst, another competent analyst could evaluate the procedures that were performed and interpret the resulting data.

2.2 Analytical Standards

2.2.1 It is the intention of the Chemistry Section to follow the minimum standards of drug identification as established by SWGDRUG (Scientific Working Group for the Analysis of Seized Drugs).

2.2.2 Analytical schemes and instrument parameters have been crafted to balance scientifically accepted requirements for recognizing compound identity with the productivity needs of the laboratory. The procedures are designed to find the presence of commonly encountered controlled substances when present at “street level” concentrations in a technically homogenous sample.

2.2.3 Adequate quality control measures will be run and quality assurance documentation will be generated in all analytical schemes and stored appropriately. See the QA section of this manual for more details.

2.2.4 When identifying a substance, preliminary color testing should be conducted when possible. In addition, two separate tests with positive results are required. At least one of the two tests must be a category “A” technique but the second test could come from categories “A” or “B”. The table below may be used as a guide:

Category A	**Category B**	**Category C**
Mass Spectrometry	Gas Chromatography	Color Tests
Infrared Spectrometry	Pharmaceutical Identifiers	
	Macroscopic/Microscopic Identifiers	

(Table adapted/abbreviated from SCIENTIFIC WORKING GROUP FOR THE ANALYSIS OF SEIZED DRUGS (SWGDRUG) RECOMMENDATIONS, Version 7.1, 2016-June-9)

2.3 Analytical Schemes

2.3.1 The following flow charts are the analytical schemes to be used for the analysis of controlled substances. These flow charts are to be used as a general guide and deviations are up to the discretion of the analyst. For deviations or with unique case/sample circumstances, the flowchart for general unknowns can be followed and any modifications noted in the case file. It should be noted that sample size or other circumstances may require rearrangement or modification of one or more steps. See the specific areas of the SOP for each individual set of procedures.

2.3.2 General Unknowns/Powders/Illicit Tablets:

Physical Examination/Description

↓

Weigh/Count

↓

Sample

↓

Color Test

↓

Sample Preparation

↓

GC-MS and/or FTIR*

(*FTIR is required for Federal cases to determine Cocaine HCl/Cocaine Base and should be run if GC-MS results fail to identify the substance)

2.3.3 Plant Material/Alleged Marijuana:

Physical Examination/Description

↓

Weigh

↓

Sample

↓

Microscopic Examination

(If appropriate based on sample type)

↓

Sample Preparation

↓

GC-MS

2.3.4 Pharmaceutical Preparations (tablets/capsules):

Physical Examination/Description

↓

Weigh/Count

↓

Pharmaceutical Identifier/Literature Reference

↓

Sample

↓

Color Test

(If appropriate based on case circumstances)

↓

Sample Preparation

↓

GC-MS

2.4 Preliminary Testing

2.4.1 Color Testing - Color tests are intended to be a screening test at the beginning of an analysis.

2.4.1.1 Color testing reagents not listed in the Appendix B may be used in casework provided they can be referenced to a literature source and positive and negative controls are run alongside the casework sample. The positive control does not have to be the same substance as the case sample as long as it is some substance that displays a reaction to the reagent being used.

2.4.1.2 The reagent may be added first to demonstrate no contamination, but this may not always be practical depending on the sample type. The order of placing the reagent and the sample will be up to the analyst's discretion.

2.4.1.3 Most color test reagents are comprised of strong acids and chemicals requiring careful handling. Appropriate safety precautions should be observed.

References

Clarke's Analysis of Drugs and Poisons, Third Edition, Volume One, Pharmaceutical Press, 2004.

Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Cobalt Thiocyanate Test Reagents. Sigma Technical Bulletin #QTCOCA.

Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Duquenois Test Reagents. Sigma Technical Bulletin #QTMJ.

Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Marquis Test Reagents. Sigma Technical Bulletin #QTOPI.

Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Simon's Test Reagents. Sigma Technical Bulletin #QTMETH.

O'Neal, Carol L., Crouch, Dennis J., and Fatah, Alim A. 2000. Validation of twelve chemical spot tests for the detection of drugs of abuse. Forensic Science International 109:189-201.

Koppenhaver, David. 1997. GHB Color Test. Microgram Journal XXX: 130.

Assorted Microgram Journals.

Assorted Journals of Forensic Science.

Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations, 4th edition, 2008.

2.4.2 Microscopic Analysis - Microscopic examination is used for plant material or suspected marijuana.

2.4.2.1 Cystolithic hairs are unicellular, 'bear claw' shaped hairs with a cystolith at the base. They are found in greatest abundance on the upper side of the leaf. Simple or covering hairs are found on the underside of the leaf. Glandular hairs have a short stalk and a bulbous end that secretes resin which contains THC. At least **two** of the above features are needed to indicate the possible presence of marijuana.

2.4.2.2 Seeds are coconut shaped, veined (with lacey markings) and have a ridge around the circumference.

References

United Nations Office on Drugs and Crime - Recommended Methods for the Identification and Analysis of Cannabis and Cannabis Products
https://www.unodc.org/documents/scientific/ST-NAR-40-Ebook_1.pdf
Microgram Bulletin article March 2009.
Journal of Mass Spectrometry (2009).

2.4.3 UV Light – Cases containing items of suspected LSD can be examined using UV light.

2.4.3.1 LSD is strongly fluorescent and will glow bluish-white under UV light.

2.4.3.2 LSD absorption is maximal at 320 nm, and LSD fluorescence is maximal at 435 nm. These values are somewhat flexible depending on the literature reference.

2.4.3.3 LSD loses its fluorescence very rapidly upon strong ultraviolet irradiation and decomposes if exposed to UV light for long periods of time. Care must be taken not to overexpose the sample.

References

Phillips, William, et al 1974. Distinction of Lysergic Acid Diethylamide and Lysergic Acid
Clarke's Analysis of Drugs and Poisons, Third Edition, Volume One, Pharmaceutical Press, 2004.

2.5 Confirmatory Testing

2.5.1 Gas Chromatography/Mass Spectrometry (GC/MS) - Gas Chromatography-Mass Spectrometry (GC/MS) is a specific method of identification for most drug substances. A liquid sample is vaporized and passed through a column, causing the sample to be separated into individual components. The time the individual components take to travel though the column is recorded and they then pass into the mass spectrometer (MS) source where they are ionized. The ions are separated according to their mass-to-charge ratios (m/z) and then collected by a detector. The detector converts the ions to an electrical current. The data system records the electrical signals and converts them into a mass spectrum. The mass spectrum is a graphical record of the different ions (m/z) and the abundance. These spectra are characteristic for individual compounds, giving specificity for most types of drug substances. MS cannot differentiate between optical isomers.

2.5.1.1 Depending on the structure of the molecule, the amount and type of fragmentation will vary. Some drugs do not exhibit a molecular ion when using electron impact MS.

2.5.1.2 Samples will be dissolved in a suitable solvent. Commonly used solvents include: methanol, chloroform, and hexane, though others may be used based on the sample type. Samples will be run using the appropriate GC/MS method based on the type of sample. See Appendix F for a list of methods and their uses.

2.5.1.3 Evaluation of the data resulting from the analysis of an unknown sample by GC/MS begins with an examination of the chromatogram/mass spectral data followed by a direct comparison of the data with that of a known reference standard.

2.5.1.3.1 In evaluating GC/MS data for identification purposes, the analyst will visually examine the GC chromatogram and address (if necessary) any problems with the peaks that are present (i.e. over/under concentration, possible co-elution, peak splitting, or other peak shape problems). The analyst will also note the significant ions present in the mass spectral data and the presence of any additional major ions in the sample spectrum that should be accounted for. Peaks present in the Total Ion Chromatogram of the sample will then be compared to a mass spectral library database so an appropriate reference standard can be selected. Depending upon the result of these searches, additional analysis may be required.

2.5.1.3.2 A similar evaluation of the reference standard will be conducted. Identification of an unknown spectrum is done by direct comparison with the known reference standard run under the same instrumental conditions. This includes comparison of peak shapes,

retention times, and mass spectral data. Note: In some cases, library spectrum or published reference spectrum may be used if a physical reference standard is not available.

2.5.1.4 Controlled substance standards will be verified by GC/MS before use.

2.5.1.5 Controlled substance standard solutions should be prepared at a target concentration of approximately 1 mg/mL in a suitable solvent. Variations in standard concentration may vary and are based on instrumental conditions, identity of material being analyzed, as well as other factors.

References
Agilent website - www.agilent.com
GC/MS owner's manual/software.

2.5.2 Infrared Spectroscopy (IR) - This method of spectral analysis is based on the molecular vibrational energies of an organic compound. As the different wavelengths of infrared light (most commonly used is the mid-infrared region which extends from 4000 to 400 cm^{-1}) pass through a molecule they are absorbed by the molecule or transmitted by the molecule according to the different vibrational frequencies within the molecule. These absorbed or transmitted frequencies can then be recorded electronically, and a resulting spectrum generated. A Fourier transform infrared (**FTIR**) spectrometer is a multiplex instrument, one in which all elements of the signal are observed simultaneously. Fourier transform is a signal decoding process used for data processing. The FTIR spectrometer is equipped with the Attenuated Total Reflectance (ATR) accessory which allows analysis with little to no sample preparation.

2.5.2.1 A FTIR spectrum will be obtained to determine between Cocaine HCl and Cocaine Base when a substance is suspected to be cocaine and is requested or is part of a federal prosecution.

2.5.2.2 FTIR spectrum should be collected when an item is a complete unknown, i.e., has not been identified by GC/MS analysis, and is a suitable item for FTIR testing.

2.5.2.3 Collecting an FTIR spectrum for other cases will be left up to the discretion of the analyst.

2.5.2.4 Samples will be identified by comparison to known reference standards or literature references if in-house standards are not available.

References
Thermo Scientific website - www.thermofisher.com
FTIR owner's manual/software.

3 Measurement Practices

3.1 Introduction

This section will cover guidelines on sampling, weighing practices, and uncertainty of measurement (UOM).

3.2 Sampling

3.2.1 Sampling is a process whereby a portion of an evidence item is taken for testing to serve as a representative of the whole. To ensure that analyses are thorough and to answer the requirements of North Carolina law, sampling in multiple unit cases/mixtures should be regulated and defined. Each sampling plan represents the minimum to be sampled. An analyst may always choose to sample more items.

3.2.2 The two types of sampling in use are **Non-statistical** sampling and **Statistical** sampling**:**

Non-statistical sampling can be used for:

- single item/unit of evidence,
- when a number of samples within a multiple unit case will be tested to reach a statutory threshold with respect to weight or dosage units as defined by North Carolina General Statutes §90, Article 5 – The North Carolina Controlled Substances Act,
- when there is either no statutory threshold with respect to weight or dosage units, or
- when the material to be tested does not reach or exceed that statutory threshold.

Statistical sampling can be used within a multiple unit case when the total weight or number of dosage units exceeds the statutory threshold. In statistical sampling, a sufficient number of units will be selected and tested using the hypergeometric sampling method with a 95% confidence level.

3.2.3 Non-statistical Sampling Plan/General Sampling Plan

3.2.3.1 Distinctive items/units shall be separated for analysis/sampling.

3.2.3.2 Distinct packages with obvious cross contamination may be combined at the discretion of the analyst.

3.2.3.3 Whenever possible the analyst shall leave a portion of the original sample for potential re-analysis and court viewing. Note: this may not be possible for items containing trace amounts of material.

3.2.3.4 For cases where the item contains only one unit, that unit will be fully analyzed.

3.2.3.5 For cases with multiple units within an item, like units may be combined, but visually different units should be separated (where possible) by common characteristics (size, color, shape, physical consistency, etc), to obtain a homogenous population.

3.2.3.6 Professionally manufactured **pills/tablets/capsules (or other pharmaceutical preparations)** which contain a pharmaceutical identifier can be combined into one item by identical physical characteristics and/or pharmaceutical identifier. Do not physically combine distinctly separated or packaged sub-units.

3.2.3.6.1 When applicable, use the pharmaceutical identifiers to determine the probable identity of any controlled substance present.

3.2.3.6.2 Determine the total weight of the submission and net weight of one pill.

3.2.3.6.3 A full analysis will be conducted on at least one unit, more if necessary to reach a statutory threshold. (See 3.2.3.9 and 3.2.3.10).

3.2.3.6.4 Only the items that were fully analyzed will have their weights reported.

3.2.3.6.5 Pharmaceutical preparations in which the imprint, markings, or physical characteristics cannot be identified using an approved reference, or whose results do not match that of the reference, shall be analyzed as if they were clandestine preparations.

3.2.3.6.6 Pharmaceutical preparations whose results match the imprint, markings, and/or physical characteristics of the reference and whose total net weight of all units does not exceed a statutory threshold, may be analyzed by fully testing only one unit (exempt from 3.2.3.12).

3.2.3.7 Bulk materials (e.g., bricks of compressed powder, bales of plant material) should be broken or cored to ensure a homogeneous sample is obtained. Depending on the size of the material, samples from several locations may be sampled to obtain a representative sample.

3.2.3.8 Co-mingled clandestine tablets may be separated into distinct sub-groups based on their physical characteristics including color, shape, imprint, and physical consistency.

3.2.3.9 When encountering different size units in an item, the larger size units may be chosen for analysis prior to the smaller sized units.

3.2.3.10 If the total net weight of the units analyzed, when added to the calculated net weight of the unanalyzed units results in exceeding a statutory threshold, additional units of unanalyzed material will be tested to exceed that statutory threshold.

3.2.3.11 If the uncertainty of measurement lowers the total net weight to below a statutory threshold, then further analysis on additional units must be performed until the threshold has been met.

3.2.3.12 When the total weight/count of the units in an item do not exceed a statutory threshold, a full analysis will be performed on a minimum of 5 (if 5 or more are present) units. The examiner has the discretion of choosing which units to test.

3.2.3.13 A full analysis of only one unit may be conducted at the analyst's discretion for items where a weight won't be reported (i.e. Food products or certain types of liquids) or where the sample/sample matrix is detrimental to the instruments being used for analysis.

3.2.3.14 All untested units in an item may be administratively grouped together and reported in totality as "Not Analyzed" or equivalent.

3.2.3.15 Deviations from 3.2.3.10 & 3.2.3.11 may be initiated at the discretion of the analyst due to circumstances related to the case (excessive number of units, time/labor intensive testing, etc). Deviations will be approved with or notified to the customer and included in the case file.

3.2.3.16 If analysis is inconclusive or reveals no controlled substance present in illicit tablets, additional units may be tested to include different sub-groups at the discretion of the analyst. If no controlled substance is present or confirmed in the additional units and not all sub-groups have been sampled, the customer will be contacted to determine if additional analysis is required.

3.2.4 Statistical sampling - Hypergeometric Sampling Method

3.2.4.1 Hypergeometric sampling is a statistically based model involving a defined confidence level with an associated probability of a particular finding within an item or unit.

3.2.4.2 Full instrumental analysis of at least one unit may be conducted before the hypergeometric sampling plan is adopted.

3.2.4.3 Based on the reference(s) in Appendix D an appropriate number of specimens within the population will be randomly selected to give a 95% confidence level that at least a 75% or higher proportion of the population contains the analyte in question. The analyst will select the appropriate proportion percentage based upon the circumstances of the case (i.e., population size, net weight, and the statutory threshold of the analyte) and indicate what proportion percentage parameter was utilized on the worksheet and/or the report.

3.2.4.4 When utilizing the excel calculator referenced in Appendix D, the analyst will follow the instructions under the “Hypg_Proportion” tab and will enter the values using the guidelines below:

- population size (total number of units that the hypergeometric sampling plan is being applied to)
- the appropriate proportion (k) of positives in the population (minimum of 0.75)
- the expected number of negatives (r) will be set to 0 (see 3.2.4.7)
- confidence level of 95% (0.95).
- the resulting sample size (n) determines the minimum number of units to be sampled/analyzed.

3.2.4.5 Each unit sampled, as determined from the reference(s) in Appendix D, will be fully analyzed.

3.2.4.6 Hypergeometric sampling will not be used for nonhomogeneous samples.

3.2.4.7 The hypergeometric sampling method can be used on an item if the reported analyte is present in all sampled units. If testing reveals any of the sampled units are negative for the reported analyte, the sampling method will revert to a non-statistical sampling method and noted in the case file.

3.2.4.8 The population net weight of the item using a hypergeometric sampling plan will be reported.

3.3 General Weighing Practices

3.3.1 The **Gross Weight** (GW) is defined as the weight of an item/unit including packaging and/or other miscellaneous materials.

3.3.2 The **Net Weight** (NW) is the weight of an item/unit without any packaging. The net weight will be determined in the most accurate manner possible based on the size, packaging, and physical condition of the evidence items.

3.3.3 Wet evidence should be dried prior to being weighed.

3.3.4 An attempt will be made to minimize sample loss during the removal of an item/unit from its packaging.

3.3.5 The controlled substance worksheet shall reflect the actual reading(s) from the balance. Weights shall be reported in the units and significant digits recorded from the balance.

3.3.6 Any calculations of net weight and/or conversion between units must be fully documented on the worksheet and/or report.

3.3.7 A net weight should be reported (when applicable) in addition to the tablet count or amount for both illicit and pharmaceutically identified tablets/capsules.

3.3.8 The net weight of capsules shall include the weight of capsules and contents.

3.3.9 No weights shall be reported for liquids or on drugs saturated into a secondary medium; instead, a count or amount (e.g., 3 sugar cubes, 5 units of blotter paper, approximately 25 mL of liquid, or 4 brownies) shall be reported.

3.3.10 Any material present in an amount that is less than 0.03 grams may be reported as “trace amount” or “residue”.

3.3.11 Cases involving quantities that exceed the capacity of the personal balances should be weighed using the bulk analytical balance and following that balance’s use protocols (verification check at the time of weighing).

3.3.12 Deviations from the weight requirements above must be approved by the Chemistry Section supervisor and noted in the case file.

3.4 Statutory/Trafficking Limits

3.4.1 It may be necessary to analyze additional items to satisfy weight requirements mandated by law or at the request of the DA's office.

3.4.2 In instances where statutory requirements or state sentencing guidelines designate weight thresholds, sufficient specimens will be weighed and analyzed to exceed the threshold, see Appendix E. The method used to generate the reported value must have an uncertainty that can be defined, and (if sufficient sample exists) the data reported can have no reasonable possibility of being below the legal limits. Care must be taken to ensure that the uncertainty of measurement (see 3.5) for the balance used is taken into account when determining the number of units to analyze.

3.4.3 If analysis reveals a mixture of drugs with different trafficking weights/penalties (such as Heroin mixed with methamphetamine), the analyst will reach the statutory requirements for both drugs, the one with the higher schedule, or contact the prosecuting agency to determine if additional analysis is required.

3.5 Uncertainty of Measurement (UOM)

3.5.1 Estimation of uncertainty shall be determined where the testing contains measurement results that are quantitative, reported, and may reasonably be expected to be used by an immediate or extended customer (anyone in the judicial process) to determine, prosecute, or defend the type or level of criminal charge(s).

3.5.2 The uncertainty measurement will include at a minimum the identification and assessment of the major sources of uncertainty in the procedure which are of importance to the process. This may include the methods, instrument/equipment, special environmental conditions, the types of evidence tested, and the reference standards used and the operator. The sources of uncertainty will be classified as Type A or Type B components.

3.5.3 An uncertainty budget table shall be completed to include both Type A and Type B components of uncertainty for the process of weighing controlled substance samples.

3.5.4 The uncertainty of measurement shall be re-evaluated on an annual basis. The technical records, supporting documentation, and calculations used for the uncertainty of measurement evaluation shall be technically reviewed by a second authorized analyst. The reviewer will submit the approved uncertainty of measurement documentation to the Chemistry Section Supervisor for review and final approval. All approvals will be documented and retained in the Chemistry section.

3.5.5 The units of uncertainty values shall be measured (converted) in the same units. The expanded uncertainty calculated for each of the individual balances will be averaged together and the result will use conventional rounding rules to determine the final UOM (the Sartorius Miras 2 balance will have its UOM calculated separately).

3.5.6 The final UOM shall not exceed two digits after the decimal for the individual balances or three digits after the decimal for the Sartorius Miras 2 balance. The reported UOM will be the final UOM multiplied by the number of weighing events.

3.5.7 The 95.45% confidence interval will be used for reporting measurement of uncertainty as it pertains to weighing of controlled substances.

References

ANSI-ASQ National Accreditation Board (ANAB) manual.
ASCLD/LAB-*International* Guidance on Measurement Traceability, May 2013.
ASCLD/LAB-*International* Guidance on Measurement Traceability-Measurement Assurance, May 2013.
ASCLD/LAB-*International* Guidance on the Estimation of Measurement Uncertainty – Overview, May 2013.
ASCLD/LAB-*International* Guidance on the Estimation of Measurement Uncertainty –Annex B Drug Chemistry, May 2013.
Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Supplement Document SD-3, Examples of Measurement Uncertainty for Weight Determinations, 7-7-2011.
ASCLD/LAB Measurement Confidence 100 and 200 level courses, 2013.
ISO/IEC 17025: 2005

4 Documentation and Reporting Procedures

4.1 Introduction

This section will cover guidelines on case documentation and the reporting of results.

4.2 Case Files / Electronic Case Records

4.2.1 The case file or electronic case record will contain all pertinent documentation related to a request for analysis and all significant communication about that case, including but not limited to:

- Laboratory request forms (if available)
- Laboratory worksheets (if available)

- Substantive case related conversations/correspondence (if any)

The file in PLIMS shall contain, upon completion, a copy of the issued report, any addendum/supplemental reports, and corrected/amended reports.

Each page within the case file should have at a minimum the Complaint or PLIMS Lab # for the case and the analyst's initials.

The laboratory case files shall be maintained in secured areas of the Crime Laboratory.

4.3 Case Notes, Worksheets, & Photographs

4.3.1 Notes will be generated by the analyst describing the submissions, tests performed, discrepancies in the submission as received, and any other pertinent remarks.

4.3.2 If a sampling plan other than the "General Sampling Plan/Non-statistical Sampling Plan" is used, the specific sampling plan used will be documented in the case notes.

4.3.3 Worksheets will be used and all administrative information is required to be complete.

4.3.4 All notes will be neat, legible, clear, and concise.

4.3.5 Any drawings will be representative of the object or logo.

4.3.6 All handwritten records will be generated in ink.

4.3.7 All data collected should have analyst initials, date, lab number, etc.

4.3.8 Any photographs taken of evidence items that are printed out for inclusion in the case file shall have the lab number, the date, and the analyst's initials on them.

4.3.9 The "Date Started" field on the case notes is defined as the date the evidence was opened/examined for that specific case assignment.

4.4 Reports

4.4.1 Scheduled and/or non-controlled substances will be reported after confirmation.

4.4.2 The methods for testing will be included on the report.

Examples include:

- Item(s) ___was/ were analyzed by color test(s) and Gas Chromatography-Mass Spectrometry (GC-MS).
- Item(s) ___ was/were analyzed by color test(s), Gas Chromatography-Mass Spectrometry (GC-MS), and Infrared Spectroscopy (FTIR).
- Item(s) ___ was/were identified/analyzed by their pharmaceutical imprint and Gas Chromatography-Mass Spectrometry (GC-MS).
- Item(s) ___ was/were analyzed by microscopy and Gas Chromatography-Mass Spectrometry (GC-MS).

4.4.3 Net weights will be reported when an item has been fully analyzed and found to be a controlled substance. Gross weights of unanalyzed items or for the total weight of multiple items may be included at the discretion of the analyst.

Example: From 20 bags of white powder with a total gross weight of 150.30 grams, 4 bags were analyzed and found to contain cocaine, net weight 30.05 grams.

4.4.4 The net weight does not need to be reported when the item has been determined to be non-controlled, has a quantity insufficient for analysis, or if the item is not suitable for analysis. The Gross Weight may be included at the discretion of the analyst.

4.4.5 Any material present in an amount less than 0.03 grams may be reported as “trace amount” or “residue”.

4.4.6 Weights recorded when checking the calibration of the balance will be the same units used for analyzing and reporting the weight. Any conversions listed on the report will be calculated based on the calibrated unit (See conversions in Appendix C) or otherwise noted in the case file.

4.4.7 Tablets/capsules, both pharmaceutically prepared and illicit, should be reported to include the count, amount, and/or weight.

4.4.8 Numbered items of evidence that are received but not analyzed will be listed on the report or otherwise noted in the casefile.

4.4.9 The following (or its equivalent) are possible options for reporting results:

- Identity of controlled substance found
- No controlled substance detected
- Inconclusive (reason listed on work sheet and report)
- Not Analyzed

Additional reporting options may be used with the approval of the Chemistry supervisor.

4.4.10 On reports where a weight which includes an uncertainty of measurement is reported the following statement will be included in the comments section:
"All (+/-) values are reported at a confidence interval of 95.45 %."

4.4.11 If a sampling plan other than the "General Sampling Plan/Non-statistical Sampling Plan" is used, the specific sampling plan used will be documented on the final report including confidence levels and information about the sampled population (i.e., *A hypergeometric sampling plan was utilized resulting in a 95% confidence level that at least 75% of the {sample population} contain {Controlled Substance Reported}).

4.4.12 Official Reports will not be issued for presumptive testing on suspected controlled substances in time sensitive investigations. Results of the testing will be documented on the Controlled Substances Preliminary Testing Results Notification Form and attached to PLIMS per QM 5.10.1.

4.4.13 The date(s) of performance of laboratory activity will be present on the final laboratory report.

The start of laboratory activity date will be taken from the "Date Started" section of the case notes and will be displayed on the report in the comments section:

"Analysis of the above evidence was started on {Date} by {Analyst name}."

The final report date will be used as the end of laboratory activity date.

5 Quality Assurance

5.1 Introduction

This section will cover quality assurance practices for instruments/equipment, reagents, standards, blanks, color tests, gases, the peer review process, and the validation of testing procedures.

5.2 Reagents

5.2.1 Reagents and solutions that affect the quality of work will be prepared utilizing materials of the highest practical purity.

5.2.2 When a reagent/solution is made, the lot numbers will be recorded on the Lab Solution Form and the reagent should be assigned a new lot number

consisting of the date it was made and the analyst's initials (example 062811AC). Preparer and supervisor or designee will initial and date the form to denote the reagent/solution is suitable for use.

5.2.3 Expiration of reagents/solutions may be one year from date of creation unless otherwise noted on the Lab Solution Form.

5.3 Standards

5.3.1 The Chemistry Section keeps both primary and secondary reference standards.

5.3.2 A primary standard is one obtained from a reputable manufacturer. When a primary standard is received into the Chemistry section, it must be marked in permanent ink the date received and the receiver's initials. When it is opened, that date and opener's initials must also be reflected on the container.

5.3.3 All primary standards must be authenticated at time of use. Anytime a standard is authenticated a "Drug Standards - Authentication and Usage Form" must be completed after being reviewed.

5.3.4 The Standard Authentication packet of a primary standard should contain at least the following:

- Manufacturer's certificate of analysis
- At least one set of category A instrumental printouts
- Drug Standards - Authentication and Usage Form

5.3.5 A secondary standard is one that is obtained from a reputable source e.g. includes but not limited to, other forensic laboratories, police agencies etc. Samples of unknown origin are not acceptable as standards.

5.3.6 When a secondary standard is obtained, it should be labeled with an identification number of the formatting: analyst's initials, abbreviation of standard, date of authorization (example for cocaine JSLCO012510), the date it was obtained, and the obtainer's initials. A note must be recorded in the authentication package of where the standard came from.

5.3.7 Secondary standards are considered authenticated when they have been confirmed by at least one category A analytical technique and a second technique from categories A or B (or C at the discretion of the Chemistry Supervisor).

5.3.8 Data obtained from the secondary standard should match literature data as well as that of a primary standard that has been entered into the instrument search libraries before it can be used in casework.

5.3.9 The Standard Authentication packet of a secondary standard should contain at least the following:

- Category A instrument print outs
- Additional printouts from a category B analysis
- Literature reference to support authenticity

5.3.10 When a standard is used to verify results in a case, data for that standard should accompany the sample data in the case file.

5.3.11 Drug Standards - Authentication and Usage forms and packets will be kept for at least five years and archived as needed.

5.3.12 Standards will be stored in either the controlled refrigerator/freezer or in the drug safe in the controlled supply room of the Chemistry section.

5.3.13:

Expiration Dates
All standard containers should be labeled with an expiration date. (QM 4.6.2)
1. Standards (solids)…….…….…..…. expire 5 years from date opened*
2. Standards (liquids)…… expire 2 years from date opened*
3. Positive Controls (e.g. Methamphetamine/Cocaine mix) expire 1 year from date prepared
4. Negative Controls (i.e. Oregano & Inositol)…..…… expire 5 year from date opened
5. 1 mg/mL solutions. ……..… expire 1 year from date prepared*
*Use the manufacturer's expiration date if provided
Re-validation
Standards should be re-validated with the same method in which they were initially validated.
All standards should be re-validated only one time.
1. Standards (solids)……..……..Extended for 2 years
2. Standards (liquids)…….. Extended for 1 year
3. Positive controls (e.g. Marihuana)…….…….……..Extended for 6 months
4. Negative Controls (e.g. Oregano & Inositol)…..……..Extended for 6 months
5. 1 mg/mL solutions…….. Extended for 6 months

5.4 Blanks

5.4.1 Blanks will be run in all analytical schemes to eliminate the possibility of carry over and the resulting data will be stored with the case file.

5.4.2 Running GC/MS Blanks

5.4.2.1 A blank consisting of the same solvent used to prepare the samples should be run before every case sample. A solvent blank should be run before every standard used for confirmation.

5.4.2.2 The solvent blank must be run at the same or lower split ratio as the sample.

5.4.2.3 Any significant peaks in the blank chromatograms must be properly investigated and documented in the case file.

5.4.2.4 A blank should be re-run if a peak is observed in the blank within ±0.05 minutes of a peak of interest in the sample/standard.

5.4.3 Running FTIR Blanks

5.4.3.1 A blank spectrum of the clean ATR accessory will be collected before every case sample. If the blank spectrum is not acceptable another blank spectrum will be collected after cleaning the accessory.

5.5 Color Tests

5.5.1 Quality control checks will be recorded in the POU log or in the case notes of the case file.

5.5.2 Quality control checks will be performed on each commonly used color test reagent on a weekly basis or at least prior to use in casework.

5.5.3 Reagents that are not used often in casework will also be tested with positive and negative controls prior to use in casework.

5.5.4 Reagents should be made in quantities to minimize waste. The shelf-life of a reagent may vary; see the reagents solution form for the expiration date. Unexpired reagents are considered to be viable as long as they react appropriately in the QC check. Reagents that fail to react will be discarded regardless of expiration date.

5.5.5 When a reagent is made, the lot numbers of its components will be recorded and the reagent will be assigned a lot number consisting of the date it was made and the analyst's initials (example 071718AO).

5.5.6 If an analyst is unable to do a weekly test of the reagents or if no casework is completed that week, an explanation will be written for that week in the POU log.

5.5.7 Quality control checks will consist of a positive and negative control. See the table below as a guide:

Check Compound				
Reagent	**Positive Control**	**Positive Result**	**Negative Control**	**Negative Result**
Cobalt Thiocyanate	Cocaine	Blue	Inositol	No reaction
Marquis	Methamphetamine	Orange	Inositol	No Reaction
Marquis	Heroin	Purple (Violet)	Inositol	No reaction
Other color reagents	See reference literature for appropriate choice			

References

Clarke's Analysis of Drugs and Poisons, Third Edition, Volume One, Pharmaceutical Press, 2004.

Sigma Chemical Company 1993. Forensic Chemistry Drug Stat Kit-Cobalt Thiocyanate Test Reagents. *Sigma Technical Bulletin* #QTCOCA.

5.6 Equipment Maintenance

5.6.1 Any equipment used for measuring that fails a performance check or calibration must be taken immediately out of service and reported to the section supervisor or designee per QM 5.5.7. External Calibrations shall be performed by an ISO 17025 accredited company when available. When equipment is checked or calibrated by an external organization, the procedures used will meet the Chemistry section's criteria, documentation of the check or calibration shall be provided, and the certificate and scope of the provider is kept with the Quality Assurance Manager.

5.6.2 Weights

5.6.2.1 Transportation and Handling of Weights:

- All weights will be handled with gloves, tweezers, or tongs.
- All weights will be transported and stored in their original box/container.

5.6.2.2 Definitions:

Primary mass standards – Certified NIST traceable weights used to verify secondary mass standards. Primary mass standards will

meet or exceed ANSI/ASTM E617 Class 1 standard (ex. UltraClass).

Secondary mass standards – Weights used in routine balance calibrations. Secondary mass standards will meet or exceed ANSI/ASTM E617 Class 2 standards.

Surrogate Check weights – check standard similar in nature and close in material content to simulate actual material encountered in normal casework. (ex. plant material and powder).

5.6.2.3 All UltraClass traceable weights (primary mass standards) will be re-certified every three years by an accredited external calibration service supplier (QM 5.6.3.1). The report of the external recertification of each primary mass standard will be evaluated to determine if the reported actual values meet acceptable criteria. If the reported actual weight of the standard falls within the tolerance of the mass value, the standards are suitable for use in the annual performance checks of the secondary calibration weights.

ANSI/ASTM E617 Class 1 tolerances	
Nominal value	**Tolerance**
1 gram	0.034 mg
10 grams	0.050 mg
100 grams	0.25 mg

5.6.2.4 All weights used to verify balance calibration (secondary mass standards) shall be evaluated annually to determine if the weight meets acceptable criteria - see QM 5.6.3.1.

5.6.2.5 If at any time a primary mass standard is dropped, becomes damaged or potentially altered in any manner or does not meet acceptable tolerance level, it must be immediately taken out of service. The affected mass standard must be recertified by an external agency prior to being placed back into service or replaced.

5.6.2.6 Process for checking Secondary weights:

For each secondary mass standard being checked (on an annual basis or otherwise necessary), a primary mass standard is weighed. That weight is recorded. The balance must provide a reading that is within the acceptable tolerance listed for that weight. Once the balance has been determined to be sufficiently

accurate using the primary mass standard, the secondary calibration weight is weighed, and that weight is recorded. If the measured weight of the secondary calibration standard falls within the declared tolerances of the primary calibration standard (i.e., within ± 1%), the standard is suitable for use in performing the routine calibration of balances. If the measured weight does not meet the criteria, it must be replaced or adjusted and recertified by an external agency. If at any time a secondary calibration weight is dropped or potentially altered in any manner, it must be rechecked using this procedure.

5.6.3 Verification of Balances

5.6.3.1 Each analyst is responsible for performance checking any balances he/she uses during the course of casework. Additional calibration/verification will be performed if the balance has been relocated, had non-routine maintenance performed, or any time the operability of the balance is called into question.

5.6.3.2 The performance of each personal balance will be verified using the following guidelines:

- Personal balances will be checked weekly using a set of secondary mass standards (minimum ASTM class 2).
- A monthly performance check will be done using surrogate weights (plant material and/or powder).
- Each personal balance will be checked quarterly using the primary mass standards (Ultraclass weights).
- Each personal balance will be calibrated annually by a certified vendor that is ISO 17025 accredited. The balances shall meet the manufacturer's specifications and the mean of the repeatability measurements shall be +/- 1.00% of the expected value. Casework may resume once the balance is performance checked.
- Once the certified vendor provides a certificate of the calibration, the certificate shall be reviewed by the Chemistry Section Supervisor or designee to verify all criteria has passed. The calibration certificate will be initialed and dated by the reviewer and retained in the Chemistry Section.

5.6.3.3 The actual readings of all performance checks will be recorded in the analyst's point of use (POU) log or the balance log. Weight measurements should be within current acceptable ranges (*see Balance Performance Verification Log or POU for acceptable*

ranges of each balance). If a weight measurement exceeds the acceptable range, confirm that the balance is level and/or perform the internal balance re-calibration then repeat all weight measurements again. If the weight measurements still fall outside the acceptable range the section supervisor will be notified and the balance will be placed out of service. Maintenance and/or calibration will be performed by an accredited vendor and verified prior to placing back into service.

References:
ASTM E617 – 13 Standard Specifications for Laboratory Weights and Precision Mass Standards.

5.6.4 Other Equipment

5.6.4.1 Microscope and alternate light source (ALS) repairs are made as needed.

5.6.4.2 Oven, Refrigerator, and Freezer:

- The temperature of the oven, refrigerator, and freezer are monitored and recorded. The section supervisor or designee will check all the temperature entries monthly.
- For refrigerators, the temperature shall be between 2 – 8 º C.
- For freezers, the temperature shall be below -5 º C.
- If the temperature should fall just outside the acceptable range, the thermostat should be adjusted accordingly to bring the temperature back into the acceptable range. The adjustment will be documented on the temperature log and the temperature of the unit will be monitored to make sure the adjustment was effective.
- For extreme temperature changes (e.g., freezer above 0°C, refrigerator below 0° C or greater than room temperature), all biological evidence and critical reagents will be removed immediately from the affected unit and placed in alternative refrigerators and/or freezers. The refrigerator or freezer should be placed out of service, labeled as such until it can be repaired and the repair should be documented on the temperature log.
- Once a year each thermometer is checked against a NIST certified thermometer.

5.6.4.3 Pipettes:

- Clean monthly or as needed.
- Adjustable volume and single volume pipettes should be cleaned by wiping the surface with Ethanol or 10% chlorine bleach solution and allowed to air dry.
- For the precision liquid processor (i.e. Hamilton Microlab Diluter) use a cleaner that is compatible with the fluids previously run through the system. It should have its surface wiped with 10% chlorine bleach solution and deionized water and then allowed to air dry. Purge, clean, and decontaminate all portions of the tubing and syringes. Tubing that is in poor condition should be replaced. Clean monthly or as needed. The precision liquid processor should be calibrated and/or performance verified once a year by a certified vendor and in-house performance verification should be done every year.

5.7 Validation of instruments, procedures, and methods

5.7.1 Prior to being incorporated into the Chemistry analytical procedures, laboratory developed, and standard methods/procedures will be validated by the Chemistry section *(see validation binder and/or the R drive for method validation).*

5.7.2 The validation process will ensure the expected results are obtained from the method/procedure and required instrumentation *(QM 5.4.5).*

5.7.3 Techniques used in validation may be one or a combination of the following:

- the use of reference standards/material
- comparison of results with those achieved using other methods (concordance studies)
- systematic assessment of the factors influencing the result
- assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.

5.7.4 Instruments located within the Chemistry section will only be operated by authorized and trained personnel. A list of equipment and software used for drug analysis can be found in Appendix H.

5.7.4.1 Each instrument will have a logbook and QC binder. Additionally, GC/MS instruments will have monthly Maintenance Log sheets. FTIR quality control check documentation and GC/MS

Maintenance Logs will be checked, initialed, and dated monthly by the supervisor or designee.

5.7.4.2 Any instrument that fails the quality control checks or gives suspect results will be placed 'out of service' until the necessary repairs are made to correct the problem. The 'out of service' designation should be reserved for instances in which general/routine maintenance does not immediately rectify the issue and may be given at the discretion of the analyst.

5.7.4.3 Any repairs will be recorded in the instrument logbook.

5.7.4.4 After an instrument has been repaired or non-routine maintenance has been performed, the instrument must pass quality control checks before being placed back 'in service'.

5.7.4.5 Commercial software in general use within the Chemistry section will be considered to be sufficiently validated as long as it is used within the application it is designed for.

5.7.4.6 Commercial software that has been altered by the Chemistry section will be validated prior to use.

5.7.4.7 Test equipment/instrumentation with settings that can be adjusted will be safeguarded against unintentional changes following and during testing by only allowing authorized personnel to operate the instrument.

5.8 Gas Chromatograph/Mass Spectrometer (GC/MS)

5.8.1 Each GC/MS system will be identified from the other systems by use of a unique identifier.

5.8.2 A list of suggested methods can be found in the Appendix F. Any changes made to these methods will be documented. Temporary changes in these methods or the use of other methods not listed will be documented in the affected case file.

5.8.3 New or significantly changed methods will be checked by running standard materials and case materials by both the old and new processes and comparing the data. New methods must be verified by the Chemistry supervisor.

5.8.4 Tuning the Mass Spectrometer

5.8.4.1 The mass spectrometer will be tuned using 'instrument appropriate tune' at least once a week or before use and after repairs.

5.8.4.2 Acceptance criteria for the tune requires evaluation of several areas on the tune print out (may be printed as an electronic document): *see Tune acceptance criteria chart below.*

Tune Acceptance Criteria (use Atune or Stune)

Tune Parameters	Specific Parameter	Stune	Atune
Ion Abundance/Ratios	69	= Base Peak	100%
Ion Abundance/Ratios	219	≥40% - ≤85%	>40%
Ion Abundance/Ratios	502	≥1.0% - ≤5.0%	≥2.4
Mass Assignments	69.00, 219.00, 502.00 amu	0.55 ± 0.10 amu	0.6 ± 0.10 amu
Isotope Ratios	Ratio of mass 70 to 69	≥0.5 – ≤1.6%	≥0.5 – ≤1.6%
Isotope Ratios	Ratio of mass 220 to 219	≥3.2 – ≤5.4%	≥3.2 – ≤5.4%
Isotope Ratios	Ratio of mass 503 to 502	≥7.9 – ≤12.3%	≥7.9 – ≤12.3%
Electron Multiplier		400 volts of previous setting & <3000	400 volts of previous setting & <3000
Air/Water Peak		<20% abundance	<20% abundance

References

Agilent website.
Instrument owner's manual/software.

5.8.4.3 An acceptable tune will be saved on the GC/MS computer file system under the appropriate WKCAL folder and the maintenance log sheet will be initialed by the analyst.

5.8.4.4 Additional analyses of controls may be analyzed at the discretion of the analyst.

5.8.4.5 If the instrument is not suitable for use, then maintenance and troubleshooting will be performed.

5.8.5 Evaluating Chromatography Performance

5.8.5.1 Chromatographic performance will be evaluated weekly by tuning the GC/MS and running mixed standards as positive controls using the drug scan method (DRGSCN) or equivalent. This is recorded on the GC/MS Maintenance Log and checked monthly by the supervisor or designee.

5.8.5.2 Chromatographic performance will also be evaluated after repairs to the instrument.

5.8.5.3 The GC/MS test mix can contain any of the following but not limited to: methadone, caffeine, cocaine, codeine, delta-9-tetrahydrocannabinol, 6-monoacetylmorphine, heroin, etc. The positive control is considered acceptable if all expected components elute with adequate separation and retention times are within ±0.05 minutes of the previously run standard mix (Note: some maintenance procedures performed on the instrument may affect retention times and would be exempt from the ±0.05 minute parameter).

5.8.5.4 The negative (blank) control must not produce unacceptable artifacts, excessive column bleed, or peaks of target analytes. Easily recognizable column/septum bleed peaks are 207, 221, 267, 281, 327, 341, 355, 385, 415, and 429 m/z.

5.8.5.5 Acceptable positive and negative control data will be saved on the GC/MS computer file system under the appropriate WKCAL folder and the maintenance log sheet will be initialed by the analyst. Method conditions should mimic those used in a general screen run by analyst. Any performance discrepancies or degradation should be reported immediately to a supervisor.

5.8.5.6 Once the chromatographic performance evaluation and tuning of the MS has been accepted, the instrument will be considered acceptable for use.

5.8.6 Acceptable results for case work:

5.8.6.1 Retention time of target analyte must be within ± 0.05 minutes of the reference standard.

5.8.6.2 To determine if two spectra match, the scientist must carefully examine the ion clusters and the fragmentation patterns. The overall fragmentation pattern and relative ratios of the ions within the spectrum are compared for consistency. Positive mass spectral results may be recorded on the analytical worksheet by listing the drug identified.

5.8.6.3 Data from chemical reference standards, library matches, or published reference spectra used for comparison and to support positive conclusions will be included in the case file.

5.8.6.4 The spectra for all significant peaks present or a notation that all significant peaks in the TIC have been examined will be included in the case file, including the identity of any controlled substances.

5.8.6.5 When no controlled substances are detected the results can be considered negative or further analysis of the sample using a different method (or instrument) may be necessary if other testing indicates the possibility of a controlled substance.

5.8.6.6 The mass spectrum of a compound that does not meet the minimum requirements will be deemed inconclusive. Examples of this include spectra that are too weak, spectra of co-eluting compounds, or spectra that have no apparent matches.

5.9 Fourier Transform Infrared Spectrometer (FTIR)

5.9.1 The FTIR and ATR accessory will be verified using the Val-Pro module of the OMNIC software. The instrument should be verified once a week and/or after non-routine maintenance has been performed.

5.9.1.1 Insert the Smart OMNI-Transmission accessory and check desiccant.

5.9.1.2 Open the OMNIC software. Select the Analyze tab and click Val-Pro Qualification.

5.9.1.3 Select the Nicolet iS10 KBr-EP qualification test from the drop-down menu and click Qualify to run the Val-Pro report.

5.9.1.4 Insert the ATR accessory (Smart iTX) and run the Val-Pro qualification using the Smart iTX accessory-EP qualification test. Follow the on-screen instructions to place the ATR polystyrene standard on the accessory when prompted.

5.9.2 The instrument must pass all of the parameters listed in the Val-Pro quality control report.

5.9.3 The Val-Pro reports will be printed, initialed/dated, and kept in the instrument QC binder. The ATR Val-Pro printout should list the serial number and expiration date of the polystyrene standard used. The Val-Pro reports are automatically recorded in the OMNIC software upon completion of the Val-Pro check.

5.9.4 If any quality control fails to meet acceptable criteria, the bench may be re-aligned or the accessory cleaned (whichever is appropriate) and the Val-Pro will be repeated.

5.9.5 Once a month (or whenever deemed otherwise necessary) an additional quality control check using a polystyrene standard on the ATR accessory shall be conducted.

5.9.5.1 Run the weekly Val-Pro qualifications as described above.

5.9.5.2 Create a folder for the monthly check data (if necessary) and with the ATR accessory inserted select the 'QC' experiment.

5.9.5.3 Collect a background spectrum. Print and save the background spectrum as 'BKG MMDDYY' or equivalent using the current date.

5.9.5.4 Collect a blank spectrum with nothing on the accessory. Print and save the blank spectrum as 'BLK MMDDYY' or equivalent using the current date.

5.9.5.5 Collect a spectrum of the polystyrene standard using the ATR accessory. Print and save the spectrum as 'PS MMDDYY S_N-NNNN Exp MM-DD-YY' or equivalent where MMDDYY current date, S_N-NNNN is the polystyrene cards serial number and MM-DD-YY is the expiration date. Ensure the serial number and expiration date of the standard is included somewhere on the page.

5.9.5.6 Label the peaks by using the find peaks option on the menu. Print the spectrum with the labeled peaks.

5.9.5.7 The following guidelines should be used in determining the success of the monthly check:

5.9.5.7.1 Background collected successfully.
5.9.5.7.2 Blank clear of any major extraneous peaks.
5.9.5.7.3 Polystyrene values range:

841 ± 10 cm^{-1}
905 ± 10 cm^{-1}
1068 ± 10 cm^{-1}
1154 ± 10 cm^{-1}
1600 ± 10 cm^{-1}
2848 ± 10 cm^{-1}
3024 ± 10 cm^{-1}
3059 ± 10 cm^{-1}

5.9.5.8 Values that fall outside of the ranges for the PS will require equipment repairs. Exceptional changes in the BKG, BLK, or PS will be discussed with staff.

5.9.5.9 After a successful completion of weekly and/or monthly quality checks, the instrument will be considered acceptable for use. If the instrument is not suitable for use, then maintenance and troubleshooting will be performed.

5.9.5.10 The data from the Val-Pro and quality check will be kept in a QC binder by the instrument and archived as needed.

5.9.6 Acceptable results for case work:

5.9.6.1 The sample spectrum should compare favorably with a spectrum of a known standard in both its overall appearance and in the presence and location of the major peaks.

5.9.6.2 Data from chemical reference standards, library matches, or published reference spectra used for comparison and to support positive conclusions will be included in the case file.

5.10 Gases

5.10.1 Gases used for instrumentation analysis shall of the highest available quality. GC/MS instruments require ultra-pure Helium to be used as the carrier gas so as not to interfere in the analysis of items.

5.10.2 All gas cylinder pressures will be recorded on the Gas Cylinder and Pressure Log at least weekly.

5.10.3 Any gas cylinder pressure at or below 300 psi should be changed that day.

5.10.4 The Chemistry supervisor will be notified when gas cylinders need to be replaced/reordered.

5.11 Peer Review

5.11.1 The final inspection of reports generated by the chemistry section consists of two stages, administrative and technical review. The administrative and technical reviews will be documented in the case file or electronically using the case review functions of the PLIMS system.

5.11.2 Administrative review will be conducted on 100% of cases to ensure the completeness and correctness of the reports issued. An administrative review will be completed by someone other than the assigned analyst or technical reviewer. The administrative reviewer shall check for accuracy and clarity by reviewing:

- The report for typographical or grammatical errors, misspellings, incorrect dates, omissions, identifying case information errors, or data transfer errors.

- Each page of the examination documentation has the unique case identifier, initials of the analyst, and no improper corrections.

- Any supporting administrative documentation or case communication contains the unique case identifier and the initials of the analyst.

- The materials present in the physical case file also exist in the electronic case file.

5.11.3 Technical review will be conducted on a minimum of 99% of cases. The technical reviewer shall check for accuracy and clarity by reviewing:

- The examination documentation to ensure the case notes, worksheets, photographs and other data support the conclusions.

- The conclusions are reasonable and within the acceptable opinions of peers within the discipline.

- All manual calculations and data transfers are correct.

- All appropriate procedures and controls are performed and documented.

- General initialing, dating, corrections, and unique case identifiers are correct and appropriate.

- The worksheets are properly utilized and filled out correctly.

- All requested examinations have been performed and addressed on the report or documented by case communication.

5.11.4 If a deficiency is detected during the review process, it shall be corrected and, if necessary, a new report generated.

Appendix A- Commonly Used Abbreviations

Refer to the Official Abbreviations for Chemistry Section document for a comprehensive list.

Appendix B - Color Reagents

The following lists some color tests and examples of how they react with various drugs. This list is by no means exhaustive as many color tests cross react with several drugs. Amounts listed below may be altered, however the ratio of components should remain consistent.

Cobalt Thiocyanate – for cocaine and cocaine base

Preparation:

a. Dissolve 2 grams of cobalt thiocyanate in 100 milliliters of water and 100 milliliters of glycerin.

OR

b. Dissolve 5 grams of cobalt thiocyanate in 250 milliliters of deionized water. Add 25 mL of Hydrochloric Acid.

Expires **one year** after preparation. Discard if reagent has changed colors.

Procedure: Place a small amount of reagent on a spot plate add sample.
Results:
a. Cocaine HCl – blue
b. Cocaine base – may initially be negative or faint blue.
c. PCP – blue, greenish blue

Duquenois-Levine – for marijuana

Preparation:

Reagent 1:

a. Dissolve 2.0 grams of vanillin to 100 milliliters of 95% ethanol add 2.5 milliliters of acetaldehyde.

OR

b. Dissolve 1.2 grams of vanillin in 60 milliliters of ethanol. Add 15 drops of acetaldehyde.

Expires **one year** after preparation. Discard if reagent has changed colors.

Reagent 2: concentrated hydrochloric acid
Reagent 3: chloroform
Procedure: Place 2-3 drops of reagent 1 in well. Add a small amount of sample. Add 1-2 drops of reagent 2. Add 1-2 drops of reagent 3.
Results: Marijuana – after reagent 2 is added a purple color will result, after reagent 3 is added the purple color diffuses into the chloroform layer

Liebermann's – for methcathinone, mephedrone and analogues of methcathinone

5 grams of Sodium Nitrite to 50 mL of Sulphuric Acid with cooling in water bath and swirling to absorb brown fumes.

Expires: Made only when needed.

Marquis – for opiates, phenethylamines, and general drug screening

Preparation:

a. Mix 10 mL of formaldehyde with 90 mL of concentrated sulfuric acid.

Expires **six months** after preparation. Discard if reagent has changed colors.

Results:

a. Opiates – purple
b. Amphetamine/methamphetamine – orange/brown
c. MDMA/MDA – black
d. Aspirin – pink to deep red
e. Bk-MDEA – yellow

Simons – for secondary amines (does not react with primary amines)

Preparation:

Reagent 1:

a. Prepare a 2% sodium carbonate in distilled water solution. OR
b. Dissolve approximately 12 grams of sodium hydrogen carbonate in 100 milliliters of distilled water to form a saturated solution.

Expires **one year** after preparation. Discard if reagent has changed colors.

Reagent 2:

a. Dissolve 1 gram of sodium nitroprusside in 100 milliliters of water and add 2 milliliters of acetaldehyde to the solution with thorough mixing.

OR

b. Dissolve 1.0 gram of sodium nitroprusside (sodium nitroferricyanide dehydrate) in 100 milliliters of distilled water. Add 10 milliliters of acetaldehyde.

Procedure: Place 2-3 drops of reagent 1 in well. Add sample. Place 2-3 drops of reagent 2 in well.

Results: secondary amines (MDMA, methamphetamine) – blue/purple

Van Urk's (Ehrlich's) Test (p-Dimethylaminobenzaldehyde): for Mushrooms and LSD

Preparation:

Dissolve 1 g p-Dimethylaminobenzaldehyde in 100 mL of 95% Ethanol and 10 mL of concentrated HCl. Mix well. Store in refrigerator in the dark. Expires **3 months** after preparation. Discard if reagent has changed colors.

Procedure:
Place sample in spot plate well (or other suitable container) and add reagent.

Results: Lysergic Acid Diethylamide (LSD) = purple
Psilocin, Psilocybin = purplish / bluish

Benzocaine, Procaine = yellowish

Simon's Test

Preparation:
Reagent A - Dissolve 1 gram of Sodium Nitroprusside in 100 ml of D.I. water.
Add 10 ml of Acetaldehyde. Expires **6 months** after preparation.
Discard if color reagent changes.

Reagent B - Dissolve 5 grams of Sodium carbonate in 100 ml D.I. water.

Procedure:
Place sample in spot plate well (or other suitable container) and add equal drops of reagent A and reagent B to the well. Note the color change.

Results:
Secondary Amines (e.g. Meth) = Blue
2,3-MDA and 2,3-MDMA = rose
3,4-MDA and 3,4-MDMA = gray-brown
Steroids = orange or yellow
Thioxanthenes = red or orange

References

Clarke's Analysis of Drugs and Poisons, Third Edition, Volume One, Pharmaceutical Press, 2004.

Bonin, Michael. June 1983. Isolation and Identification of Psilocybin and PsilocinMicrogram Journal XVI: 94-98

Appendix C - Unit Conversions

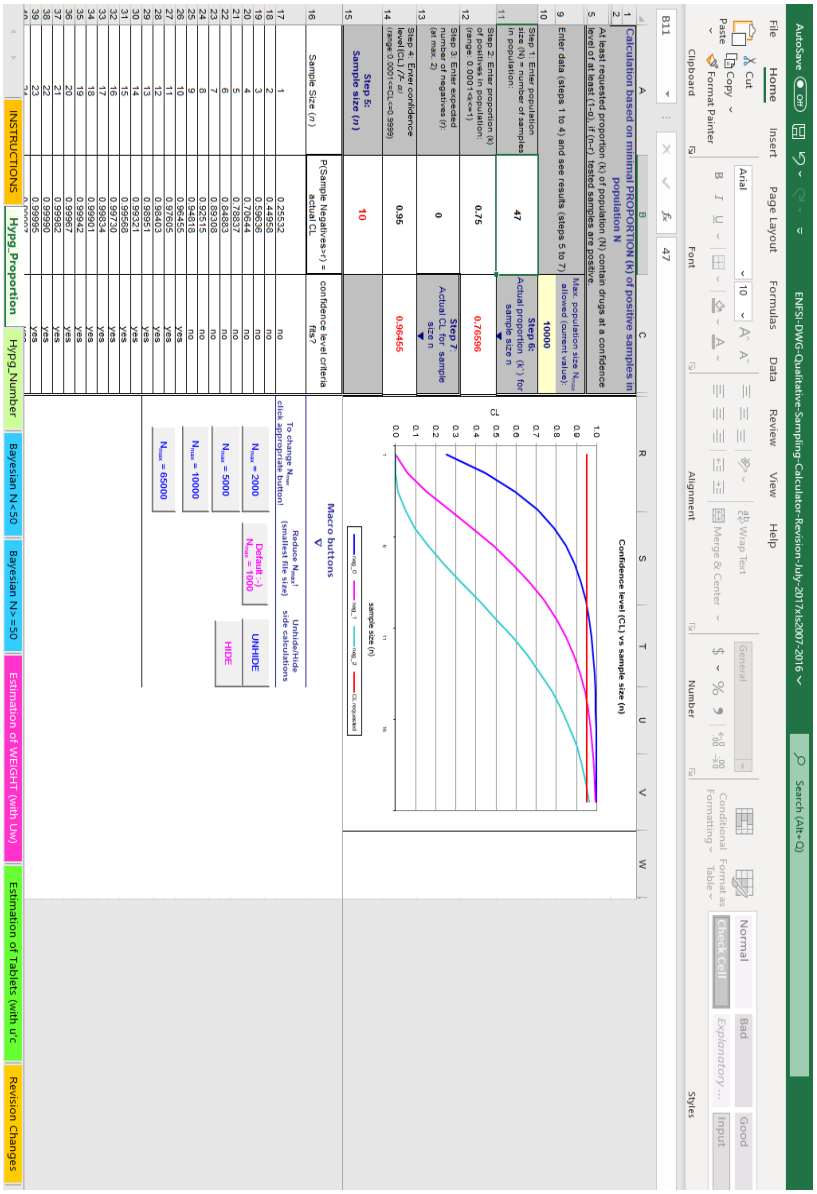
1 oz = an Avoirdupois ounce = 28.35 grams
1 pound (lb) = 453.59 grams
1 pound (lb) = 0.4536 kilograms (kg)
1 kilogram (kg) = 2.20 pounds (lb)
1 cc = 1 milliliter (mL)

Appendix D - Hypergeometric Sampling Table & Excel Calculator

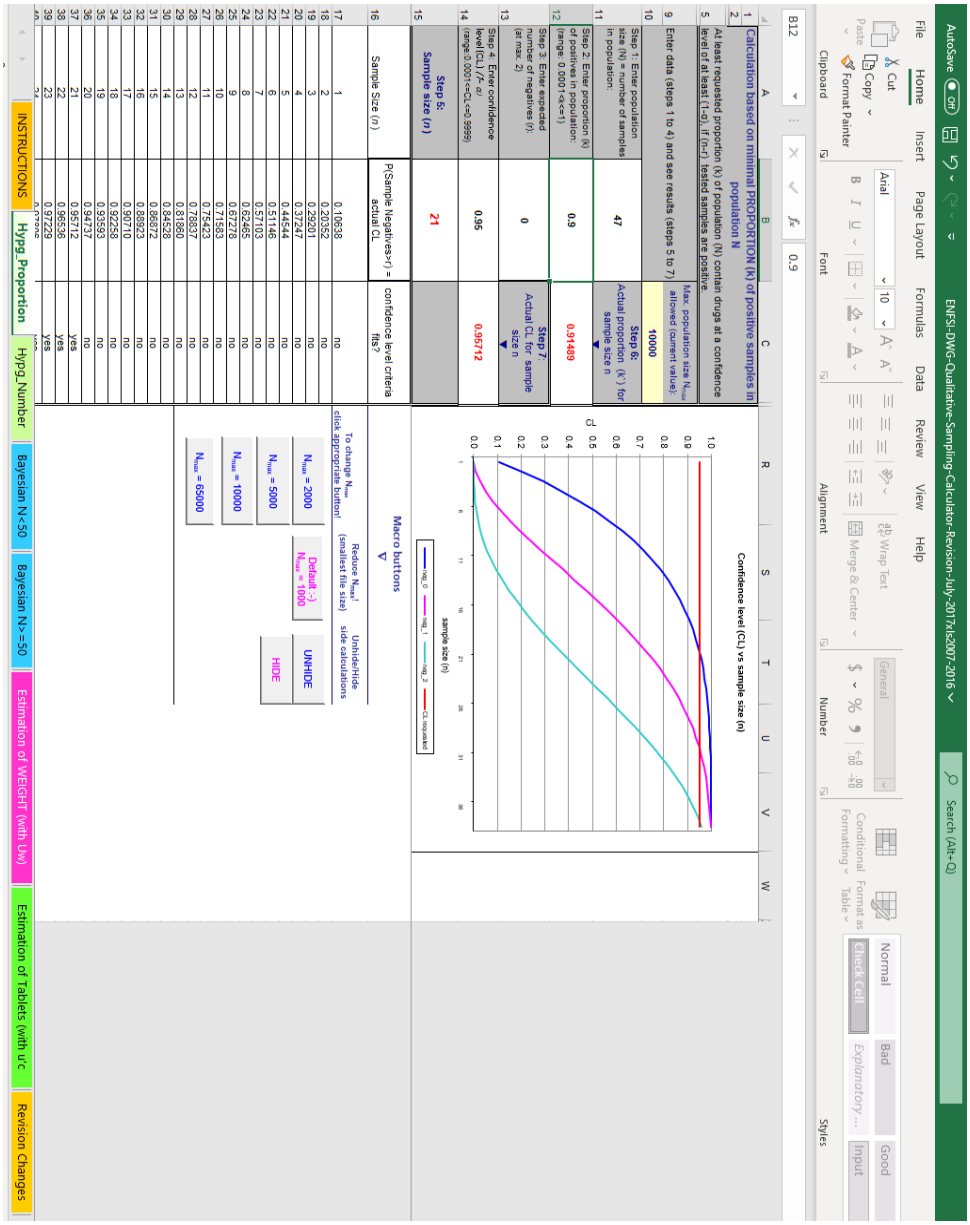
The table and excel screenshots in this appendix are for illustrative purposes only. Figures should be entered into the Excel calculator located on the shared drive at the following location: R:\Department\Crimelab\Sensitive\Lab Documents\Chem\Hypergeometric Sampling\ENFSI-DWG-Qualitative-Sampling-Calculator-Revision-July-2017xls2007-2016.xlsm

Sample Population Size (N)	95% confidence interval	
	k = 0.75	k = 0.9
10	6	8
20	7	12
30	9	15
40	9	18
50	9	19
60	9	20
70	10	21
80	10	22
90	10	23
100	10	23
200	10	26
300	11	27
400	11	27
500	11	28
600	11	28
700	11	28
800	11	28
900	11	28
1000	11	28
5000	11	29
10000	11	29

(Table derived from ENFSI – Guidelines on Sampling of Illicit Drugs for Qualitative Analysis-Second Edition 2016)



(Hypergeometric Sampling Excel Calculation of 47 pills at a 95% confidence interval that 75% of the population contains the analyte in question)



(Hypergeometric Sampling Excel Calculation of 47 pills at a 95% confidence interval that 90% of the population contains the analyte in question)

References:[European Network of Forensic Science Institutes Drug Working Group (ENFSI DWG) "Guidelines on Sampling Illicit Drugs for Qualitative Analysis" Second Edition 2016 and the Qualitative Sampling Calculator Revision July 2017 (xls2007-2016) https://enfsi.eu/docfile/enfsi-dwg-qualitative-sampling-calculator-revision-july-2017-xls2007-2016/].

Appendix E - Statutory/Trafficking Limits in North Carolina (as of November, 2024) [https://www.ncleg.net/EnactedLegislation/Statutes/HTML/ByArticle/Chapter_90/Article_5.html]

Marijuana

Possession - Class 3 misdemeanor
Exceeds one-half of an ounce (14.18 grams) - Class 1 misdemeanor.
Exceeds 1.5 ounces (42.53 grams) - Class I felony.

Trafficking
Excess of 10 pounds, but less than 50 pounds
50 pounds or more, but less than 2,000 pounds
2,000 pounds or more, but less than 10,000 pounds
10,000 pounds or more

Hashish (extracted resin of marijuana)

Possession
Exceeds one-twentieth of an ounce (1.42 grams) – Class 1 misdemeanor.
Exceeds three-twentieth of an ounce (4.25 grams) – Class I Felony

Synthetic Cannabinoids (check NC statute for regular update - http://www.ncga.state.nc.us/gascripts/statutes/statutelookup.pl?statute=90-94)

Trafficking – dosage unit = 3 grams
Exceeds 50 dosage units, but less than 250 dosage units.
Exceeds 250 dosage units, but less than 1250 dosage units.
Exceeds 1250 dosage units, but less than 3750 dosage units.
3750 dosage units or more

Cocaine

Trafficking
28 grams or more, but less than 200 grams
200 grams or more, but less than 400 grams
400 grams or more

Methamphetamine or amphetamine

Trafficking
28 grams or more, but less than 200 grams
200 grams or more, but less than 400 grams
400 grams or more

Opium, opiate, or opioid (Heroin, Oxycodone, Hydrocodone, Fentanyl)

Trafficking
4 grams or more, but less than 14 grams
14 grams or more, but less than 28 grams
28 grams or more

MDMA, MDA

Trafficking
By weight
28 grams or more, but less than 200 grams
200 grams or more, but less than 400 grams
400 grams or more

By tablet count
100, but less than 500 tablets, capsules, or other dosage units
500, but less than 1,000 tablets, capsules, or other dosage units
1,000 or more tablets, capsules, or other dosage units

LSD (Lysergic Acid Diethylamide)

Trafficking
100 or more dosage units, but less than 500 dosage units, or equivalent
500 or more dosage units, but less than 1,000 dosage units, or equivalent
1,000 or more dosage units

Any substituted cathinone, as defined in G.S. 90-89(5)(j), or any mixture containing such substance

Trafficking
28 grams, but less than 200 grams
200 grams, but less than 400 grams
400 grams or more

Appendix F – Commonly used Instrument Methods

METHOD NAME	SUGGESTED USE / NOTES
SHORT	Cocaine, Heroin, Marijuana
DRGSCN	Unknowns, Mushrooms, Heroin, Tablets, QC
SLEEP	End of sequence/End of day
STEROIDSCN	Steroids, synthetic Cannabinoids etc

METHOD NAME	INIT TEMP (in °C)	INIT TIME (min.)	RATE 1 (°/min.)	TEMP 1 (in °C)	HOLD TIME (min.)	RATE 2 (°/min.)	FINAL TEMP (in °C)	FINAL TIME (min.)	TOTAL RUN TIME (min.)	MODE	INLET TEMP (in °C)
SHORT*	255	3.20	75	N/A	N/A	N/A	300	2.50	6.30	SPLIT	255
DRGSCN	100	1.75	25	200	0.25	15	300	5.33	18.00	SPLIT	250
SLEEP	100	999.99	N/A	N/A	N/A	N/A	100	N/A	999.99	SPLIT	255
STEROIDSCN	150	1.0	20	N/A	N/A	N/A	300	10	18.5	SPLIT	250

**Samples that render a negative result on the GC-MS "SHORT" method must be run again on a GC-MS " DRGSCN" or an appropriate temperature program method.*

These methods are not all inclusive. Other methods may be developed for specific uses. Final times listed above are the minimum time requirements. Final times can be extended to include late eluting compounds as necessary.

Appendix G – Special Procedures

Not all procedures are addressed in this SOP. At times there may be a new drug, analog, or drug mixture that requires a new procedure to be implemented. Any literature references can be used to develop a new method or process for analysis. These procedures once shown to be reliable can be added to this SOP or appendix as a valid method/process. Refer to QM 5.4 for more details on validating methods for use in case work.

Appendix H – Equipment

Software (Optimized in accordance with vendor recommendation of revision changes).

GC-MS A2: GC 7890B
MS 5977B
Autosampler 7683
Chemstation/Mass Hunter F.01.03.2357

GC-MS B2: GC 7890
MS 5975C
Autosampler 7693
Chemstation E.02.02.1431

GC-MS C2: GC 8890
MS 5977C
Autosampler 7693A
Chemstation F.01.03.2365

GC-MS Galileo: GC7890A
MS 5975C
Autosampler 7693
Chemstation E.02.02.1431

Chromatography Data System - Agilent Technologies GC ChemStation™ software
Columns: Agilent J&W, Restek (DB-5MS) 30m, 0.25 mm i.d., 0.25 µm film thickness or equivalent.

FTIR Nicolet iS10/SMART iTX
Omnic 9.8.372
Data System –Thermo Fisher Scientific Inc. software.

A more detailed equipment list is maintained under the Lab Documents folder in the Sensitive folder in the Crime Lab folder of the R drive.

Appendix I – Uncertainty of Measurement

Definitions for Uncertainty of Measurement

Balance:		Reference Standards:		Facility:		Procedure:		Staff:		Properties of Sample:	
range		calibration uncertainty	B	temperature	A	static vs. dynamic weighing	A	training	A	moisture	A
readability	B	integrity (storage and handling)	A	humidity	A	weighing vessels	A	experience	A	density	A
calibration uncertainty	B	temperature	A	air currents	A	placement of sample on balance	A	multiple staff performing same measurement	A	size	A
linearity	B	magnetism	N/A	vibrations	A	stabilization (time to record reading)	A	operator technique	A	physical stability	A
drift	A			electrical quality	A			distraction	A	air bouyancy effects	A
corner-loading	A			barometric pressure	A			attitude or mood	A	magnetism	A
hysterisis	SOP			static	A						
cleanliness	SOP			physical limitations (workspace size)	A						

Definitions

Type A evaluation of uncertainty is a method by statistical analysis of a series of observations.

Type B evaluation of uncertainty is a method by means other than the statistical analysis of a series of observations.

The mean is defined as the sum of the measured values divided by the total number of values:

$$X = \frac{\sum x_i}{n}$$

Where X is the mean, x_i is the different measured values,
n is the number of measured values

The standard deviation measures how closely the data are clustered about the mean. The standard deviation is defined as

$$s = \sqrt{\sum (x_i - X)^2 / n-1}$$

where s is the standard deviation
X is the mean, x_i is the different measured values
And n is the number of measured values

The confidence interval is an expression stating that the true mean, μ, is likely to lie within a certain distance of the measured mean, X. The confidence interval of μ is given by:

$$\mu = X \pm \frac{ts}{\sqrt{n}}$$

where s is the measured standard deviation,
n is the number of measured values, and
t is a number from the Student's t table for a certain confidence interval.

Sometimes the values are more likely to fall near the average than further away. This is typical of a *normal* or *Gaussian* distribution. This is graphically represented by a bell curve. Normal distribution uses the above equations for determining measured standard uncertainty.

When the measurements are quite evenly spread between the highest and the lowest values, a *rectangular* distribution is produced. The standard uncertainty for a rectangular distribution is:

$$\frac{a}{\sqrt{3}}$$

where a is the semi-range (or half-width) between the upper and lower limits.

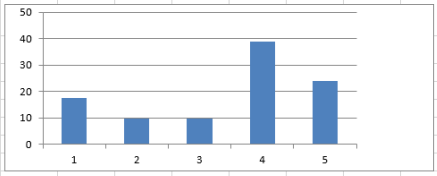
Estimating the Uncertainty of Measurement

The factors to be considered in determining the uncertainty of the measurement are the measurement process reproducibility(historical data), readability at zero, readability at load, linearity, and the balance calibration uncertainty(determined by outside calibration vendor).

Example of an Individual Balance Budget table:

Measurement Uncertainty Estimation Form

Measurement:	Weighing on the Mettler Balance PM2000(N11031) which reads to 0.01 grams								Year: 2024	
Range of measurement values:	0 - 2100 grams									
Procedure name and revision:	Drug ID SOP, 6-23									
Estimation prepared by:	MAJ						**Date Prepared:**	Dec-23		
Line Item	**Uncertainty Component**	**Value**	**Units**	**Distribution**	**Type**	**Divisor**	**Degrees Freedom (n-1)**	**Standard Uncertainty**	**Component Contribution %**	
1	Measurement Process Reproducibility	0.00516398	grams	normal	A	1.00	9	0.005163978	17	
2	Readability at Zero	0.005000	grams	rectangular	B	1.73	infinite	0.002886751	10	
3	Readability at Load	0.005000	grams	rectangular	B	1.73	infinite	0.002886751	10	
4	Linearity	0.020000	grams	rectangular	B	1.73	infinite	0.011547005	39	
5	Calibration uncertainty	0.01430	grams	-	B	2.00	9	0.007150000	24	
	Combined Standard Unc							0.015092686	100	
	Expanded Unc	*U* (*k*=2)						0.030185372		



NOTE: Regardless of the number of digits that are showing in a cell, Excel carries the maximum number of significant figures in the background and will use the entire number for further calculations

The basis for the data above:

1	Measurement Process Reproducibility takes into account all components listed as Type A Evaluation on the Uncertainty Component Tab
2	Balance resolution at zero is 0.01; any rounding will be from half of that interval above or below the reading. Therefore value is readability divided by 2
3	Balance resolution at zero is 0.01; any rounding will be from half of that interval above or below the reading. Therefore value is readability divided by 2
4	Linearity is taken from the manufacturer's specification for this individual model.
5	Accredited Vendor's Uncertainty at k=2 (largest uncertainty value from calibration certificate)
6	
7	
8	
9	
10	

Revision: Dec-23

Calculation of Combined Uncertainty

The combined uncertainty will be determined when more than one standard uncertainty is shown to be a major contributor to the uncertainty of measurement. The combined uncertainty will be calculated using the Root Sum Squares (RSS) method. The RSS equation is defined as the square root of the sum of the squares of the data points. All significant figures will be carried throughout this calculation.

$$U_{combined} = \sqrt{u_1^2 + u_2^2 + u_3^2}$$

Calculation of Expanded Uncertainty

The expanded uncertainty is used as a measure of uncertainty that defines a confidence interval about the measurement result. It is the combined uncertainty multiplied by the confidence factor (k). All significant figures will be carried throughout this calculation.

$U_{expanded} = U_{combined}$ (k) k = 2, at the 95.45% confidence level

$$U_{expanded} = 0.015092686 \text{ g } (2) = 0.030185372 \text{ g}$$

The expanded uncertainty calculated for each of the individual balances will be averaged together and the result will use conventional rounding rules to determine the final uncertainty of measurement. The final uncertainty for individual balances will only use two digits after the decimal while the Sartorius Miras 2 balance will use three digits after the decimal (the Sartorius Miras 2 balance will have its UOM calculated separately). Assuming the value from the above example:

$$U_{final} = 0.03 \text{ g}$$

A weighing event is defined as the actual weighing of a sample, not to include the zeroing/taring of the balance as a weighing event. For casework, where n is the number of weighing events.

$$U_{reported} = n * U_{final}$$

For the single item example with only one weighing event, the final uncertainty equals the reported uncertainty at the 95.45% confidence level:

$$U_{reported} = 1* 0.03 \text{ g} = 0.03 \text{ g}$$

For a multiple items example where the number of items and/or weighing events is 5, the reported uncertainty at the 95.45% confidence level is:

$$U_{reported} = 5* 0.03\text{g} = 0.15 \text{ g}$$

Recording and Reporting Weights with Uncertainty

The measurement of uncertainty will be recorded in the case notes and on the report as the total weight ± the reported uncertainty at the 95.45% confidence level.

References

Sampling
NC v Hayes (7Dec96)
NC v Anderson (3Sep85)
NC v Wilhelm (1Nov82)
NC v Wooten (16Jan74)
NC v Riera (1Mar70)
Journal of Forensic Science (38/4:885, 38/3:641, 37/6:1541, 36/2:350, 35/3:713, 29/2:493)
Uncertainty of Measurement
ASCLD/LAB **–** *International* Standard ISO/IEC 17025:2005(E) 5.4.6 Estimation of Uncertainty of Measurement.
ASCLD/LAB **–** *International* Estimating Uncertainty of Measurement Policy (AL-PD-3008-Ver 2.1 effective date: March 9, 2007).
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Jeffrey S. Taylor

Digitally signed by Jeffrey S. Taylor
Date: 2024.12.10 09:20:10 -05'00'